THE JOURNAL OF INDUSTRIAL AND ENGINEERING CHEMISTRY

PUBLISHED MONTHLY BY THE AMERICAN CHEMICAL SOCIETY
At Eschenbach Printing Company, Easton, Pa.

Volume 12 JULY 1, 1920 No. 7

Editorial Offices:
One Madison Avenue, Room 343
New York City
Telephone: Gramercy 613–614

Cable Address: JIECHEM

Advertising Department:
170 Metropolitan Tower
New York City
Telephone: Gramercy 3880

CONTENTS

Editorials:

Original Papers:

Laboratory and Plant:

Addresses and Contributed Articles:

Willard Gibbs Medal Award:

Chemical Reading Courses:

Scientific Societies:

Notes and Correspondence:

Subscription to non-members, $6.00; single copy, 60 cents, to members, 50 cents. Foreign postage, 75 cents, Canada, Cuba and Mexico excepted
Entered as Second-class Matter December 19, 1908, at the Post Office at Easton, Pa., under the Act of March 3, 1879
Acceptance for mailing at special rate of postage provided for in Section 1103, Act of October 3, 1917, authorized July 13, 1918.
Subscriptions and claims for lost copies should be referred to Charles L. Parsons, Secretary, 1709 G Street, N. W., Washington, D. C.

EDITORIALS

ROPE MAKING

Tearing to pieces a bit of hemp rope may indicate summer idling, but it has induced two thoughts—first, the individual bast fibers stand out preëminently as strong, hardy vegetable cells; second, the act of intertwining the individual fibers produces a rope of a strength multifold that of the sum of the individual fibers. What has this to do with chemistry? Quite a bit, in the light of coming events.

During the summer and early fall of 1921 four strong chemical organizations will hold their annual meetings in America, namely, the Society of Chemical Industry (British), the Convention of Canadian Chemists, the American Chemical Society, and the Seventh National Exposition of Chemical Industries.

These are all lusty bast fibers, each strong in itself as such strength goes, and there they lie individually on the table of 1921. Let's get at the business of rope making, and thereby fashion for the Anglo-Saxon world a binding power which will hold fast for our respective countries those new chemical industries which have developed because of the war and whose importance to the national welfare cannot be overestimated.

Why not bring these meetings together, and thereby enable all to partake of the benefits of such a gathering of Anglo-Saxon chemists? It is recognized, of course, that exposition week is an unfortunate time for scientific gatherings, for the distractions of the Exposition militate against a successful scientific program, but the whole problem could be solved if the American Chemical Society would hold its annual meeting a week or two later than usual, and the British organization could postpone its visit to this country until September, 1921. Our Canadian friends would certainly enjoy New York more in September than during the heated spells of July. On the basis of a two weeks' program it would be possible for all to share in the meetings of the chemical societies, and to attend the Exposition. At the same time, the opportunities for informal conferences and mutual acquaintance during exposition week would make possible a meeting to immediately follow the Exposition which would bring to more definite conclusion many policies which would be originated and informally discussed during the early days of the joint gathering. Aside from the visits to the Exposition, that week would offer exceptional opportunity to our British and Canadian guests to visit in small groups many of the industrial plants in the vicinity of New York City. The journey to Canada at the closing of such an eventful gathering would attract many. Thus would strength be increased multifold by this intertwining of chemical forces.

It is remarkable how similar are the problems affecting chemistry in our three countries. There is the same fundamental need of public understanding of what our efforts mean; there is the same consequent difficulty in securing favorable legislative action; salaries are inadequate in all alike; progress during the last four years has been appreciably the same. Much still remains to be done. 1921 offers a great opportunity.

As a coördinating agency in this matter, we would suggest that the officers of the Chemical Exposition take this subject up at once with the officers of the several organizations concerned with a view to making such adjustments as will insure this joint gathering. Thus would be assembled a large group of chemists, inspired with the common purpose of making the Anglo-Saxon race supreme in the field of chemistry, to take part in deliberations which should prove historic for our science.

CANADIAN HOSPITALITY

At the recent joint annual meeting of the Canadian Section of the Society of Chemical Industry and the Canadian Institute of Chemistry, it was our good fortune to be the guest of Canadian chemists. Those last few words tell a story of most delightful hospitality, of charming entertainment, and of unaffected good fellowship. The meeting furnished opportunity for seeing Canadian chemists in vigorous action, progressive, wide-awake, developing policies suited to conditions of their country, and imbued through and through with a spirit of patriotic devotion. We can never forget the experience. We understand now the glorious record of Canadian soldiers throughout the war. And the long boundary line from the Atlantic to the Pacific has and needs no military guard of either nation.

ONE-SIDED GREASING

The National Prohibition Act is a two-wheeled cart, and not a monocycle. It provides against the use of alcohol as a beverage and at the same time in specific terms declares that the use of alcohol in research and in the industries is to be encouraged and facilitated. The Volstead Act exists for enforcing the provisions of the Prohibition Act and for carrying out the intent of Congress. One wheel of this cart, the prohibition side, has been abundantly greased and is running smoothly and efficiently in spite of the obstacles deliberately thrown in the roadway, while the other wheel has received but scant attention. It is screaking badly and friction is developing.

In such situations this friction usually finds its expression in loud-mouthed complaints, but fortunately that does not hold true in the present case. There is always a spirit of coöperation hovering around, if we only try to find it. It has been found in the case of alcohol for research and the industries. On June 17, 1920, there gathered in the office of the Commissioner of Internal Revenue in Washington, Lt. Col. Amos A. Fries of the Chemical Warfare Service, Mr. George W. Patterson, designated by Secretary Daniels to represent the Navy Department, representatives of the American Chemical Society, the Manufacturing Chemists' Association, the American Dyes Institute, the American Pharmaceutical Association, the American Drug Manufacturers' Association, the National Association of Retail Druggists, the National Whole-sale Druggists' Association, the Manufacturing Perfumers' Association, the Proprietary Association of America, the Drug Trade Conference, and individual manufacturers and users of industrial alcohol.

Owing to the illness of Commissioner Williams the hearing was conducted by Mr. Paul Myers, assistant to the Commissioner, in the presence of several officials of the Bureau. Brief addresses were made by the representatives present, the burden of which was that it was fully realized how pressing had been the problems before the Bureau in the administration of the Act, and how great an organization had had to be quickly built up. The great importance of alcohol in the chemical, pharmaceutical, and drug industries was clearly pointed out. Likewise the large demands of the Government for alcohol in smokeless powder and poison gas manufacture in time of war were demonstrated by the officials present. Statistics were exhibited showing the present greatly reduced output of alcohol.

In view of these representations it was urged that a special officer of the Bureau possessing technical qualifications be designated to have direct supervision of this feature of the Act. It was further suggested that if the Commissioner desired such aid, he invite the various national organizations to nominate representatives to an unofficial civilian board which would act in an advisory capacity to the Commissioner in working out satisfactory solutions of the various technical questions involved.

A fine spirit pervaded the meeting, and should the Commissioner adopt the suggestions made at the meeting the friction of that other wheel will be diminished, the industries will prosper, and the intent of Congress be fully carried out.

AN UNFINISHED STORY

Congress has adjourned. The expected "recess" instead of adjournment did not materialize. Satisfactory assurance from Democratic leaders that no attempt would be made to create political capital by a presidential reconvening of Congress, the call of the two great national conventions, and the desire of both Republicans and Democrats to get into the great American quadrennial game of president-making led to adjournment. The record as to legislation affecting directly the chemical industries is only a half-told tale.

First and foremost—the future existence of the Chemical Warfare Service is assured. Both Senate and House voted favorably upon its independent character, and in the conference committee the Senate members accepted the wording of the bill as it passed the House, defining the duties of the Service. This was important, for without it there would always have been the possibility of a side-tracking of the Service by departmental rulings. There remains only one matter of vital importance, the appointment of the Chief of the Chemical Warfare Service. The situation in regard to this is so clear, the individual with the necessary experience and demonstrated ability so easily recognized and so unique in the possession of these qualifications, that it is impossible to conceive of any other appointment by the Secretary of War and the Chief of Staff than that of Colonel Amos A. Fries, the present Chief of the Service. With Colonel Fries in command, bearing again the title of Brigadier General which he won in the field with the A. E. F., the enthusiastic coöperation of civilian chemists may be relied upon.

Second—The closely related dye bill remains in the position recorded in our last issue. The bill was not brought up for consideration by the Senate during the hurried closing days of the session. Senator Frelinghuysen, that indomitable fighter, read into the record an important communication from all of the smaller manufacturers of dyes, showing that the failure of Congress to enact adequate protective legislation for the industry would react destructively first upon these smaller manufacturers. Senator Wolcott of Delaware presented a lucid explanation of the Levinstein agreement, which showed that no world monopoly was contemplated or indeed possible under the agreement. The dye bill holds its place upon the Senate calendar, in position to be called up whenever the Senate desires during the short session beginning next December. At that time the same senators will be present to vote upon it, and, for the same reasons given in our last issue, we again predict that before this Senate adjourns finally on March 4, 1921, the dye bill will be passed and the industry thoroughly safeguarded. Meanwhile, the Sundry Civil Appropriation bill carries a significant item of $25,000 for the continuance of the work of the War Trade Board Section of the Department of State. The protection of the coal-tar chemical industry which has been afforded by the Trading with the Enemy Act since the blockade was raised will be maintained.

Third—The Bacharach bill never reached consideration by the Senate. It is a strange outworking that this Congress, controlled in both Houses by that party which has always boasted of its policy of protection for American industries, should have left without adequate protection, and for the most part on an absolutely free-trade basis, those industries which were called into being by the nation's need in time of war and upon the strong urging of officials of our Government. These industries, for which skilled labor has had to be developed in every department, must for the next few months rely solely upon the patriotic support of American consumers. There are to-day strange things going on which threaten their very existence.

Fourth—The Nolan bill, providing relief for the Patent Office, passed the Senate, but as it carried an amendment providing for assignment to the Federal Trade Commission of patents by government employees the bill had to go to conference, and there it remains, for the conductor was calling "All aboard for Chicago," and the engineer of the San Francisco special was getting up steam. We've seen the boys in college get just as badly demoralized just before a big football game. We always have maintained that campus life is but a miniature reflection of the greater world outside. Well, when it's all over we'll be as good Americans as ever, and our faith is still strong in the present Senate, though it sure is taking plenty of time to pass a few, plain, simple, necessary measures.

NOTES

It is with pleasure we announce that, beginning with the August issue, THIS JOURNAL will carry a monthly London letter from Dr. Stephen Miall, LL.D., B.Sc. Dr. Miall is the co-secretary and treasurer of the Federal Council for Pure and Applied Chemistry, and is also managing director of the Brimsdown Lead Company. It is hoped that through Dr. Miall's exceptional opportunity for close observation American chemists will be kept in touch with the thought and policy of British chemists and with the most recent developments in the field of industrial chemistry in Great Britain.

Announcement of the recess appointment of Professor Marston Taylor Bogert of Columbia University as a member of the Tariff Commission brings a comfortable feeling to all chemists. It has long been felt that the intricate character of the questions involved in the tariff as applied to chemicals demanded the presence on the Commission of one thoroughly familiar with matters chemical. Professor Bogert's high attainments in chemistry make his appointment an appropriate one. The country is under many obligations to him for patriotic service rendered throughout the war, and his work on the Tariff Commission will constitute a further obligation of this nature. Dr. Bogert has served as President of the American Chemical Society and of the Society of Chemical Industry. He represented chemistry at the original White House conference on conservation called by President Roosevelt. It is peculiarly fitting that one who is above all else an organic chemist should have been appointed to the Commission at a time when organic chemical industries are being so rapidly developed. Congratulations to both Dr. Bogert and the Tariff Commission.

It is a matter of rejoicing that Arthur Lachman has returned to his home in San Francisco none the worse for wear during his absence of several months while suffering from amnesia. In fact, he seems to have spent his time profitably, for according to a communication from a friend, he returned in the possession of a new suit of clothes and a hundred dollars earned in the automobile industry, which is a pretty good proof of chemical amnesia in these days. All of Dr. Lachman's friends join in the hope for a speedy recovery of his usual good health.

Synthetic camphor is rapidly finding its place on the map. From time to time references have been made in these columns to the necessity of the development of this industry as a measure of economic independence. It is welcome news that three of our most prominent chemical manufacturers are now engaged in the manufacture of camphor from spirits of turpentine, while we hear rumors of another project using a method which gives promise of very valuable results. From America's raw material America's supplies of camphor will be produced, thus gaining another step forward in the march of economic independence.

Honors are coming thick and fast to Dr. F. G. Cottrell, who organized the Research Corporation and assigned to it the patents on his process of electrical precipitation for the threefold purpose of insuring sound commercial development of the process, of dedicating its profits to scientific research, and of insuring his own freedom from the usual business cares connected with patents in order to devote his time uninterruptedly to scientific work. Within the past few months he has been appointed Director of the U. S. Bureau of Mines, has been chosen by his colleagues chairman of the Division of Chemistry and Chemical Technology of the National Research Council, and has been presented the Gibbs medal by the Chicago Section of the AMERICAN CHEMICAL SOCIETY.

Warmest possible welcome is given to the first bulletin of the Society of Chemical Industry News Service, of which Dr. E. H. Tripp, Central House, Finsbury Square, London E. C. 2, is Director. This bulletin contains the announcement of the award of the medal of the Society of Chemical Industry to Monsieur Paul Kestner, who "was born in Alsace prior to the German occupation in 1871; he was one of the chief founders and the first president of the Société de Chimie Industrielle in France, which was established in 1917. He has been connected with engineering as applied to chemical industry throughout his career, and among his more notable achievements are the use of forced draught in acid towers, automatic acid elevators, the climbing film evaporator, the scale-less water-tube boiler, and several inventions in connection with beet-sugar manufacture. Very shortly before the armistice M. Kestner gave an address of outstanding interest and importance to the London Section of the Society on 'The Alsace Potash Deposits and Their Economic Significance in Relation to Terms of Peace.' " The problem of public education regarding chemistry is the same in Great Britain as in America. This new medium for distribution of reliable information concerning the British chemical industry will perform a useful public function. We have tried it. We know.

There are those who for reasons easily understood have sought eagerly for justification of complaints about the handling of the reparation dyes by the War Trade Board and the Textile Alliance, Inc. These complainants have willfully ignored the retarding effect of Rhine overflows and the prolonged strike of dock laborers at Rotterdam, and have continually claimed that the British textile industry was getting a jump on the American industry because of tardy methods of our officials. To all such critics it must be somewhat confusing to read in the report of the U. S. Consul General in London (*Commerce Reports*, May 3, 1920, page 677) the following:

> After a long and weary wait the first batch of reparation dyes has been distributed in Yorkshire and has been very heartily welcomed.

But after all—facts are not wanted by those who have axes to grind.

RESPONSIBILITIES OF THE CHEMIST AS A CITIZEN[1]

By **H. W. Jordon**

Solvay Process Company, Syracuse, N. Y.

Chemists, engineers, and technical men, associated equally with capitalists, have created a new world of industry during the past fifty years, through scientific research applied to the mechanic arts. The automobile, the aëroplane, the submarine, and scores of Jules Verne fairy wonders have grown bit by bit through experimentation into huge, international industries. Our science, applied chemistry, is their foundation. It is not necessary to prove it. It must be admitted. This transformation of the material world has produced intense, breaking stress in the mental world. These industrial changes have brought social conditions which present serious, swiftly growing difficulties. It is our duty to lead in solving the social problems which exist, and helping to direct aright the tendencies of the day, for which we are responsible.

The mental activities, expressed in government, education, and religion, have been stagnant, unresponsive or hostile to the changes in social life. Those to whom we have entrusted government have taken it for granted that methods of administration, drafted for thirteen agricultural, seaboard states in the infancy of the steam engine and forty years before the first railroad, are equally suitable for the engineering age of 1920. Public school education clings to the long summer vacation and the program of the early nineteenth century, when ninety per cent of our people lived on farms where the food, clothing, and all the essential materials of life were grown or manufactured at home. The churches, struggling along with alembic methods in a triple effect age, complain of their declining influence, destroyed by selfish, materialistic city life. To-day seventy per cent of our people live in overcrowded cities, as employees of the industries which arose from our scientific research. Villages and farms have telephones, electric lights, phonographs, and automobiles. Life has been completely reversed, yet we chemists and engineers, prosperous in dollars acquired in creating the causes of world social turmoil, live in the midst of it in indifference. As a civic force, we are ciphers.

NEED OF ACTIVE CITIZENSHIP

First, we should become active citizens, each in his own community. Among civic activities, those relating to modernizing city government, public school education, reform of taxation, reorganization of agriculture, and revivification of the church, are among the most urgent and promising. Many feeble volunteer societies struggle spasmodically with these problems, and attain trifling results. But technical men, immersed in industry, have devoted little time or effort to public affairs. We chemists cannot make social progress by scattered efforts, united only during thirty days before election, against opposing forces of stagnation and reaction that are on the job three hundred and sixty-five days a year.

The entrance of women into government presents a promising force for social improvement. War taught women public work. In peace they have the time to apply their power to reforms, in which they are more practical than men, because they are moved by a deeper sense of idealism. Locally, men can best advance social progress by devoting their personal assistance and resources to support of level-headed, common-sense reforms in government, education, and sociology which women undertake.

Our constructive civic work must be devoted primarily to a new order of city life; to the upbuilding of personal character, through the home, the school and the church; and to what Roosevelt called civic righteousness. Good government of our cities is fundamental to that of the nation. The whole equals the sum of its parts. The character and strength of a nation are the aggregate of its cities, which in turn are the sum of the character of the citizens. Character and the finest qualities of the mind have been smothered in gross materialism. "Never was Carlyle's image of a basket of serpents, each struggling to get its head above the rest, so expressively precise a picture of humanity as it is to-day." The old order of individual selfishness, necessary to self-preservation in the stone age and in colonial days, is impossible in this industrial age. As Samuel Adams said, "Unless we hang together, we will hang separately."

LAW OF MENTAL MASS ACTION

We have evolved the engineering industries by coöperative scientific research. The social world is disintegrating because we have not attempted to build a modern social order by research methods like those through which we created the engineering industries. It has been the notion that man is an individual, free to do what he chooses, and that no laws govern his mental action. But in recent years the combined study of history, statistics, anatomy, psychology and allied sciences, has begun to disclose that man is subject to law as distinct as that of gravity or of chemical reaction. Babson, Carlton Parker, Crile, Cannon, Veblen, and others have shown that mankind acts in mass movements similar to those of the tides; that close relationship exists between the physical body and the action of the mind, and that physical animal life action can exist, entirely dissociated from mentality. These studies indicate that we are at the threshold of a century of mental scientific discovery, succeeding the nineteenth century of disclosure in material applied science.

Had any one in 1820 predicted that within one hundred years man would fly from America to Europe, and that his skill in flying would greatly excel that of the birds, or that with simple instruments he would send messages through the air from Paris to Honolulu, or that he would transmit and accurately reproduce the human voice over a copper wire from New York to San Francisco, a town which did not then exist, he would have been thought in danger of hell-fire, preceded by the fate of the Salem witches.

Sir Oliver Lodge's belief in and hope of development in the mental sphere are conservative, compared with any prophet who, one hundred years ago, might have predicted things as they are to-day.

We are learning that the human mind, in mass action, moving in waves through distinct cycles of years, expresses itself in financial inflation followed by panic, or in war followed by gross speculation and extravagance, which turns into industrial and financial depression and deep distress. Bolshevism, the mania for strikes, desertion of the farms and neglect of agriculture, overcrowding and insufficient housing in the cities, declining birthrate,—all these and scores of similar international wave activities prove man to be almost fluid in his mass response to social law.

The surgical investigators have disclosed that physical degeneration of the brain, the liver, the adrenal, and other glands is coincident with what we had formerly presumed to be purely mental emotions of anger, fright, and worry. As one writer says: "When stocks go down, diabetes goes up." The biologist maintains pulsating life for three months or more by transferring embryo heart tissue of a chick to a suitable culture medium. Sections of veins, arteries, and nerves, dissected from the body and

---

[1] Read at a meeting of the Syracuse Section, American Chemical Society, April 30, 1920.

preserved for weeks in alcohol, and then grafted in place of tissue destroyed by accident or in war, begin nerve and blood circulatory functions and ultimately attain normal transfer of blood and nerve action through the alcohol-preserved tissue. These studies prove that human physical and mental life is subject to laws, as definite and as discoverable as those of chemistry or electricity. They await our research.

We can no longer expect to attain progress in government and social life by watchfully drifting. We must organize agencies for profound, experimental social research, applied to the mental departments of life. If we do not inaugurate such social research, and maintain it with increasing vigor similar to that of industrial research, our industries, our personal investments, our life insurance, our homes, art, music, and all intellectual and spiritual attainments will be destroyed in political and social chaos, as in Russia. There is chaos because the two wings, the material and the mental, do not flop together. The mental wing is atrophied, and we are headed for a tail spin.

NECESSITY FOR SCIENTIFIC SOCIAL RESEARCH

Eighty per cent of our people lack means of effective public coöperation. Our highest mentality, the graduates of our engineering schools, universities, and colleges, and all intellectual people, have no agency for analyzing and testing their theories of social construction or for registering their opinions in practical, effective public action. Mediocrity is triumphant, because it is organized and aggressively led. In self-defense, we chemists and technical men should establish within our own industries, Departments of Social Public Relations, to educate our own staffs, to study and solve local social problems, and to coöperate with university men and women, with all intellectual people and with the public, in concerted, persistent social progress, based on science.

Next, we should unite our engineering industries with the technical schools and universities, in Federations for Social Research, to undertake the work of evolving the highest attainable individual and national growth in government, education, and religion, by scientific social research. Our fundamental principle should be that of Babson, that action and reaction are equal in the mental world as they are in the physical world; that we get out of life what we put into it. We are getting Bolshevism, class misunderstanding and hatred now, because for the past thirty-five years we have put little else into life than materialism and selfishness. The research would be conducted, under our direction, by engineering schools and universities, through the city, state or national government, to assure authority to our work. Our results would be given full publicity to put the facts before the people, whose judgment is correct if they know the truth. If the truth be frankly and fearlessly presented, there is little need of reform activities. Most abuses are the result of suppression and secrecy.

REVISION OF SOCIAL PUBLIC RELATIONS

Among the possibilities of public service through scientific research are:

(1) There should be reorganization of secondary education. "The war has clearly shown that the popular education of tomorrow must be entirely different from that of to-day," says Anatole France. The Omaha mob which burned the courthouse and nearly lynched the mayor was composed of youths sixteen to twenty-five years old. They committed these crimes "just because they'd nothing else to do." Their school training had been so empty and purposeless that life suggested no other more interesting adventure.

In New York State alone there are about one-half million children who left school at fourteen, and who are employed in ineffective ways in the industries, or more frequently are demoralized through lack of any occupation whatsoever. They are the Omaha mob. Our coöperative organizations of social public relations should develop a plan of citizenship education to combine the resources of agriculture and the industries, in conjunction with the municipal activities of our cities, as the basis of education for this neglected half million. In this age of practical science we have rigidly excluded all children from observation of science applied in agriculture and industry, yet all of us earn our living in these activities. If we would swim, we must get into the water. The city children should be taken on study trips to see the inspiring and interesting phases of life in operation, and become familiar with them. That which they see and observe should be made the basis of broad, liberal education. In this way, we can put daily life and the work of the world back where they were thirty years ago, when the boy and girl got the better part of their education from their daily tasks. Farm and home life in those days compelled growth of observation, attention, and reasoning, and gave us self-reliant Yankees. That was before "Passing the buck" and "Let George do it" were invented.

The training should be designed to make better citizens, and not mere factory operatives. If our people be educated to high ideals of life, if they be raised to high mental and spiritual standards, broad understanding of the interrelation of industries, trade and world commerce, of agriculture and nature, and of the love of books, art and music, they will automatically acquire wholesome home and community interests. If they know the land and its processes, and if they own their homes, they will be good citizens. Few men would fight for a rented flat, but nations have fought for their farms.

(2) Working with the state and federal government we can study to bring about new conditions in the production, distribution, and preparation of food and clothing. We can make individual home ownership and personal thrift a national habit and establish a new social order, adapting the industries and the home to each other. The home and a family of pure racial blood should be the primary motive of our nation, and raising a family should be made more attractive and profitable than its avoidance. We should lay the technical journals on the shelf for awhile and read Madison Grant's "The Passing of the Great Race," and Lothrop Stoddard's "The Rising Tide of Color."

(3) Industry in recent years has been the victim of lack of truthful publicity. In conjunction with the universities and other agencies representing highest national intellectuality, we should present the truth through the press, so that the public may realize that our research is solving the baffling and complex problems of social life. The packers and milk distributors are spending money lavishly to put the truth in the minds of the people by explanatory advertising, one to ten years belated. If we are sincere, we can secure coöperation of great journals, daily, weekly, and monthly, and through them place our work before the nation in a manner so convincing that it will insure our ultimate, complete success. Truth and publicity are absolutely destructive to social abuses. The evil results of venereal disease are being steadily diminished by frank, dignified publicity. We need to study the problem of "Liberty and the News," as analyzed by Walter Lippman in his recent book of that title.

(4) We should develop a new class of engineers trained at the technical schools and universities to occupy positions in city, state, and national government similar to those occupied by engineers in industry. Their work should assure a lifelong career, with tenure of office based on merit, and transfer or promotion free from political, partisan, or sectarian influence. They should be the men who execute and maintain the findings of our social research. Through these engineers of social technology, we would rescue government from corroding reaction and put it on a modern basis of progressive, applied engineering. We would substitute scientific economics for the existing order of government by selfish, unenlightened, partisan politics.

The work proposed, though apparently highly idealistic, is merely the self-preservation of business and social life from the dangers of swiftly progressive mediocrity. The four examples cited are only a few of the possibilities of social scientific research, attainable by a federation of the chemical and engineering industries, working in conjunction with the engineering schools and universities through the authority of city, state, or national government. A nucleus already exists in the National Research Council, which includes departments of medical sciences, of biology and agriculture, and of anthropology and psychology. We should quickly expand that work by undertaking the most urgent tasks of social research in several industrial cities.

The object sought has been well described by Professor George F. Swain: "Continued progress and the interest of the social organism as a whole require that individual initiative and ability shall be encouraged to the utmost and allowed to enjoy the reasonable fruits of its exercise; that property should be protected; that taxation shall be equable and uniform; that leaders shall be chosen from those most capable and judicious; and that those who are only fitted for manual labor shall not acquire a distaste for it or look down upon it as inferior in dignity to other occupations; that waste and extravagance shall be reduced to a minimum."

AGRICULTURAL *vs.* INDUSTRIAL EPOCH

As an illustration of the splendid possibilities of scientific social research, let us outline a research study of agriculture in conjunction with education. The United States is a powerful, unique nation. Our close contact with nature, the sea and the wilderness, and the individual ownership of our farms and homes, made us intensely independent and highly individualistic. Each one was free to develop his ability within his natural limitations, in a country of vast resources. Coincident with exhaustion of the free land, we entered the industrial era and our population shifted from 80 per cent agricultural to 70 per cent city dwelling, and a vast influx of Slavs, Semetics, Latins, Southeastern Europeans and Western Asiatics diluted our English and Northern European stock to a minority. Thus we have land and racial problems, in addition to those of congested city industrialism.

Formerly the child learned life by living. The household arts which used to supply home technical training have been almost completely superseded by factory manufactured food and clothing. Our light and fuel for cooking are delivered to our homes through pipes or over wires, displacing the kerosene lamp and the wood pile, the old oaken bucket and the chain pump, and the bucksaw. The water wheel at the brook, the wind wheel on the woodshed, the figure-four trap and the muzzle loading shotgun have been supplanted by Mary Pickford, Douglas Fairbanks, and Charlie Chaplin, witnessed seated, in a dark room. Healthful play at home is rarely possible, for there is neither room nor play space. Play is transferred to city parks, where unmarried women of advanced years are hired to *teach* children to play.

The schools of former days and the long summer vacation were designed for New England village and farm life. The red schoolhouse education of that time was adequate, because the training in practical life which children received from their daily work at home produced self-reliant Yankees. The industrial changes during the past thirty-five years have robbed children of active experience in life, and have given them passive, commercialized amusement instead. Migration from the farms is making us a restless, discontented, purposeless nation, flabby in muscle and in mind. We are being transformed from red-blooded, rigid vertebrates, to water-white, sloppy, physical and mental mollusks, encased in a shell of organized labor.

With this topsy-turvy condition in education, there came similar inversion in farm life and food production, aggravated by unenlightened and wasteful methods of food distribution. And finally we have entered upon a deplorable decline in the influence of religion. The religious spirit, based upon close touch with nature and ownership of the land, degenerates and dies when a nation falls under the control of commercialism or industrialism. The people leave the farms they own and become city tenants. They flock to Babylon, Athens, Rome, New York, where they succumb to unimaginative materialism. They lose their love and fear of nature, their respect for law and authority, and their reverence for the Deity. They no longer have chivalrous regard for womanhood. Marriage, the home, and the family cease to be the prime motives in life, the pursuit of pleasure and luxury becomes dominant, and there is general subsidence in art, music, and the finer qualities of the mind. Spiritual indifference follows, ending in the decline and death of religion.

FIELD FOR SOCIAL EXPERIMENTATION

One of our most urgent social tasks, perhaps the most urgent, is scientific social research upon the interrelation of agriculture and country life, and the industries and city life in their practical bearing upon education and religion. Therefore, let us conduct a social experiment to give our city boys some measure of American education by contact with nature and the land, like that which our sturdy Yankee grandfathers received.

An experimental group of boys, about one hundred, of ages eighteen, nineteen, and twenty, would be assembled and assigned to farm life combined with school training. The periods would be for the farm growing season of seven months, April to October, inclusive, of their eighteenth and nineteenth years, and for the entire time of their twentieth year of age. The boys would be quartered in a suitable cantonment, in the center of a typical farm district to do the work on the farms, as many of our older chemists did when they were boys. They would be paid as we were, by their board, clothes and education, with some, preferably a small amount of, spending money.

Associated with their work, these boys would be given a generous amount of education in agriculture by simple, interesting, scientific instruction through lectures, farm and field demonstrations, and moving pictures. The fundamental principles of raising crops, preparation of the land, selection of seed, production and use of fertilizers and care of the crops from sowing to harvest should be taught, as well as the storage of the crops and their distribution to market. The breeding of poultry, cattle and all live stock should be taught, with special emphasis laid on the economic importance of pure breeds and the selection of the most productive individuals for breeding, as proved by the experience of Holstein-Friesian breeding for milk production. Good breeding pays, whether of Holsteins, hogs or humans.

There would be abundant opportunity to teach many scientific facts of the out-of-doors relating to production of fruit and garden vegetables, protection against insect pests, cultivation of flowers and practical forestry. A multitude of interesting topics regarding nature would occur to any alert teacher.

This instruction would afford not only pleasing recreation from manual labor, but also would give our boys broad and liberal education in agriculture and nature, and create an interest which would attract many of them permanently to the farm. It would give all of them robust health and keen, lifelong interest in out-of-door life, and bring them into educational balance with the other ten per cent of high school boys. The educational portion of the experiment could be in charge of the state agricultural college.

The abundant supply of dependable labor from this source would permit farm work to be done in two shifts of six or eight hours, covering the entire day from sunrise to sunset. This arrangement of shifts would remove all cause for objection by the farmer to daylight saving, because he could direct his work to best advantage at all hours of the day.

As the boys would be in excellent condition from not more than eight hours' labor, they could multiply the production of the farms by thorough, intense cultivation. Wasteful use of land could be ended. Inferior pasture or bush land could be brought into fruitful production or planted as forests. Rich bog lands could be drained. Scrub growth corners could be made permanent forests. Roadsides could be beautified and used for pasturage and fruit. Sheep-raising could be promoted to fertilize abandoned farms and to produce mutton and wool.

A further advantage would be that tractors could be used intensely, by sending them from farm to farm and keeping them in action sixteen hours a day. In the rush season, tractors drawing plows or harrows could be used twenty-four hours a day, with the help of artificial light supplied by the tractor engine, and operated by eight-hour shift labor.

Housing the boys in central camps would relieve the farmer's wife of the drudgery she now endures during the growing season, and would interfere in no way with the home life of the farmer. The boys could be trained in the Swiss military system, if desired. A steady hand and keen eye would be developed by competitive rifle marksmanship.

REDUCTION OF "H. C. L." ASSURED

Three great fundamentals of living are food, clothing, and housing. Food is the product of agriculture. Wool, cotton, leather, and linen, the raw materials of clothing, are products of agriculture. Lumber, an important material in housing and in furniture, is a product of forestry, which is tree agriculture. The high cost of living can be reduced by abundant, cheap production of food, clothing, and housing. That result can be assured if this system of coöperation between education and agriculture be evolved and brought into general practice, as the result of our scientific research.

The most highly perfected social organization in the world is that of the bees, who produce not only sufficient food for themselves but a liberal surplus for export to mankind, by their thoroughly organized food production. The plan proposed is essentially identical with that of the bees.

Country life compels attention, observation, reasoning, foresight, self-reliance and sturdy health. The Elkhorn ranch transformed Roosevelt from a sickly youth to our best example of vigorous physical and mental American life; and boyhood on the farm was the basis upon which Lincoln and most of the great American mental leaders built their lives.

The trends of civilization are swiftly flowing into a vortex of highly socialized life, directed by government. And government is becoming an aggregate of applied science through engineering. The situation is analogous to that in industry when Cornell, the Massachusetts Institute of Technology, and other engineering schools, were founded to educate engineers of applied material science as directors of industry. An extraordinary opportunity and duty confronts us: that we build a new social, intellectual, and spiritual structure, commensurate with our material one. For, as Anatole France recently said: "Whether we like it or not, the hour is at hand in which we must choose between being citizens of the world, or spectators at the death of civilization."

CHEMICAL INDUSTRIES AND TRADE OF NORWAY

By O. P. Hopkins

1824 Belmont Road, Washington, D. C.

Norway is a thinly populated country with only a few notable natural resources, chief of which are the forests, fisheries, and water power. Of minerals, iron ore and cuprous pyrites are the only items that need be considered. Not more than 3 per cent of the area of the country is under cultivation, and it is estimated that fully three-fourths of that area are absolutely unsuited for agricultural development. Another serious handicap is the lack of native coal.

There has been, however, a great accumulation of wealth as a result of the war, for timber, fish, and electrochemicals have been exported in large quantities at very advantageous prices, and Norwegian shipping, in spite of all vicissitudes, has earned splendid dividends. This wealth will be used to develop native industries and will be put into hydroelectric projects, into the development of a real iron and steel industry, and into agriculture. Before the war Norway had been exporting a few dairy products, but importing cattle feed and cereals, and the disadvantages of such an arrangement have been amply demonstrated during the last few years. Much of the wealth will naturally be spent outside of the country for equipment for the new developments and for a long list of necessities and luxuries that were difficult to obtain during the war. The American chemical industry will be interested in the extent to which the manufacture of electrochemicals is to be developed and in the extent to which dependence will be placed upon outside sources for other chemical products.

ELECTROCHEMICAL INDUSTRIES

It is estimated that the water power available in Norway is not less than 15,000,000 horse power, of which about 1,500,000 horse power are now actually utilized. It is widely distributed and, in the absence of coal, electricity is commonly used to an extent and for a variety of purposes that would be surprising even to an American. The greatest development has been along electrochemical lines, but there is an increasing call for current in the metallurgical industries, and it is used liberally for power, lighting, and heating.

Calcium carbide was the first product of the Norwegian electrochemical industry and production was so greatly stimulated by the war that the capacity has been wonderfully developed. The total output in 1915 was estimated at 100,000 tons, but large plants have been erected since that time. In that year one concern produced 60,000 tons and another 30,000 tons. A considerable proportion of the carbide is now used in the manufacture of cyanamide.

As every chemist knows, it was cheap Norwegian electricity that made possible the first commercial utilization of atmospheric nitrogen. Between 300,000 and 400,000 horse power are now used in Norway for the production of nitric acid, calcium nitrate (commonly known as "Norwegian saltpeter"), and other nitrates and nitrites. The future of this industry is considered very promising.

There was a time when a great deal of iron was smelted in Norway from the native ores, but even before the war this industry was declining with the rise in the price of coal, and after hostilities began most of the remaining plants were obliged to close. Much attention is therefore being given to the establishment of electric-smelting plants and there seems to be a good future in that direction. One plant that was in operation when the war began found it very difficult to obtain electrodes, but these are now manufactured in the country. Plans are under way for the development of an electric steel industry in connection with the smelters.

Electricity is also used in the production of aluminum, much of which is used in connection with air-nitrate plants, and in the manufacture of ferrosilicon. A good idea of the character and quantity of articles produced electrolytically can be obtained by studying the table of exports in this article.

IMPORTANCE OF FISHERIES

In value, the exports of fish and fish products amount to about half of the goods shipped out of the country. Cod, herring, whale, mackerel, and salmon form the principal catches, and the by-products of the industry are of interest to the chemist. Great quantities of oil are obtained from the livers of the cod, while the heads are used for fodder and fertilizer. Oil is also obtained from the blubber of the whale, the bones are crushed for use as fertilizer, and the flesh is used for both fodder and fertilizer.

OTHER CHEMICAL INDUSTRIES

As in Sweden, the production of wood pulp has grown to striking proportions in Norway, and the manufacture of paper has become increasingly important. Exports of pulp amounted to nearly 700,000 tons in 1913, the greater part going to England. Import restrictions in various countries and difficulties in shipping decreased the sales abroad, but the general demand since peace was declared bids fair to continue and means renewed prosperity to the Norwegian manufacturers.

Oxalic acid is made from sawdust and is an important item of export, the United States being one of the best customers.

Matches also go into the export trade in large quantities, although the industry has not reached the proportions attained in Sweden.

With supplies of native apatite, the manufacture of superphosphate has been developed to the point where more than 50,000 tons can be produced annually, as compared with a pre-war production of 20,000 tons.

Before the war, Norway imported something like 1,400,000 lbs. of soap of various kinds, a large quantity when the small population is considered. As there is a scarcity of oils suitable for the manufacture of soap, it is likely that a good market will continue for imported soap. Before the war, England supplied the demand to a large extent, but during the last few years American manufacturers have been doing about 40 per cent of the business and their products are enjoying popular favor.

STATISTICS OF PRODUCTION

There are no figures available to show the quantity and value of the production of chemicals and allied products in Norway, but the following table will serve to show the effect of the early years of the war upon such industries. It has been compiled from official returns issued by the Government Bureau of Insurance:

Some Effects of the War upon the Chemical Industries

	1913			1917		
Industries	Factories	Operatives	Average daily wage. Dollars	Factories	Operatives	Average wage per hour. Cents
Bone grinding	2	7	1.09	2	5	15.3
Calcium carbide	5	1,063	1.40	4	1,654	24.4
Candles	1	27	1.07	1	14	25.7
Cement	1	297	1.37	4	404	24.4
Chemicals:						
Heavy	7	219	1.15	11	381	24.4
Minor	27	137	1.00	32	243	18.8
Colors	10	73	1.09	13	162	19.0
Electrochemicals	12	2,497	1.50	12	4,847	31.9
Explosives	7	268	1.16	8	570	21.7
Fertilizers	14	140	0.82	15	106	18.0
Gas works	11	143	1.03	9	87	19.8
Glass	12	1,064	1.21	16	864	18.0
Glue	5	75	0.92	6	95	16.3
Insulating materials	5	28	1.29	3	58	16.9
Matches	4	919	1.29	4	928	20.4
Oils and by-products	42	663	1.02	51	907	22.0
Paper and cardboard	42	4,920	1.13	47	5,390	18.5
Perfumes	1	8	0.64	1	5	20.4
Pharmaceuticals	12	76	0.77	12	92	19.6
Refining oil	6	256	1.16	6	426	21.2
Refining petroleum				1	69	22.5
Soap	19	116	0.98	17	109	18.8
Soap, candles, perfumes	13	205	1.00	12	265	16.6
Tanneries	78	574	1.03	83	736	20.4
Tar paper	5	25	1.00	9	52	20.9
Train oil	78	360	0.91	82	295	18.8
Treatment of ores	11	617	1.25	17	1,455	30.6
Varnishes	6	41	0.98	6	42	23.3
Wood pulp:						
Chemical	27	5,261	1.17	27	5,099	20.9
Mechanical	69	3,735	1.03	67	3,010	17.4

As Norway is seeking to equip herself to refine and work her own minerals, the following table is inserted to show the materials available. The statistics are issued by the Norwegian Bureau of Statistics.

Production of Minerals

Minerals	1913 Metric Tons	1915 Metric Tons	1916 Metric Tons
Silver and silver-bearing ores	5,411	8,431	7,515
Copper ores	70,349	56,097	28,670
Pyrites (iron and copper)	441,291	513,335	295,354
Nickel ore	49,990	77,018	79,903
Iron ore	544,686	714,917	417,899
Zinc and lead ores	897	1,829	1,017
Chrome ore	.....	350	2,757
Molybdenite and wolfram	12	97	137
Rutile	77	170	103
Bismuth ore	.....	10	.....
Graphite	300	.....	.....
Apatite	757	1,901	2,236
Feldspar	40,842	8,778	12,811
Other	1,500	380	5,640
Total	1,156,112	1,383,313	854,042
Total Value	$5,753,500	$11,020,400	$8,227,900

THE MARKET FOR IMPORTED CHEMICALS

As already shown, Norway is displaying great activity along certain chemical lines, but it should be borne in mind that the variety of products is decidedly limited and that almost total dependence is placed upon outside sources for the bulk of the articles usually described as chemicals and allied products. In some of these lines American manufacturers have successfully entered the market since the war started and their wares have made a uniformly good impression. The character and extent of the requirements of the market can be determined by a study of the following table, which is based upon official figures published by the Norwegian Central Bureau of Statistics:

IMPORTS OF CHEMICALS AND ALLIED PRODUCTS

	1913 Pounds	1916 Pounds	1917 Pounds
CHEMICALS, DRUGS, DYES, MEDICINES:			
Acids:			
Boric	23,869	40,816	41,010
Carbolic, pure	15,084	32,222	23,215
Carbonic, liquid	4,328	13,827	882
Citric and tartaric	34,535	104,824	49,653
Muriatic	604,200	546,265	604,364
Nitric	61,632	386	.....
Sulfuric	6,473,698	3,239,748	1,555,427
Germany	1,128,712	649,189	1,940
Netherlands	3,879,346	.....	.....
Sweden	1,390,133	2,567,256	1,539,532
United Kingdom	176	15,322	2,756
United States	.....	.....	10,648
Other acids	175,532	338,488	365,187
Alum	6,886,796	7,001,413	3,762,519
Germany	.....	135,473	250,578
Sweden	.....	2,700,150	1,774,809
United Kingdom	.....	3,082,670	796,354
United States	.....	1,083,120	940,778
Ferric	(1)	408,918	773,485
Ammonia, anhydrous	(2)	35,171,042	18,327,136
Ammonia compounds: salt of hartshorn	232,830	467,671	264,246
Other	.....	4,431	3,217
Baking powder	30,875	38,331	27,935
Bromides, iodides, and cyanides of metals	(1)	68,112	6,484
Bromine	.....	.....	70
Camphor	11,380	10,523	21,332
Carbon disulfide	.....	8,437	12,216
Casein	31,557	10,335	10,692
Chemicals, n. e. s	238,333	888,520	104,608
Chloride of lime	7,418,788	5,169,590	2,339,568
United Kingdom	.....	2,282,488	.....
United States	.....	2,584,496	2,132,553
Chlorides of metals, n. e. s	(1)	57,494	6,371
Drugs, n. e. s	1,350,547	1,613,540	1,284,386
Dyes and dyestuffs:			
Aniline and alizarin dyes	669,985	633,192	789,319
Germany	532,700	577,191	766,402
Netherlands	119,386	822	.....
Switzerland	441	10,198	.....
United Kingdom	635	11,041	.....
United States	.....	18,631	805
Chemicals, herbs, roots, etc., used in dyeing	225,908	83,224	109,724
Cochineal	2,987	126	.....
Dyewood extracts	206,536	454,787	1,325,637
Indigo	12,493	4,266	6,665
Germany	8,670	4,156	6,665
Explosives:			
Gunpowder, smokeless, including ballistite	1,243	17,884	.....
Other; dynamite and other explosives	133,092	719,493	205,851
Fireworks	21,449	17,008	19,361
Matches of wax, stearin, etc	(1)	24	.....
Other	(1)	3,988	6,890
Miners' fuses	112,590	109,225	262,172
Friction tinder boxes	(1)	24	55
Glycerol	941,306	1,262,378	324,628
Germany	216,449	.....	.....
Netherlands	380,163	936,157	225,535
Sweden	.....	111,945	54,077
United Kingdom	328,672	211,921	45,012
Glycerol dip	2,116	3,307	.....
Hydroxides, n. e. s	(1)	8,792	23,391
Insect powder	60,803	92,022	115,020
Magnesium chloride	(1)	700,785	992,907
Oxides, n. e. s	(1)	743,018	202,580
Oxygen	9,694	710	134
Perfumery and cosmetics:			
Creams and paste, solid or liquid	24,230	16,034	11,041
Fumigating materials	37	.....	342
Hair oil	2,050	1,669	2,560
Pomatums and cosmetics	18,371	28,911	26,118
Toilet waters, alcoholic	20,161	32,262	32,132
Non-alcoholic	1,775	12,110	9,156
Phosphate of lime (for cattle feed)	462,133	77,18	560,415
Phosphorus	67,251	73,753	40,977
Potash	1,262,089	2,128,410	1,238,976
Germany	953,842	1,910,418	835,993
Sweden	154	32,584	11,354
United Kingdom	300,796	131,990	.....
United States	.....	53,418	391,430
Caustic; potash lye	2,472,350	3,237,252	2,688,510
Potassium chlorate	820,552	2,139,720	23,589
Chloride	(1)	1,118,434	701,692
Nitrate	1,151,771	20,811	.....
Germany	1,133,606	.....	.....
United Kingdom	12,059	.....	.....
United States	.....	20,811	.....
Rat poison	10,538	7,987	12,762
Rennet; cheese coloring	85,165	92,096	75,923
Salt prepared for medical use	34,917	7,030	1,296
Salts of metals, n. e. s	(1)	6,806	1,120
IMPORTS OF CHEMICALS AND ALLIED PRODUCTS (*Continued*)	1913 Pounds	1916 Pounds	1917 Pounds
CHEMICALS, DRUGS, ETC. (*Concluded*):			
Soda, calcined	36,161,414	57,011,948	61,174,100
Austria-Hungary	.....	8,158,321	.....
Belgium	11,454,109	.....	.....
France	21,826	3,322,552	10,999,125
Germany	19,458,041	8,538,107	76,390
Sweden	52,161	228,529	15,630
United Kingdom	5,108,058	36,341,751	43,455,460
United States	.....	359,703	6,627,495
Caustic	3,790,848	4,861,575	3,969,600
Germany	.....	854,000	356,002
United Kingdom	.....	2,968,610	742,025
United States	.....	920,900	2,865,257
Sodium biborate	237,288	1,380,540	304,026
Bicarbonate	325,930	437,591	189,487
Sulfate	24,803,470	28,490,575	13,244,467
Sulfite and other salts	2,296,504	635,196	838,970
Soluble glass	954,548	966,893	878,807
Sulfates of copper, iron, zinc	381,513	1,075,670	490,297
Sulfates of metals, n. e. s	(1)	509,014	480,449
Sulfur	33,974,910	16,010,650	8,974,180
Italy	16,307,327	8,627,745	2,183,280
Spain	4,288,144	540,132	.....
United Kingdom	12,016,360	6,734,438	335,014
United States	.....	108,335	6,455,862
Tanning materials:			
Barks and extracts	13,333,796	13,677,670	8,048,239
Argentina	.....	3,611,157	2,614,902
South America, other	.....	1,831,361	.....
East Africa	.....	2,088,043	595,468
France	585,343	5,776	8,818
Germany	7,292,311	23,214	.....
Russia	2,098,382	110,230	478,403
Sweden	1,123,420	399,100	1,208,860
United Kingdom	1,337,266	2,709,520	765,379
United States	51,257	1,960,242	309,440
Catechu and gambier	532,786	1,876,556	638,900
Divi-divi	589,201	631,717	248,108
Gallnuts and valonia	88	.....	.....
Tartar	37,192	75,532	44,033
Tin salts	875	33	159
Fertilizers:			
Ammonium sulfate and other salts	19,839,740	16,677,323	21,452,700
Sodium nitrate	2,948,211	14,166,208	98,757
Cyanamide	.....	10,653,705	7,272,168
Kainite and other potash salts	46,541,553	26,123,121	25,138,310
Germany	46,138,800	22,781,388	25,138,310
Portugal and Madeira	.....	2,909,852	.....
Sweden	.....	420,858	.....
Superphosphates	9,683,860	24,566,960	82,524,595
France	.....	756,178	.....
Portugal and Madeira	.....	23,810,716	.....
United States	.....	.....	73,555,080
Thomas phosphate	76,622,686	18,414,792	.....
Portugal and Madeira	.....	4,589,803	.....
United Kingdom	18,726,393	13,636,934	.....
Belgium	38,455,625	.....	.....
Other fertilizers	4,011,093	711,138	887,735
PAINTS, PIGMENTS, COLORS, VARNISHES:			
Black: bone black, lampblack, graphite, pot-lead	1,065,074	972,459	1,163,512
Blacking, shoe and stove polish, leather creams	281,691	293,205	254,934
Chalk, ground; baryta, china clay	32,138,497	37,580,367	17,678,007
Denmark	.....	.....	8,678,011
Germany	.....	.....	3,464,211
Sweden	.....	.....	3,912,015
United Kingdom	.....	.....	309,595
Ink, liquid	89,851	63,107	53,852
Powder; India ink	4,588	8,896	7,915
Printers', of all kinds	312,226	279,933	108,855
Lead, red	992,665	1,253,756	583,564
White	193,013	177,735	230,163
Ocher, amber, English red	3,877,578	3,337,930	2,730,160
Paint boxes; paints in tubes, vases	26,468	33,860	33,014
Paints ground in oil	206,617	288,760	194,205
Compounded with tar, including paint for ships' bottoms	299,762	396,453	330,274
Germany	.....	62,280	2,183
United Kingdom	.....	313,032	325,579
United States	.....	2,227	375
Paints, liquid, n. e. s	67,941	22,754	33,107
Mixed, in packets	34,586	78,484	29,723
Sugar coloring for brandy	8,768	.....	3,241
Varnishes containing alcohol	52,714	37,716	11,360
Other	820,949	585,520	454,902
Zinc white and dutiable dry paint	5,322,213	5,990,998	5,514,162
Belgium	280,271	11,486	.....
Germany	3,576,126	5,576,076	4,436,547
Netherlands	1,370,907	214,773	12,932
Sweden	7,121	34,577	1,016,765
United States	110	8,516	33,676
SOAP AND CANDLES:			
Candles, stearin and tallow	69,685	9,015	60
Wax	6,537	1,327	1,043
Soap, green; unscented soap powder	119,981	70,946	94,545
Sweden	85,212	12,588	.....
United Kingdom	13,754	53,492	72,298
United States	4,978	4,729	21,890
Transparent, or perfumed	87,476	105,340	181,273
Germany	38,773	1,294	240
United Kingdom	35,078	70,437	134,634
United States	2,293	18,585	36,980

[1] Not shown separately.

[2] Under "fertilizer" in 1913.

IMPORTS OF CHEMICALS AND ALLIED PRODUCTS (*Continued*)

	1913 Pounds	1916 Pounds	1917 Pounds
SOAP AND CANDLES (*Concluded*):			
Other	1,358,590	1,024,967	2,645,027
Germany	29,160	.....	.....
United Kingdom	1,296,373	891,761	1,442,467
United States	3,997	125,391	1,198,012
OILSEEDS:			
Copra	9,711,714	17,239,044	10,939,250
Linseed	32,510,186	27,542,437	13,385,652
Argentina	17,219,290	11,438,926	11,461,633
South America, other	5,070,741	.....	.....
British India	.....	7,009,795	1,922,430
Russia	4,791,526	.....	.....
United States	2,889,245	5,643,370	.....
Rapeseed	2,124,572	236,800	655,125
Belgium	2,003,318	.....	.....
British India	.....	.....	651,774
United Kingdom	.....	236,337	.....
OILS, FATS AND WAXES:			
Fatty oils:			
Butter coloring	41,048	73,940	57,609
Castor oil, resin oil, and other fatty oils, n. e. s	7,314,694	4,255,075	14,358,088
Denmark	410,369	51,080	9,155,312
France	934,981	1,527,230	1,672,559
Germany	2,963,850	.....	.....
Netherlands	2,341,176	674,085	57,232
United Kingdom	182,388	640,928	3,064,668
United States	302,253	1,319,290	394,760
Coconut oil	8,016,866	5,974,902	2,182,069
Asia, n. e. s	.....	1,937,950	1,755,760
British India	5,002,177	1,865,110	.....
France	453,403	547,562	265,965
Germany	1,208,022	.....	.....
United States	93,652	339,798	32,870
Corn oil	30,336	76,787	.....
Cottonseed oil	11,563,817	23,680,157	27,433,308
Brazil	.....	9,634	356,046
Germany	572,981	.....	.....
Netherlands	2,383,611	304,567	.....
United Kingdom	1,572,182	564,736	59,304
United States	6,842,860	22,744,493	26,557,742
Hempseed oil	31,570	3,042	31,195
Linseed oil, raw	1,409,547	1,446,717	2,830,360
Boiled	1,918,109	851,270	1,262,345
Netherlands	497,407	73,193	159,593
United Kingdom	1,399,494	762,910	336,557
United States	.....	11,640	766,195
Olive oil	2,408,439	7,392,606	3,863,050
France	1,011,878	239,576	71,496
Italy	701,643	48,060	2,072
Spain	569,873	7,080,167	3,723,300
Palm oil	174,341	107,740	211,357
Rapeseed oil	482,592	210,078	195,991
Sperm oil, olein, lard oil	578,934	340,702	1,234,897
Belgium	65,323	.....	.....
Germany	128,816	.....	.....
Netherlands	301,482	18,342	.....
United Kingdom	38,251	257,853	825,036
United States	.....	35,120	323,572
Whale oil, cod liver oil, fish oil	4,607,727	3,301,245	7,720,124
Linseed oil, raw; rapeseed oil, palm oil	2,066,480	1,764,535	3,237,710
Germany	528,823	882	.....
Netherlands	539,714	1,587	523,664
United Kingdom	794,965	1,568,875	910,818
United States	375	40,300	1,803,228
Volatile oils:			
Turpentine, juniper, birch oils	362,833	597,667	298,528
Other	25,693	33,272	32,392
Blubber, cod liver, etc	.....	13,073	992
Lye of fat or train oil; degras	937,560	218,720	181,396
Stearin, palmitin, paraffin, spermaceti	1,628,670	4,553,665	1,372,964
Germany	499,305	82,487	.....
Netherlands	273,635	56,720	.....
United Kingdom	759,374	3,648,858	642,564
United States	3,770	743,554	729,706
Tallow, oleomargarine and other greases	18,237,497	26,127,486	24,843,734
Argentina	227,054	3,010,985	3,846,603
Germany	2,357,270	2,359	1,124
Netherlands	1,357,562	327,739	.....
United Kingdom	2,410,666	2,252,838	349,100
United States	8,730,040	19,895,196	18,259,542
Wax	82,500	740,826	85,504
ORES:	Metric tons	Metric tons	Metric tons
Aluminum oxide, bauxite, cryolite	4,363	12,071	10,291
Chrome ore	.....	2,897	4,677
Nickel ore	569	.....	.....
Pyrite	.....	600	.....
Pyrite slag	.....	323	.....
Zinc ore	.....	.....	330
VARIOUS PRODUCTS:	Pounds	Pounds	Pounds
Glass	9,766,930	13,984,685	29,613,365
Glue:			
Capsules of gelatin, filled	505	258	298
Isinglass and gelatin in sheets	37,811	31,561	10,880
Liquid glue, etc	43,930	38,620	32,205
Other	492,450	438,973	475,920
Sizing	643,443	459,880	220,855
Paper, except printed matter	18,058,300	25,054,937	31,267,910
Pitch and coal pitch	1,463,160	3,078,050	1,664,755
Tar:			
Coal tar	2,210,905	1,857,504	871,245
VARIOUS PRODUCTS (*Concluded*):			
Tar (*Concluded*):			
Wood and peat tar	4,124,320	5,848,775	2,449,200
Creosote, creosote oil, raw phenol	5,511,620	3,222,894	1,961,188
Turpentine	1,373	1,204	3,461
Rosin and similar gums	8,103,970	11,074,500	2,053,958
Woodpulp, straw pulp	69,132	91,853	54,103

EXPORT TRADE

The extent to which the Norwegian chemical industries have exceeded the needs of the home market in certain specialties is made evident in the next table, compiled from figures published by the Central Bureau of Statistics:

EXPORTS OF CHEMICALS AND ALLIED PRODUCTS

	1913 Pounds	1916 Pounds	1917 Pounds
CHEMICALS:			
Acids:			
Caprylic	120,364	4,493	.....
Carbonic	.....	4,980	.....
Nitric	4,630	8,887,340	3,575,170
Oxalic	1,220,030	960,505	736,533
France	.....	278,590	97,074
Japan	.....	139,255	148,428
United Kingdom	134,938	29,388	237,597
United States	1,026,638	379,955	170,089
Sulfuric	507	.....	611,531
Aluminum sulfate	.....	66,216	688,082
Ammonium nitrate	20,078,315	131,481,474	140,165,745
France	.....	74,249,430	102,418,975
Russia	.....	36,815,515	15,300,784
United Kingdom	.....	16,926,780	21,390,790
United States	.....	1,427,427	156,815
Calcium carbide	147,513,146	128,822,470	101,559,524
Australia	29,473,568	7,569,680	782,860
Denmark	1,414,574	2,421,072	8,136,687
Germany	46,023,063	63,952,370	53,763,924
Netherlands	4,903,190	4,320,290	2,249,640
United Kingdom	21,715,543	39,969,690	34,912,727
Casein	249,160	132,487	305,160
Drugs, n. e. s	1,603	12,075	2,046
Explosives:			
Gunpowder and other explosives	38,228	32,337	2,491
Dynamite	4,253	1,292	.....
	Metric tons	Metric tons	Metric tons
Fertilizers:			
Ammonium sulfate	138	.....	50
Cyanamide	22,111	13,152	2,313
Germany	12,188	.....	.....
Netherlands	1,591	1,253	.....
United Kingdom	3,444	10,172	1,320
Norway saltpeter	70,927	46,001	35,932
Denmark	4,512	.....	1,118
France	8,738	1	.....
Germany	45,238	19,805	16,279
Sweden	139	1,130	1,425
United Kingdom	4,874	25,062	17,110
	Pounds	Pounds	Pounds
Glycerol, raw	277,483	309,756	66,615
Iodine, raw	26,784	2,493	2,601
Magnesite, calcined	1,446,232	3,673,120	1,054,603
Matches	12,106,628	11,331,030	8,916,990
British India	3,498,736	1,374,030	965,404
Russia	5,790	4,409,840	2,946,940
United Kingdom	3,467,870	3,405,237	185,607
United States	2,291,264	1,494,293	3,579,579
Nickel sulfate	.....	11,043	.....
Oxygen	.....	.....	3,142
Phosphorus	.....	.....	37,285
Potassium chlorate	.....	628,364	.....
Rat poison	.....	11,840	17,857
Salt of lemons (bioxalate of potassium)	90,072	62,333	64,534
Silicon carbide, durubit, etc	3,638	2,196,355	1,485,520
Russia	.....	457,690	76,170
Sweden	.....	1,534,395	1,122,946
United Kingdom	.....	44,820	211,798
Soda crystals (washing soda)	1,262,080	905,900	.....
Sodium bisulfate	3,760,149	551,156	.....
Chlorate	.....	27,550	174,665
Nitrate	2,483,000	32,590,820	50,069,622
France	.....	.....	5,236,573
Sweden	.....	2,969,956	2,300,413
United Kingdom	.....	29,143,233	42,498,947
United States	.....	170,615	.....
Nitrinitrate	4,448,910	14,198	.....
Nitrite	18,085,920	13,468,633	7,795,743
Germany	2,829,774	6,806,154	662,864
Netherlands	13,561,570	65,852	15,917
United Kingdom	699,313	3,295,360	5,774,347
United States	721,273	3,054,813	1,196,338
Peroxide	(1)	53,694	48,127
Soluble glass	202,023	34,260	.....
Tanning materials:			
Oak bark	114,640	836,826	.....
Other barks	24,612	27,370	.....

(1) Not shown separately.

Exports of Chemicals and Allied Products (*Continued*)

	1913 Pounds	1916 Pounds	1917 Pounds
Paints, Pigments, Etc.:			
Copper paints for ships' bottoms	527,068	28,984	.....
Paints ground in oil	25,850	67,228	23,931
Zinc white and white lead	106	676,334	354,944
Other paints and colors	75,422	1,453	1,576
Varnishes	46	538	.....
Oils, Fats and Waxes:			
Coconut oil	(1)	2,119,720	.....
Linseed oil, rapeseed oil	25,413	10,211	606
Train oil, gallons	7,086,670	5,397,964	1,003,690
Austria-Hungary	44,804	128,232	.....
Germany	3,574,378	3,230,420	585,708
Netherlands	931,411	7,582	238
Russia	56,323	90,428	206,323
United Kingdom	813,826	878,681	115,763
United States	260,664	81,552	78,038
Turpentine oil	.....	.....	19,758
Vegetable oils, solid	53,215	690,190	.....
Bone fat	535,975	660,152	191,780
Blubber	53,398	8,201	.....
Stearin, gallons	175,600	24,991	14,054
Germany	100,335	23,961	3,963
Sweden	18,149	53	.....
United Kingdom	23,538	.....	10,091
Tallow	212,680	852,849	.....
Whale fat, solid	14,386,000	13,823,400	440
Soap and Candles:			
Soap	40,785	336,368	1,170
Germany	.....	309,392	33
Russia	5,534	14,270	789
Sweden	19,484	10,988	326
Stearin candles	584	1,730	904
	Metric tons	Metric tons	Metric tons
Ores:			
Bismuth ore and other ores, n. e. s.	.....	10	.....
Chrome ore	20	2,737	2,395
Copper ore	74	2,175	809
Copper and nickel ore	.....	.....	.....
Iron ore and concentrates	373,071	187,805	150,960
Germany	189,596	152,454	135,814
Netherlands	90,345	.....	.....
United Kingdom	92,827	35,086	9,956
Briquettes	195,692	216,896	46,875
Germany	74,861	.....	.....
Sweden	2,512	.....	.....
United Kingdom	118,319	216,896	46,875
Lead ore	35	10	.....
Manganese ore	.....	22	.....
Molybdenum ore	5	141	201
Nickel ore	.....	.....	.....
Pyrite (iron and copper)	425,876	253,362	212,909
Belgium	26,251	.....	.....
Germany	40,892	84,511	4,105
Netherlands	46,773	10,145	8,914
Sweden	89,518	115,488	101,554
United Kingdom	138,134	21,844	50,770
Slag (rich in copper)	35,035	47,523	41,367
Belgium	399	.....	.....

[1] Not shown separately.

Exports of Chemicals and Allied Products (*Concluded*)

Articles	1913 Pounds	1916 Pounds	1917 Pounds
Ores (*Concluded*):			
Pyrite slag (*Concluded*):			
Germany	2,974	4,031	3,925
Sweden	31,662	43,492	37,307
Rutile	51	82	114
Wolfram	.....	.....	2
Zinc ore	285	1	540
Paper:			
Pounds	410,976,947	453,177,497	185,718,270
Value	$ 8,642,491	$15,601,272	$13,122,674
France	$7,826	$ 2,338,059	$615,944
Germany	$542,352	$25,380	$9,300
United Kingdom	$ 4,926,644	$ 6,090,809	$ 5,009,081
Australia	$618,356	$ 1,969,184	$ 1,945,439
British India	$191,406	$834,472	$ 1,303,257
China	$929,370	$762,808	$106,101
South America	$542,807	$ 1,699,120	$ 1,408,956
Pulp and Cellulose:	Metric tons	Metric tons	Metric tons
Cellulose, dry	209,544	216,800	133,564
Argentina	8,832	1,450	25
Belgium	12,064	.....	.....
France	22,803	32,839	6,905
Germany	7,613	.....	.....
Italy	3,666	8,111	3,206
Japan	5,124	8,923	.....
Mexico	3,344	51	.....
Netherlands	5,604	4,640	1,866
Spain	2,929	2,957	158
Sweden	2,952	3,966	1,260
United Kingdom	49,175	109,430	98,640
United States	77,777	40,331	19,855
Wet	1,634	5,540	3,169
Belgium	611	.....	.....
France	267	1,211	.....
United Kingdom	510	3,949	2,899
United States	.....	.....	30
Woodpulp, dry	14,661	12,962	17,319
Argentina	3,585	680	102
France	4,133	5,559	2,786
Spain	1,505	2,728	695
United Kingdom	1,608	1,303	11,195
United States	1,672	207	1,489
Wet	481,080	455,522	250,099
Belgium	46,992	.....	.....
Denmark	19,773	20,416	9,748
France	95,730	105,470	57,035
Netherlands	21,163	14,316	10,862
Spain	41	7,707	3,498
United Kingdom	295,210	302,418	148,861
United States	184	4,823	19,881
Various Products:			
Glass	1,366,605	3,522,987	2,415,200
Glue	453,896	198,872	51,194
Pitch	549,718	606,644	251,569
Tar:			
Coal	6,392,898	1,394,732	1,064,877
Wood	106,854	119,975	21,605
Tar oil	9,057	20,948	.....
Vinegar and acetic acid	626	77	.....
Leather cream	1,433	109,188	9,085

ORIGINAL PAPERS

SOME CATALYSTS WHICH PROMOTE REACTION BETWEEN ANILINE AND ETHYL ALCOHOL[1]

By T. B. Johnson, A. J. Hill and J. J. Donleavy

Department of Chemistry, Yale University, New Haven, Conn.

Received February 24, 1920

The facts and experimental data recorded in this publication have been obtained as the result of a preliminary research dealing with the interaction of ethyl alcohol and aniline hydrochloride. Our main object has been to obtain information leading to a better knowledge of the experimental conditions governing the formation of diethylaniline by direct alkylation with alcohol. The details of our investigation and the results obtained are discussed in the experimental part of the paper.

It has been known for several years that aromatic amines, of which aniline is the prototype, can undergo reactions with aliphatic alcohols if these reagents are heated together at high temperatures and under pressure. They interact to give products of two different types, namely, nitrogen substituted amines like monomethyl- and dimethylaniline, which are prepared commercially in large quantities, or true carbon derivatives resulting from substitution of the alkyl group in the aromatic nucleus of the amine. Para-ethylaniline is a representative of this second type of compound.[1] The molecular changes productive of these different compounds can be brought about by applying the alkylation reaction to a salt of the aromatic amine like aniline hydrochloride,[2] and also by heating the free amine with an alcohol in the presence of a strong dehydrating agent like zinc chloride,[3] or of certain catalysts which have been shown to promote

---

[1] Researches on Amines, VII. The previous papers of this series have been published in the *Journal of the American Chemical Society*. Paper VI, *J. Am. Chem. Soc.*, **38** (1916), 2507.

[1] Hoffmann, *Ber.*, **5** (1872), 720; **7** (1874), 527; **13**, 1729; **15** (1882), 1011, 1646, 2895; Pictet and Bunzl, *Ibid.*, **22** (1889), 1847; Studer, *Ann.*, **211** (1882), 237; Senkowski, *Ber.*, **24** (1891), 2975; Anschutz and Beckerhoff, *Ibid.*, **28** (1895), 407; Willgerodt and Bergdolt, *Ann.*, **327** (1903), 286; Merz and Weith, *Ber.*, **14** (1881), 2346; Calm, *Ibid.*, **15** (1882), 1642; Louis, *Ibid.*, **16** (1883), 105, 116; **17** (1884), 1221; **21** (1888), 1159; Romburgh, *Rec. trav. chim.*, **3** (1884), 392; Sampaio, *Ber.*, **14** (1881), 2172; Benz, *Ibid.*, **15** (1882), 1646.

[2] Staedel, *Ber.*, **16** (1883), 30.

[3] Willgerodt and Bergdolt, *Loc. cit.*

such transformations.[1] The most important factors which regulate the course of the reaction with a given amine and alcohol are, apparently, the catalyst, and the temperature employed to bring about the change.

While the operation of alkylating aniline directly with methyl alcohol to dimethylaniline has been developed to a high degree of efficiency, and is applied successfully on a very large scale commercially by heating the sulfate or hydrochloride of the amine with wood alcohol, the application of a similar reaction with amines and the higher aliphatic alcohols apparently has not been very successfully applied. In the case of aniline and ethyl alcohol, which is of particular interest to us at this time, Walther[2] writes as follows: "On the contrary, diethylaniline cannot be prepared with sulfuric acid." On the other hand, Staedel[3] claims to have obtained this tertiary amine in a nearly theoretical yield by heating the hydrobromide or hydriodide of aniline with ethyl alcohol at 150° C. Schultz and Julius[4] state that the same change can be brought about by using aniline hydrochloride for the reaction, but the product is less pure and contains a considerable amount of monoethylaniline.

There is apparently a definite temperature for each amine-alcohol mixture, above which it is impossible to obtain nitrogen alkylation if the reactions are applied in acid solution or in the presence of zinc chloride. This temperature reaches its maximum when alkylating with the lowest alcohol, namely, methyl alcohol. Application of the reaction at temperatures above this maximum leads to the formation of nucleus-substituted amines due to rearrangement, and it seems to be true that the tendency to rearrange from nitrogen to carbon is influenced by the size of the alkyl group of the alcohol. In other words, the larger the alkyl group of the alcohol the greater the tendency to rearrange in acid solution, and consequently the lower the temperature at which the formation of tertiary amines can be accomplished successfully.[5] These rearrangements have been the subject of much investigation, especially in the case of the action of methyl alcohol on aniline and its homologs.[6] The migrations in the majority of cases so far carefully examined are generally brought about by applying the reactions at temperatures above 250° C.

A careful review of the major researches on amine alkylation by heating with alcohols reveals the interesting fact that the investigator hitherto has generally proceeded to apply his reaction at a high temperature, namely, in the range of 250° to 300° C., or in some cases at much higher temperatures. The result has been that the product of his reaction has always been a nucleus-substituted compound, and in some cases the temperature has been sufficiently high to actually alter the constitution of the aliphatic group of the alcohol used. It seems also to have been quite general practice, when heating an amine directly with an alcohol, to introduce zinc chloride to accelerate the alkylation. This apparently promotes the splitting off of water and leads to the same results as are obtained by heating the salts of the amines with alcohol. In the case of reactions involving the use of methyl alcohol, which has received the most attention, the experimental data obtained indicate that temperatures below 230° C. are favorable for the formation of nitrogen derivatives. In the case of the higher alcohol homologs the optimum temperature for successful nitrogen alkylation in acid solution will undoubtedly be found to be lower than 230° C. The results obtained by Niementowski,[1] who investigated the behavior of aniline hydrochloride towards ethyl formate and ethyl acetate at 225° C., are in conformity with this statement.

If we confine our attention to the chemistry of aniline it appears, therefore, that any experimental conditions which favor the formation of alkyl halides as intermediate products of the reaction will lead to the production of nucleus-substituted amines if the alkylation reaction is applied at a higher temperature than 250° C. On the other hand, when experimental conditions are adopted which exclude the possibility of intermediate alkyl halide formation, it is possible to heat aniline with alcohols at much higher temperatures than 250° to 300° C. and obtain easily nitrogen derivatives without rearrangement of the alkyl group to the benzene nucleus. For example, Mailhe and de Gordon[2] have actually shown that mixtures of methyl alcohol and aniline vapors, when passed over thorium oxide, aluminum oxide or zirconium oxide at 400° to 500° C., interact smoothly to give mono- and dimethylaniline. They state that aluminum oxide is the best catalyst of the three oxides. In other words, under these conditions aniline interacts with alcohol in a manner similar to that of the corresponding reduced amine, hexaphenylamine, which was investigated by Sabatier and Mailhe.[3]

In this connection it is of special interest to call attention to the interesting observations made by by Knoevenagel[4] who has shown that the presence of traces of iodine promotes the reaction between aromatic amines and alcohols with formation of nitrogen alkyl derivatives. He obtained, for example, excellent yields of dimethyl- and diethylaniline by heating aniline with methyl and ethyl alcohol, respectively, in the presence of traces of iodine at 200° to 230° C. Not only did he apply his reaction successfully with aniline and amyl alcohol under these conditions, but he also showed that α- and β-naphthylamines behaved in a manner similar to aniline. In other words, no rearrangement of alkyl from nitrogen to carbon was observed in any case examined. These interesting observations of Mailhe and Knoevenagel suggest that quaternary nitrogen combinations functionate as intermediate products in the molecular rearrangements of secondary and tertiary aromatic amines into their isomeric carbon-substituted compounds.

In the light of these results of Knoevenagel it seemed

---

[1] Knoevenagel, *J. prakt. Chem.*, **89** (1914), 31.

[2] *Chem.-Ztg.*, **34**, 641.

[3] Staedel and Reinhardt, *Ber.*, **16** (1883), 30.

[4] "Organic Coloring Matters," p. 25.

[5] Niementowski, *Ber.*, **30** (1897), 3071.

[6] Hoffmann, *Loc. cit.*

[1] *Loc. cit.*

[2] *Compt. rend.*, **166** (1918), 467.

[3] *Ibid.*, **148** (1909), 898; **153** (1911), 621.

[4] *Loc. cit.*

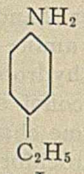
probable to us that the reaction between aniline hydrochloride and ethyl alcohol might be stimulated by the introduction of certain reagents. So far as we are aware, no attention has been paid to the question of catalytic influences in such reactions. A research in this field seemed, therefore, very desirable both from a theoretical and a practical standpoint. The successful development of a method of operating which would be productive of diethylaniline in good yield, without recourse to ethyl bromide or ethyl chloride for alkylation, would be of the greatest practical interest. The results of our preliminary work on this problem are recorded in the following experimental part. The investigation is being continued and in our next paper we shall discuss experimental data obtained by working with the higher homologs of aniline.

EXPERIMENTAL

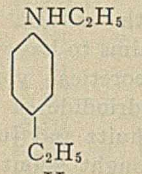
The experiments described in this paper were carried out in an iron autoclave of 1.7 liters capacity. This was constructed of heavy iron pipe and was furnished with a standard pressure gauge, thermometer well, and a glass or copper inset to avoid corrosion. The charge was heated by suspension of the autoclave in a bath of cottonseed oil. The procedure which was followed in all the experiments herein described was as follows: After the completion of the heating period the autoclave was allowed to cool and opened. The contents were transferred to a distilling flask and the excess of alcohol removed by heating at 100° C. under diminished pressure. A large part of the excess alcohol could thus be profitably recovered. The residual oil was then made strongly alkaline with sodium hydroxide in order to liberate the aromatic bases from their hydrochlorides, and steam-distilled. The amines were separated from the aqueous distillate by extraction with ether and the ether solution dried over fused sodium sulfate. After removal of the ether the oil was fractionally distilled at atmospheric pressure. The correct boiling point of monoethylaniline is 204° C. and that of diethylaniline is 215.5° C., at 760 mm.

During the preliminary experiments, and before the degree of alkylation of the aniline was observed to be notably raised by catalytic influence, a tolerably good idea of the success of any given experiment was gained by noting the range of distillation of the crude oil. We found, however, that a boiling point was no criterion of purity when oils containing 25 per cent and less of monoethylaniline were distilled. Constant boiling mixtures were always formed and it was absolutely necessary to analyze such oils.

The method which was used for the estimation of primary and secondary amines involved the use of acetic anhydride as a reagent. In brief this method is as follows: About one gram of the oil is weighed into a small flask having a ground glass neck. An equal, carefully weighed amount of acetic anhydride is added, and the flask, after being attached to a condenser provided with a ground glass connection, is heated for 30 min. on the water bath. Fifty cc. of distilled water are then carefully added through the condenser tube, and the mixture heated for 30 min. to hydrolyze the excess of acetic anhydride. After careful dilution in a graduated flask of convenient capacity an aliquot part is titrated with standard alkali solution in order to determine the excess of acetic anhydride. A blank experiment run under similar conditions standardizes the latter. The difference between the two analytical results is a measure of the acetylizable impurity in the oil. This is then calculated in terms of monoethylaniline. The aromatic bases which theoretically must be considered here together with diethylaniline are unaltered aniline, monoethylaniline, and nucleus-substituted amines such as ethylaniline and its monoethyl derivatives represented by Formulas I and II, respectively.



I



II

The proportion of these nucleus-substituted bases is increased by heating aniline with ethyl alcohol at high temperatures.

ACTION OF ETHYL ALCOHOL ON ANILINE IN THE PRESENCE OF CALCIUM OXIDE—Theoretically, the simplest procedure for the ethylation of aniline is that involving the interaction of this amine with alcohol. This reaction is typified by the equation

$$C_6H_5NH_2 + 2C_2H_5OH = C_6H_5N(C_2H_5)_2 + 2H_2O$$

and theoretically should be facilitated by the addition of a reagent capable of combining with water. So far as the writers are aware only one investigator has examined the influence of a basic oxide like calcium oxide on the activity of these two reagents. Calm[1] tried to bring about a reaction between amyl alcohol and aniline by heating these reagents in the presence of calcium oxide and also calcium chloride but reports that no change was effected by their presence. We have repeated this experiment with ethyl alcohol and our first set of preliminary experiments was therefore made with this oxide present as the dehydrating agent.

The autoclave charge was as follows:

Aniline	72 g.
Alcohol[2]	180 g.
Calcium oxide	100 g.
Time of heating	8 hrs.

Four runs were made at different temperatures and the results are recorded in Table I. Autoclave pressures were obtained as high as 500 and 700 lbs., but in no case was alkylation obtained under the conditions

TABLE I

No.	Aniline G.	Alcohol G.	Lime G.	Temperature ° C.	Time Hrs.	Product
1	72	180	100	150	8	Aniline
2	72	180	100	175	8	Aniline
3	72	180	100	200	8	Aniline
4	72	180	100	220–230	8	Aniline
5	72	180	No lime 10 g. KI and 2 g. iodine	150	8	Aniline

---

[1] *Ber.*, **15** (1882), 1642.

[2] The alcohol used in all the experiments described in this paper contained about 1 per cent or less of water. It was prepared from ordinary 95 per cent alcohol by prolonged digestion with an excess of quicklime, until the desired gravity was attained.

described. In Expt. 5 potassium iodide and iodine were substituted as catalytic agents in place of calcium oxide and the mixture heated at a temperature of 150° C., but in this case also we obtained no evidence of the formation of alkylated derivatives.

ACTION OF ETHYL ALCOHOL ON ANILINE HYDROCHLORIDE—In the light of the results obtained by previous investigators, who have examined the behavior of ethyl alcohol toward aniline oil, we decided to confine our work to a study of the action of aniline hydrochloride on ethyl alcohol. Here we have a condition favorable for the production of ethyl chloride which is known to react with aniline with formation of alkyl derivatives. Aniline hydrobromide is far more reactive than the hydrochloride but is too expensive a reagent to use in manufacturing operations, while alkylation of aniline in the presence of sulfuric acid is not practical, due to the tendency of alcohol to undergo dehydration in the presence of this reagent giving ether and ethylene.[1]

While the question of alkylation of aniline by interaction of the halide salts of this amine with various alcohols has received much attention, no really satisfactory results have been obtained with ethyl alcohol, if one is to draw conclusion from what has been published in the chemical literature, as only generalized statements are available. The most productive results are those described in a publication by Staedel,[2] and which were obtained by application of a process patented by him.[3] He states that diethylaniline may be obtained in nearly quantitative yields by heating aniline hydrobromide or the corresponding iodide with a slight excess over two molecular proportions of ethyl alcohol at a temperature of 145° to 150° C. No description of a systematic series of alkylation experiments is recorded.

Concerning the use of aniline hydrochloride, only rather generalized statements are available in the literature in so far as they pertain to the synthesis of diethylaniline. For example, Schultz and Julius[4] state that diethylaniline is obtained by heating aniline hydrochloride and aniline with rather more than two moles of ethyl alcohol. The product contains a considerable quantity of monoethylaniline and in order to obtain the diethyl compound it is usually treated with sufficient ethyl bromide to complete the reaction. According to them this base is obtained more readily and in nearly theoretical yield by heating aniline hydrobromide with 2.2 moles of ethyl alcohol at 145° to 150° C.

In respect to the preparation of monoethylaniline these authors state that it is obtained by heating aniline hydrochloride or a mixture of aniline and hydrochloric acid with rather more than one mole of ethyl alcohol at 200° C. Cain[5] states the conditions for the preparation of mono- and diethylaniline as follows: For monoethylaniline, 95 parts of aniline hydrochloride and 28 parts of ethyl alcohol are heated together in an enameled autoclave at 180° C. for some hours. The product contains 70 to 73 per cent of monoethylaniline which crystallizes out as the hydrochloride. Diethylaniline may be prepared like monoethylaniline using aniline hydrochloride and ethyl alcohol. The product, however, contains a considerable amount of monoethylaniline.

In our experiments with aniline hydrochloride we first operated at the temperature recommended by Staedel in his work on the corresponding hydrobromide and hydriodide, namely, 150°. We applied the reaction under various conditions, using in some cases zinc chloride and calcium chloride as dehydrating agents, and also incorporating several reagents which we believed would exert a catalytic influence. It was found that the nature of the product was very much influenced by the presence of certain reagents. The results of our preliminary experiments are recorded in Table II.

TABLE II—INTERACTION OF ANILINE HYDROCHLORIDE AND ETHYL ALCOHOL

100 g. of aniline hydrochloride used in each experiment
Temperature, 150°
Time of heating, 8 hrs.
When $ZnCl_2$, $CaCl_2$, NaBr or KI were introduced as catalysts, 10 g. were always used

Expt.	$ZnCl_2$ or $CaCl_2$ 10 G.	NaBr or KI 10 G.	Moles of Alcohol	Special Catalyst	Total Yield	Distillate								
						195–200	200–204	204–206	206–208	208–210	210–214	214–216	216–218	218–220
1	...	...	2.2	....	87	..	24	24	27	9	..	..	..	..
2	...	...	5	....	87	40	26	8	6	..	..	..	..	..
3	...	NaBr	5	....	96	7	38	26	12	2	..	..	..	..
4	...	KI	5	....	96	6	37	26	14	2	..	..	..	..
5	$CaCl_2$	KI	5	....	..	..	..	72	5	..	..	..	..	..
6	$ZnCl_2$	KI	5	....	..	..	..	75	..	..	..	..	..	..
7	$ZnCl_2$	...	5	....	..	..	70	..	..	..	..	..	..	..
8	$ZnCl_2$	KI	10	....	..	..	..	..	73	..	14	..	..	..
9	$ZnCl_2$	KI	5	Copper lining	92	..	..	..	..	..	65	..	..	..
10	$ZnCl_2$	KI	5	Copper lining	93	..	..	..	..	..	70	..	..	..
11	$ZnCl_2$	KI	5	Copper strips	..	..	80	..	..	..	..	..	..	..
12	$ZnCl_2$	KI	5	5 g. CuO	..	..	63	..	..	..	17	..	..	..
13	$ZnCl_2$	KI	5	5 g. CuO + 8 g. HCl	..	..	65	..	19	..	..	..	..	..
14	$ZnCl_2$	KI	5	5 g. CuCl	..	42	3	..	..	..	..	..	..	..
15	$ZnCl_2$	KI	5	5 g. CuCl + 8 g. HCl	..	..	72	..	..	..	..	..	..	..
16	$ZnCl_2$	KI	5	3 g. $(C_6H_5)_2NH$	..	..	88	..	..	..	10	..	..	..
17	$ZnCl_2$	KI	5	5 g. $CuCl_2$	..	..	..	..	2	..	93	..	..	..
18	$ZnCl_2$	KI	5	5 g. $CuCl_2$	..	..	..	4	..	..	91	..	..	..
19	$ZnCl_2$	KI	5	5 g. $CuCl_2$ + 8 g. HCl	..	..	78	..	8	..	..	..	..	..
20	...	KI	5	5 g. $CuCl_2$	..	..	38	..	8	..	..	..	..	..
21	$ZnCl_2$	...	5	5 g. $CuCl_2$	..	..	..	..	16	..	46	..	..	..
22	...	...	5	5 g. $CuCl_2$	..	..	..	80	11	..	..	..	..	..

Expt. 1,[1] Table II, shows the result of substituting aniline hydrochloride for aniline hydrobromide under the conditions used by Staedel in the case of the latter salt. A theoretical yield of monoethylaniline would have been 92 g. Our product was very largely monoethylaniline. Nine grams of material boiling at 208° to 210° C. were obtained. Nothing, however, distilled within the range of diethylaniline. It is rather astonishing, in the light of Staedel's work, that diethylaniline was not more predominant in the reaction mixture. The use of 5 molecules of alcohol (Expt. 2) did not give as good a result. This is in some degree contradictory, for in the presence of a catalyst as well as at higher temperatures an increase of alcohol up to a certain limit is decidedly favorable in its effect.

---

[1] Walther, *Chem.-Ztg.*, **34**, 641.

[2] *Loc. cit.*

[3] D. R. P. 21,241.

[4] *Loc. cit.*

[5] "The Manufacture of Intermediate Products for Dyes," p. 66.

[1] Except in special cases, the writers have considered it unnecessary to record in the tables the results obtained by duplication of their experiments. It should be understood that all experiments have been carefully checked, in particular where abnormalities occur. In Expts. 8, 19, and 20 the fractions indicated under column 206°–208° were collected from 206°–210°, and in Expt. 22 the fraction of 80 g. was collected between 202°–206°.

With the idea of obtaining a static condition similar to that existing when aniline hydrobromide or iodide is employed, 10 g. of sodium bromides (Expt. 3) and a similar amount of potassium iodide (Expt. 4) were introduced into the reaction mixture. An increased total yield was the result. The addition of 10 g. of calcium chloride as a dehydrating agent (Expt. 5) was likewise very favorable and slightly better than the former case, the total yield of monoethylaniline being of the order of 80 g. The substitution of zinc chloride (Expt. 6) for calcium chloride did not materially affect the result, whereas the omission of the iodide or bromide (Expt. 7) materially decreased alkylation. Increasing the alcohol factor to 10 moles (Expt. 8), together with the use of potassium iodide and zinc chloride, gave decidedly the best result yet obtained. For the first time a distillate boiling above 210° C. was obtained.

To summarize, the results of the first eight experiments recorded in Table II reveal the following facts:

1—That aniline hydrochloride when heated with 2.2 molecules of alcohol at 150° C. gives a product consisting largely of monoethylaniline, in contradistinction to Staedel's results, in which the use of aniline hydrobromide (or iodide) gave a product composed essentially of diethylaniline.

2—That the alkali bromides and iodides (NaBr and KI) used in conjunction with the dehydrating effect produced by either calcium or zinc chloride, catalyze to a certain degree the ethylation of aniline.

3—That a large excess of alcohol is favorable for ethylation when used in the presence of the metallic salts.

During the course of our preliminary experiments it became necessary to employ a copper inset or lining in the autoclave, where a glass one had been used hitherto. The first two experiments (9 and 10) carried out with the copper lining gave rather good yields of diethylaniline. Since this lining had previously been used for other operations we had good reason to believe that the catalytic effects produced in our reaction might possibly be attributed to one of the following substances:

1—Metallic copper
2—Hydrochloric acid
3—Cuprous chloride
4—Diphenylamine
5—Cupric chloride

Experiments were therefore designed to establish the identity of the catalyst and were carried out in a glass inset; and a basic charge of 10 g. potassium iodide, 10 g. zinc chloride, 100 g. aniline hydrochloride, and 200 g. alcohol was used, together with the catalytic substance whose effect was sought.

The result recorded in Expt. 11 is typical of those obtained when strips of clean copper were placed in the charge, or when a thoroughly scoured copper inset was used. No catalytic effect similar to that observed in Expts. 9 and 10 was produced. Expt. 12 shows the effect of the addition of 5 g. of cupric oxide. The larger proportion of the reaction mixture was monoethylaniline. Expt. 13 was similar to 12 except in the fact that a little free acid was added (conc. HCl). The yield of monoethylaniline was increased, thus demonstrating the inhibitive effect of free hydrochloric acid on the final alkylation reaction.

A most striking inhibitive action was observed in Expt. 14 when cuprous chloride was employed. No diethylaniline was apparently formed and much unchanged aniline was present; this being evidenced by the fact that the bulk of the distillate boiled below 200° C. Somewhat more alkylation was observed in Expt. 15 when a small quantity of concentrated hydrochloric acid was added to the reaction mixture employed in Expt. 14. Expt. 16 shows the somewhat more favorable effect of diphenylamine as a catalyst. However, the yield of diethylaniline was insignificant.

Expt. 17 demonstrated very clearly the nature of the catalyst in that the addition of 5 g. of cupric chloride gave a most excellent yield of diethylaniline. This experiment was repeated (Expt. 18) and a similar result obtained. It is our conclusion, therefore, from these observations that the results obtained in Expts. 9 and 10 were due to the presence of cupric chloride in the slightly corroded containers used in these operations. The unfavorable effect of moisture, even when cupric chloride was employed, is shown in Expt. 19 where a small amount of concentrated hydrochloric acid was added to the charge.

An endeavor was made to obviate the use of either zinc chloride (Expt. 20) or potassium iodide (Expt. 21) when cupric chloride was used. Neither reagent could be omitted from the charge. However, the experiments did show the greater value of the zinc chloride as a catalyst as compared with the potassium iodide. The omission of both zinc chloride and potassium iodide (Expt. 22) gave no oil distilling within the range for diethylaniline.

The experimental work thus far reviewed established the fact that the autoclave charge when heated for 8 hrs. at a temperature of 150° C. would give a product consisting largely of diethylaniline. The autoclave pressures recorded in all these experiments carried out at 150° C. averaged about 190 lbs.

10 g. of zinc chloride
10 g. of potassium iodide
5 g. of cupric chloride
100 g. of aniline hydrochloride
200 g. of ethyl alcohol (99 per cent)

To summarize, the following substances were found either without appreciable effect, or slightly inhibitive in their action on the alkylation of aniline.

Without Effect	Inhibitive	Positive
Copper	Moisture-free HCl	Cupric chloride
Cupric oxide	Cuprous chloride	Diphenylamine

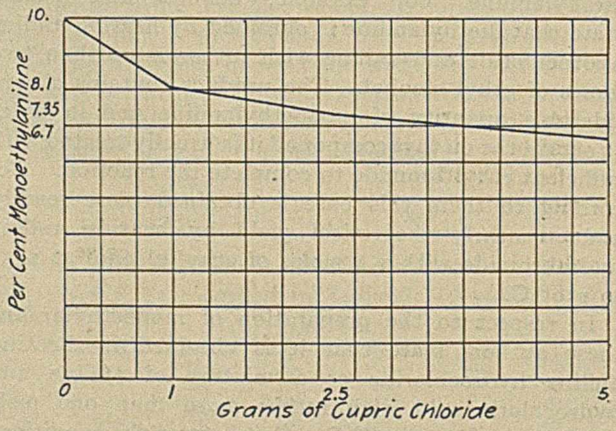


Fig. 1

The next phase of our investigation which received attention was the proper temperature for efficient alkylation. Several experiments were run under the best conditions stated above and it was found that more consistent results, attended with better yields of diethylaniline, could be obtained by working at a constant temperature of 175° to 180° C. instead of 150° C. The results recorded in Expts. 1 and 2 of

TABLE III

Expt.	$C_6H_5NH_2.HCl$. G.	$ZnCl_2$ or $CaCl_2$. G.	NaBr or KI. G.	Moles of Alcohol	Temperature. ° C.	Time	Special Catalyst G. $CuCl_2$	Total Yield	DISTILLATE								Monoethylaniline Per cent
									M. E. A.					D. E. A.			
									195–200	200–202	202–204	204–206	206–210	210–212	212–218	218–220	
1	100	10 $ZnCl_2$	10 KI	5	175–180	8	5	96	..	..	..	..	..	..	88	..	...
2	100	10 $ZnCl_2$	10 KI	5	175–180	8	5	96	..	..	..	..	..	..	88	..	...
3	100	10 $ZnCl_2$	10 NaBr	5	175–180	8	5	99	..	..	..	..	..	..	92	..	...
4	100	10 $CaCl_2$	10 NaBr	5	175–180	8	5	112	..	..	..	..	..	..	93	..	...
5	100	10 $CaCl_2$	10 NaBr	5	175–180	8	5	97	..	..	..	..	..	5	89	..	6.79
6	100	10 $CaCl_2$	10 NaBr	5	175–180	8	None	83	..	..	..	..	2	..	72	..	10.00
7	100	10 $CaCl_2$	10 NaBr	5	175–180	8	2.59	105	..	..	..	..	3	..	95	..	7.35
8	100	10 $CaCl_2$	10 NaBr	5	175–180	8	1	96	..	..	..	..	3	..	88	..	8.10
9	100	10 $CaCl_2$	10 NaBr	8	175–180	8	5	103	..	..	..	..	..	..	90	..	6.70
10	100	10 $CaCl_2$	10 NaBr	10	175–180	8	5	100	..	..	..	..	1	..	94	..	5.00
11	100	10 $CaCl_2$	10 NaBr	14	175–180	8	5	90	..	..	..	..	..	..	83.5	..	7.60
12	100	10 $CaCl_2$	1 NaBr	10	175–180	8	5	85	..	..	..	..	3	..	77	..	11.08
13	100	10 $CaCl_2$	25 NaBr	10	175–180	8	5	90	..	..	..	..	3	..	84	..	11.6
14	100	10 $CaCl_2$	5 NaBr	10	175–180	8	5	92	..	..	..	..	2	..	87	..	7.7
15	100	10 $CaCl_2$	15 NaBr	10	175–180	8	5	100	..	..	..	..	..	..	84	..	7.1
16	100	20 $CaCl_2$	10 NaBr	10	175–180	8	5	105.5	..	..	..	..	..	..	92.5	..	10.5
17	100	5 $CaCl_2$	10 NaBr	10	175–180	8	5	100	..	..	..	..	..	..	87	..	9.4
18	100	10 $CaCl_2$	10 NaBr	10	175–180	4	5	95	..	..	..	..	..	22[1]	70	..	10.6
19	100	10 $CaCl_2$	10 NaBr	10	175–180	16	5	98	..	..	..	..	..	3	91.6	..	6.8
20	100	10 $CaCl_2$	10 NaBr	10	190	8	5	90.6	..	..	..	..	..	..	85	..	8.3
21	100	10 $CaCl_2$	10 NaBr	10	220	8	5	94	..	..	..	..	..	..	82	..	13.0
22	15 36	10 $CaCl_2$ Aniline	10 NaBr	10	175–180	8	5	..	..	..	69	..	..	..	11	..	M.E.A.
23	100	Aniline	10 NaBr	95% Alc.	175–180	8	5	96	..	..	24	..	..	..	91	..	12.24
24	100	20 $CaCl_2$	10 NaBr	95% Alc.	175–180	8	5	88	..	..	..	..	..	..	83	..	11.58

[1] The fraction was collected from 208–212.

Table III are representative of the effect of operating under such conditions.

Another phase of the investigation was concerned with the replacement of zinc chloride and potassium iodide by sodium bromide and calcium chloride (Table III). In Expt. 3, the substitution of sodium bromide for potassium iodide was productive of results wholly comparable with those obtained when potassium iodide was used, nor was the calcium chloride found to be less efficient than zinc chloride (Expt. 4). In other words, our results indicated that the conditions for reaction and the relative proportions of reagents best adapted for the preparation of diethylaniline were the following:

Calcium chloride	10 g.
Cupric chloride	5 g.
Sodium bromide	10 g.
Alcohol	200 g.
Aniline hydrochloride	100 g.
Temperature	175°–180° C.
Time	8 hrs.

Adopting a unit charge represented above as the basis for further work, a series of experiments were conducted in which the factors of concentration, time, and temperature were thoroughly investigated. The amount of cupric chloride productive of the best results was first ascertained, the other ingredients of the reaction mixture being kept constant in quantity. From inspection of Table III, as well as the curve developed from the same (Fig. 1), it may be seen that the effect of introducing cupric chloride is quite apparent even when small quantities are used (0–1 g.), the effect becoming gradually less pronounced up to 5 g., when a product containing but 6.79 per cent of monoethylaniline was obtained. This was the purest sample of diethylaniline that we had thus far obtained in our experiments. However, at this point it should be noted that the total yield as well as the 212° to 218° fraction was falling off in amount, due to the initiation of some decomposition from the presence of the excess of copper. This amount of copper chloride, however, was deemed the most efficient because it had promoted such a high degree of alkylation.

Four experiments were carried out, using alcohol as the variable factor. Five, eight, ten, and fourteen molecular proportions of alcohol were employed, corresponding to 160, 288, 360, and 504 g. of this reagent, respectively.

The curve for alcohol (Fig. 2) remains practically flat up to 8 moles; from here it falls sharply to 10 moles, where the monoethylaniline content of the oil was only 5 per cent, *the best product obtained in all our experiments.* Striking was the result, however, with the use of 14 moles, the purity of the product falling off to the extent of 7.9 per cent monoethylaniline. This is shown by the sharp rise in the curve from 10 to 14 moles.

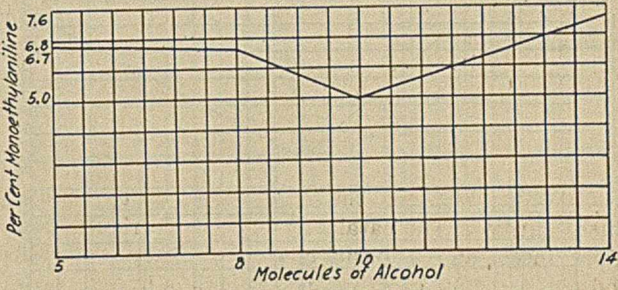


FIG. 2

With the following factors constant, sodium bromide was made the variable:

Aniline hydrochloride	100 g.
Alcohol	360 g.
Cupric chloride	5 g.
Sodium bromide (variable)	10 g.
Calcium chloride	10 g.
Temperature	175°–180°
Time	8 hrs.

Using but one gram of sodium bromide, the curve (Fig. 3) commences at a purity of 11 per cent monoethylaniline, rising slightly at 2.5 g. (presumably due to mechanical errors), then falls steadily with 5 and

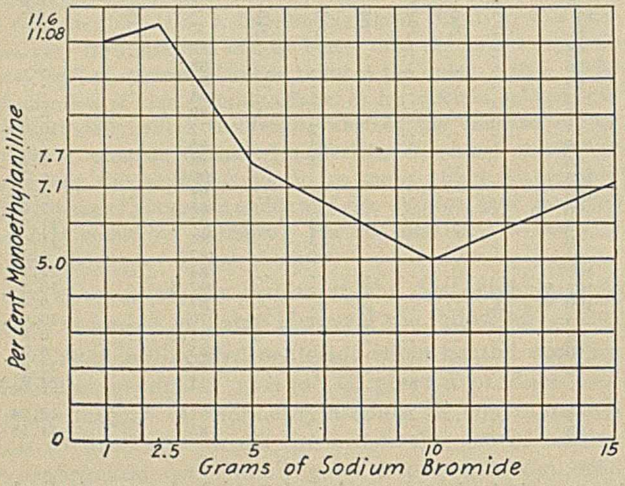


FIG. 3

10 g. of sodium bromide, reaching its lowest point with the latter figure. Further introduction of sodium bromide did not better the result, but if anything affected the purity, since the monoethylaniline content rose from 5 to 7.1 per cent. The effect of doubling the normal amount of calcium chloride is evidenced in the curve (Fig. 4) by an abrupt rise, denoting an increase in the acetylizable impurities, probably in the nature of *p*-ethyl substituted anilines. Using half the normal amount of calcium chloride was also unfavorable since incomplete alkylation was plainly manifest. In respect to concentration the following proportions of the catalysts were therefore the most efficient in their effect:

	Grams
Sodium bromide	10
Cupric chloride	5
Calcium chloride	10

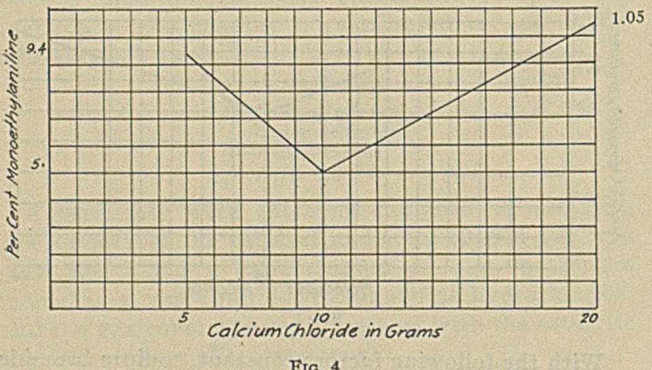


FIG. 4

The effect of time on the degree of alkylation is best shown in Fig. 5, where the highest point on the curve represents the percentage of monoethylaniline when heated for a period of 4 hrs. Incomplete reaction is evident in comparison with 8 hrs., the lowest point on the curve. Extension of the time factor beyond 8 hrs. was not productive of favorable results: in fact, at 16 hrs. the per cent of diethylaniline fell off slightly, showing the initiation of a reaction presumably typified by the expression

$$C_6H_5N(C_2H_5)_2 \longrightarrow C_2H_5.C_6H_4.NHC_2H_5.$$

In this case, in contradistinction to the 4-hr. period, the acetylizable material very probably is a nucleus-substituted derivative of aniline and not unchanged aniline.

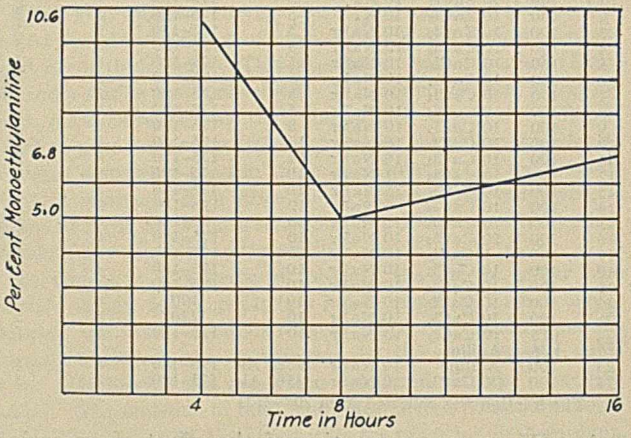


FIG. 5

It was our experience that temperatures below 175° C. made for results not as consistent as those obtained when the higher temperature was employed. The question as to whether the optimum temperature was above 175° C. was next investigated. The temperature of 190° C. (see Fig. 6) caused a rise in the acetylizable impurities. This fact could be attributed only to rearrangement of the tertiary amine through the influence of the metallic salts at the elevated temperature. This effect was even more striking at 220° C., when the impurity calculated as monoethylaniline rose to 13 per cent.

To summarize, the results obtained in our experimental work have led us to conclude that sodium bromide, cupric chloride and calcium chloride jointly functionate as catalytic agents in the alkylation of aniline when the hydrochloride of this base is heated with ethyl alcohol. The relative proportions of the various reagents and the time and temperature factors which are productive of the maximum yield of the tertiary base—diethylaniline—are given in the following table representing a unit autoclave charge. A unit charge run at a temperature of 175° to 180° is productive of pressures in the autoclave varying between 280 and 310 lbs.

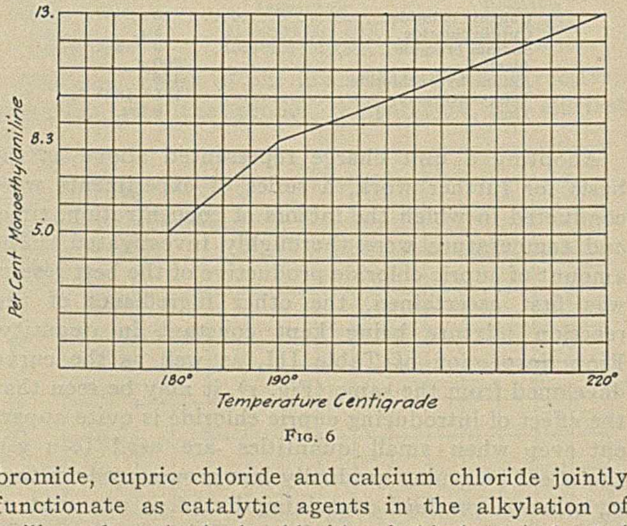


FIG. 6

Aniline hydrochloride	100 g.
Alcohol	10 moles (360 g.)
Sodium bromide	10 g.
Cupric chloride	5 g.
Calcium chloride	10 g.
Time	8 hrs.
Temperature	175°–180° C.

We have run through a great many experiments under the above conditions and they have all been productive of good yields of diethylaniline. The yields have always been very consistent. The total yield is more frequently above (*i. e.*, 104–105 g.) than below 100 g. Complete alkylation, together with complete extraction of the alkylated material, should produce the theoretical yield of 115 g. Therefore, notwithstanding the many opportunities for mechanical losses, decomposition through autoclave heat, to say nothing of the purity of the commercial aniline hydrochloride, crude yields of the order of 87 to 91 per cent are consistently obtainable.

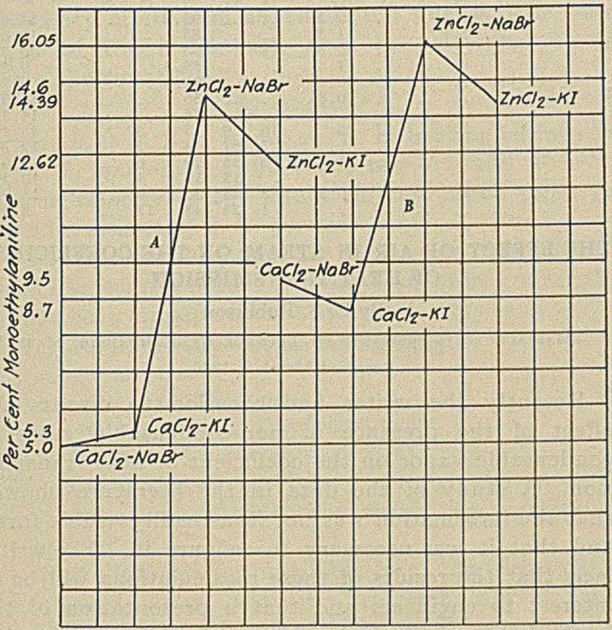


FIG. 7

Generally speaking, in the distillation of the crudes resulting from these best operative conditions, no oil, other than a drop or two, is obtained below 212° C. The loss of material in distillation is largely accounted for in the residue left in the flask, no doubt resulting from some decomposition during the distillation. The product, purified by distillation, is always a clear, highly refractive oil, possessing a slight yellow color.

An attempt was made to lower the cost factor in the production of diethylaniline by our method, through the incorporation of some aniline base in place of the hydrochloride, and also through the use of 95 per cent alcohol. The substitution of aniline oil for an equivalent amount of the hydrochloride in our best unit charge gave a product consisting largely of monoethylaniline (Expt. 22). The use of 95 per cent alcohol (Expt. 23) instead of the product dehydrated over lime lowered the purity about 7 per cent; nor could this be prevented by the incorporation of double the usual amount of calcium chloride as a dehydrating agent (Expt. 24).

As previously stated, calcium chloride and sodium bromide had been utilized in place of zinc chloride and potassium iodide, respectively, purely from the standpoint of economical procedure. In the light of this fact it seemed of interest to ascertain the relative efficiency of these salts. The efficiency of zinc chloride as compared with calcium chloride is shown in Fig. 7 (also Table IV), where Graph A represents the effects produced when the usual amounts of the catalysts are used. The second Graph (B) represents the effect when the amount of calcium chloride is reduced one-half, and also the sodium bromide or potassium iodide. The nature of the two results should check. At the peak of each curve stands zinc chloride and sodium bromide with zinc chloride and potassium iodide just below it, while at the inception of Curve A stands calcium chloride and sodium bromide, with calcium chloride and potassium iodide slightly above it. In Graph B the conditions are reversed. The results may therefore be summed up in the statements, first, that zinc chloride is much less efficient than calcium chloride in alkylation, and, second, that the iodide is but slightly different in its effectiveness as compared with the bromide. Placing more emphasis on the zinc chloride results, the conclusion may be reached that the potassium iodide functions somewhat more favorably than the sodium bromide.

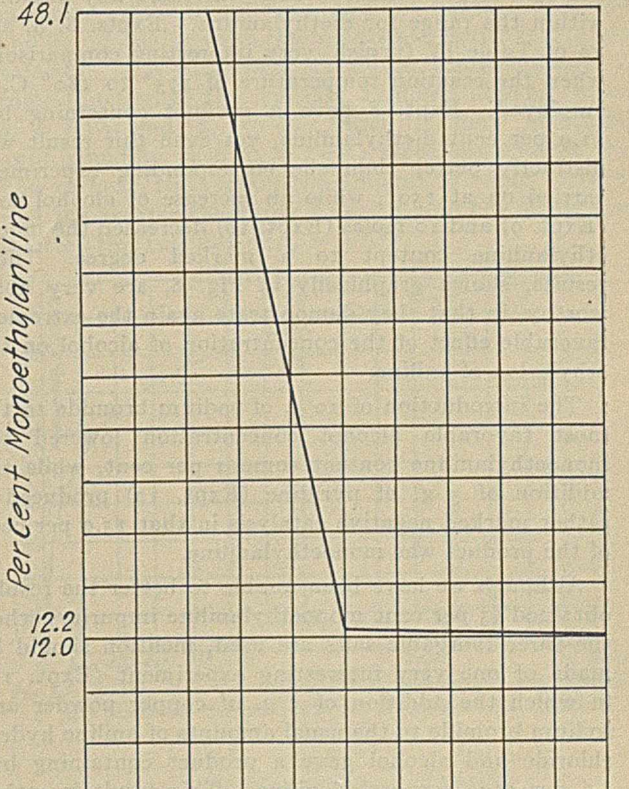


FIG. 8

In regard to the zinc chloride, it may be said in explanation of its action, that in all probability it

TABLE IV

100 g. of aniline hydrochloride were used in each experiment. The quantity of $ZnCl_2$ and $CaCl_2$ introduced was always 10 g., and the time of heating the autoclave was 8 hrs. In each experiment the temperature was held between 175°–180° C.

Expt.	$ZnCl_2$ or $CaCl_2$	Grams NaBr or KI	Moles of Alcohol	Grams Special Catalyst	Total Yield. G.	Distillate											Monoethylaniline Per cent
						M. E. A.						D. E. A.					
						195–200	200–202	202–204	204–206	206–208	208–210	210–212	212–214	214–216	216–218	218–220	
1	$ZnCl_2$	10 NaBr	10	5 $CuCl_2$	101.3	..	..	..	..	..	2.5	..	..	95.8	..	..	14.6
2	$ZnCl_2$	10 KI	10	5 $CuCl_2$	90	..	..	..	..	..	1·3	..	..	85	..	..	12.62
3	$ZnCl_2$	10 KI	10	5 $CuCl_2$	100.4	..	..	..	..	..	0.8	..	..	95	..	..	5.3
4	$ZnCl_2$	5 NaBr	10	2.5 $CuCl_2$	94	..	..	..	..	..	1.6	..	..	89.3	..	..	9.5
5	$ZnCl_2$	5 KI	10	2.5 $CuCl_2$	96	..	..	..	..	..	0.3	..	..	91.8	..	..	8.7
6	$ZnCl_2$	5 NaBr	10	2.5 $CuCl_2$	96.3	..	..	..	..	..	6.8	..	..	86	..	..	16.05
7	$ZnCl_2$	5 KI	10	2.5 $CuCl_2$	80	..	..	..	..	..	..	..	..	75	..	..	14.39
8	....	....	2.2	....	80	..	..	..	..	12	26	16	9	3	3	..	48.1
9	....	....	5	....	93	..	..	..	..	..	..	7	57	4	2	..	12.2
10	....	....	10	....	103	..	..	..	..	..	1	6	55	29	6	..	12.0
11	....	10 NaBr	10	....	102	..	..	..	..	..	..	..	55	39	4	..	10.2
12	....	....	10	5 Pyridine	99	..	..	..	..	1	0.5	8	44	35	9	..	15.9
13	....	....	10	5 Cu Powder	93	..	..	..	..	..	..	1	24	48	11	..	8.6

functions at a high temperature as a de-alkylating, as well as a dehydrating agent, and the smaller yield of diethylaniline produced by it in comparison with calcium chloride may be due to the fact that this salt rearranges some of the diethylaniline formed to a nucleus-substituted amine, such as the monoethylated derivative of *p*-ethylaniline or similar combinations.

Expts. 1 and 2 of Table II are illustrative of the results obtained by heating aniline hydrochloride alone with alcohol at 150° C. In these experiments the concentration of the alcohol was 2.2 and 5 moles, respectively. In neither case was there any distillate within the range for diethylaniline. Expts. 8, 9, and 10 of Table IV furnish very interesting comparisons, when the reaction temperature of 175° to 180° C. is employed. Expt. 8 gave a product containing but 51.9 per cent diethylaniline, yet even this result was distinctly better than the corresponding experiment carried on at 150°, while an increase of alcohol to 5 (Expt. 9) and 10 moles (Expt. 10) decreased the monoethylaniline content to a marked degree. These results, shown graphically in Fig. 8, are very noteworthy, in that they demonstrate again the extremely favorable effect of the concentration of alcohol on the ethylation of aniline.

The introduction of 10 g. of sodium bromide to the most favorable alcohol concentration lowered the monoethylaniline content some 2 per cent, while the addition of 5 g. of pyridine (Expt. 12) produced a rather marked negative catalysis in that 15.9 per cent of the product was monoethylaniline.

Although we have been unable to better the results obtained (5 per cent monoethylaniline impurity) when the three inorganic salts are used, mention should be made of one very interesting experiment (Expt. 13) in which the addition of 5 g. of copper powder and sodium bromide to the usual amounts of aniline hydrochloride and alcohol gave a product containing but 8.6 per cent monoethylaniline. This result is rather interesting in the light of the experiment mentioned in the first part of the paper, when clean copper strips or a thoroughly cleaned copper inset were apparently without catalytic effect. The difference in results may of course be attributed to the state of division of the copper.

THE EFFECT OF AIR IN STEAM ON THE COEFFICIENT OF HEAT TRANSMISSION

By C. S. Robinson

Massachusetts Institute of Technology, Cambridge, Mass.

Received March 9, 1920

Recently the writer had occasion to discuss the effect of the presence of non-condensable gases in condensable vapor on the coefficient of heat transmission. A study of the data in the literature[1] showed that the information was not in a readily usable form, and that it was necessary to refigure it. The writer feels that the results of these recalculations will be of interest to engineers and that a presentation of the methods of calculation used may be of assistance to others who have similar problems to be solved. It should be emphasized that the relations hereinafter derived are approximate, and are based on very incomplete data. Until more information is available, they are, however, the best to be had.

The flow of heat from one point to another is inversely proportional to the thermal resistance of the material between the two points. Where the material in the path of flow is not homogeneous, but is made up of successive sections having different resistances, the method used in calculating the over-all resistance is the same as that used in electricity, that is, the over-all resistance is the sum of the resistances of the successive sections.

In the special cases of the surface condenser and the evaporator, the heat flows from the condensing vapor to the separating wall, through this wall, and from the further side of the wall to the cooling fluid, which in the first case is the condenser water, and in the second, the boiling liquid. The coefficient of heat transfer from the vapor to the cooling fluid may be calculated, providing the resistances of the successive sections of the path are known. These sections are: first, the film resistance on the vapor side; second, the resistance of the metal; and third, the film resistance on the liquid side.

The conductivity of metals has been carefully studied, and it is possible to obtain values of the

---

[1] E. W. Kerr, "Tests upon the Transmission of Heat in Vacuum Evaporators," *Trans. A. S. M. E.*, **35** (1913), 731; G. A. Orrok, "Air in Surface Condensation," *Ibid.*, **34** (1912), 713; J. A. Smith, "Effect of Air in Feed Water," *London Engineering*, Oct. 7, 1904.

coefficient of the metal from such handbooks as Kent and Marks. The film resistance on the liquid side has also been the subject of careful experimental work. It has been found that this resistance is largely a function of the liquid velocity, and recent work done at the Massachusetts Institute of Technology (not yet published) would indicate that the coefficient of conductivity of the film on the liquid side, h_e (the reciprocal of the resistance), may be calculated closely from the equation

$$h_e = 450\overline{V}^{0\cdot 8},$$

where h_e is in B. t. u. per sq. ft. of heating surface per deg. F. temperature difference per hr., and $\overline{V}$ is the linear velocity of the liquid through small pipes (up to 2 in. diameter) in ft. per sec.

The film coefficient on the vapor side, however, has had less careful study. It has long been known that it has been largely affected by such factors as

1—Steam velocity past the heating surface
2—Steam pressure
3—Rate of condensation
4—Rate of removal of condensed steam
5—Presence of non-condensable gases

The last of these factors is of very great importance and is the one in which the writer was particularly interested.

Of the information available from the three articles mentioned above, that in Professor Kerr's article proved to be the only one with the data in such condition that it could be analyzed with respect to the film conductivity on the steam side, and the following calculations are based almost entirely on his data. His experiments were made with an experimental, vertical, tubular evaporator, with water inside the tubes. The pressure and temperature of the vapor in the steam belt were measured, and from the latter the partial pressure of the water vapor in the mixture of steam and air was determined with the aid of steam tables. The ratio of the partial pressure of the water vapor to the total pressure was calculated and tabulated against the coefficient of heat transmission of the evaporator. This ratio multiplied by 100 is equal to the percentage by volume of the water vapor in the mixture, and in Table 1 it is so recorded.

TABLE 1

Steam by Volume Per cent	Coeff. of Heat Transmission	Film Coeff. Steam Side	y	Film Coeff. Water Side
100	594	3000 (assumed)	4.55	757
100	533	3000 (assumed)	4.35	658
99.7	510	2500 (calculated)	4.45	650
97.3	464	2080 (calculated)	4.45	606
93.1	487	2270 (calculated)	4.45	628
91.6	408	1720 (calculated)	4.45	541
91.3	386	1510 (calculated)	4.45	523
90.9	300	1030 (calculated)	4.45	427
82.2	296	1010 (calculated)	4.45	421
81.1	284	970 (calculated)	4.45	405
74.2	258	830 (calculated)	4.45	377
74.4	241	740 (calculated)	4.45	360
72.4	215	640 (calculated)	4.45	324
72.1	240	750 (calculated)	4.45	356
67.6	179	500 (calculated)	4.45	281
66.8	147	377 (calculated)	4.45	242

The film coefficient of heat conductivity on the steam side is calculated from the expression

$$\frac{1}{K} = \frac{1}{h_s} + \frac{1}{k_m} + \frac{1}{h_e},$$

where K is the over-all coefficient of the evaporator, h_s is the film coefficient on the steam side, k_m is the coefficient of the given metal wall, h_e is the film coefficient on the liquid side.

In the data referred to, K only is known. It is, however, possible to approximate values for k_m and h_e. The metal wall used was copper 0.05 in. thick. The heat transmission of copper is 220 B. t. u. figured as per foot of thickness. Therefore,

$$k = \frac{220 \times 12}{0.05} = 53{,}000\text{—a relatively large quantity.}$$

The film coefficient for steam containing no gases is stated by various writers to lie between 2000 and 4000. It is known that vertical tubular evaporators, when boiling water under test conditions, have given over-all coefficients of 1500. This would indicate that the film coefficient on the steam side was not less than double 1500, or 3000. The writer has taken this last value as the mean of the limits noted above, and as very probably near the true one for the conditions of the experiment.

The first test would then give the expression

$$\frac{1}{594} = \frac{1}{3000} + \frac{1}{53000} + \frac{1}{h_e}.$$

The phenomena occurring in a vertical evaporator tube are well understood. The liquid at the lower end contains no vapor, and has relatively low velocity. The evaporation and therefore the volume of vapor increases as the liquid rises, and the mixture of vapor and liquid issuing from the top does so under maximum velocity. The film conductivity on the liquid side is some function of this velocity as noted above. Since $h_e = f(\overline{V})$ and $K = f'(\overline{V})$, then it may be stated approximately that $h_e = f''(K)$ since the greater the flow of heat, the greater the amount of vapor formed, and the greater the vapor velocity, and the writer has selected this function as $h_e = yK^{0\cdot 8}$.

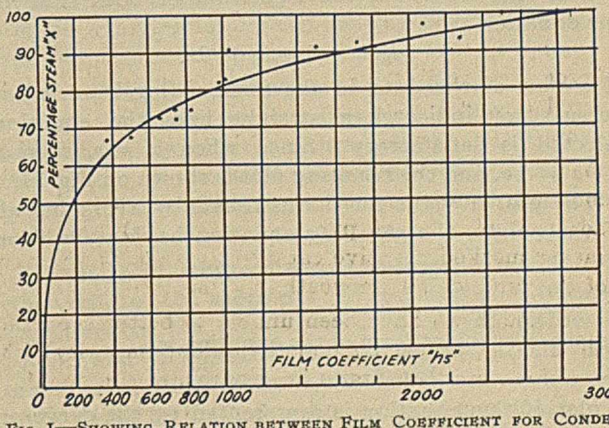


FIG. I—SHOWING RELATION BETWEEN FILM COEFFICIENT FOR CONDENSING VAPOR AND PERCENTAGE STEAM IN STEAM-AIR MIXTURES

$$\frac{1}{594} = \frac{1}{3000} + \frac{1}{53000} + \frac{1}{y(594)^{0\cdot 8}}$$

$$\therefore y = 4\cdot 55$$

Repeating this calculation for the second test

$$y = 4\cdot 35.$$

The writer selected the mean, 4.45, as the value of y to use in the balance of the tests when air was present in the steam. Test 3 would then give the following:

$$\frac{1}{510} = \frac{1}{h_s} + \frac{1}{53000} + \frac{1}{4.45(510)^{0.8}}$$

$$h_s = 2500$$

Table 1 gives the results of the calculation for all of the tests. Fig. I shows values of percentage of steam plotted against film coefficient—h_s.

The data include steam percentages down to 66 per cent only. The film coefficient for zero per cent, that is, all air, has been the subject of much careful work, the values of h being a function of the velocity and varying between 1 and 20. Since no information was given in Professor Kerr's experiments as to the rate of removal of the air, the writer selected a mean value of 10 for this point in order that it might be possible to derive a suitable empirical equation to fit the above calculation.

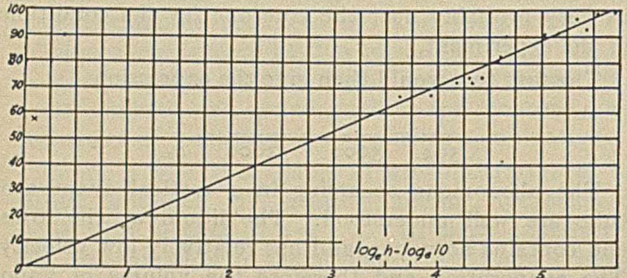


FIG. II—EFFECT OF AIR ON "L" FOR STEAM: x = PER CENT STEAM BY VOLUME, h = FILM COEFFICIENT

Inspection of the curve on Fig. I indicates that it has the form indicated by the exponential equation

$$h = ae^{\beta x},$$

where a and β are constants to be determined and x is per cent steam by volume. In order to test the applicability of this type of equation, it may be rectified as follows. Take the logarithm of both sides of the equation

$$\log_e h = \log_e a + \beta x.$$

If a plot of the data in the form of ($\log_e h - \log_e a$) versus "x" indicates a straight line, the equation selected is satisfactory. Since when $x = 0$, $h = a$, $\therefore a = 10$, and the resulting plot is shown on Fig. II.

The proper value for β was found by averaging all of the points, the resulting equation having the form

$$\log_e h = 2.303 + 0.0566x$$

or

$$h = 2.303\, e^{0.0566x}$$

a more convenient form of the equation is

$$\log_{10} h = 1 + 6.0240x.$$

A plot of this equation is represented by the curve on Fig. I.

THE PRODUCTION OF PHOSPHORIC ACID BY SMELTING PHOSPHATE ROCK IN A FUEL-FED FURNACE

By William H. Waggaman and Thomas B. Turley

BUREAU OF SOILS, U. S. DEPARTMENT OF AGRICULTURE, WASHINGTON, D. C.

[PRELIMINARY REPORT]

Received March 1, 1920

Ever since the early days of the war when the high cost and scarcity of acid phosphate seemed to menace our agricultural interests, the Bureau of Soils has given considerable attention to the production of soluble phosphates by means other than the usual method of treating phosphate rock with sulfuric acid.

Perhaps the most attractive and in many ways the most promising of these processes is that in which an intimate mixture of a natural phosphate, silica, and coke is smeltered at a high temperature, with the result that silicates of lime are formed and the phosphoric acid volatilized and subsequently collected in some suitable manner. This process is really based on the method long in use for the manufacture of phosphorus, and in order to separate completely the phosphoric acid from the lime it is apparently necessary to have a sufficient quantity of reducing agent present to produce elementary phosphorus according to the equation

$$Ca_3(PO_4)_2 + 3SiO_2 + 5C = 3CaSiO_3 + 2P + 5CO.$$

The phosphorus produced is subsequently burned or oxidized within or outside of the furnace by carbon dioxide or air thus

$$2P_2 + 5O_2 = 2P_2O_5$$

or

$$P_2 + 5CO_2 = P_2O_5 + 5CO.$$

It has been generally believed that the temperature and other conditions necessary to volatilize completely phosphorus and phosphoric acid from natural phosphates could be attained only in the electric furnace,[1] and the Bureau's early experiments were therefore conducted in furnaces of the arc type. As the results of these investigations have been described in previous articles, only a brief outline of the work is given in the present paper.

The first experiments were carried on in a rather crude electric furnace constructed at the Bureau's experimental laboratories at Arlington Farm, Va., and here the Cottrell method of electrical precipitation was first successfully applied to the collection of the volatilized phosphoric acid. Ross, Carothers and Merz[2] showed that by this means there could be obtained an acid of such high concentration that the added cost of manufacturing phosphoric acid by the volatilization process would be partly offset by the saving in transportation charges in shipping and distributing the product. Later a larger and more complete furnace with auxiliary apparatus was constructed at Hoboken, N. J.

In reporting on this work Carothers[3] showed that, assuming the price of electric power at $25 per h.-p. yr., phosphoric acid (P_2O_5) could be produced and recovered at a cost of 3.37 cents per pound exclusive of interest on investment, taxes, and royalties. By using the acid thus obtained to treat more phosphate rock, however, a double superphosphate could be produced which brought down the price of the unit of soluble phosphoric acid very appreciably. The figures indicated that under the abnormal conditions existing at that time, the cost per unit of phosphoric acid produced by the volatilization process compared favorably with the cost of this ingredient in ordinary superphosphate obtained by the sulfuric acid method.

---

[1] F. S. Washburn, U. S. Patents 1,044,957 (1912); 1,100,639 (1914).

[2] THIS JOURNAL, **9** (1917), 26.

[3] *Ibid.*, **10** (1918), 35.

TABLE I—VOLATILIZATION OF PHOSPHORIC ACID FROM MIXTURES OF TRICALCIUM PHOSPHATE, SILICA, COKE AND ALUMINA BY SMELTING IN OPEN AND CLOSED CRUCIBLES IN A DENTAL FURNACE BY MEANS OF ILLUMINATING GAS AND AN AIR BLAST

Sample No.	Crucible	Proportions of Materials Used in Charge				Percentage Composition					Temperature Attained ° C.	Length of Time Heated Hrs.	P_2O_5 in Slag Per cent	P_2O_5 Volatilized Per cent of Total	Character of Slag
		$Ca_3(PO_4)_2$ Grams	SiO_2 Grams	Al_2O_3 Grams	Coke Grams	SiO_2	CaO	Al_2O_3	P_2O_5	C					
12 A.....	Closed (Clay)	20.0	16.6	...	5.2	40.7	25.9	0.7	21.9	10.8	Above 1400	0.75	3.24	89.8	Fluid, grayish black
12 G....	Open (Clay)	20.0	16.6	...	5.2	40.7	25.9	0.7	21.9	10.8	Above 1400	1	7.20	76.2	Viscous gray. White bloom on surface[1]
$12\ N_1$....	Open (Graphite)	20.0	16.6	6.6	5.2	35.1	22.4	14.3	18.9	9.3	1200	1	4.92	80.4	Viscous black
$12\ N_2$....	Open (Graphite)	20.0	16.6	6.6	5.2	35.1	22.4	14.3	18.9	9.3	Above 1400	2	None	100.0	Fluid, black
12 O.....	Open (Clay)	20.0	16.6	...	4.8	41.1	26.2	0.7	22.1	9.9	Above 1400	1	3.36	89.3	Fluid, light gray
12 P.....	Open (Clay)	20.0	16.6	6.6	5.0	40.9	26.1	0.7	22.0	10.3	Above 1400	1	2.22	93.0	Fluid, light gray; bloom on surface[1]
12 Q.....	Open (Clay)	20.0	16.6	6.6	5.4	40.7	25.8	0.7	21.8	11.0	Above 1400	0.75	4.65	84.9	Fluid, light gray; bloom on surface[1]
12 R....	Open (Clay)	20.0	16.6	6.6	5.6	40.5	25.7	0.7	21.7	11.4	Above 1400	0.75	2.45	92.2	Fluid, light gray; bloom on surface[1]
12_1......	Open (Clay)	20.0	16.6	6.6	5.2	40.7	25.9	0.7	21.9	10.4	1300	1	Trace	100.0	Fluid, light gray; large bloom[1]
12_2......	Closed (Clay)	20.0	16.6	6.6	5.2	40.7	25.9	0.7	21.9	10.4	1300	1	Trace	100.0	Fluid, all slag
12_{11}......	Open (Clay)	20.0	16.6	2.0	5.6	38.7	24.5	5.2	20.7	10.9	1300	1	5.28	81.3	Viscous light gray; bloom on surface[1]
12_{12}......	Closed (Clay)	20.0	16.6	2.0	5.6	38.7	24.5	5.2	20.7	10.9	1300	1	1.91	93.6	Viscous dark; no bloom
12_{15}......	Open (Clay)	20.0	20.0	2.0	4.8	43.6	23.2	4.9	19.6	8.7	1300	1	4.26	83.7	Viscous gray; large bloom[1]
12_{16}......	Closed (Clay)	20.0	20.0	2.0	4.8	43.6	23.2	4.9	19.6	8.7	1300	1	3.52	86.6	Viscous gray; no bloom
12_{17}.....	Open (Clay)	20.0	22.0	2.0	4.8	45.9	22.2	4.7	18.8	8.4	1300	1	3.90	84.3	Viscous gray; large bloom[1]
12_{18}.....	Closed (Clay)	20.0	22.0	2.0	4.8	45.9	22.2	4.7	18.8	8.4	1300	1	3.75	84.8	Viscous gray; no bloom

[1] An analysis of the bloom or white crust on the surface of slags obtained in open crucibles shows a considerably higher percentage of phosphoric acid than in the original mixture. The proportion of lime to phosphoric acid coincided very closely with that in calcium pyrophosphate ($Ca_2P_2O_7$).

In a later investigation, Waggaman and Wagner[1] pointed out that by using the "mine-run" phosphates of Florida, which in their natural state contain impurities which preclude their treatment with sulfuric acid until they have been put through an elaborate washing and screening process, a great saving in phosphate could be effected and the cost of the soluble phosphoric acid produced therefrom very materially reduced.

The high cost of electric power in this country, however, made it appear very desirable to test out the commercial possibilities of producing phosphoric acid for fertilizer purposes in a fuel-fed furnace, and, since crude oil is the cheapest and most accessible fuel for the phosphate regions of Florida, it was decided to undertake experiments with a view to producing phosphoric acid by this means.

LABORATORY EXPERIMENTS

Preliminary work in the laboratory, where various mixtures were heated in a dental furnace by means of gas and an air blast, showed that contrary to general opinion the nearly complete evolution of phosphoric acid from a charge of calcium phosphate, carbon and quartz flour was perfectly feasible provided that reducing conditions were maintained until a fusible slag was produced, and a temperature of approximately 1500° C. was continued throughout the operation.

Table I shows in part the results obtained by heating such mixtures, with and without the addition of small amounts of aluminum oxide, in both open and closed fire-clay crucibles.

An inspection of Table I will show that as a rule considerably better results were obtained where the crucibles were kept covered so that the oxidizing gases were not allowed to come into contact with the charge. When the crucibles were left open, it was noticed that a white crust or "bloom" almost invariably formed over the slag and no matter how long the high temperature was maintained this crust gave no sign of melting. The quantity of this crust in a number of instances was equal to that of the underlying slag. It is assumed that the phosphoric acid distilling from the lower part of the mass recombined with or was fixed by the lime at the surface where oxidizing conditions prevailed, forming calcium pyrophosphate according to the following equation:

$$2Ca_3(PO_4)_2 + P_2O_5 = 3Ca_2P_2O_7$$

It was found, however, that when the fusion of the charge was well under way and the carbon or coke thus protected in the mass of molten slag, the covers of the crucibles could be removed without the formation of this crust and that the reaction continued in spite of the oxidizing conditions at the surface of the slag. The addition of small amounts of alumina to the charge seemed to aid the fusion somewhat, it being a well-known fact that the presence of this substance in limited amounts lowers the melting point of both acid and basic slags.

In most of these mixtures the ratio of silica to lime was approximately 39 to 61 per cent, but it was found later, in dealing with the natural phosphates of Florida, that better results could be obtained by varying these proportions according to the composition of the mineral used.

These laboratory experiments pointed almost conclusively to the necessity of maintaining reducing conditions in the phosphatic charge until fusion has begun, and it appeared at first sight that the most practical method of doing so in a mass containing much finely divided material, such as the pebble phosphates and the mine-run phosphates of Florida, was to heat the mixture in a separate chamber so that the oxidizing gases from the burning fuel would not come into contact with the charge until the latter had been brought to a state of incipient fusion.

LARGER SCALE EXPERIMENTS

Accordingly in order to test this process on a semi-commercial scale, a fire-brick furnace of the type shown in Fig. 1, described by the senior author and

---

[1] THIS JOURNAL, 10 (1918), 353.

others in U. S. Patent 1,282,994, was constructed at Arlington Farm, Va. This furnace comprised a central or inner chamber (holding about 150 lbs. of charge) open both at the top and bottom but constricted somewhat at its lower end to prevent the charge from working through too rapidly. This chamber was supported on arches of carborundum brick above a hollow hearth intended to receive the molten silicate. The whole was surrounded by an outer chamber into the opposite walls of which were set two oil burners so placed that their flames played upon and around the lower part of the inner chamber, heating the charge by radiation through the 4-in. walls. Any fumes which were evolved from the smelting of the mass were to be drawn down through the charge chamber and passed together with the gases of combustion onto the Cottrell precipitator in order to collect the phosphoric acid.

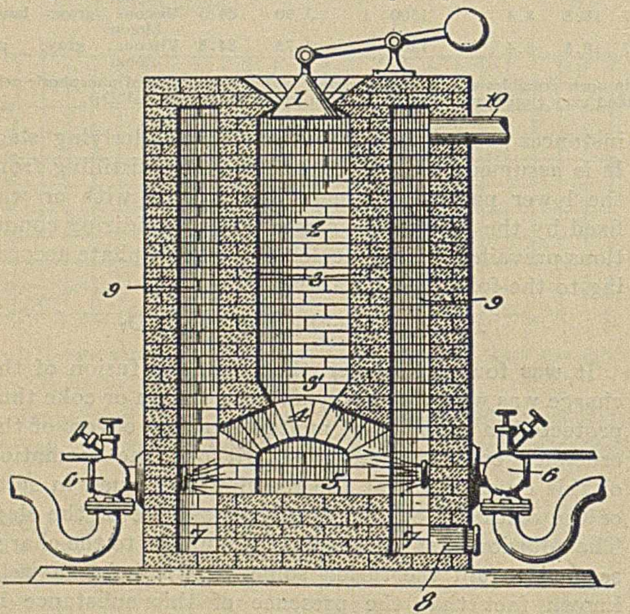


Fig. 1—Furnace Used in First Experiments

This indirect method of heating the charge proved so inefficient and entailed such a loss of heat that after several trials lasting from 18 to 24 hrs. it was abandoned as impractical, and no molten slag was obtained, although a certain amount of phosphoric acid was driven off, and a sintered product obtained in the charge chamber. The experiments showed quite clearly that in order to make the process economically practicable the full calorific power of the fuel must be utilized, which can be done only by heating the charge directly in the flame. This conclusion only served to emphasize the problem of how to maintain the reducing conditions necessary for the volatilization of the phosphoric acid, when the maximum efficiency of the crude oil flame, or that of any other fuel, can only be obtained under oxidizing conditions. Moreover, the phosphates with which the experiments were being conducted are of such a character that it is impossible to handle them in a plant of the blast furnace type, as a flame cannot be forced through a mass of such finely divided material.

At the suggestion of Professor Whitney, chief of this Bureau, the plan of spraying the phosphatic charge into the furnace along with the fuel was then tried; one oil burner was entirely cut off and a concave baffle of carborundum brick built opposite the other burner. The charge was placed in a hopper from which it was mechanically fed through a screw conveyor and blown into the furnace against the baffle in a spray under an air pressure of 30 lbs. to the square inch. It was found that if the charge was fed into the furnace very slowly a certain amount of slag low in phosphoric acid formed upon and ran down the wall of the baffle, but the vast bulk of the material, owing to its finely divided condition, was carried out of the furnace along with the gases of combustion and lost. After several trials with material of various degrees of fineness this method was also abandoned.

BRIQUETTING THE PHOSPHATE CHARGE

It was then thought that perhaps the nodulizing or briquetting of the phosphate charge might present a solution of the problem, and experiments were begun on the briquetting of mixtures of finely ground pebble phosphate, sand and coke, using various binders such as solutions of magnesium chloride, calcium chloride, calcium sulfate, sodium chloride, sodium silicate, phosphoric acid, and an acid sludge from the refining of petroleum. Short cylindrical briquettes 1 in. thick and 1 in. in diameter were made in a mold under a pressure of about 1 ton, but none of the binders employed proved satisfactory since the briquettes (either air dried or oven dried) shattered when dropped upon a stone table from a height of 1 to 3 ft. In the Florida hard-rock regions, however, the phosphate deposits in their natural state contain much soft phosphate and clay-like material of considerable plasticity, and it was thought that possibly the binding qualities of this mine-run phosphate might prove sufficiently effective without the addition of any other ingredient.

Table II—Mechanical Analysis of Finely Ground Florida Pebble Phosphate, Mine-Run Phosphate, and Furnace Charges Made up from These Phosphates

	Percentage of Various Sized Particles in Samples					
Materials Analyzed	Fine Gravel and Coarse Sand Diam. 2–0.5 Mm.	Medium Sand Diam. 0.5–0.25 Mm.	Fine Sand Diam. 25–1.1 Mm.	Very Fine Sand Diam. 1–0.05 Mm.	Silt Diam. 0.05–0.005 Mm.	Clay Diam. 0.005–0.000 Mm.
1—Florida pebble phosphate (finely ground)..........	0.0	0.0	20.4	24.8	36.0	18.8
2—Mine-run phosphate from hard rock regions......	0.0	0.2	30.2	14.6	21.0	34.0
3—Sample 1 mixed with finely ground silica and coke....	3.2	6.4	36.6	20.2	27.6	6.2
4—Sample 2 mixed with finely ground silica and coke....	0.4	0.6	35.0	18.9	19.5	25.5

In order to compare the fineness of the washed and ground pebble phosphate with that of the mine-run phosphate, samples of each were ground in a ball mill and then submitted to the mechanical analysis employed in this Bureau in connection with the classification of soil types. Samples of these two phosphates mixed with the proper proportions of finely ground sand and coke to produce a charge suitable for furnace treatment were also analyzed in the same way. For results of these mechanical analyses see Table II.

This table will show that the percentage of clay particles present (upon which the plasticity of the material largely depends) was nearly twice as great in the mine-run phosphate as in the sample of pebble phosphate, even after the two materials had been ground for several hours in a ball mill and passed through a 60-mesh sieve.

Experiments were then undertaken in briquetting samples of the original mixture except that the finely ground pebble phosphate was replaced in part and finally wholly by the mine-run phosphate. These charges in each case were mixed with 10 per cent of water and made into short cylindrical briquettes (1 in. in diameter) under a pressure of one ton.

The results of these tests[1] are given in Table III.

TABLE III—SHATTER TESTS ON BRIQUETTES

Ratio between the Two Phosphates in Charge, Per Cent		Height at Which the Briquette Shattered, Feet	
Pebble Phosphate	Mine-Run Phosphate	Air Dried Briquette	Oven Dried Briquette
100.0	0.0	3	3
90.0	10.0	3	3
75.0	25.0	4	4
50.0	50.0	4	6
25.0	75.0	6	7
0.0	100.0	9	10

It is evident that a charge containing from 20 to 25 per cent of finely divided material classed as clay can be formed into very satisfactory briquettes, but it was decided that the size of those used in the first tests was somewhat small for furnace treatment, so a larger mold was made and a round briquette 2 in. in diameter used in the next furnace experiment. The tediousness of preparing a sufficient number for a protracted run, coupled with a desire to test thoroughly the efficiency of the natural binder, prompted the writers to make arrangements with the Lehigh Coal and Navigation Co., of Lansford, Pa., to use their coal briquetting machinery on this phosphate mixture. This company kindly consented to turn over their briquetting plant for the experiment, and accordingly a half ton of the mixture was crushed to pass a 10-mesh screen, thoroughly mixed with 10 per cent of water and shipped to Lansford in air-tight barrels. Without any further treatment it was charged into a screw conveyor and fed directly to the hopper above a press of the Belgian Roll type. Very satisfactory briquettes in the shape of eggets (2.25 x 2′ x 1.5) were produced which on drying withstood a drop of 6 ft. upon a cement floor without shattering.[2]

DIRECT HEATING OF BRIQUETTED CHARGE

The furnace was then so modified that a portion of the flames and hot gases of combustion from the oil burners would play up through the central shaft and thus heat the charge of briquettes directly. The burners were lighted at 8 A. M. and run steadily until 6 P. M., when a reading with an optical pyrometer was made which showed a temperature of over 1300° C. on the hearth. A few lumps of coke were then dumped into the shaft followed by 32 lbs. of briquettes. Within 10 min. fumes of phosphoric acid began to be evolved, and this continued for over an hour. At 7.45 P. M. a few more lumps of coke were added followed by 32 lbs. of briquettes. Copious fumes were evolved about 15 min. later which gradually grew less dense until at 8.50 P. M. (one hour later) another charge of 34 lbs. was added. This was followed by a fourth charge of 33 lbs. at 9.03 P. M. The heat was continued until 12:00 midnight when the optical pyrometer gave a reading of 1470° C. on the hearth. The burners were then shut off and an attempt made to tap the furnace, without success, due to the viscous nature of the slag produced.

After the furnace had cooled down, the slag was dug out and its phosphoric acid content determined. An average sample of the slag showed 11.52 per cent P_2O_5 while that which was more glassy and better fused contained only 3.53 per cent of P_2O_5. It was found that some of the briquettes which had not been fused were covered with a thin white glaze. This glaze evidently protects the carbon in the mass from the oxidizing gases until fusion takes place, for upon breaking the briquettes the coke was found unaltered, although they had been in the furnace several hours.

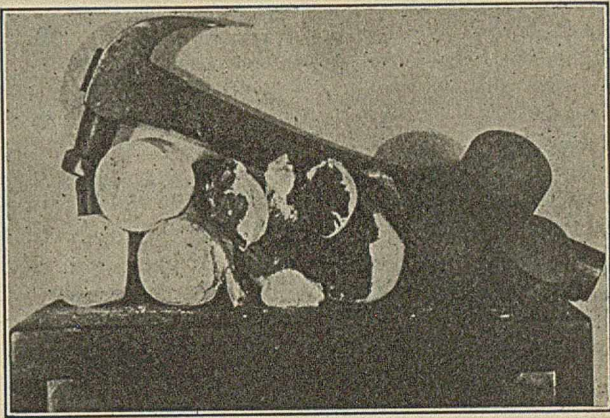


FIG. 2—EFFECT OF THE DIRECT OIL FLAME UPON PHOSPHATIC BRIQUETTES JUST BEFORE FUSION TAKES PLACE. NOTE THE UNALTERED COKE CONTAINED WITHIN THE MASS

The effect of this direct heating on the briquettes before they are actually fused can be seen in Fig. 2. On the right are the briquettes as they appear before being charged to the furnace, and at the left of the picture are shown those which have been exposed to a high temperature for several hours but not sufficiently high to cause them to melt. In the center are shown some of these latter briquettes broken open. It will be noted that there is a sharp line of demarkation between the thin glaze of the oxidized exterior of the briquettes and the interior containing the unaltered coke.

It was unfortunate that the furnace used was of such a type as to be ill adapted to the change from the indirect to the direct method of heating the charge. This fact accounts in part for the long time required in attaining a smelting temperature. With a view to overcoming this difficulty, firebrick "annexes" were built on each end of the furnace and the burners thus placed further from the charge chamber so that the

---

[1] These early briquetting experiments were made by Mr. L. A. Steinkoenig, who has since resigned from the Department of Agriculture.

[2] The writers wish also to express their appreciation of the courtesy extended by the General Briquetting Co., of New York City. This company not only made a number of experiments in briquetting the phosphate charge but also briquetted a ton of material used in later experiments.

oil would have a better chance for combustion. The iron plates holding the burners were replaced by cast iron water jackets to avoid the danger of injuring the burners, and a coil of pipe imbedded in a coke fire was also placed between the blower or fan and the furnace so that the air required for the combustion of the oil could be preheated before it was delivered to the burners.

The first test (No. 2, Table IV) made with these changes in the furnace was unsuccessful due to the cracking of the cast iron water jacket around one of the burners which made it necessary to close down after a few hours' run. But the second run (No. 3, Table IV) was very encouraging, since phosphoric acid was copiously evolved but the temperature attained was not quite high enough. The third test (No. 4, Table IV) was more successful while it lasted, but during the last hour of the experiment the charge chamber collapsed and the burners had to be shut off before the last of the charge was fully smelted.

TABLE IV—RESULTS OBTAINED IN SMELTING BRIQUETTES MADE OF MINE-RUN PHOSPHATE, SAND AND COKE IN A SHAFT FURNACE HEATED BY CRUDE OIL

Test	Briquettes Charged Lbs.	Time after First Charge, Hrs.	Temp. Air to Oil Burner, ° C.	Temp. Attained in Furnace, ° C.	P_2O_5 in briquettes Per Cent	P_2O_5 in Slag Per Cent	P_2O_5 Volatilized, Per Cent of Total
1	153	6.5	15	Over 1400	20.46	11.52[1] 3.53[2]	49.12 85.82
2	97	1	100 to 150	Approx. 1400	20.46	11.36	50.19
3	110	6	200 to 270	1500	20.46	11.09[1] 10.66[2]	51.51 53.56
4	105	3.5	250 to 300	1500	20.46	8.64[1] 1.63[3]	63.24 93.55

[1] Bulk of slag.
[2] Evidently better fused slag.
[3] Part of this slag was left in the furnace from the previous test and its phosphoric acid content further reduced by the last heating of the furnace.

While it was only in the last run of the furnace that a slag was obtained nearly free from phosphoric acid, it appeared evident that by exposing the charge to a high temperature for a sufficient length of time nearly complete evolution of phosphoric acid could be brought about.

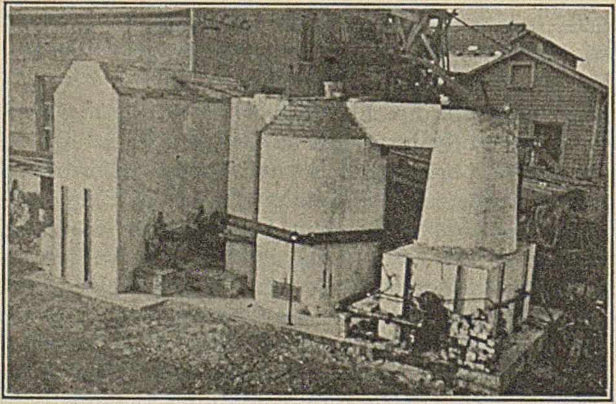


FIG. 3—NEW OIL BURNING FURNACE WITH AUXILIARY APPARATUS

The erection of a furnace better adapted for the treatment of the material and for the more efficient utilization of the heat of the burners was then begun. Auxiliary apparatus for burning of any combustible gas evolved and heating of the air to the burners by waste gases was constructed, and the whole system connected with a Cottrell precipitator for the collection of the volatilized phosphoric acid. This furnace (shown in Fig. 3) holding 700 lbs. of charge was completed early in January 1920, but adverse weather conditions and certain necessary changes in the equipment have delayed its running. Only one short test has been made so far, and the breaking of the belt to one of the motors made it necessary to cut off the burners before the experiment was completed. The results obtained in this short run, however, have convinced the workers that the form of the furnace is well adapted for the purpose and the principle used apparently sound. It is expected that interesting and valuable data will be obtained within the next few months which will show the commercial practicability of this process.

ECONOMIC CONSIDERATIONS

It is too early yet to prophesy the cost of producing phosphoric acid from mine-run phosphates by this new process, or to compare this method with that long in use for manufacturing soluble phosphates for fertilizer purposes. Certain advantages of this new process over the old sulfuric acid method, however, are so evident that they hardly need pointing out. For example, only high-grade phosphate rock containing a maximum of 4 or 5 per cent iron and aluminum oxides are at present used by the fertilizer manufacturers in producing acid phosphate. In order to obtain rock of this grade in Florida an elaborate washing and screening process is employed. This combined washing and screening process entails great losses of finely divided phosphate material which is discharged along with the impurities contained in the phosphate beds. The losses of phosphate thus entailed are fully twice as great as the quantity marketed and although much work and considerable money has been spent upon the problem, no mechanical or chemical method has yet been devised which will separate the phosphate from these impurities. It appears peculiarly fortunate that much of the material in the hard rock regions of Florida as it occurs in the mines has a composition almost ideally fitted for this furnace treatment. Instead of requiring reinforcement or enrichment by the addition of higher grade phosphate much of it must be brought to the desired composition by the addition of silica or sand, of which there is an unlimited supply at hand throughout the phosphate area. It will be seen, therefore, that the saving which can be effected by cutting out the expensive washing and screening plants, coupled with the advantage of prolonging the life of the phosphate deposits, will go far towards offsetting the additional expense, if any, involved in the preparation of the briquettes and the use of oil fuel rather than sulfuric acid as a reagent to convert the phosphoric acid into a soluble or available condition. Moreover, the product obtained is so concentrated that it can stand the expense of much longer shipments than the ordinary acid phosphate (which contains at best only from 16 to 18 per cent of soluble phosphoric acid) and by combining this concentrated phosphoric acid with ammonia and potash, fertilizers of the highest grade can be produced.

INVESTIGATIONS ON ZIRCONIUM[1]

By J. W. Marden and M. N. Rich

U. S. Bureau of Mines, Golden, Colorado

Received March 29, 1920

I—PREPARATION OF ZIRCONIUM OXIDE

In the past two years much interest has been shown in zirconia as a refractory and in zirconium alloys. The demand for purified zirconium oxide has exceeded the very limited production. The following material is an abstract of a portion of a report[2] submitted on this subject.

Although there is some supply of zircon sand to be had in this country, most of the experimental work recorded below has been done on the Brazilian ore (zirkite). The American zircon sand is relatively impure (40 to 60 per cent ZrO_2), and is much more difficult to get into solution than the Brazilian ore which consists of a mixture of zircon ($ZrSiO_4$) and baddeleyite (ZrO_2), having about 73 to 75 per cent ZrO_2.[3]

The following methods have been employed in decomposing zirconium ores:

1—Fusion with $NaHSO_4$, commercial niter cake, etc.[4]

2—Fusion with caustic soda or soda ash together with sodium peroxide.[5]

3—Fusion with $NaHF_2$ or treatment with hydrofluoric acid.[6]

4—Ignition with charcoal and subsequent chloridizing.[7]

5—Electric arc fusions of zircon, CaO and carbon, leaching with water and chloridizing of the residue.[8]

Table I—Efficiencies of Various Fusion Agents on Zirkite

Fusion Mixture	Ratio of Fusion Mixture to Ore	Time of Fusion	Per cent Decomposed
Na_2CO_3 KNO_3	5 1 : 1	1 hr. Clear liquid fusion	50
Conc. H_2SO_4, 180°–90° C.	10 : 1	Digested 0.5 hr.	15
Conc. H_2SO_4, boiling	4 : 1	Digested 5 hrs. and heated to dryness	80
Conc. HF	10 : 1	Boiled to dryness twice	70
$Na_2S_2O_7$	10 : 1	1 hr. Clear liquid fusion	60 Incomplete
NaOH Na_2O_2	3 1 : 1	20 min. Clear fusion	100
NaCl	10 : 1	1 hr. Clear fusion	Very Incomplete
Na_2CO_3 NaOH	1 2 : 1	0.5 hr. Clear fusion	90
Na_2CO_3 NaOH	1 2 : 1	1 hr. Clear fusion	100
Na_2CO_3 $Na_2B_4O_7.10H_2O$ (borax)	4 1 : 1	High temperature and continued fusion necessary for complete decomposition	...
NaF	5 : 1	0.5 hr.	100

After the ore is decomposed (with one exception) the zirconium is brought into solution and the hydroxide precipitated and converted into the oxide by ignition. The oxide is further purified by dissolving in acids and reprecipitating, or by recrystallization of a salt such as the oxychloride. Many trials of these methods were made on quantities of ore varying from one gram up to 10 lbs. (See Table I.)

These trials were made on small samples. On larger samples a longer time of fusion was necessary and in some cases stirring hastened decomposition.

On a commercial scale, the use of fluorides and saltpeter is expensive. The treatment of the ore with lime and coke and subsequent treatment with chlorine was attended with poor results, due partly to the small amounts of zirconium made soluble and partly to the difficulty of finding apparatus which would withstand the chlorine at the high temperature required for satisfactory chlorination.

The results of several trials in which zirkite ore has been fused and ZrO_2 subsequently produced with some success are described below.

(1) CAUSTIC SODA, SODA ASH FUSION AND PRECIPITATION WITH SO_2—One pound of zirkite (100 mesh) was sifted into a thoroughly fused mixture of 3 parts of Na_2CO_3 and 3 parts of NaOH. The fusion was continued for 1 hr., the mass cooled, crushed, and lixiviated three times with hot water. The filtered, washed residue was digested with 1 : 1 HCl, the solution filtered and diluted. The acid was neutralized as nearly as possible with soda ash without incurring precipitation and SO_2 passed into the hot solution. The precipitate yielded 0.5 lb. of nearly white oxide containing 98 per cent ZrO_2, about 70 per cent of the theoretical quantity. The total cost for this production, exclusive of labor and equipment, is about forty cents a pound.

(2) PHOSPHATE METHOD OF PRODUCTION OF ZrO_2—Ten pounds of niter cake were fused and one pound of zirkite (100 mesh) was sifted into the liquid melt. The fusion was continued for 1 hr., the mass cooled, crushed, and extracted with hot water. The liquid was filtered, some sodium peroxide and a small calculated excess of sodium phosphate added. The zirconium phosphate was filtered in a filter press, dried, and fused with 3 parts of caustic soda, crushed, leached with water, filtered, and the residue treated with diluted HCl to remove the iron and aluminum. The residue was dried and ignited. It gave a white oxide of 98 per cent purity at a cost of about forty cents a pound. A 60 per cent yield was realized.

(3) BASIC SULFATE METHOD—One pound or a larger quantity of ore was slowly heated with four parts of concentrated H_2SO_4 until free of fumes and the temperature gradually raised to 650° C. After cooling the flour-like residue was extracted with 50 parts of cold water, filtered, and sodium carbonate added to neutralize any free acid until the zirconium hydroxide just began to form. The solution was allowed to stand for 3 or 4 days, during which time the basic sulfate $4ZrO_2.3SO_3.14H_2O$ gradually separated out. When this was ignited a very high-grade ZrO_2 was obtained, which in one case contained 99.84 per cent ZrO_2 and was free from any determinable trace of SiO_2. Exclusive of labor and equipment the cost for the production

---

[1] Published by permission of the Director of the Bureau of Mines.

[2] The full report will be published by the U. S. Bureau of Mines in the near future. Thanks are due to Dr. R. B. Moore and Dr. S. C. Lind for their aid and advice during this work.

[3] For a discussion of the mining and concentration of the Brazilian ore see Meyer, *Foote Mineral Notes*, November 29, 1916.

[4] Franz, *Ber.*, **3** (1870), 58.

[5] Berzelius, *Ann. Mines*, [1] **12** (1826), 297; Schiotz ("Suggested Use of Na_2O_2 for Analytical Work"), *Tidskrift. Kem. Farm. Terapi*, **14** (1917), 256.

[6] Rose, *Ann. chim. phys.*, [3] **60** (1860), 260; Marignac, *Ibid.*, [3] **60** (1860), 257.

[7] Troost, *Compt. rend.*, **116** (1893), 1227, 1428; Moisson and Lengfeld, *Ibid.*, **122** (1896), 651.

[8] Troost, *Loc. cit.*

of 1 lb. of oxide by this method was about twenty cents.

(4) OXYCHLORIDE CRYSTALLIZATION METHOD—This method was tried in several cases for the purification of zirconium compounds but it was found to be both too tedious and expensive for commercial work.

II—PREPARATION OF PURE POTASSIUM FLUORZIRCONATE AND THE CRYSTALLINE ZIRCONIUM ALUMINUM ALLOYS

Before pure metal can be prepared, the principal object of the investigations, pure zirconium oxide or zirconium salts, must first be obtained. Even the oxide prepared by the basic sulfate method of crystallization contains various impurities. The quickest and most satisfactory method of purification is by means of successive crystallization of K_2ZrF_6, previously described by Marignac[1] and others.

The K_2ZrF_6 is prepared by dissolving ZrO_2 in hydrofluoric acid. This was done in the present work in lead vessels made for the purpose having a capacity of one to two gallons.

After heating, the oxide gradually dissolves and a clear solution is obtained. This is diluted and filtered (using a rubber funnel) into a clean lead dish. It should not come in contact with glass on account of the formation of fluorsilicates.

A filtered solution of C. P. potassium carbonate is added in sufficient quantity for the complete formation of all the K_2ZrF_6, but not enough to neutralize all of the free acid. If too much K_2CO_3 is added the zirconium is precipitated as the hydroxide, and more hydrofluoric acid is needed. Potassium carbonate should be used in place of potassium hydroxide because of its greater freedom from sodium salts. The sodium fluorzirconate is very insoluble and cannot be easily recrystallized.

The solubility of the potassium salt varies considerably with the temperature, as is shown from the following figures:

	G. in 100 cc. H_2O	
Temp. ° C.	K_2ZrF_6	Na_2ZrF_6
100	25.0	1.67
20	15.0	..
18	..	0.38
0	0.78	..

After cooling the hydrofluoric acid solution prepared above, the crystals of K_2ZrF_6 are filtered off. The crystals are redissolved in hot water containing a little hydrofluoric acid, filtered while hot, and allowed to cool. The crystals of K_2ZrF_6 again separate.

Starting with 98 per cent ZrO_2, three crystallizations yield a perfectly white crystalline salt which analyzed 32.10 per cent (theoretical: 32 per cent) Zr. A one-gram sample did not contain enough of either iron or titanium to give distinguishable colorimetric reactions after the fluorides had been removed and the salt was considered to be of at least 99.99 per cent purity.

In the production of zirconium metal the so-called "crystalline zirconium" alloy has also been used. This alloy has often been mistaken for zirconium metal on account of its great stability toward chemical agents and oxidation. It has been prepared by the method of Weiss and Newman.[1]

Recrystallized K_2ZrF_6 is fused in a graphite dish, in a gas furnace. In this investigation bored-out graphite sticks in the shape of deep cylindrical vessels were used. A temperature of about 900° C. is necessary to get a clear liquid. When the fusion is complete 1.5 times its weight of aluminum are added, a little at a time. During these additions a vigorous reaction takes place. When the reaction is finished the heating is continued for 10 to 20 min., the whole mass covered with a layer of sodium and potassium chlorides, and allowed to cool. When the dish is broken the aluminum alloy is found collected free of salt in the bottom of the dish. Flat plate-like, apparently monoclinic, crystals are to be seen on the surface. The alloy is carefully cleaned off, broken up with a chisel and treated with strong solutions of caustic soda until no more aluminum will dissolve. The residue is separated by decantation and filtration, then boiled with successive portions of 1 : 1 hydrochloric acid to remove the zirconium hydroxide which forms.

On filtering and drying the residue it is found to consist of silver-white crystals about 0.5 to 1 mm. across, and about as resistive to chemical action as zirconium metal itself. The more silicon impurity present the larger the crystals. The following is an analysis of this material:

	Per cent
Al	30
Zr	68
Fe, Si, small amts. Ti, etc.	1.0
TOTAL	99.0

A much purer product showed the following analysis:

	Per cent
Al	29.9
Zr	70.00
Fe	0.03
Si	0.05
Ti, not enough to determine	
TOTAL	99.98

Since the K_2ZrF_6 can be prepared in very pure condition, the purity of the crystals is governed very largely by the purity of the aluminum metal used. The so-called C. P. aluminum metal rarely runs over 99.5 per cent Al, the principal impurities being iron and silicon.

This aluminide (Zr_3Al_4) may be prepared in impure condition directly from zirkite by fusing the ore with sodium fluoride and then adding the aluminum. The treatment of the alloy (with caustic soda) after cooling is as above. Following is an analysis of this product, where a part of the aluminum is replaced by silicon:

	Per cent
Al	13.10
Zr	74.47
Fe	0.84
Si	11.15
Ti, not determined	
TOTAL	99.76

A crystalline silver zirconium alloy may also be prepared by the alumino-thermic reduction.

---

[1] *Loc. cit.*

[1] *Z. anorg. Chem.*, **65** (1910), 248.

III—PREPARATION OF ZIRCONIUM METAL

The literature refers to four varieties of zirconium metal: amorphous, crystalline, graphitoidal, and sintered. The properties given for these varieties of metal by various authors differ widely. The historical references have been omitted for the most part for the sake of brevity.

The following methods have been employed for the production of this metal:

A. AMORPHOUS—(1) Reduction of K_2ZrF_6 with sodium metal in an iron tube;[1] (2) Passing $ZrCl_4$ over heated sodium metal;[2] (3) Reduction of ZrO_2 with calcium metal.[3]

B. CRYSTALLINE[4]—Reduction of K_2ZrF_6 with large excess aluminum metal in a graphite dish. This so-called crystalline zirconium has been shown to be an alloy of aluminum and zirconium.

C. GRAPHITOIDAL—The interaction of sodium zirconate and iron is said to yield this variety[5] of the metal. After repeated failures in attempting to prepare it the conclusion is drawn that there is no such variety.

D. SINTERED[6]—In most cases the authors have introduced the word "coherent" for this variety of metal.

1—Alumino-thermic reductions produce coherent metal.

2—Heating the amorphous product obtained in A-3 yields small lumps or granules of partly coherent metal.

3—Reduction of ZrO_2 with carbon yields Zr metal mixed with carbide.

In the experimental work, all of these methods have been tried in the production of the amorphous and coherent varieties of zirconium.

The amorphous metal has been prepared by the following methods:

1—Reduction of K_2ZrF_6 in an evacuated iron tube with a small excess of sodium. This takes place on heating the tube to a dull red heat. The mass is cooled and extracted with dilute hydrochloric acid until all the salts are removed. Samples were prepared containing 98 per cent zirconium.

2—Reduction of K_2ZrF_6 with aluminum in the Arsem vacuum furnace, in which the excess salts were distilled away, yielded a good grade of amorphous zirconium contaminated with some aluminum.

3—Reduction of ZrO_2 with aluminum will under certain conditions yield an impure amorphous metal.

PROPERTIES OF AMORPHOUS ZIRCONIUM

PHYSICAL

Atomic weight: 90.6 (taken from literature).

Alloys: With one exception does not form alloys under ordinary conditions. Can be alloyed by heating with aluminum. Does not form amalgams.

Boiling point: Very high.

Corrosion: Remains unchanged at ordinary temperatures although when suspended in water it apparently reacts slowly to form the hydroxide.

Color: Black; heated above 1000° C. gray, metallic.

Electrical conductivity: Very low: black amorphous nonconductor.

Fusibility: According to Wedekind, it partly sinters at 1000° C. although we have never observed fusion at any such low temperature. The material apparently runs together to form the white coherent metal, looking like iron steel, a little above 1600° in a high vacuum in an atmosphere of exceedingly dry and pure hydrogen. If a small amount of air or oxygen is present, this fusion does not take place. The melting point of the zirconium is in the neighborhood of 1600°. When heated to 1000° C. the amorphous black metal changes to gray-colored material, taking a metallic polish.

Hardness: Soft, velvety. Ignited above 1000° C. granular.

Luster: Metallic when polished. When pressed into sticks at a high pressure and polished it has the appearance of a solid gray metal.

Melting point: About 1600° C.

Occlusion of gases: This material absorbs or occludes gases very readily. It also seems to have much affinity for moisture.

Specific gravity: About 4.0. The specific gravity varies with the amount of heating. The amorphous metal when heated in the high vacuum furnace gradually shrinks and the specific gravity increases until coherent metal is obtained, which has a specific gravity of more than 6.0. The amorphous metal as ordinarily prepared has a specific gravity of about 4.0.

Volatility: Does not volatilize at 2000° C. in a vacuum of 1 to 2 mm.

CHEMICAL

Combines readily with chlorine at low red heat; in nitrogen it burns to nitride at dull red heat. When heated in the air to a dull red heat, it burns to the oxide with a white incandescence. It is readily soluble in hydrofluoric acid even when dilute. It is slowly soluble in 1 : 1 hydrochloric acid. It is slowly soluble in nitric acid but more readily soluble in sulfuric acid. It dissolves in the ordinary fusion agents such as potassium bisulfate, caustic soda, etc. Amorphous zirconium formed by the reduction of K_2ZrF_6 with sodium will dissolve in water, forming bluish colloidal solutions which readily pass through the filter paper.

The coherent metal was prepared alumino-thermically in small beads which showed the following analyses:

	Per cent	
Zr	99.40	99.50
Ti	0.45	0.40
Fe	0.01	0.02
Al and Si, by difference	0.14	0.08
TOTAL	100.00	100.00

A typical charge for this alumino-thermic reduction was made as follows:

ZrO_2	90 g.
$KClO_3$	30 g.
Al	40 g.

All the materials were first purified, thoroughly mixed and finally fired in a fire-clay crucible.

The coherent metal was also prepared in the Arsem furnace in an evacuated atmosphere of very pure, very dry hydrogen by the reduction of K_2ZrF_6 with aluminum. This method is that used in the reduction to the amorphous, but this amorphous metal cannot be made to cohere unless the evacuated atmosphere in the furnace is entirely free from moisture and air. The following is an analysis of this metal:

	Per cent
Fe and Si	0.2
Al	Absent
Zr	99.5
TOTAL	99.7

Coherent metal was also prepared by arcing pressed sticks of the crystalline zirconium-aluminum alloy in an evacuated atmosphere of hydrogen.

PROPERTIES OF COHERENT ZIRCONIUM

PHYSICAL

Alloys: Does not dissolve in lead, tin, or silver by simply heating above the melting point of the respective metal.

---

[1] Berzelius, *Ann. Phys. Chem.* (Pogg.), **4** (1825), 124; **8** (1826), 186.

[2] Troost, *Compt. rend.*, **61** (1865), 109.

[3] Wedekind, *Ann.*, **395** (1913), 149.

[4] Weiss and Newman, *Z. anorg. Chem.*, **65** (1910), 248; *Am. J. Sci.*, [4] **29**, 457; *J. Soc. Chem. Ind.*, **29**, 218.

[5] Troost, *Loc. cit.*

[6] Kuzel and Wedekind, U. S. Patent 1,088,909 (1914).

Will alloy with silver, aluminum, and iron when heated above the melting point of zirconium in the Arsem furnace. Alloys of silver, iron, nickel, or aluminum are easily made by simultaneous reductions at high temperatures (Goldschmidt's process). Does not form amalgams.

Corrosion: Does not rust or tarnish on long standing in the laboratory but when polished remains bright white like polished nickel.

Color: White metallic.

Electrical conductivity: Fairly good conductor. Probably less than that of iron.

Fusibility: Can be readily fused under the proper conditions away from the air at about 1600° C.

Hardness: About 6–7 on the Mohs scale. The hardness varies with the method of preparation and purity of samples. The pure samples were less hard than the more impure metal. In all cases the metal would readily scratch glass. As would be expected with a metal of this degree of hardness it is also quite brittle and can easily be broken by lightly pounding with a small hammer. The metal is still very hard although less brittle at a red heat. The hardness on the Shore scleroscope is 40 to 45.

Luster: White metallic. Takes a fine polish like nickel.

Melting point: About 1600° C. The U. S. Bureau of Standards has found the melting point of the metal from 1400° to 2200° C. as it was prepared by various authors. In recent trials the metal prepared appears to melt very close to 1600° C. in an atmosphere of pure dry hydrogen.

Occlusion of gases: When samples of zirconium are fused in pure dry hydrogen and rapidly cooled, a phenomenon similar to the so-called "spitting" of silver is observed. When silver is fused it dissolves oxygen and on cooling the oxygen is evolved, causing the "spitting" and leaving the metallic mass porous. The zirconium, when similarly treated in hydrogen, shows the same porous appearance. This porosity is not observed in samples prepared by the Goldschmidt process.

Specific Gravity: Partially sintered amorphous zirconium shows a specific gravity of 4.39. The specific gravity of the alumino-thermic, 99.5 per cent zirconium is 5.55, while the specific gravity of 99.5 per cent coherent metal prepared by the reduction of K_2ZrF_6 with aluminum was found to be 6.06. The specific gravity of zirconium, as with other metals, varies with the method of treatment.

CHEMICAL

The white metal is much less chemically active than the amorphous metal. It does not react with chemical reagents readily at ordinary temperatures. This variety of the metal in a solid piece can be heated to a bright red heat in the blast flame with no more than a very thin bluish superficial surface oxidation. It can be burned in the air by heating to a white heat in the finely divided condition. It is insoluble in all acids except aqua regia and hydrofluoric acid. The metal dissolves easily in hydrofluoric acid (1 : 1) but it takes at least 5 hrs. to dissolve one gram of the solid metal by digesting in an excess of aqua regia. The metal is not readily dissolved by fusion with bisulfates, caustic soda, etc.

IV—COMPARISON OF THE METHODS OF ANALYSIS

On attempting the analysis of zirconium ores by the different methods in the literature very conflicting results were obtained. For example, a sample of zirkite ore gave the following percentages of ZrO_2 by different methods:

TABLE II—ANALYSIS OF ZIRKITE ORE BY DIFFERENT METHODS

Method Employed	Weight of Ore (or Aliquot) G.	Weight of Phosphate G.	Weight of ZrO_2 G.	ZrO_2 Per cent
Phosphate..............	0.0853	0.1215	0.0626	73.3
	0.0707	0.1010	0.0520	73.5
Sodium thiosulfate......	0.2000	....	0.1740	87.0
	0.2000	....	0.1700	85.0
Sodium iodate..........	0.2000	....	0.0947	47.35

The results obtained by the phosphate method are very nearly the correct ones but an inexperienced worker would have trouble in deciding just what percentage of ZrO_2 the ore should have. The thiosulfate precipitate is high, due to occlusion of large amounts of sodium salts, while the iodate results are low due to too strong acidity of the solution for precipitation.

The following methods in more common use are listed and comparisons are made as to their relative merits. The more important methods for the estimation of zirconium are as follows.

1—The phosphate precipitation. Hillebrand, U. S. Geol. Survey, *Bull.* **73** (1900); Lundell and Knowles, *J. Am. Chem. Soc.*, **41** (1919), 1801; Nicolardot and Reglade, *Compt. rend.*, **168** (1919), 348.

2—The thiosulfate or SO_2 precipitation. Baskerville, *J. Am. Chem. Soc.*, **16** (1894), 475; Ferguson, *Eng. Min. J.*, **106** (1918), 356, 793.

3—The iodate separation. Davis, *Am. Chem. J.*, **11** (1889), 25.

4—The peroxide precipitation. Bailey, *J. Chem. Soc.*, **149** (1886), 481; Walker, *J. Am. Chem. Soc.*, **20** (1898), 513.

5—The phenylhydrazine precipitation. Allen, *J. Am. Chem. Soc.*, **25** (1903), 421.

6—The cupferron precipitation. Ferrari, *Atti ist. Venata Scienze Lettere ed arti*, **73** (1914), 445; Brown, *J. Am. Chem. Soc.*, **39** (1917), 2358.

7—The fluoride separation. Headden, *Proc. Colo. Sci. Soc.*, **11** (1917), 185.

In a study of these methods it was found that the fluoride separation is incomplete and the peroxide precipitation takes place only when the concentration of the peroxide is very high in the solution. Methods 4 and 7 were therefore given but scant attention.

TABLE III—COMPARISON OF RESULTS BY DIFFERENT METHODS

Weight of ZrO_2 Taken Grams	Weight of ZrO_2 Found	Variation
Phosphate Method:		
0.0865 ZrO_2	0.0875	
0.0357 Fe_2O_3	0.0877	+0.0011
0.0471 Al_2O_3		
	Av., 0.0876	
Sodium Thiosulfate Method:		
0.0865 ZrO_2	0.0845	—0.0018
No Fe_2O_3 or Al_2O_3	0.0850	
	Av., 0.0847	
0.0865 ZrO_2	0.0988	No check; large amts. iron and alum in precipitate
0.0643 Al_2O_3	0.1873	
0.1514 Fe_2O_3		
Phenylhydrazine Method:		
0.0865 ZrO_2	0.1370	ZrO_2, 0.0865
0.0357 Fe_2O_3	0.1388	Al_2O_3, 0.0471
0.0471 Al_2O_3		
	Av., 0.1380	0.1336 + 0.0044
Cupferron Method:		
0.0865 ZrO_2	0.1213	ZrO_2, 0.0865
0.0357 Fe_2O_3	0.1219	Fe_2O_3, 0.0357
0.0471 Al_2O_3	0.1213	
		0.1222 — 0.0007
	Av., 0.1215	

The phosphate is best precipitated from about 10 per cent sulfuric acid to which H_2O_2 is added to retain the titanium in solution. An excess of disodium phosphate should be used, the solution heated to boiling and allowed to stand, before filtration. Due to the gelatinous nature of the precipitate and difficulties in washing no larger quantity than 0.05 to 0.1 g. of ZrO_2 should be precipitated from 200 cc. of solution at one time.

It has been found from Table III and similar tables that:

(1) The phosphate gives fairly good results in the presence of iron and aluminum but a 50 per cent excess of ammonium phosphate should be used when the acidity lies between 10 and 20 per cent H_2SO_4.

(2) The sodium thiosulfate method cannot be used in the presence of much iron and aluminum salts.

(3) Phenylhydrazine precipitates the zirconium together with the aluminum, away from the iron, but this precipitate is slimy and difficult to wash free of impurities. The results by this method should equal the weight of ZrO_2 + the weight Al_2O_3 but are too high *for this reason.* If done in dilute solution the phenylhydrazine separates the Zr and Al from the iron.

(4) Cupferron serves to separate Zr and Fe from aluminum with a fair degree of exactness. The cupferron precipitate is a desirable one to handle.

(5) Although not shown here, the sodium iodate method can be used with accuracy in carefully neutralized solutions. It is subject, however, to somewhat the same limitations as the sodium thiosulfate method.

A method of analysis for zirconium which could be applied to alloys must be such as to include separation from Fe, Al, V, Cr, Ni, Co, Ti, Si(?), and perhaps other elements. The method of analysis of ores is not complicated, as elements such as V, Cr, Ni, and Co are not usually found in zircon ores.

RECOMMENDED METHOD OF ANALYSIS

PREPARATION OF SAMPLE—The alloys, which are for the most part brittle, are pulverized as far as possible in a steel mortar, while the ores are pulverized to pass a 100- to 200-mesh sieve before analysis is undertaken. The thorough grinding of samples of the ore is very important as much of the ease with which the samples go into solution in various fusion agents depends on the fineness of subdivision of the sample.

METHOD OF SOLUTION AND SEPARATION OF THE SILICA—Alloys should be dissolved in aqua regia using about 0.5 to 1 g. of the alloy and, after the solution has been made, diluting it up to 500 cc. or a liter so that a suitable aliquot of 100 cc. would represent about 0.05 g. of ZrO_2 for analysis.

If the percentage of zirconium and silicon in the alloy is high, the process of dissolving it in aqua regia will be slow. It is occasionally necessary to use hydrofluoric acid in a platinum dish in preparing the solution. The use of this is objectionable when the percentage of silicon is desired and the following procedure is to be preferred:

After treatment with aqua regia and evaporating the solution nearly to dryness to separate the silica, if there is an insoluble residue other than silica remaining, the solution is diluted, filtered, and the residue ignited in a platinum crucible. This is then fused with sodium or potassium bisulfate, the fused mass cooled, dissolved in water, and the insoluble silica again filtered and washed. The filtrate is combined with the filtrate from the aqua regia treatment in a 500-cc. volumetric flask. The silica is determined by the usual method of volatilization with HF and H_2SO_4, and if there is any weighable residue remaining after this treatment, this must be brought into solution by means of a second fusion with bisulfate, the solution from this fusion being combined with the other filtrates in the volumetric flask. Suitable aliquots of this solution, made up to 500 cc. and containing 5 per cent H_2SO_4, are taken for the zirconium determination. In no case should more than 0.1 g. of ZrO_2 be taken.

In the case of ores, 1 g. of the ore is fused with four parts of NaOH and one part of Na_2O_2 in a nickel crucible to a clear, dull red liquid fusion for 15 or 20 min. The fused mass is cooled and dissolved out from the crucible with warm water to make a volume of about 100 cc. The zirconium and most of the titanium are in the form of insoluble sodium zirconate and sodium titanate suspended in the solution. The solution is then made about 10 per cent acid with sulfuric acid and heated to dissolve all the zirconium and titanium. After boiling down nearly to dryness the solution is again made up to about 200 cc. and the silica filtered off, together with any insoluble residue. The filtrate is reserved in a 500 cc. volumetric flask, while the silica is determined in the residue by the customary H_2SO_4 and HF treatment. If there is a residue left after the hydrofluoric-sulfuric acid treatment this must be again fused with caustic soda and sodium peroxide and the solution obtained by the subsequent acid treatment combined with the filtrate in the volumetric flask for the analysis.

It is frequently the case that two and even three fusions are necessary in order to obtain a complete solution. It might be further suggested in the determination of the silica that the residues for this purpose be first ignited with a little sulfuric acid and weighed before the hydrofluoric acid treatment is attempted. If this is not done even where there is a small percentage of silica sometimes the weight will be found to increase instead of decrease as it should.

ANALYSIS OF SOLUTION CONTAINING Fe, Al, Cr, Ni, Mo, W, Ni, Co, Zr, Ti, AND P—The acid solution which is now free of silica and has been drawn off in a suitable aliquot to contain about 0.05 g. ZrO_2 is heated nearly to boiling (about 90° C.) and ammonium hydroxide added until a slight permanent precipitate is formed. Dilute HCl (1 : 1) is added from a burette drop by drop until the precipitate which has formed just redissolves and the solution is perfectly clear. About five drops more of the HCl are added to the solution, which should now have a volume of about 200 cc. Three cc. of phenylhydrazine which have previously been dissolved in 10 cc. of hot water are added, and the solution stirred vigorously and filtered immediately through quantitative paper. After washing thoroughly with hot water, the paper and precipitate are dried and ignited in a platinum crucible. This procedure separates practically all of the iron from the zirconium, titanium, and aluminum. In order to separate further the zirconium and titanium from the aluminum the following method is employed: Fuse the residue in the platinum crucible with 5 to 10 g. of Na_2CO_3 and one g. KNO_3; after cooling disintegrate the fused mass with hot water, filter, and wash. The residue on the paper now contains zirconium and titanium free from all interfering substances such as aluminum, vanadium, etc., with the possible exception of traces of iron. The paper containing the sodium zirconate and titanate is dried and ignited in a platinum crucible and the titanium and zirconium obtained in solution by cautiously fusing with 5 to 10 g. of $KHSO_4$, bringing the temperature up to a dull red heat for about one-half hour and using a cover on the crucible to avoid spattering during the process of fusion. The zirconium may be determined by practically any of the methods mentioned above, using this solution, provided that the quantity of zirconium in the solution is not greater than 0.1 g. calculated as ZrO_2. Most precipitates of zirconium are more or less gelatinous, bulky, and difficult to filter and wash, and therefore if larger quantities are taken, accurate results cannot be attained.

If the zirconium alone is desired and there is no need of separating the iron, aluminum, etc., from the solution, the phosphate method is the only one which gives accurate results and a clean separation from both the above-mentioned elements.

The solution which has been freed from silica, containing not over and preferably less than 0.05 g. ZrO_2, is diluted to 200 cc. volume, made 10 per cent acid with H_2SO_4, 2 cc. hydrogen peroxide added, heated nearly to boiling and a slight excess of disodium phosphate solution added. The zirconium precipitate is a white or greenish white flocculent, gelatinous precipitate. After allowing to stand for about 2 hrs. or longer the precipitate is filtered onto an 11 cm. filter paper and thoroughly washed with hot dilute sulfuric acid. The paper and contents are then transferred to a platinum crucible, ignited, and finally blasted to remove as much of the carbonaceous matter as possible. The proper ignition of this precipitate is one of the most difficult steps of the whole procedure. The weight of zirconium pyrophosphate, ZrP_2O_7, multiplied by the factor 0.4632 yields the weight of ZrO_2.

Iron is determined in the solution from the silica filtration by the method of precipitation as the sulfide from ammonium tartrate solution. This method is well known. To 100 cc. portions of the silica-free solution 0.5 g. of tartaric acid is added, the solution heated to boiling, made strongly alkaline with NH_4OH, and H_2S added. The iron precipitates as the sulfide, is filtered out, washed, and weighed as Fe_2O_3.

The titanium may also be determined on a separated portion of the original solution (if all HF or HNO_3 has been first removed) by Weller's colorimetric method with H_2O_2 or by titration with methylene blue.

Aluminum is usually determined by difference, the aluminum, zirconium and titanium being precipitated with sodium thiosulfate, SO_2, or phenylhydrazine practically free from iron, if the percentage of iron in the original is not high. In certain cases it is necessary first to remove the iron by the above-mentioned method and destroy the ammonium tartrate by the ordinary methods of wet combustion with nitric and sulfuric acids before the precipitation of the others is attempted.

V—BIBLIOGRAPHY

Before much work on zirconium was attempted, all of the methods for the production of the metal, etc., described in the literature were reviewed and tried. At the start there were many conflicting data regarding the properties and reactions of zirconium and its compounds. A complete bibliography of the subject contained about five hundred references, from some forty-one different journals.

It has been arranged in alphabetical order with regard to names of authors under the following headings:

1—Zirconium Minerals
2—Preparation, Properties, and Uses of Zirconium Salts
3—Analytical Chemistry of Zirconium
4—Zirconium Metal
5—Zirconium Alloys

The bibliography also includes references on the use of ZrO_2 as a refractory and as an opaquing agent in enamels, etc. All of the technical uses have been faithfully included. Section 5 has been devoted to the preparation and properties of zirconium alloys.

A CHEMICAL STUDY OF FROZEN FISH IN STORAGE FOR SHORT AND LONG PERIODS

By E. D. Clark and L. H. Almy

Food Research Laboratory, Bureau of Chemistry, Philadelphia, Pa.

Received January 26, 1920

The present investigations upon fish commercially frozen and stored were undertaken in the hope of furnishing exact information upon which the public could base its opinion concerning the wholesomeness of frozen fish as food. The part of these investigations of practical interest to the trade has already been published in *Departmental Bulletin* 635 of the U. S. Department of Agriculture, and entitled "The Commercial Freezing and Storing of Fish."

The magnitude of the frozen fish industry of this country is indicated by the fact that over 120,000,000 lbs. of food fish,[1] valued at more than $12,500,000 to the wholesaler, entered cold storage in 1918. Depending upon the locality, the amount of frozen fish in storage during a year represents from 4 to 15 per cent of the annual catch.[2] Furthermore, there are over 190 fish-freezing and storage plants in the United States, and the number is increasing. These statistics show that the practical advantage and economic necessity of cold-stored fish have long been realized by those familiar with the industry. Until recently, however, no chemical investigation has been made of the effect upon the food value of the freezing and storing of fish.

HISTORICAL

In 1913 several Dutch scientists[3] made experiments with ice-chilled and frozen fish to see what changes in microscopical appearance occurred under refrigeration and what part bacteria played in the process of deterioration. This commission found that certain changes take place in the reaction of the tissues to litmus, the flesh of some species being acid, and, of

---

[1] Estimate based on U. S. Dept. of Agriculture, Bureau of Markets, Report of Stocks of Frozen and Cured Fish, December 15, 1918; issued December 28, 1918.

[2] Hearings before the Committee on Agriculture, House of Representatives, on Cold Storage Legislation, August 14, 1919, p. 203.

[3] J. M. Bottemaine, "Notes on the Investigation of Preserving Fish by Artificial Cold." (*a*) Preliminary Report, 48 pages; (*b*) Preprint, 3 pages; 3rd International Congress of Refrigeration, 3rd Section (The Netherlands), Washington-Chicago (1913).

some, alkaline during decomposition. Some differences in the histology of fresh and frozen fish were noted. All of this work was, however, of a preliminary nature and not in any sense an exact chemical investigation.

The quantitative chemical study of changes in fish flesh upon standing or during cold storage is a new one. The researches of Richardson and Scherubel,[1] Emmett and Grindley,[2] and others upon meat during storage opened the way in that field. Pennington[3] and Wiley, *et al*,[4] have done fundamental work on the chemistry of ripening processes in the flesh of poultry and game.

The investigations of Smith[5] and of Perlzweig and Gies[6] on chemical changes in fish during cold storage are the pioneer contributions to our knowledge of this subject. They determined the chemical composition, ammonia content, etc., of fresh fluke, *Paralithys dentatus*, and flounder, *Pseudopleuronectes americanus*, before freezing, and also made the same determinations at intervals during two years on the same lot of storage fish. These fish were all handled according to the usual trade practices, except that they were probably frozen more promptly after catching than the average commercially frozen fish. Even at the end of two years of storage these investigators were unable to detect any appreciable variations in chemical composition or nutritive value attributable to freezer storage. There were no significant changes in the percentages of the ordinary food constituents, or of ammoniacal nitrogen, acidity of fats or reaction of the aqueous extracts; all of which would tend to show that neither autolytic nor bacterial influences had been active. Furthermore, two years after storage the fish were found palatable and entirely acceptable as food.

Recently, Almy[7] investigated the chemical changes in mackerel (*Scomber scombrus*) after freezing and cold storage for 9 mo. Unlike the so-called "bottom" fish used in the researches of Smith, which contain less than one per cent of fat, mackerel are tender, oily fish, very prone to rapid deterioration unless handled carefully and kept constantly under refrigeration. This investigation represented the first attempt to study the nitrogen distribution in the flesh of frozen fish, but, as the work was a continuation of that reported in the present paper, no attempt will be made to describe the methods used but only to summarize the conclusions based on the study of a typical fat-containing fish. Almy found that during storage there were in frozen mackerel slight increases in the amount of total nitrogen and coagulable nitrogen in the aqueous extracts, but no change in the content of ammonia and amine nitrogen. There were also slight increases in the acidity of the aqueous extracts of the flesh and of the fat extracted from the flesh of the cold stored mackerel. In no case, however, was there any significant change in the food value of the fish or any evidence of deterioration at the end of 9 mo. At the end of this period of storage some of the mackerel were cooked and prepared for the table. Unlabeled portions of these cooked storage mackerel and of perfectly fresh mackerel were served to a representative group of 15 people in New York City, and none of them was able to detect any difference between the fresh and frozen fish as judged by appearance and flavor.

METHODS OF THE PRESENT INVESTIGATION

HISTORY OF FISH ANALYZED

Two species were used in the present investigation: weakfish, *Cynoscion regalis*, and bluefish, *Pomatomus saltatrix*, which is carnivorous and a voracious enemy of all fish smaller than itself. The medium sized fish of both species were studied. All of the specimens were caught off the New Jersey coast and promptly landed at the shore without any contact with ice. In all the lots but one the fish were eviscerated before they were landed. As soon as possible the different lots were weighed and carted a short distance to a fish freezer, where the specimens for freezing were washed and frozen in pans on brine pipes in the usual way. The temperature of the freezing room was —5° F., and of the storage room about 0° F. This is lower than the customary storage temperature, but the isolated nature of the plant necessitated such a practice as a safeguard to allow time for repairs in case of a break in the refrigerating machinery. The fish to be analyzed immediately in the freshly frozen condition were quickly chilled and frozen, and while still at a temperature below 0° F. were taken to the laboratory in Philadelphia where they arrived hard frozen. They were then thawed slowly by standing over night in an ice-box, cleaned, and prepared at once for analysis in the laboratory. The frozen fish for long holding were shipped in cork-insulated shipping cases, and upon reaching Philadelphia were at once transferred to a storage room where a temperature of about 15° F. was maintained during the various periods of storage. Each lot was boxed in the commercial way and reglazed whenever necessary, which was about three times during a year. As indicated in the tables, some of the weakfish were not glazed. One lot was stored without removal of entrails (in the so-called "round" condition), and another lot was wrapped in commercial semi-parchment paper without any glazing. These variations from the commercial practices were made in order to study the effects of such modifications.

PREPARATION OF SAMPLES FOR ANALYSIS

The thawed fish were prepared for sampling by separating as completely as possible the edible portion from the heads, tails, skins, entrails, and bones, which were discarded. The edible part was weighed and the difference between this weight and that of the whole fish represented the refuse or inedible portion. The flesh was ground three successive times in a meat chopper, and the samples of individual fish mixed thoroughly

---

[1] This Journal, **1** (1909), 95.

[2] *Ibid.*, **1** (1909), 413.

[3] Hearings before Committee on Manufactures, U. S. Senate, on Foods Held in Cold Storage, May 25, 1911, p. 57.

[4] U. S. Dept. of Agriculture, *Bulletin* **115** (1908).

[5] *Biochem. Bull.*, **3** (1913), 54.

[6] *Ibid.*, **3** (1913), 69.

[7] L. H. Almy, unpublished report of a study of the chemical changes occurring in frozen mackerel during storage for a year. Research performed in partial fulfillment of the requirements for the degree of Doctor of Philosophy at Columbia University, 1917.

by hand. In the case of bluefish, which were of considerable size, two specimens were used; but of the weakfish three were analyzed in each lot, the edible portions being united, ground again, and well mixed before final sampling for analysis.

The following section contains a description of the various determinations of chemical changes in the fish during cold storage, and is intended to include every determination that offered any possibility of giving useful information about fish flesh and its changes. Possibly, some of these determinations are unnecessary and some may be of little value, but all should help to give information about the adequacy of the methods themselves as applied to problems of this sort. Most of the determinations have been adapted from the work of early investigators upon meat and poultry. Any new methods or modifications will be apparent to those familiar with chemical work on cold storage problems.

ANALYTICAL PROCEDURE

MOISTURE, ETHER EXTRACT, ASH AND TOTAL NITROGEN—The methods used for the estimation of the percentage of moisture, ether extract, and ash are those described in *Bulletin* 107 (revised) of the Bureau of Chemistry, U. S. Department of Agriculture. Total nitrogen was determined by the method of Kjeldahl and Gunning, using 0.1 *N* acid and alkali.

FAT-FREE SOLIDS—It is a well-recognized fact that the flesh of fat fish contains a lower per cent of moisture than that of lean fish. The reciprocal relation of the per cent of moisture and fat in the flesh of fish of the same species is such that the per cent of fat-free solids is more or less constant. An increase of 2 per cent in the fat-free solids therefore shows that the per cent of moisture has decreased by 2 per cent. The per cent of fat-free solids was computed in each case by subtracting the per cent of fat from the per cent of solids.

AMMONIA AND AMINE NITROGEN BY AËRATION—Loosely bound basic nitrogen compounds, which in fish flesh include ammonia and amine nitrogen, were estimated by a modification of the Steel-Gies method described by the authors.[1]

TOTAL SOLUBLE NITROGEN AND COAGULABLE NITROGEN—The extraction of the flesh and the determinations of soluble nitrogen and coagulable nitrogen were made by the methods previously outlined.[1]

SOLIDS—One hundred cc. portions of the water extract of the flesh were evaporated on the steam bath in weighed platinum dishes, the residue being dried at 100° C. to constant weight.

ASH—The organic matter of the soluble solids was burned off in a muffle at low red heat to avoid any loss of chlorides.

ORGANIC EXTRACTIVES—This is the difference between ash and solids already determined.

ACIDITY OF THE EXTRACTS—Owing to the presence of both proteins and phosphates in extracts of fish flesh, it is not believed that direct titrations of acidity can give results of absolute value. For comparative studies in investigations of this sort, however, such direct titrations would certainly show any significant increases in acidity due to changes occurring during a prolonged period of storage. Correction was made for the very slight acidity of the distilled water used in preparing the extracts. One hundred cc. of the original extract were titrated with 0.1 *N* sodium hydroxide, using phenolphthalein as indicator. The end-point was taken as the point at which the alkali produced a pink color, permanent for one minute. In the tables the results are stated as the number of cc. of 0.1 *N* sodium hydroxide required to neutralize the entire extract from a 50 g. sample.

NITROGEN PRECIPITATED BY TANNIN AND SALT—Proteose and peptone nitrogen were determined by the method of Bigelow and Cook.[1] Fifty cc. portions of the extract, after removal of coagulable protein, were treated in a 100 cc. volumetric flask with 15 g. of sodium chloride, and, when the latter was thoroughly dissolved, chilled to ice-box temperature (12° C.), treated with 30 cc. of a 24 per cent solution of tannic acid (cold), made up to volume with cold water, shaken, and allowed to stand at the temperature of the ice-box for 24 hrs., after which the precipitate was filtered off. The solution was kept cold, and the nitrogen in 50 cc. of the filtrate was determined according to the usual Kjeldahl procedure, except that the addition of potassium sulfate was unnecessary because of the large amount of sodium chloride present. Care was taken during the early part of the oxidation that the escaping hydrochloric acid did not cause the material to foam out of the flask. Since tannic acid invariably contains a certain amount of nitrogen, a blank determination must be made with every experiment. The per cent nitrogen in the tannin salt precipitate was obtained by subtracting the per cent of nitrogen in the tannin salt filtrate from the per cent of non-coagulable nitrogen. The results obtained by this method were sometimes erratic but are included in the table as an indication of changes in peptone and proteose nitrogen.

PROTEOSE NITROGEN BY ZINC SULFATE PRECIPITATION—For this determination 50 cc. of the filtrate from the coagulated protein were concentrated to 30 cc. in a 100 cc. beaker and the resultant solution was saturated with zinc sulfate, according to the method of Bömer.[2] When cold, the solution was filtered through a thin asbestos layer in a Gooch crucible and the precipitate washed with a saturated solution of zinc sulfate. The precipitate, together with the asbestos mat, was transferred to a Kjeldahl flask, and the nitrogen determined as usual.

NITROGEN IN PEPTONE BASES, ETC.—The figures under this heading in the tables were obtained by subtracting the sum of the amine and ammonia nitrogen, amino-acid nitrogen, proteose nitrogen, and coagulable nitrogen from the total soluble nitrogen.

AMINO-ACID NITROGEN—This determination was made according to the general method of Sörensen[3] on 50 cc. of the filtrate after removal of coagulable nitrogen

---

[1] *J. Biol. Chem.*, **33** (1918), 486.

[1] *J. Am. Chem. Soc.*, **28** (1906), 1485.

[2] *Z. anal. Chem.*, **34** (1895), 563.

[3] *Biochem. Z.*, **7** (1907), 64.

in the extract. The presence of ammonia and the amines tends to interfere with the Sörensen titrations for amino acid but the results have comparative value in detecting changes during cold storage.

AMMONIA AND AMINE NITROGEN BY AËRATION—Portions of 150 cc. of the original extract were analyzed for ammonia in the same manner as the fish flesh.

DETERMINATION OF FAT CONSTANTS—The methods employed for the gross extraction of the oil from the flesh and for the determinations of specific gravity, index of refraction, iodine number, saponification number and acid value of the oil are the same as those described by the writers.[1]

In the tables, the average of two or more concordant determinations is given in every case.

OBSERVATIONS AND RESULTS OF CHEMICAL ANALYSES

(1) *Unglazed Eviscerated Weakfish Stored without Wrapping (Series 1001)*

APPEARANCE AFTER STORAGE—These weakfish, stored with neither a glaze nor parchment wrapping, were unmarketable at the end of 4 mo. The whole fish were dried and wrinkled in appearance, while the noses and eyes were whitish and dried out.

At the end of 8 mo. this lot of fish was very uninviting in appearance and unmarketable. As they were unfit for food, no further samples were taken after 8 mo. of storage. The whole fish were badly wrinkled, and white. The eyes were also white and sunken. When cooked the flesh seemed tasteless and dry.

GROSS ANALYSIS—The quantitative variations in the composition of certain fish due to season, age, and individual differences are often very great, as shown by previous investigators and confirmed by the present writers,[2] and the differences attributable to the two latter causes must be borne in mind before giving undue emphasis to variations in the gross analysis of cold stored fish in the accompanying tables. The tendency to lose moisture during storage is common to all flesh products. The loss of moisture from these unglazed fish was greater than that from any of the other fish as shown by the chemical analysis. The percentage of fat varied greatly but this is a characteristic of weakfish and was not due to changes in cold storage. The acidity of the aqueous extract showed no definite change.

NITROGENOUS CONSTITUENTS—Any of the minor but significant changes due to bacterial or enzyme action on the nitrogenous constituents of the stored fish would be indicated by the amounts of such constituents, but more especially by their relation to the total nitrogen, commonly called the "distribution of nitrogen." In general, these unglazed weakfish at the end of 8 mo. showed no greater changes in their nitrogen distribution than the glazed eviscerated weakfish (Series 1002) at the same period. Apparently, 8 mo. of storage temperatures did not produce significant alteration in the nitrogenous constituents of the frozen weakfish even without the protection of this glaze. Ammonia and amine nitrogen increased in amount, but only at about the same rate as in the glazed weakfish. The changes in the soluble and coagulable nitrogen were irregular.

CONSTANTS OF FAT EXTRACTED FROM THE FLESH—Since there is known to be considerable variation in the chemical properties of oils from individual fish, depending on their food, the relation to time of spawning, etc., it does not seem wise to consider that any of the changes in the composition of the fat in these fish, as recorded in Table IV, were due primarily to storage except in the case of the acid value.[3] This value at the end of the 8 mo. storage period showed a considerable increase as compared with that of the oil of the fish when first frozen. However, this change in the acid value was of the same magnitude as that of glazed weakfish (Series 1002) at the same period. Although this lot of weakfish did not have the protection of either glaze or wrapping, it showed no greater signs of aging than similar glazed fish; hence, we may conclude that the slight changes in oils of these frozen fish during the 8 mo. of storage are probably not due to their oxidation by oxygen of the air.

(2) *Glazed Eviscerated Weakfish Stored without Wrapping (Series 1002)*

APPEARANCE AFTER STORAGE—Up to the end of 13 mo. the fish of this lot looked bright under their glaze and were neither white nor wrinkled, except to a slight extent on the nose. The eyes were bright but slightly sunken. After cooking, the flesh had a normal appearance and flavor.

After 2 yrs. in storage all of these weakfish were unmarketable owing to the whitish and wrinkled appearance, especially of the heads. The eyes were opaque and much sunken, and the gills yellow and dry. Some of the fins were yellowish and dry. When cooked, they were not unpalatable, but tasteless and flat.

GROSS ANALYSIS—There were no noteworthy changes in the amounts of the various constituents during storage, aside from the usual variations found in the composition of fish of the same species even when of nearly uniform size and taken from the same school. Loss of moisture based on percentage of fat-free solids continued to occur until the twelfth month of storage, after which the results do not seem to indicate further loss of moisture. There was no definite increase in the acidity of the aqueous extract of the flesh.

NITROGENOUS CONSTITUENTS—The percentage of ammonia and amine nitrogen made slight but definite increases during storage. However, these increases were not as marked or as uniform as in the case of frozen bluefish (Series 1006). The per cent of total soluble nitrogen increased up to the eighth month, then decreased. The per cent of coagulable nitrogen showed a similar rise and fall. The percentages of amino-acid nitrogen, as determined by the Sörensen titration method, showed a slight tendency to increase in the storage fish.

CONSTANTS OF FAT EXTRACTED FROM THE FLESH—The only fat constant which changed during storage was the acid value, the latter increasing at an irregular rate during the entire storage period of 2 yrs. and 1 mo. As this was interstitial fat, the increase in the acid value was probably due to enzyme action rather than to bacterial or oxidation changes which might affect external fat. From unpublished work, it is known that the flesh of properly frozen fish is practically sterile.

(3) *Unglazed Eviscerated Weakfish Stored with Paper Wrapping (Series 1003)*

APPEARANCE AFTER STORAGE—After 8 mo. these wrapped fish appeared somewhat dried out and wrinkled on the heads. The eyes were sunken and dull but not opaque. After cooking, the flesh tasted normal in every way. At the end of 13 mo. the fish were entirely unmarketable, being much wrinkled and white with opaque and deeply sunken eyes. As their condition was so bad, it was not deemed necessary to carry further the studies on these unglazed wrapped fish. When cooked, they tasted flat and stringy, with a slight bitter after-taste.

GROSS ANALYSIS—The most striking fact about the composition of the fish in this lot is that they were badly dried out at the end of 13 mo. storage. Judging by chemical analysis and by appearance, the wrapping around these unglazed fish slightly retarded their desiccation but did not keep them from becoming unmarketable and unfit for food after 13 mo. The ordinary food constituents showed no significant changes.

NITROGENOUS CONSTITUENTS—As usual, there was a gradual increase in the percentage of ammonia and amine nitrogen during storage for 13 mo. However, this increase was no more marked than that found in glazed weakfish stored for about the same period. Like the glazed fish (Series 1002) the total soluble nitrogen and the non-coagulable nitrogen increased to the eighth month, then fell.

CONSTANTS OF FAT EXTRACTED FROM THE FLESH—There was the usual increase in the acid value of the oils from the fish after 13 mo. of storage. The magnitude of this change was about the same in the case of the glazed weakfish (Series 1002) after storage for the same period.

(4) *Glazed Weakfish Stored with Entrails Intact and without Wrapping (Series 1004)*

APPEARANCE AFTER STORAGE—After storage for 13 mo., the appearance of these fish was still good, only the slightly sunken condition of the eyes indicating aging. One of the weakfish in this lot had in its stomach 4 small fish about 6 in. long. None of these ingested fish showed any signs of enzyme action on the part of digestive juices or bacteria. However, at the end of storage for 2 yrs., the fish were entirely unmarketable owing to their general wrinkled and whitish appearance and sunken dull white eyes. Upon cooking, the flesh was tasteless and poor in quality, though not inedible.

GROSS ANALYSIS—There were no noteworthy changes in the percentages of the ordinary food constituents during storage.

---

[1] *J. Biol. Chem.*, **33** (1918), 487, 493.

[2] *Ibid.*, **33** (1918), 483.

[3] Analyses of a large number of fish have shown that the acid value of the fat varies with the content of fat. In general the lean fish have a higher acid value than the fat fish of the same species. This relationship between the acid value of the fat and the state of nutrition of the fish is being studied further. Consideration has been given to this factor in arriving at conclusions in regard to changes due to storage.

TABLE I—GROSS FLESH ANALYSIS

					Per cent on Moist Basis						Per cent on Dry Basis			
No.	Description of Fish	Storage Period	Weight of Fish Analyzed G.	Edible Portion Per cent	Moisture	Fat-free Solids	Ash	Ether Extract	Total Nitrogen	Ammonia and Amine Nitrogen	Ash	Ether Extract	Total Nitrogen	Ammonia and Amine Nitrogen
	Weakfish													
1002/1	Eviscerated	Fresh	1273	55.1	75.59	19.26	1.18	5.15	3.00	0.016	4.83	21.10	12.29	0.065
1002/2		Fresh	1104	50.0	78.70	19.54	1.24	1.76	2.96	0.018	5.82	8.26	13.89	0.084
1001/1	Eviscerated, not glazed, not wrapped in paper	1 mo.	1585	51.9	75.86	20.77	1.14	3.37	3.06	0.014	4.72	13.96	12.67	0.059
1001/2		2 mo.	1840	50.3	75.98	20.67	1.08	3.35	3.12	0.018	4.50	13.94	12.98	0.073
1001/3		4 mo.	1597	57.6	71.78	20.19	1.12	8.03	3.19	0.016	3.97	28.45	11.34	0.058
1001/4		8 mo.	991	56.5	74.72	22.93	1.31	2.35	3.42	0.024	5.18	9.27	13.53	0.093
1002/1	Eviscerated, glazed, not wrapped in paper	2 mo.	2027	50.6	77.61	20.48	0.96	1.91	2.99	0.017	4.29	8.53	13.35	0.076
1002/2		4 mo.	1655	53.1	76.65	20.88	1.10	2.47	3.13	0.015	4.71	10.59	13.40	0.066
1002/3		8 mo.	1657	45.5	76.92	21.09	1.18	1.99	3.12	0.023	5.11	8.62	13.52	0.101
1002/4		1 yr. 1 mo.	1543	52.8	75.14	22.04	1.16	2.82	3.20	0.020	4.67	11.34	12.87	0.081
1002/5		2 yrs. 1 mo.	2123	51.5	75.16	21.97	1.10	2.87	3.17	0.023	4.42	11.54	12.75	0.091
1003/1	Eviscerated, not glazed, wrapped in paper	4 mo.	1660	51.7	77.12	21.53	1.16	1.35	3.13	0.019	5.07	5.90	13.67	0.083
1003/2		8 mo.	1721	56.3	72.87	20.96	1.16	6.17	3.16	0.021	4.28	22.74	11.64	0.076
1003/3		1 yr. 1 mo.	1505	52.3	75.57	22.03	1.28	2.40	3.30	0.023	5.24	9.82	13.50	0.093
1004/1	Not eviscerated, glazed, not wrapped in paper	4 mo.	1528	47.2	74.49	20.63	1.16	4.88	3.09	0.019	4.55	19.13	12.11	0.075
1004/2		10 mo.	1518	48.9	75.95	20.98	1.14	3.07	3.12	0.030	4.74	12.76	12.97	0.125
1004/3		1 yr. 1 mo.	1858	43.9	75.41	21.40	1.13	3.19	3.18	0.028	4.60	12.97	12.93	0.112
1004/4		2 yrs. 1 mo.	1863	42.8	75.77	20.52	1.13	3.71	3.12	0.032	4.66	15.31	12.88	0.132
	Bluefish													
1006/1	Eviscerated	Fresh	5432	50.0	76.02	22.85	1.34	1.13	3.44	0.019	5.58	4.71	14.34	0.081
1006/2		Fresh	2982	51.4	75.64	22.54	1.32	1.82	3.31	0.024	5.41	7.40	13.58	0.097
1006/1	Eviscerated, glazed, not wrapped in paper	4 mo.	4354	52.3	74.20	22.47	0.99	3.33	3.34	0.016	3.84	12.90	12.94	0.060
1006/2		5 mo.	5030	49.5	73.98	22.83	1.14	3.19	3.41	0.022	4.38	12.26	13.10	0.082
1006/3		8 mo.	4250	48.4	74.32	23.66	1.27	2.02	3.56	0.025	4.95	7.87	13.82	0.097
1006/4		1 yr.	4801	47.7	74.74	23.41	1.22	1.85	3.37	0.029	4.83	7.32	13.34	0.114
1006/5		1 yr. 4 mo.	4210	53.9	74.29	23.90	1.23	1.81	3.37	0.026	4.78	7.04	13.10	0.100
1006/1*a*		1 yr. 4 mo.	3930	51.4	75.67	23.70	1.26	0.63	3.56	0.021	5.18	2.59	14.63	0.067
1006/6		2 yrs. 3$^1/_2$ mo.	3796	51.0	73.78	24.89	1.23	1.33	3.63	0.028	4.69	5.07	13.85	0.107

NITROGENOUS CONSTITUENTS—With the exception of the pronounced tendency of ammonia and amine nitrogen to increase in these uneviscerated weakfish, there were no other changes different from these discussed in the case of the glazed eviscerated weakfish (Series 1002). No other lot of fish exhibited such a tendency to form ammonia and amine nitrogen as did the weakfish stored "in the round." As all the lots of weakfish came from the same school, and had a normal uniform content of nitrogen in this form at the start, it would appear that the presence of the entrails in the fish during storage was concerned in this matter. The importance of this question, however, is such that until further study has been made no definite conclusions should be drawn.

CONSTANTS OF FAT EXTRACTED FROM THE FLESH—Except for the usual increase in the acid value there were no significant changes in the composition of the fat of these fish as revealed by chemical analyses.

(5) *Glazed Eviscerated Bluefish Stored without Wrapping* (*Series 1006*)

APPEARANCE AFTER STORAGE—There were no noticeable changes in the appearance of these bluefish until the end of 16 mo. of freezer storage. When removed at the end of the sixteenth month, the lot showed a certain amount of drying out on the noses of the fish, which were wrinkled and yellowish; the eyes were somewhat sunken, but eyes and skin were fairly bright. Upon cooking, the flesh was palatable and the flavor did not noticeably differ from that of fresh bluefish. These fish were in marketable condition and only a person familiar with the appearance of frozen fish would have classed them as "old stock."

The bluefish which were in storage for 2 yrs. and 3.5 mo. were in unmarketable condition when taken out for analysis. The noses and skin were dried, wrinkled, and yellowish. The gills were dry and brown. Some of the fins were swollen and soft. The eyes were sunken and opaque. The flesh seemed soggy after thawing but there was no odor of decomposition, only a peculiarly strong fishy odor. Upon cooking, the parts of the flesh near the skin were bitter and unpalatable, while the interior portions had a good flavor but were tough. As the fish had been kept glazed during the whole period of storage, there had been no opportunity for molds to attack their surface. From every point of view these fish were unmarketable and hardly fit for food.

GROSS ANALYSIS—There were no variations in the proportions of the gross food constituents of bluefish which could not be explained as being due to a loss of moisture during the periods of storage or to natural variations in the composition of this species of fish. The difference between the average initial per cent of fat-free solids and the per cent of this constituent at the end of the several periods of storage indicates a gradual loss of moisture amounting to about 0.7 per cent at the end of one year and to about 2.2 per cent at the end of 2 yrs. and 3.5 mo., in spite of the protection afforded by the ice glaze. At the close of the latter period the physical appearance of the fish confirmed the analytical findings in this regard. The acidity of the aqueous extracts showed no change.

NITROGENOUS CONSTITUENTS—There is a slight but definite tendency for the percentage of ammonia and amine nitrogen to increase during storage, both as determined on the flesh itself and on its aqueous extract. There was a considerable decrease in the amount of coagulable nitrogen after one year of storage. The proportion of soluble nitrogen showed a less marked decrease. Proteose nitrogen, as determined by zinc sulfate precipitation, was another constituent that decreased perceptibly during storage.

CONSTANTS OF FAT EXTRACTED FROM THE FLESH—The acid value of the oil of the bluefish showed a definite increase during storage and a relatively high value at the end of over 2 yrs. In addition there appeared to be a decrease in the iodine number. This may have been due to a slight oxidation of the oil, a change which probably was wrought by the action of the air on the oil near the surface of the fish after this part of the fish had become somewhat dried out.

DISCUSSION

One of the inevitable effects of cold storage upon frozen fish is loss of moisture. This evaporation of water at temperatures from —5° to 15° F. is indeed slow, and within certain limits decreases in amount with decreasing temperatures. At the beginning of the storage period the proportion of fat-free solids in the freshly frozen weakfish averaged 19.40 per cent. After 8 mo. the fat-free solids (Table I) of the frozen weakfish, which had been stored in the eviscerated condition without a protective coating of ice-glaze or paper, were 22.93 per cent, indicating a decrease of 3.53 in the per cent of moisture. In contrast to this, the fat-free solids of the frozen weakfish, which had been stored for 25 mo. in the eviscerated condition and "sealed" with a thin layer of ice, were 21.97 per cent, corresponding to a decrease of 2.5 in the per cent of moisture. It is plain that the ice-glaze greatly retarded evaporation of water from the flesh. This slight desiccation of the fish is shown, not only by chemical analysis, but by the appearance as well. The unglazed fish stored without wrapping were unmarketable at the end of 4 mo. owing to their dried-out appearance, whereas the glazed fish were in marketable condition even at the end of 13 mo. Loss of

TABLE II—COMPOSITION OF THE COLD WATER EXTRACT OF THE FLESH

			Per cent on Moist Basis											Per cent on Water-, Ash-, and Fat-free Basis						
							Nitrogen							Nitrogen						
No.	Description of Fish	Storage Period	Solids	Ash	Organic Matter	Acidity of Extract Cc.	Total	Coagulable	In Peptone Bases, etc (by Difference)	In Tannin-Salt Precipitate	Proteose	Amino Acid	Ammonia and Amine	Total	Coagulable	In Peptone Bases, etc.(by Difference)	In Tannin-Salt Precipitate	Proteose	Amino Acid	Ammonia and Amine
	Weakfish																			
1002/1	Eviscerated	Fresh	7.49	1.04	6.45	15.4	0.858	0.547	0.233	0.040	0.010	0.054	0.014	4.75	3.03	1.29	0.221	0.055	0.298	0.082
1002/2		Fresh	7.28	1.11	6.18	15.0	0.771	0.499	0.228	0.033	0.017	0.054	0.015	4.21	2.45	1.29	0.180	0.092	0.295	0.081
1001/1	Eviscerated, not glazed, not wrapped in paper	1 mo.	5.83	1.07	4.76	15.1	0.906	0.554	...	0.029	...	0.090	0.013	4.62	2.82	..	0.250	...	0.458	0.068
1001/2		2 mo.	6.04	1.08	4.96	17.8	0.821	0.489	0.232	0.031	0.012	0.074	0.014	4.16	2.45	1.16	0.158	0.060	0.377	0.072
1001/3		4 mo.	7.28	1.05	6.23	13.6	0.793	0.482	0.202	0.027	0.020	0.074	0.015	4.15	2.52	1.06	0.144	0.103	0.389	0.077
1001/4		8 mo.	8.88	1.21	7.67	16.4	0.998	0.628	0.266	0.030	0.011	0.076	0.017	4.62	2.90	1.24	0.139	0.052	0.352	0.078
1002/1	Eviscerated, glazed, not wrapped in paper	2 mo.	6.23	0.97	5.26	18.5	0.834	0.494	0.235	0.035	0.010	0.082	0.013	4.27	2.53	1.18	0.179	0.050	0.421	0.086
1002/2		4 mo.	4.10	1.05	3.05	15.1	0.843	0.515	0.220	0.026	0.015	0.078	0.015	4.26	2.60	1.13	0.132	0.075	0.393	0.076
1002/3		8 mo.	4.22	1.14	3.08	16.0	0.949	0.631	0.216	0.045	0.010	0.073	0.019	4.77	3.17	1.09	0.234	0.047	0.365	0.095
1002/4		1 yr. 1 mo.	7.28	0.97	6.31	16.0	0.807	0.493	0.224	0.034	0.013	0.060	0.017	3.86	2.36	1.07	0.163	0.063	0.286	0.080
1002/5		2 yrs. 1 mo.	7.36	0.97	6.39	18.0	0.813	0.456	0.254	0.029	0.008	0.077	0.018	3.88	2.18	1.21	0.139	0.040	0.367	0.085
1003/1	Eviscerated, not glazed, wrapped in paper	4 mo.	4.77	1.07	3.70	16.6	0.866	0.511	0.262	0.039	0.015	0.064	0.014	4.25	2.51	1.29	0.192	0.073	0.312	0.068
1003/2		8 mo.	7.85	1.08	6.73	17.0	0.897	0.546	0.256	0.018	0.011	0.071	0.013	4.53	2.76	1.29	0.191	0.057	0.357	0.066
1003/3		1 yr. 1 mo.	7.56	1.02	6.54	18.6	0.835	0.499	0.235	0.021	0.011	0.075	0.015	4.02	2.40	1.14	0.101	0.052	0.360	0.072
1004/1	Not eviscerated, glazed, not wrapped in paper	4 mo.	4.48	1.07	3.41	16.0	0.855	0.520	0.233	0.024	0.014	0.071	0.017	4.39	2.67	1.20	0.125	0.074	0.363	0.086
1004/2		10 mo.	7.23	1.05	6.18	25.5	0.992	0.658	0.205	0.026	0.021	0.087	0.021	5.00	3.32	1.03	0.131	0.104	0.438	0.104
1004/3		1 yr. 1 mo.	7.72	0.97	6.75	15.6	0.849	0.515	0.221	0.026	0.015	0.073	0.025	4.19	2.05	1.58	0.128	0.073	0.361	0.124
1004/4		2 yrs. 1 mo.	7.67	0.96	6.71	15.5	0.805	0.452	0.228	0.045	0.015	0.084	0.026	4.15	2.38	1.14	0.232	0.077	0.421	0.132
	Bluefish																			
1006/1	Eviscerated	Fresh	7.16	1.24	5.92	35.4	0.933	0.479	0.281	0.045	0.054	0.102	0.017	4.34	2.23	1.31	0.200	0.251	0.474	0.078
1006/2		Fresh	8.50	1.30	7.20	36.0	1.064	0.580	0.310	0.054	0.050	0.107	0.017	5.01	2.73	1.46	0.254	0.233	0.504	0.079
1006/1	Eviscerated, glazed, not wrapped in paper	4 mo.	4.54	1.14	3.40	30.0	0.769	0.421	0.175	0.055	0.044	0.113	0.016	3.58	1.96	0.82	0.254	0.203	0.526	0.074
1006/2		5 mo.	5.65	1.24	4.41	27.1	0.760	0.329	0.266	0.012	0.035	0.112	0.018	3.50	1.52	1.22	0.055	0.162	0.514	0.081
1006/3		8 mo.	5.49	1.17	4.32	28.0	0.818	0.440	0.220	0.056	0.034	0.103	0.021	3.65	1.96	0.99	0.250	0.149	0.459	0.093
1006/4		1 yr.	6.80	1.19	5.61	38.0	0.849	0.311	0.329	0.050	0.044	0.143	0.022	3.82	1.40	1.48	0.227	0.197	0.645	0.097
1006/5		1 yr. 4 mo.	6.27	1.15	5.12	36.1	0.785	0.302	0.319	0.041	0.028	0.112	0.024	3.45	1.33	1.40	0.180	0.124	0.492	0.106
1006/1*a*		1 yr. 4 mo.	6.98	1.18	5.80	31.4	0.863	0.351	0.350	0.071	0.021	0.120	0.021	3.84	1.56	1.46	0.316	0.094	0.636	0.091
1006/6		2 yrs 3½ mo.	7.22	1.23	5.99	37.4	0.863	0.332	0.334	0.058	0.032	0.137	0.028	3.65	1.40	1.42	0.245	0.134	0.579	0.118

moisture is also slightly retarded by wrapping the fish in semi-parchment paper, although it is plain that the paper alone gives insufficient protection from the drying effects of the air. Glazing seals the fish in an almost air-tight manner, and is evidently a necessary precaution to preserve the bright and fresh appearance of fish when removed from storage. It is known that ordinary glazing processes add from 3 to 5 per cent to the weight of the fish, but, on the other hand, during storage both the fish and the glaze lose weight as a result of the evaporation of water.

The rate of evaporation of water from the flesh of the glazed weakfish appeared to decrease markedly after the fish had been in storage for a year (Fat-free Solids, Table I). The losses of moisture from the flesh of all the glazed fish during the first 8 or 10 mo. of storage were small and not such as to produce a material change in the appearance of the fish. Except for an occasional broken or dried fin these fish after thawing resembled closely those often found in markets and sold as fresh fish. This good showing was due to the fact that the fish were frozen in cakes, conforming to the size of the usual commercial pan used for the initial freezing, thus reducing the amount of surface exposed to the air; to the protection afforded by the ice-glaze, which was renewed as required; and to the uniformly low temperature (15° F.) at which the fish were kept. In most commercial fish freezers in this country even better results might be expected, as the average temperature of their storage rooms is 5° or 10° (F.) lower than that of the rooms in which the experimental fish were held.

There is no evidence to show that the proportions of the food constituents of fish, namely, fat, protein, and ash, underwent appreciable changes in cold storage. At first glance it would seem that the variations in the amounts of these constituents in individual fish of the same school are surprisingly great. However, this lack of uniformity in composition is due almost entirely to the variations in the fat content, which is for the most part compensated for by changes in the moisture content. When due allowance is made for this natural variation, the proportions of protein, as represented by nitrogen in the tables, and ash do not change during storage except by amounts which can be accounted for by the slight loss of moisture.

Upon autolysis or bacterial attack the flesh of fish yields both ammonia and amines. In comparative investigations, such as here presented, the sum of the ammonia and amine nitrogen, or nitrogen determined by aëration, may be used as one index of changes in the nitrogenous constituents of fish flesh during storage. It is impossible to say whether such observed changes are due primarily to enzyme action or to the growth of bacteria. In either case, the nitrogen determined by aëration represents end-products which collect and indicate aging processes in fish. During storage there seemed to be a slight tendency for ammonia and amine nitrogen to increase, but this was usually not marked until after one year of storage. Bluefish had a more pronounced tendency to form ammonia and amine nitrogen than most of the lots of weakfish. In the latter, the effects of glazing and wrapping on the production of this form of nitrogen were not apparent. However, the weakfish which were stored with entrails intact exhibited a relatively

TABLE III—NITROGEN DISTRIBUTION CALCULATED IN PER CENT OF TOTAL NITROGEN

			Per cent Nitrogen							
No.	Description of Fish	Storage Period	Total	Soluble	Coagulable	In Peptone Bases, etc. (by Difference)	In Tannin-Salt Precipitate	Proteose	Amino Acid	Ammonia and Amine
	Weakfish									
1002/1	Eviscerated	Fresh	100	28.60	18.26	7.71	1.33	0.33	1.80	0.50
1002/2		Fresh	100	26.13	15.21	8.01	1.12	0.57	1.83	0.51
1001/1	Eviscerated, not glazed, not wrapped in paper	1 mo.	100	29.61	18.07	..	1.60	..	2.93	0.44
1001/2		2 mo.	100	26.25	15.64	7.40	1.00	0.38	2.38	0.45
1001/3		4 mo.	100	24.82	15.07	6.36	0.86	0.61	2.32	0.46
1001/4		8 mo.	100	29.19	18.33	7.81	0.88	0.33	2.23	0.49
1002/1	Eviscerated, glazed, not wrapped in paper	2 mo.	100	28.07	16.63	7.78	1.18	0.32	2.77	0.57
1002/2		4 mo.	100	26.93	16.43	7.06	0.83	0.48	2.48	0.48
1002/3		8 mo.	100	30.44	20.23	6.98	1.49	0.30	2.33	0.60
1002/4		1 yr. 1 mo.	100	25.19	15.40	6.89	1.06	0.40	1.87	0.63
1002/5		2 yrs. 1 mo.	100	25.66	14.37	8.05	0.92	0.26	2.42	0.56
1003/1	Eviscerated, not glazed, wrapped in paper	4 mo.	100	27.67	16.34	8.38	1.26	0.48	2.03	0.44
1003/2		8 mo.	100	28.38	17.29	8.08	0.57	0.35	2.24	0.42
1003/3		1 yr. 1 mo.	100	25.28	13.09	9.16	0.64	0.32	2.26	0.45
1004/1	Not eviscerated, glazed, not wrapped in paper	4 mo.	100	27.66	16.82	7.54	0.79	0.47	2.29	0.54
1004/2		10 mo.	100	31.80	21.12	6.57	0.83	0.66	2.79	0.66
1004/3		1 yr. 1 mo.	100	26.72	13.07	10.10	0.82	0.46	2.30	0.79
1004/4		2 yrs. 1 mo.	100	25.82	14.81	7.09	1.44	0.48	2.62	0.82
	Bluefish									
1006/1	Eviscerated	Fresh	100	27.14	13.94	8.18	1.31	1.57	2.96	0.49
1006/2		Fresh	100	32.13	17.51	9.55	1.63	1.33	3.23	0.51
1006/1	Eviscerated, glazed, not wrapped in paper	4 mo.	100	23.00	12.60	5.24	1.63	1.30	3.38	0.48
1006/2		5 mo.	100	22.26	9.67	7.78	0.35	1.03	3.27	0.51
1006/3		8 mo	100	23.04	12.37	6.25	1.58	0.94	2.89	0.59
1006/4		1 yr.	100	25.16	9.22	9.77	1.49	1.29	4.25	0.63
1006/5		1 yr. 4 mo.	100	23.31	8.98	9.45	1.21	0.84	3.32	0.72
1006/1*a*		1 yr. 4 mo.	100	24.21	9.84	9.82	1.99	0.59	3.38	0.58
1006/6		2 yrs. $3^1/_2$mo.	100	23.79	9.12	9.26	1.60	0.87	3.77	0.77

strong tendency for this constituent to increase. In fact, the highest percentages of ammonia and amine nitrogen found in any case were in those weakfish frozen "in the round." Since these fish were hard frozen it is difficult to conceive that this increase was due to the slow diffusion of decomposition products into the flesh from the entrails or to the penetration of bacteria from the same source. Whatever the explanation may be, it does not seem impossible that these changes in the flesh are traceable to the presence of the entrails during storage. Further experimentation is necessary before a definite conclusion can be reached.

During storage the bluefish showed a decrease in the amounts of water-soluble and coagulable nitrogen. These constituents increased at first and then decreased in all of the weakfish, except those stored in the eviscerated condition without glazing or wrapping, in which the changes were irregular. The nitrogen in the form of peptone, bases, etc., did not seem to be a variable constituent in the case of any of the fish in storage. The proteose and peptone nitrogen, as precipitated by tannin and salt, did not vary appreciably in amount. Proteose nitrogen, as precipitated by the zinc sulfate method, decreased perceptibly in the bluefish during storage, but not in the weakfish. Bluefish seem to contain, naturally, about three times as much proteose nitrogen as weakfish. Bluefish also contain more amino-acid nitrogen than weakfish, and both species show a slight tendency towards the accumulation of this constituent while in storage.

The interpretation of the meaning of the slight changes in the nitrogenous constituents just described is difficult owing to the small amounts involved, the apparently conflicting tendencies, and to the fact that the role of bacteria and enzymes in such changes is unknown for fish flesh in the hard frozen condition. However, the general conclusion to be drawn from the slight changes observed is that they indicate the beginning of the process of the breaking down of the larger protein molecules into intermediate products, and the simple substances like amino acids, and ammonia. Such changes are typical of the decomposition of proteins by enzymes or bacteria in ripening processes. The flesh of fish, unlike that of beef or poultry, is known to yield considerable amounts of amines when attacked by bacteria or enzymes. The changes occurring were very slight and of such a nature as would warrant the belief that they were brought about by autolysis rather than by bacterial action.

The iodine number of the fat of the fish which were analyzed after 2 yrs. of storage seemed to be somewhat lower than that of fish which had been stored for shorter periods. This was most pronounced in the case of the bluefish and is probably due to the oxidizing action of the air on the fats near the surface of the fish which was more or less dried out at this stage.

The increase in the acid value of the oils from frozen bluefish and weakfish was the most striking effect noticed in the storage investigations. The weakfish contained a large and also more variable amount of fat when compared with bluefish. The latter did not show such large increases in acid value as the weakfish. This change began to be most noticeable after 8 mo. or a year in storage. Up to this time neither glazing nor wrapping the fish to protect them from the air seemed to have any effect on this aging process in the fat. For this reason, during this time it is probably not influenced as much by atmospheric oxidation as by the bacterial or tissue enzymes in the flesh of the fish itself. The action of such enzymes at the low temperature of storage is necessarily slow and the acids produced in the fats of these fish did not impart a "rancid" flavor to the flesh of the fish which had been stored for a year in the glazed condition.

During the first 13 or 16 mo. of storage the changes in the chemical composition of the fish which had been frozen and stored under the best conditions were not reflected in changes in the flavor of the product after cooking. During the longer periods of storage, however, an undesirable change in the flavor of the cooked flesh could be detected. However, it should be noted,

TABLE IV—ANALYSIS OF FAT

No.	Description of Fish	Storage Period	Specific Gravity 25/25° C.	Index of Refraction 30° C.	Iodine No.	Saponification No.	Acid Value
	Weakfish						
1002/1	Eviscerated	Fresh	0.9183	1.4745	142.3	185.8	5.5
1002/2		Fresh	0.9315	1.4749	138.9	182.6	12.4
1001/1	Eviscerated, not glazed, not wrapped in paper	1 mo.	....	....	...	...	..
1001/2		2 mo.	0.9188	1.4709	...	192.6	11.7
1001/3		4 mo.	0.9195	1.4726	132.6	194.8	11.4
1001/4		8 mo.	0.9216	1.4727	129.6	187.1	23.4
1002/1	Eviscerated, glazed, not wrapped in paper	2 mo.	0.9240	1.4726	...	191.6	17.9
1002/2		4 mo.	0.9207	1.4743	132.9	194.1	16.2
1002/3		8 mo.	0.9254	1.4724	124.9	187.3	25.3
1002/4		1 yr. 1 mo.	0.9266	1.4725	128.3	188.7	20.2
1002/5		2 yrs. 1 mo.	0.9333	1.4740	136.5	184.1	26.1
1003/1	Eviscerated, not glazed, wrapped in paper	4 mo.	0.9267	1.4740	...	180.5	21.3
1003/2		8 mo.	0.9221	1.4728	136.7	190.9	15.9
1003/3		1 yr. 1 mo.	0.9141	1.4704	133.6	177.9	20.1
1004/1	Not eviscerated, glazed, not wrapped in paper	4 mo.	0.9187	1.4724	124.1	194.2	12.8
1004/2		10 mo.	0.9261	1.4731	140.3	191.0	22.4
1004/3		1 yr. 1 mo.	0.9147	1.4703	128.6	182.3	20.2
1004/4		2 yrs. 1 mo.	0.9214	1.4733	128.8	184.1	25.2
	Bluefish						
1006/1	Eviscerated	Fresh	0.9310	1.4786	126.0	178.9	38.9
1006/2		Fresh	....	1.4745	136.4	172.8	16.3
1006/1	Eviscerated, glazed, not wrapped in paper	4 mo.	....	....	...	...	..
1006/2		5 mo.	0.9325	1.4723	137.9	187.9	21.8
1006/3		8 mo.	0.9148	1.4735	126.5	...	27.4
1006/4		1 yr.	0.9286	1.4745	133.4	188.9	34.1
1006/5		1 yr. 4 mo.	0.9078	1.4694	127.5	172.6	32.0
1006/1*a*		1 yr. 4 mo.	0.9269	1.4745	118.8	176.1	40.4
1006/6		2 yrs. 3½ mo.	0.9204	1.4710	118.5	181.3	41.6

in connection with the longer storage periods, that the holding temperature (15° F.) is higher than is ordinarily used commercially.

SUMMARY

Two species of salt water fish, *i. e.*, bluefish, *Pomatomus saltatrix*, and weakfish, *Cynoscion regalis*, were frozen by the commonly used air process, stored in a holding room whose temperature remained near 15° F. and examined chemically and organoleptically at the conclusion of holding periods varying in duration from 1 to 27.5 mo. The bluefish before freezing were eviscerated and were kept "glazed" with an ice covering during storage. The weakfish were divided in four lots, each of which was prepared for storage in a different manner in order to determine the effects of glazing, paper wrapping, and the presence of entrails on the keeping quality of these fish.

I—Bluefish and the weakfish stored in the glazed condition remained palatable and wholesome during storage for 16 and 13 mo., respectively.

II—Weakfish, which had not been glazed or otherwise protected against loss of moisture, dried out so rapidly that at the end of 4 mo. they were considered unmarketable.

III—The loss of moisture in weakfish was checked but slightly by wrapping the fish in semi-parchment paper. Paper wrapping is by no means an adequate substitute for the ice-glaze.

IV—The keeping quality of glazed uneviscerated weakfish in storage up to 13 mo. was practically the same as that of the glazed eviscerated weakfish.

V—Both bluefish and weakfish became unpalatable after 2 yrs. of storage. The desiccation and consequent ruining of the appearance of the fish after long periods of holding were more marked than they would have been if the temperature of the storage rooms had been nearer the average commercial holding temperature.

VI—The following changes in the composition of the flesh of the fish during storage were noted:

(*a*) Water-soluble and coagulable nitrogen decreased in the bluefish, and increased at first, then decreased in all weakfish except those stored in the eviscerated condition without glazing or wrapping. In the latter the changes were irregular.

(*b*) Proteose nitrogen decreased in the bluefish but underwent no distinctive change in the weakfish.

(*c*) Amino-acid nitrogen increased slightly in both bluefish and weakfish.

(*d*) Ammonia and amine nitrogen increased markedly in the glazed uneviscerated weakfish and only slightly in the other fish.

(*e*) The acid value of the extracted fat increased in all samples.

(*f*) The iodine number of the extracted fat decreased in all samples, the change being greatest in the bluefish.

SOLID PHASES OBTAINED BY THE EVAPORATION OF CERTAIN SOIL EXTRACTS[1]

By M. S. Anderson and William H. Fry

BUREAU OF SOILS, U. S. DEPARTMENT OF AGRICULTURE, WASHINGTON, D. C.

[PRELIMINARY PAPER]

Received February 9, 1920

In recent years a very considerable amount of investigation in the Bureau of Soils has been devoted to the nature and functions of soil solution and to water extracts of soils. The results are published in the various bulletins of the Bureau.[2] For the most part these investigations have concerned themselves with the total material extracted and its relation to plant growth and to the so-called plant food constituents. Similarly in various other publications are found more or less extensive accounts of the relation of water to soil dealing with concentration,[1-8,*] movement,[9,10] methods of extraction and materials extracted by water.[11,15] There can also be found in the literature an enormous amount of material dealing with the mineral composition of sea water, saline lakes and deposits, well and drainage waters, etc. These publications are sum-

---

[1] Published by permission of the Secretary of Agriculture.

[2] See various publications of the Bureau of Soils, U. S. Department of Agriculture, Washington, D. C.

* Numbers refer to Bibliography, p. 668.

marized and references found in Clarke's "Data of Geochemistry."[16]

In all these articles there is found almost no reference to the actual character of the materials deposited from soil extracts upon evaporation. The usual forms of expression are the relative quantities of ions or radicals found, or a recalculation of these quantities according to conventional formulas supposed to represent the compounds present. In an ordinary soil extract there are, in addition to the organic material present, many inorganic substances other than the so-called plant foods. It is not to be doubted that these may profoundly affect the properties of the soil solutions, and this is particularly true when it is considered that soil solutions become very concentrated as the soils become air-dried and the aqueous films approach or reach saturation. It therefore seemed of interest to determine the nature of the actual salts crystallizable from soil extracts and to determine if possible the variations in these in different types of soils. Many difficulties are encountered in an investigation of this sort, among them being the large volumes of solution and of soil which must be used to secure a workable amount of deposit. The extract contains not only relatively large amounts of organic matter but also considerable quantities of highly colored, non-crystallizable material which very seriously interfere with the petrographic examination of the deposits. Failure to properly estimate the difficulties in advance has rendered the results reported in the following pages somewhat fragmentary and this communication is to be regarded as a preliminary report upon the subject. A more comprehensive investigation is now under way, but the data at hand are of such character as to render their present publication desirable.

EXPERIMENTAL WORK

PROCEDURE—The general plan of operation, with such modifications as seemed advisable as the work developed, was to prepare extracts of the various soils by agitating large quantities with quantities of water ranging from 3 to 5 parts of water to one of soil. The water extract was filtered through Pasteur-Chamberland filters and a portion of the extract evaporated to dryness. The soil and the dry extract were analyzed by the same methods. The main body of the extract was concentrated on a steam bath in a tin-lined copper kettle until crystallization began to take place. Considerable quantities of amorphous and undeterminable material separated before this point was reached. This was filtered off and subjected to complete analysis. The filtrate was allowed to evaporate spontaneously until a crop of crystals appeared. These were filtered off and examined with a petrographic microscope by the ordinary method.

PRELIMINARY WORK—A certain amount of preliminary work was done upon a sample of Chester silt loam from Oakton, Va. The quantity of soil extracted was about 100 kg. and the ratio of soil to water was one to five. After filtering, a portion of the solution was evaporated to dryness and the extract dried at 105° C. This extract was analyzed and the following table gives the composition as compared with a sample of the original soil.

ANALYSIS OF CHESTER SILT LOAM, OAKTON, VA., AND WATER EXTRACT

Analysis by L. A. Steinkoenig

	SOIL	WATER EXTRACT
Loss on ignition	5.30	57.51
Silica, SiO_2	75.37	4.14
Titanium, TiO_2	1.51	0.02
Iron, Fe_2O_3	3.31	0.46
Alumina, Al_2O_3	–12.08	1.78
Manganese, MnO	0.27	0.11
Lime, CaO	0.30	10.52
Magnesia, MgO	0.35	4.84
Potash, K_2O	1.44	11.36
Soda, Na_2O	0.38	8.71
Phosphoric acid, P_2O_5	0.23	0.13

The greater portion of the extract was evaporated in an open kettle until a separation of solid material began, when it was transferred to a porcelain dish and evaporated under a pressure of 15 mm. As soon as a separation of a few grams of solid material had taken place the evaporation was interrupted and the solid material removed by filtration or decantation. In this manner 13 successive fractions were obtained, the last being a pasty mass of the total remaining material. The examination of the fractions of solid material was very difficult because of the precipitation of amorphous and highly colored inorganic matter as well as of large quantities of organic substances. Nevertheless by petrographic examination, the following crystalline compounds were identified:

Gypsum, $CaSO_4.2H_2O$
Calcite, $CaCO_3$
Thenardite, Na_2SO_4
Sylvite, KCl
Kieserite, $MgSO_4 + H_2O$
Picromerite, $MgSO_4K_2SO_4 + 6H_2O$
Mirabilite, $Na_2SO_4.10H_2O$
Epsomite, $MgSO_4.7H_2O$
Carnallite, $KMgCl_3.6H_2O$
Soda niter, $NaNO_3$
Halite, NaCl

In view of these interesting results it seemed desirable to pursue the investigation by a comparison of the salts deposited from the water extract of an alkali soil with those deposited from extracts from soils of humid areas.

EXAMINATION OF SAMPLES OF SOIL EXTRACTS—A sample of Yolo clay, secured from near Los Baños, Cal., was subjected to detailed examination. The sample was an alkali crust together with about 6 in. of underlying soil. This soil is described[17] as follows: "The type is a chocolate-brown clay 6 ft. or more in depth—wheat and barley are the principal crops grown. Alfalfa is successfully grown on this type." This soil was selected because, as an alkali soil, the soluble salt content is relatively high.

The solution was prepared by treating 448 lbs. of the air-dry soil with water using 16 lbs. of soil for each extraction. After extraction the solution was filtered and evaporated in a steam-jacketed kettle. The total amount of solution used was 985.6 liters with a soluble salt content of about 2 g. per liter. After evaporation to about one-tenth of its volume the solution was filtered and returned to the kettle for further concentration. The residue was found to contain considerable quantities of carbonate but no estimation of its quantity or definite composition was made. The solution, which had become of an amber color, was slowly concentrated until a sample on cooling showed a deposition of crystals.

The whole of the solution, now about 12 liters in volume, was placed in a porcelain-lined pan and allowed

TABLE I—WHITE ALKALI SOIL

Yolo clay loam 0–4 in. deep from near Los Baños, California. 448 lbs. of air-dry soil—442 lbs. of dry soil—were treated with 985.6 liters of water equivalent to 440 per cent of moisture calculated on the weight of soil used. On evaporation calcites, gypsum, amorphous mineral and organic matter separated out down to a volume of 13,260 cc., when the following minerals separated out in crystalline form.

Microscopical work by W. H. Fry

Crop of Crystals	Sp. Gr. of Solution	Temperature ° C.	Volume of Solution. Cc.	Solution on Basis of Original Soil Per cent	Mirabilite, $Na_2SO_4.10H_2O$	Thenardite, Na_2SO_4	Halite, NaCl	Thermonatrite, $Na_2CO_3.H_2O$	Hanksite, $9Na_2SO_4.2Na_2CO_3.KCl$	Epsomite, $MgSO_4.7H_2O$	Picromerite, $MgSO_4.K_2SO_4.6H_2O$	Leonite, $MgSO_4.K_2SO_4.4H_2O$	Aphthitalite, $(KNa)_2SO_4$	Vanthoffite, $3Na_2SO_4.MgSO_4$	Blodite, $MgSO_4.Na_2SO_4.4H_2O$	Loweite, $MgSO_4.Na_2SO_4.2.5H_2O$	Langbeinite, $K_2SO_4.2MgSO_4$	Tri-sodium Phosphate, Na_3PO_4	Kainite, $MgSO_4.KCl.3H_2O$	Soda Niter, $NaNO_3$	Sodium Carbonate, Na_2CO_3	Carnallite, $KMgCl_3.6H_2O$
0	1.103	23	34,084	17.0[1]	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..
1	1.247	25	13,260 4.400	6.6 2.2	x	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..
2	1.315	24	3,700	1.8	..	x	x	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..
3	1.318	23.5	3,353	1.6	..	x	x	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..
4	1.304	21.5	2,660	1.3[2]	..	x	x	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..
5	1.301	23	2,312	1.2	..	x	x	x	..	..	..	..	..	..	..	..	..	..	..	..	..	..
6	1.288	21	1,920	0.9	..	x	x	x	..	..	..	..	..	..	..	..	..	..	..	..	..	..
7	1.287	23	1,607	0.8	..	x	x	x	..	..	..	..	..	..	..	..	..	..	..	..	..	..
8	1.273	24	1,246	0.6	..	x	x	x	..	..	..	..	..	..	..	..	..	..	..	..	..	..
9	1.294	22	852	0.4	..	x	x	x	x	x	..	..	..	..	..	..	..	..	..	..	..	..
10	1.323	23	447	0.2	..	x	x	x	x	x	x	..	..	..	..	..	..	..	..	..	..	..
11	1.330	21	255	0.1	..	x	x	x	x	x	x	x	x	x	..	..	..	..	..	..	..	..
12	1.375	25	90	0.04	..	x	x	x	x	x	x	x	x	x	x	x	..	..	..	..	..	..
13	1.404	25	42	0.02	..	x	x	x	x	x	x	x	x	x	x	x	x	x	..	..	..	..
14	...	..	24	0.01	..	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x

[1] Optimum water content. [2] Air-dry condition in laboratory.

to cool over night. A crop of well-formed and quite pure crystals of hydrated sodium sulfate separated out, together with a considerable quantity of a gray sediment. The sediment, about 5 g., washed free from soluble sulfates was analyzed and showed the following composition:

Analysis by M. S. Anderson

	Per cent
Loss on ignition (including CO_2)	37.75
Silica, SiO_2	13.05
Iron oxide, Fe_2O_3	Trace
Alumina, Al_2O_3	2.03
Titanium oxide, TiO_2	None
Lime (CaO)	35.30
Magnesia, MgO	9.38
Potash, K_2O	None
Soda, Na_2O	1.78
Carbon dioxide, CO_2	26.80

The evaporation was now allowed to continue, at room temperature, in porcelain-lined dishes. No salt other than hydrated sodium sulfate separated until a concentration of 5,400 cc. was reached and from this point only traces of any solid phase separated until the concentration had reached a volume of 4,400 cc. The whole deposit to this point is described as Crop 1 in Table I.

A second crop of crystals was recovered when a volume of 3,700 cc. was reached. The specific gravity of the mother liquor was also determined. The evaporation was continued at room temperature until, in all, 14 fractions of crystals were obtained. These were examined petrographically and the compounds identified as indicated in Table I.

In Column 5 are given the percentages of the water in each fraction from which crystals were obtained, calculated on the basis of the original soil. It will be noted that no salts crystallized at a moisture content corresponding to the optimum for the soil and that no salts other than sodium sulfate and sodium chloride assumed the solid phase until the concentration became greater than that corresponding to air-dry soil. The residual liquid from Fraction 14 was a thick brown, pasty mass of such character that further petrographic examination was impracticable.

After the work with the extract from 448 lbs. of soil was completed, a 16-lb. sample of the same soil was extracted with water and the extract evaporated to determine the point at which the sodium sulfate began to crystallize. This was found to be at a concentration of 6.6 per cent of the original soil weight. The total solid separated out at this point amounted to 0.02898 g. Its composition was as follows:

Analysis by W. O. Robinson

	Per cent
Silica, SiO_2	13.37
Alumina, Al_2O_3	5.53
Iron oxide, Fe_2O_3	0.58
Calcium carbonate, $CaCO_3$ (calculated)	32.62
Magnesium oxide, MgO	2.45
Potassium oxide, K_2O	0.34
Sodium oxide, Na_2O	15.50
Sulfur trioxide, SO_3	12.70
Phosphorus pentoxide, P_2O_5	Trace
Ignition loss	16.91

The solution from which the solid was removed had a volume of 1,234 cc. with a specific gravity of 1.112 at 28° C. Its composition was found to be

Analysis by W. O. Robinson

	Grams per Liter
Silica, SiO_2	0.34
Alumina, Al_2O_3	Trace
Ferric oxide, Fe_2O_3	Trace
Lime, CaO	0.25
Magnesium oxide, MgO	0.14
Potassium oxide, K_2O	0.48
Sodium oxide, Na_2O	74.49
Phosphorus pentoxide, P_2O_5	0.019
Chlorine	2.34
Sulfur trioxide, SO_3	56.99
Carbon dioxide, CO_2	1.37

HUMID SOILS—In order to contrast the alkali soil extract with a humid soil a sample of sassafras silt loam[17] was collected in the grounds of the Smithsonian Institution in Washington, D. C.

About 400 lbs. of this soil were extracted and 1,250 liters of solution prepared, filtered through Pasteur-Chamberland filters, and evaporated in the steam kettle. The solid material which separated out when the volume was reduced to 33 liters was collected and analyzed. Evaporation was continued to a volume of 1.56 liters and the solid material again collected and analyzed.

The volume of the solution remaining when the more soluble salts began to crystallize was so small that it was not practicable to determine the volume and specific gravity of each successive fraction from which crystal crops were examined.

Analysis by W. O. Robinson

	Residue at Optimum Water Content Volume 33 L. Per cent	Residue at Air-Dry Water Content Volume 1.56 L. Per cent
Loss on ignition	25.13	8.73
Silica, SiO_2	27.91	2.95
Alumina, Al_2O_3	16.84	2.06
Iron oxide, Fe_2O_3	5.41	1.48
Manganese oxide, MnO	0.43	0.06
Copper oxide, CuO[1]	5.08	Trace
Calcium oxide, CaO	8.80	33.62
Magnesium oxide, MgO	2.28	0.48
Potassium oxide, K_2O	0.66	0.68
Sodium oxide, Na_2O	0.54	1.34
Sulfur trioxide, SO_3	3.91	45.15
Phosphorus pentoxide, P_2O_5	0.23	0.05

[1] From steam kettle.

The results of the petrographic examination of the crystal crops are given in Table II, Column 8. The last fractions contained such relatively large quantities of organic matter that petrographic examination was impossible. As a means of getting this material into workable condition it was dried and charred at as low a temperature as possible and the organic material thus destroyed or rendered insoluble. The residue was dissolved in a small amount of warm water and filtered. The filtrate was allowed to evaporate and the fractions of crystallized material examined. In similar manner Crowley silt loam subsoil from Crowley, La., was examined,[17] and also a sample of Stockton clay adobe from Stockton, Cal.[17] One hundred pounds of each soil were used and 375 liters of each solution prepared. The results of these examinations are shown in Table II, Columns 9 and 10.

TABLE II—SALTS FOUND IN SALINE DEPOSITS AND OBTAINED FROM WATER EXTRACTS OF SOILS

Salts Found	(1) Stassfurt Deposits	(2) Sea Water	(3) Great Basin Region	(4) Searles Marsh	Analysis by W. H. Fry (5) White Alkali Soil	(6) Chester Silt Loam	(7) Various Alkali Crusts	(8) Smithsonian Soil	(9) Stockton Clay Loam	(10) Crowley Silt Loam
SINGLE SALTS										
Chlorides										
Sylvite—KCl	x	x	..	..	..	x	x	x	..	..
Halite—NaCl	x	x	x	x	x	x	x	x	x	x
Bischofite—$MgCl_2.6H_2O$	x	x	..	..	..	..	..	..	..	..
Sulfates										
Gypsum—$CaSO_4.2H_2O$	x	x	x	x	x	x	x	x	..	x
Anhydrite—$CaSO_4$	x	..	x	x	..	..	..	..	..	..
Celestite—$SrSO_4$	x	..	x	x	..	..	..	..	..	..
Epsomite—$MgSO_4.7H_2O$	x	x	..	..	x	x	..	..	..	..
Kieserite—$MgSO_4.H_2O$	x	x	..	..	..	x	..	..	..	..
Thenardite—Na_2SO_4	..	x	x	x	x	x	x	x	x	x
Mirabilite—$Na_2SO_4.10H_2O$	..	..	x	..	x	x	x	x	..	..
$MgSO_4.6H_2O$	..	x	..	..	..	..	..	..	..	..
Nitrates										
Soda niter—$NaNO_3$	..	..	x	x	x	x	x	..	..	..
Niter—KNO_3	..	..	x	..	..	..	..	..	..	..
Nitrocalcite—$Ca(NO_3)_2.nH_2O$	..	..	x	..	..	..	..	..	..	..
Nitromagnesite—$Mg(NO_3)_2.nH_2O$	..	..	x	..	..	..	..	..	..	..
Carbonates										
Calcite—$CaCO_3$	x	x	x	x	x	x	x	x	x	x
Aragonite—$CaCO_3$	..	..	..	..	..	..	..	..	..	x
Magnesite—$MgCO_3$	..	..	..	..	x	..	..	..	..	x
Thermonatrite—$Na_2CO_3.H_2O$	..	..	x	..	x	..	..	..	..	..
Natron—$Na_2CO_3.10H_2O$	..	..	x	x	..	..	..	..	..	..
Sodium carbonate—Na_2CO_3	..	..	..	..	x	..	..	..	x	x
Phosphates										
Tri-sodium phosphate—Na_3PO_4	..	..	..	..	x	..	..	..	..	..
DOUBLE SALTS										
Chlorides										
Carnallite—$KCl.MgCl_2.6H_2O$	x	x	..	..	x	x	x	x	..	..
Douglasite—$2KCl.FeCl_2.2H_2O$	x	..	..	..	..	..	..	..	..	..
Tachydrite—$CaCl_2.2MgCl_2.12H_2O$	x	..	..	..	..	..	..	..	..	..
Sulfates										
Aphthitalite—$(KNa)_2SO_4$	x	x	..	..	x	..	x	x	..	..
Picromerite—$K_2SO_4.MgSO_4.6H_2O$	x	x	..	..	x	x	x	x	x	x
Leonite—$K_2SO_4.MgSO_4.4H_2O$	x	x	..	..	x	..	..	x	x	..
Langbeinite—$K_2SO_4.2MgSO_4$	x	..	..	..	x	..	..	..	..	..
Blodite—$Na_2SO_4.MgSO_4.4H_2O$	x	x	..	..	x	..	..	x	x	x
Loweite—$2Na_2SO_4.2MgSO_4.5H_2O$	x	..	..	..	x	..	..	x	..	x
Vanthoffite—$3Na_2SO_4.MgSO_4$	x	..	..	..	x	..	..	x	..	..
Glauberite—$Na_2SO_4.CaSO_4$	x	..	x	x	..	..	..	..	..	..
Kalinite—$K_2SO_4.Al_2(SO_4)_3.24H_2O$	..	..	x	..	..	..	..	..	..	..
Sulfates and Chlorides										
Kainite—$KCl.MgSO_4.3H_2O$	x	x	..	..	x	..	..	..	..	..
Sulfohalite—$3Na_2SO_4.2NaCl$	..	..	x	x	..	..	..	..	..	..
Carbonates										
Trona—$Na_2CO_3.NaHCO_3.2H_2O$	..	..	x	x	..	..	x	..	..	..
Gaylussite—$CaCO_3.Na_2CO_3.5H_2O$	..	..	x	x	..	..	x	..	..	..
Dolomite—$MgCa(CO_3)_2$	..	..	x	x	..	..	..	..	..	..
Pirssonite—$Na_2Ca(CO_3)_2.2H_2O$	..	..	x	x	..	..	..	..	..	..
Silicates										
Natrolite—$Na_2Al_2Si_3O_{10}.2H_2O$	..	..	..	..	..	..	..	..	..	..
TRIPLE SALTS										
Sulfates										
Polyhalite—$K_2SO_4.2CaSO_4.MgSO_4.2H_2O$	x	..	..	..	..	..	..	..	..	..
Krugite—$K_2SO_4.4CaSO_4.MgSO_4.2H_2O$	x	..	..	..	..	..	..	..	..	..
Chlorides and Aluminates										
Koenenite—$2MgCl_2.3MgO.Al_2O_3.6H_2O$ (or $8H_2O$?)	x	..	..	..	..	..	..	..	..	..
Sulfates, Carbonates and Chlorides										
Hanksite—$9Na_2SO_4.2NaCO_3.KCl$	..	..	x	x	x	..	x	..	x	..
Carbonates and Chlorides										
Northupite—$MgCO_3.Na_2CO_3.NaCl$	..	..	x	x	..	..	x	..	..	..
Carbonates and Sulfates										
Tychite—$2MgCO_3.2Na_2CO_3.Na_2SO_4$	..	..	x	x	..	..	..	..	..	..
BORATES										
Boracite—$Mg_7Cl_2B_{16}O_{30}$	x	..	x	..	..	..	..	..	..	..
Pinnoite—$MgB_2O_4.3H_2O$	x	..	..	..	..	..	..	..	..	..
Hydroboracite—$CaMgB_6O_{11}.6H_2O$	x	..	..	..	..	..	..	..	..	..
Heintzite-hydrous borate of Mg and K, formula doubtful	x	..	..	..	..	..	..	..	..	..
Ascharite—$3Mg_2B_2O_5.2H_2O$	x	..	..	..	..	..	..	..	..	..
Sulfoborite—$2MgSO_4.4MgHBO_3.7H_2O$ or $3MgSO_4.2Mg_3B_4O_9.12H_2O$	x	..	..	..	..	..	..	..	..	..
Borax—$Na_2B_4O_7.10H_2O$	..	..	x	x	..	..	..	..	..	..
Colemanite—$Ca_2B_6O_{11}.5H_2O$	..	..	x	x	..	..	..	..	..	..
Ulexite—$NaCaB_5O_9.8H_2O$	..	..	x	..	..	..	..	..	..	..
Howlite—$H_5Ca_2B_5SiO_{14}$	..	..	x	..	..	..	..	..	..	..
Neocolemanite—$Ca_2B_6O_{11}.5H_2O$	..	..	x	..	..	..	..	..	..	..

The Crowley silt loam extract had the composition as shown by the following analysis:

Analysis of Crowley Silt Loam Extract

Analysis by W. O. Robinson

	Parts per Million of Solution	Parts per Million of Soil
Total solids	103.8	856.0
Silica, SiO_2	17.5	143.0
Alumina, Al_2O_3	Trace	Trace
Iron oxide, Fe_2O_3	Trace	Trace
Lime, CaO	20.4	168.0
Magnesia, MgO	1.9	9.7
Potassium oxide, K_2O	0.5	4.1
Sodium oxide, Na_2O	10.1	83.3
Sulfur trioxide, SO_3	3.7	30.5
Chlorine, Cl	1.8	14.9

ALKALI SOIL CRUSTS—Since complex salts were identified at solution concentrations corresponding to a moisture content a little below that of the air-dry condition of the alkali soil it seemed probable that in soils containing a large soluble mineral content the same minerals should be present in air-dry soils in the solid phase. To determine if this were true 14 alkali soil crusts from Ventura County, Cal., and from Arizona were selected and examined by means of the petrographic microscope. The soils were examined without other treatment than pulverization and no attempt was made to secure especially dry samples. The highest number of soluble salts identified in any one soil was 8 and the total list of different minerals is found in Table II, Column 7.

Table III

Percentage of Salts Found in Water Extract Calculated to Hypothetical Combinations

Minerals Actually Observed by Petrographic Method in the Same Soil or Crust

	Analysis by J. C. Ripperton									*Analysis by W. H. Fry*													
No.	$CaCl_2$	$CaSO_4$	$MgSO_4$	K_2SO_4	Na_2SO_4	NaCl	Na_2CO_3	$NaHCO_3$	$NaNO_3$	$Na_2SO_4.10H_2O$	Na_2SO_4	$Na_2CO_3.NaHCO_3.2H_2O$	KCl	$CaSO_4.2H_2O$	NaCl	$CaCO_3.Na_2CO_3.5H_2O$	$CaCO_3$	$MgSO_4.K_2SO_4.6H_2O$	$MgCO_3.Na_2CO_3.NaCl$	Na.NO_3	$Na_2(SO_4)_4.Na_2CO_3$	$KMgCl_3.6H_2O$	$(KNa)_2SO_4$
1	...	22.74	5.75	1.69	...	51.33	...	3.33	..	..	x	..	..	x	x	..	x	..	..	..	..	..	..
2	...	11.22	19.65	3.55	47.66	14.22	1.59	1.89	0.22	x	x	x	x	x	x	x	x	..	..	..	..	..	..
3	...	1.89	1.50	5.02	16.69	51.45	17.84	4.46	1.05	x	..	..	x	x	x	x	x	..	..	..	..	..	..
4	...	7.00	13.30	8.18	59.70	9.79	1.10	0.85	..	x	x	..	..	x	x	..	x	..	..	..	..	..	..
5	...	18.18	11.15	9.08	28.09	33.78	2.14	1.67	..	x	..	..	..	x	x	..	x	..	..	..	..	..	..
6	...	2.75	0.75	12.31	51.80	20.82	4.47	7.04	..	..	x	..	x	x	x	..	x	..	..	..	..	..	..
			$MgCl_2$	KCl																			
7	8.60	10.29	15.56	15.52	...	49.69	...	..	..	..	..	..	x	x	x	..	x	..	..	..	..	..	..
8	...	7.68	6.40	4.37	2.44	77.12	0.42	0.69	0.99	..	..	..	..	x	x	..	x	..	..	..	..	..	..
9	...	4.59	20.55	3.55	60.09	9.24	...	1.19	0.45	x	..	..	x	..	x	..	x	x	..	..	x	x	..
10	...	3.74	28.40	2.19	51.98	12.01	...	1.41	..	x	..	..	x	x	x	..	x	x	..	..	..	..	..
11	...	2.95	6.95	7.56	17.73	27.47	27.19	9.84	..	..	..	..	x	x	x	..	x	..	x	..	x	x	x
12	...	0.24	1.60	1.87	10.70	64.69	14.97	3.06	2.22	..	x	..	..	x	x	x	x	..	x	..	x	..	..
			$MgCl_2$	KCl																			
13	37.58	6.46	8.32	14.08	...	24.98	5.04	3.40	..	..	..	..	..	x	x	..	..	..	..	x	..	..	..
14	...	5.34	2.40	8.96	53.61	12.18	9.67	6.85	0.90	x	x	..	..	x	x	..	x	..	..	..	..	..	..

Water extracts of these soils were made, the extracts analyzed and the analyses calculated to the hypothetical combinations according to the usual method. Table III contains the results, together with the minerals found in the air-dry soils.

DISCUSSION OF RESULTS

The existence of complex salts in the crystalline material deposited from the extracts of both alkali and humid soils renders interesting a comparison between these and the natural deposits of saline material and in Table II is given a summarization of the minerals found in Stassfurt deposits[16,18], the Great Basin region,[19] Searles marsh,[16] and obtained from the evaporation of sea water.[4]

An inspection of the tables reveals the fact that halite, NaCl, and calcite, $CaCO_3$, are the only minerals uniformly present, while gypsum, $CaSO_4.2H_2O$, and thenardite, Na_2SO_4, are next in order of frequency. One of the more notable facts is the occurrence of the more soluble carbonates in the soil extracts and their absence from the deposits from sea water and from the Stassfurt deposits. It is of course not to be expected that the extract from any single soil would deposit the whole range of salts obtained from sea water or in the natural deposits from it since the saline deposits represent the soil extract from the entire globe. It is indeed remarkable that so great a similarity exists. The most notable difference between the soils and soil products is the entire absence of the borates in the latter. This would not be the case, of course, were soils containing borates subjected to examination. The Stassfurt deposits are marked by the absence of thenardite, Na_2SO_4, while carrying large amounts of sulfates in the form of more complex salts. Both the chemical and petrographic analyses indicate that the sulfates, chloride, and carbonates of sodium form by far the greater portion of the soluble material of the soil extract. Hanksite, $9Na_2SO_4.2Na_2CO_3.KCl$, is probably next in abundance, though even this is in very small quantity. The remaining substances identified occur only as traces in the material examined. Although chemical analysis shows the presence of phosphoric acid and of nitrates, it gives no indication of the complexity of the compounds which separate out. It is probable that these salts are present in such small amounts that they may never be present in the solid phase under ordinary soil conditions. As will be seen from Table I, none of the soluble salts crystallize at a concentration corresponding to the optimum moisture content for the alkali soil, and until a concentration corresponding to the air-dry state is reached only sodium sulfate and chloride separate out. It is even more certainly true that in the humid soils the more soluble salts are constantly in solution in the soil moisture film.

It is to be noted with respect to the order of crystallization indicated in Table I for the Yolo clay extract that this order may be expected to vary with different soil extracts and with the temperature at which crystallization takes place. Van't Hoff[21] has given the order of crystallization of minerals from a system containing sodium, potassium, magnesium, sulfate and, chloride in certain proportions as shown in Table IV.

TABLE IV

	HALITE	KIESERITE	KAINITE	CARNALLITE	BISCHOFITE
1.........	95.4	..	..	..	..
2.........	4.42	1.05	2.02	..	..
3.........	0.03	0.35	..	0.1	..
4.........	0.15	0.38	..	0.08	7.62
	100.	1.78	2.02	0.18	7.62
	NaCl	3.8 $MgSO_4$	2.2 KCl	7.8 $MgCl_2$	

In this system halite separates first and continues to come down throughout the process, and the other salts as indicated above, the temperature of the system being maintained at 25° C. The soil solution system is much more complex than this, not only in respect to composition but also in the condition of crystallization. It is not therefore to be expected that the order of crystallization or even the composition of the salts would be just the same as that given in van't Hoff's summarization.[20]

The following geological profile of the Stassfurt deposits arranged in the order of the occurrence of the salts, from the bottom of the deposits upward, has been compiled from the data given by Clarke,[20] and serves as a convenient means of comparison with the results shown in Table I.

- Limestone
- Anhydrite
- Gypsum
- Halite
  - Glauberite
  - Langbeinite
- Polyhalite
- Kieserite
- Carnallite
  - Sylvite
  - Douglasite
  - Tachhydrite
  - Bischofite
- Kainite
  - Blodite
  - Loweite
  - Picromerite
  - Leonite
  - Aphthitalite
  - Vanthoffite
- Epsomite

The marked similarities which are observed are that halite having once begun to crystallize continues to do so during the entire process of evaporation. In the Stassfurt deposits we find a kainite group consisting of a mixture of kainite, blodite, loweite, picromerite, leonite, aphthitalite, and vanthoffite. This entire group is present, and in a comparatively narrow and contiguous range of evaporation in the white alkali extract. In these two respects at least the evaporation products from the white alkali extract very closely resemble the Stassfurt deposits.

All the results given in Tables I to IV indicate that complex salts separate out from soil solutions when evaporation takes place to a sufficient extent and in Table IV data are given which indicate that the conventional method of expression of the analytical data of solution is not only improper by reason of the extensive ionization which takes place in dilute solutions, but is also not justifiable, at least in concentrated solutions, because of the formation of complex salts not represented by the conventional methods of calculation. This observation is not so important, probably, as the following.

There is an accumulating mass of data which indicates that the condition of salts in solution is more complex than we are ordinarily accustomed to consider it, not only by reason of the formation of hydrates but also because of the formation of complex salts and ions, some of which are markedly different in properties from their components. It is not therefore without significance that as soil solutions become more concentrated the tendency toward the formation of complex ions increases and, with saturation, the deposition of complex salts takes place. These therefore may be, and probably are, present in soil moisture as the soil approaches the air-dry condition. The complex salts may very reasonably be assumed to play a role in the fertility of soils. What this role may be, whether beneficial or injurious to plant growth, is not apparent, but that it is of no consequence is not to be assumed. It is also possible that this question may be related to the much vexed one of the part played by fertilizers in soils.

It is to be regretted that this investigation was limited by its conditions in such a way as not to include a study of the organic constituents of the extract and a more detailed examination of the salts present in quantities less in amount or greater in solubility than those identified.

SUMMARY

(1) The salts deposited on the evaporation of the water extract of soils are much more complicated in character than is indicated by a simple statement of the ions existing in solution.

(2) There is a marked general similarity between the salts obtained on the evaporation of water extracts of soils and those obtained by both natural and artificial evaporation of sea water. No soil can be expected to furnish all the salts occurring in natural deposits of saline material because these represent crystallization from a composite soil extract.

(3) Since complex salts begin to separate from a water extract at a point not far from the concentration of an air-dry soil, it follows that the roots of growing plants may be in contact with solutions of these salts. Stated obversely, it is probable that the complex salts obtained from soil extracts may be present in the soils only in solution in soil moisture and never in the solid phase.

BIBLIOGRAPHY

1—G. J. Bouyoucos and M. M. McCool. "The Freezing Point Method as a New Means of Measuring the Concentration of the Soil Solution Directly in the Soil," Michigan Expt. Sta., *Tech. Bulletin* **24** (1915), 592.

2—G. J. Bouyoucos and M. M. McCool. "Further Studies on the Freezing Point Lowering of Soils," Michigan Sta., *Tech. Bulletin* **31** (1916), 637.

3—G. R. Stewart. "Effect of Season and Crop Growth in Modifying the Soil Extract," *J. Agr. Res.*, **12** (1918), 300.

4—D. R. Hoagland. "The Freezing Point Method as an Index of Variations in the Soil Solution Due to Season and Crop Growth," *J. Agr. Res.*, **12** (1918), 369.

5—G. P. Hoch. "The Effect of Sterilization of Soils by Heat and Antiseptics upon the Concentration of the Soil Solution," *Soil Science*, **3** (1917), 525.

6—C. Colemore and E. W. Hilgard. "The Amount and Kind of Salts Present in Different Portions of an Alkali Spot, from the Center to the Circumference." California Station, *Report* 1893–1849, 140.

7—E. W. Hilgard. "The Distribution of the Salts in Alkali Soils," California Station, *Bulletin* **108** (1895), 14.

8—A. H. Church. "Notes on 'Soluble Salts' in Soil," *Agr. Student's Gaz.*, **7** (1896), 212.

9—H. Puchner. "Concerning the Transport of Soluble Salts by the Movement of Water in the Soil," *Forsch. Geb. agr. phys.*, **18** (1895), 1.

10—J. J. Pwovarov. "On the Eolian Origin of Soil Salts," *Pedologie*, **1906**, 67.

11—A. Von. Sigmond. "The Principles Involved in the Preparation of Soil Solutions for Chemical Analysis." II Conf. Agrogeol. Internat., Stockholm, Resumé [1] **1910**, p. 25

12—S. A. Zkharoo. "Soil Solutions: Their Role in Soil Formation, Methods of Their Examination, and Their Significance for the Characterization of Soil Types," *Russ. J. Expt. Landw.*, **7** (1906), 388.

13—J. F. Morgan. "The Soil Solution Obtained by the Oil Pressure Method," *Soil Science*, **3** (1917), 531.

14—C. B. Lipman. "A New Method of Extracting the Soil Solution," Univ. of Cal. Pubs., *Agr. Sci.*, **3** (1918), 131.

15—John S. Burd. "Water Extractions of Soils as Criteria of Their Crop Producing Powers," *J. Agr. Res.*, **12** (1918), 297.

16—F. W. Clarke. "The Data of Geochemistry," U. S. Geol. Survey, *Bulletin* **616** (1916), 58.

17—C. F. Marbut, H. H. Bennett, J. E. Lapham, and M. H. Lapham. "Soils of the United States," U. S. Dept. of Agr., Bureau of Soils, *Bulletin* **96** (1913); Yolo Clay, p. 683; Smithsonian Soil, p. 290; Crowley Silt Loam, p. 288; Stockton Clay Loam, p. 685.

18—E. Erdman. "Die Chemie aud Industrie der Kalisalze," p. 2; Deutschlands Kalibergban, **1907**.

19—G. J. Young. "Potash Salts and Other Salines in the Great Basin Region," U. S. Dept. of Agr., *Bulletin* **61** (1914), 69.

20—J. H. van't Hoff. "Zur Bildung der Ozeanischen Salzablagerungen," [1] **1905**, p. 72–75, [2] **1909**, p. 79–81.

DETERMINATION OF NITRATE NITROGEN IN NITRATES AND FERTILIZERS[1]

By H. C. Moore

CHEMICAL LABORATORY, ARMOUR FERTILIZER WORKS, ATLANTA, GA.

The determination of the nitrate value in nitrate of soda and potash is an important one. Oftentimes a sample represents several thousand tons, and at the present price of these materials it is obvious that wide discrepancies lead to controversies. There have been many methods proposed for this determination, but the majority of them have only a historic interest. Descriptions of these earlier methods, which are scarcely heard of recently, may be found in the literature.[2]

The methods which have been in more or less use recently may be referred to briefly as follows:

I—CONVERSION TO NITRIC OXIDE:
- (1) Nitrometer methods[3,4]
- (2) Schloesing[3] and Schloesing-Wagner[5] methods

II—DETERMINATION AS AMMONIA:
- (1) Reduction in alkaline solutions
  - (*a*) Reduction with aluminum[3]
  - (*b*) Reduction with zinc dust and iron powder[3,6]
  - (*c*) Reduction with Devarda alloy[4]
- (2) Reduction in acid solution
  - (*a*) Reduction with reduced iron powder[3,6]
  - (*b*) Reduction with reduced iron powder and zinc[3]
- (3) Phenol-sulfonic acid method (Kjeldahl[2,3,4,5,6]-Gunning)

III—WEST COAST OR REFRACTION METHOD: Determining method water-insoluble matter, chlorine and sulfur trioxide, and calculating as the chloride and sulfate of soda. The difference between the sum of these four results and 100 is called sodium nitrate.

Of the above, the nitrometer methods, or some of those under II, have been in quite general use. Unfortunately the West Coast, or Refraction, method has been very much used, being especially prescribed in contracts governing the imported Chilean nitrate. Doubtless this method would be very accurate if the four impurities named were the only ones found in commercial nitrate, but other impurities also are sometimes present, such as KNO_3, Na_2CO_3, $NaClO_3$, $NaClO_4$, Fe_2O_3, Al_2O_3, CaO and MgO.[1] It is obvious, therefore, that the method favors only the seller, the buyer paying for all ingredients other than the four above named as sodium nitrate.

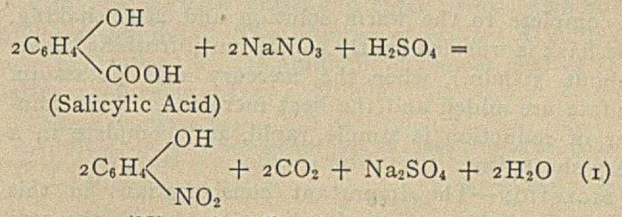
Probably the methods which are in most general use at present are the Nitrometer, Devarda, and Modified Kjeldahl-Gunning. In the fertilizer laboratories either the Modified Kjeldahl or Modified Gunning, or the Modified Kjeldahl-Gunning methods fit most conveniently into the routine. Mr. W. S. Allen[2] described a modification of the Devarda method by which correct results may be obtained. He also reported the analysis of 69 samples by this method and by the West Coast method, and showed that for all of these samples the latter method gave higher results as follows:

SAMPLES	$NaNO_3$ Agreement within per cent
5	0.1
11	0.3
19	0.5
32	0.8
35	1.0
54	1.5
63	2.0
67	2.5
69	3.0

He gives the complete analysis of several samples, showing that when proper allowance for all impurities was made, results by both methods agreed closely. It will be noted that for six of the above samples the buyer would lose from $1.00 to $2.00 per ton by the West Coast result.

MODIFIED KJELDAHL-GUNNING METHOD

The Modified Kjeldahl-Gunning method will be considered here, and when correctly applied will yield extremely accurate results. The method is based upon the following reactions:



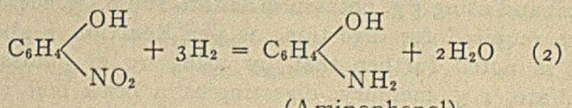
$$2C_6H_4\begin{cases}OH\\COOH\end{cases} + 2NaNO_3 + H_2SO_4 =$$

(Salicylic Acid)

$$2C_6H_4\begin{cases}OH\\NO_2\end{cases} + 2CO_2 + Na_2SO_4 + 2H_2O \quad (1)$$

(Nitrophenol)

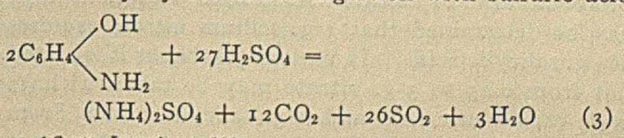
This nitrophenol is next reduced to aminophenol. Just what this reaction is when using "hypo" is not known, since there are present SO_2, H_2S, and free sulfur. The reaction with zinc where hydrogen is liberated is probably as follows:



$$C_6H_4\begin{cases}OH\\NO_2\end{cases} + 3H_2 = C_6H_4\begin{cases}OH\\NH_2\end{cases} + 2H_2O \quad (2)$$

(Aminophenol)

And finally by continued digestion with sulfuric acid:



$$2C_6H_4\begin{cases}OH\\NH_2\end{cases} + 27H_2SO_4 =$$

$$(NH_4)_2SO_4 + 12CO_2 + 26SO_2 + 3H_2O \quad (3)$$

---

[1] Presented at the 58th Meeting of the American Chemical Society, Philadelphia, Pa., September 2 to 6, 1919.

[2] Lunge, "Technical Methods of Chemical Analysis," **1**, Part 1, pp. 309–11; Fresenius-Cohn, p. 573; Sutton, "Volumetric Analysis," Third Edition, p. 100

[3] Lunge, *Loc. cit.*

[4] Scott, "Standard Methods of Chemical Analysis," p. 306.

[5] Wiley, "Principles and Practice of Agr. Anal.," **2**, p. 397.

[6] U. S. Dept. of Agr., Bur. of Chem., *Bulletin* **107**, 10.

[1] Scott, *Loc. cit.*, p. 303.

[2] *Proc. 8th Intern. Congr. Appl. Chem.*, **1**, 19.

Reaction 3 is completed by adding mercury as in the Modified Kjeldahl method or potassium sulfate as in the Modified Gunning or by adding both as in the Modified Kjeldahl-Gunning method.

The four important considerations in the process of analysis by the Modified Kjeldahl-Gunning method are as follows: Solution, reduction, digestion, and proportion of reagents.

SOLUTION—The sample of nitrate must be dissolved in the salicyl-sulfonic acid (salicylic acid dissolved in sulfuric acid) before the reduction is begun. As usually accomplished this solution is a slow process requiring from one-half hour to a day or more with frequent shaking. It has been generally believed that this must be done in the cold and some have even kept the flasks cooled with ice water, hoping thereby to avoid loss of nitrogen as nitric acid. The loss of time occasioned by dissolving the sample, even at room temperature, was so great as to make burdensome the amount of work contemplated. Attempts were made to hasten solution by heating gently, when it developed that solution could be effected very rapidly. The heating may be done by immersing flask in boiling water, or more easily over low flame or electric heater and with frequent shaking, heating the flask not much hotter than the hand can bear. A peculiar hissing sound is observed when the heat is applied, and the solution completed in from one to three minutes without the slightest loss of nitrogen. Results were even more concordant than when allowing to dissolve by long standing at room temperature.

REDUCTION—While zinc dust is used by some analysts for the reduction, the writer has found sodium hyposulfite, "hypo," more convenient and also that it gives more reliable results. This may be due to inexperience with zinc dust, which has given low results. The results reported herein were obtained using hypo. The hypo is added as soon as solution is complete to the warm solution and, after shaking, the flask is warmed gently at first until frothing ceases (about 5 min.), when the mercury and potassium sulfate are added and the heat increased. This manner of reduction is simple, rapid, and complete in a very short time.

DIGESTION—The important considerations in this stage are the length of the digestion period, the rate of boiling or amount of heat applied, and the amounts of reagents employed, as will be noted from a study of Tables I to IV. It is noted that at least 45 cc. to 50 cc. salicyl-sulfonic acid are required for 0.8517 g. sodium nitrate and that as much as 4 to 8 hrs. at gentle boil and using 5 g. potassium sulfate and 0.5 g. mercury are required for digestion, as per Reaction 3 above. It is noted further that by using 10 g. potassium sulfate, 1 g. mercury, digesting at a brisk boil 1 hr. after solution had become clear, is sufficient.

PROPORTION OF REAGENTS—From Reaction 1 it may be determined that 1 g. sodium nitrate requires 1.64 g. salicylic acid. In various textbooks it is stated that from 0.25 to 5 g. nitrate may be taken with 30 to 35 cc. sulfuric acid and 2 g. salicylic acid. From this it may be understood why some analysts have not obtained concordant and correct results on account of failure to use the correct proportion of reagents.

In 1911, the Committee on Nitrogen, Division of Fertilizer Chemists, AMERICAN CHEMICAL SOCIETY, received reports from fifteen analysts from coöperative work on a sample of nitrate of soda sent out by the committee, and the committee in its report noted the tendency to higher results when a 0.25 g. sample of nitrate was used than when using larger quantities. This is due to the fact that a more nearly correct proportion of reagents was employed when the smaller sample was used. Hardly any two analysts described the same details of analysis.

TABLE I

REAGENTS: 0.8517 g. sodium nitrate 1 (20.01 per cent NH_3—accepted value); 50 cc. salicyl-sulfonic acid (containing 2 g. salicylic acid) warming to effect solution; 5 g. hypo; 5 to 6 g. potassium sulfate; 0.5 g. mercury; 1 g. sodium sulfide, etc.

No. determinations..	4	2	7	8	15	7	15	...
Digestion after solution is clear, boiling gently, hrs.[1]......	1	1.5	2	3	4	5	6	8
Max. result, per cent	19.86	19.95	19.99	20.02	20.02	20.02	20.04	...
Min. result, per cent	19.81	19.91	19.81	19.86	19.92	19.93	19.91	...
Av. of all, per cent..	19.84	19.93	19.90	19.95	19.97	19.97	20.00	...
Same as Above, but Added 10 g. Potassium Sulfate								
No. determinations..	25	...	14	17	27	...	8	7
Max. result, per cent	20.04	...	20.05	20.01	20.03	...	20.02	20.06
Min. result, per cent	19.81	...	19.91	19.86	19.93	...	19.96	19.92
Av. of all, per cent..	19.95	...	19.97	19.95	19.99	...	20.00	20.01
Same, but after Adding 10 g. Potassium Sulfate and 0.5 g. Mercury at Brisk Boil[2]								
No. determinations..	...	...	4	4	15	5	...	...
Digestion, hrs.......	...	...	0.25	0.5	1.0	1.5	...	...
Max. result, per cent	...	...	19.95	19.98	20.03	20.05	...	...
Min. result, per cent	...	...	19.88	19.93	19.98	19.96	...	...
Av. of all, per cent..	...	...	19.92	19.95	20.01	20.00	...	...

[1] By "gentle boil" is meant sufficient heat to keep contents of flask just boiling.

[2] By "brisk boil" is meant sufficient heat to keep contents of flask boiling briskly, and to keep the neck of the flask washed down by the sulfuric acid condensing therein.

Probably most analysts use not over 0.5 g. sample of sodium nitrate for the analysis, and from other results obtained it was found that for 0.5 g. sample 35 cc. of salicyl-sulfonic acid, containing 1 g. salicylic acid, 5 g. hypo, 5 to 6 g. potassium sulfate, 0.5 g. mercury, 1 g. sodium sulfide, and digesting for 1 hr. after clear at a slow boil, gave correct results. It thus appears that the proportion of reagents is very important. It was found further that 1 g. sample of sodium nitrate could be used with the same proportion of reagents as used in this work with 0.8517 g. Table III shows that at least 45 cc., and preferably 50 cc., salicyl-sulfonic acid are required for 0.8517 g. sample, and other reagents used, and Table 4 shows that for 1.7034 g. sample 70 cc. are required. Why it is necessary to use so great an excess of sulfuric acid with increase in sample is not apparent. It is not to prevent loss of ammonia from ammonium sulfate, as was proved by using a sample of pure ammonium sulfate, weighing out 0.4259 g., 0.8517 g., and 1.7034 g. portions, digesting with 25 cc., 30 cc., 35 cc., and 40 cc. sulfuric acid, and adding 10 g. potassium sulfate, 0.5 g. mercury, 1 g. sodium sulfide, 1 g. sugar, etc., and digesting for 2 hrs. after clear at gentle boil. Results were practically the same with all portions and for all amounts of sulfuric acid.

EXPERIMENTAL WORK

The samples used for this experimental work, as reported partly in Tables I to IV, were as follows:

(1) Sodium nitrate 1, a sample of sodium nitrate

TABLE II

REAGENTS: 1.0134 g. potassium nitrate (equivalent to 0.8517 g. sodium nitrate); 50 cc. salicyl-sulfonic acid (containing 2 g. salicylic acid) warming to effect solution; 5 g. hypo; 5 to 6 g. potassium sulfate; 0.5 g. mercury; 1 g. sodium sulfide, etc.

No. determinations	6	...	...
Digestion after solution is clear, boiling gently, hrs.	4	6	8
Maximum result, per cent	20.00	...	...
Minimum result, per cent	19.93	...	...
Average of all, per cent	19.98	...	...

Same, but 10 g. Potassium Sulfate

No. determinations	9	8	8
Maximum result, per cent	20.03	20.08	20.03
Minimum result, per cent	19.96	19.93	19.89
Average of all, per cent	19.99	20.01	19.95

Same, but 10 g. Potassium Sulfate, 1 g. Mercury and 2 g. Sodium Sulfide Digesting for One Hour after Clear

	Gentle Boil	Brisk Boil
No. determinations	4	26
Maximum result, per cent	20.07	20.07
Minimum result, per cent	20.00	19.99
Average of all, per cent	20.03	20.036

NOTE—20.038 per cent ammonia is theoretical for pure sodium nitrate.

of high purity, ground and dried at 130° C. The value was accepted as 20.01 per cent ammonia, being the actual result obtained as shown in tables and by comparison with the sample of potassium nitrate.

(2) Potassium nitrate of high purity was recrystallized twice from water, dried at 100° C. and finally at 210° C. This was found free from sulfate, chloride, etc., prepared as per Scott.[1] In using this sample 1.0134 g., equivalent to 0.8517 g. sodium nitrate, were used and results calculated as if pure sodium nitrate was used, containing 20.038 per cent ammonia.

As some laboratories use copper sulfate instead of mercury, some determinations were made using 0.5 g. copper sulfate, but the few results obtained were practically the same though slightly less concordant. The same applies when using 0.5 g. each of mercury and copper sulfate. Copper sulfate offers no advantage over mercury and seems slower in action. Sodium sulfate was found to give the same results as when potassium sulfate was used. The effect of sodium chloride was noted by adding from 0.1 g. to 0.5 g. sodium chloride without effect on the accuracy of the method.

The effect of using moist samples was noted by adding from 0.1 cc. to 0.5 cc. water without affecting the results. At times, however, adding the salicyl-sulfonic acid to moist samples has caused some heating and puffs of nitric acid could be noted, so dry samples are preferable. If not dry the sample may be dried after weighing, except for the difficulty of weighing a moist sample, by rotating flask over a low heat.

The details of analysis for samples of nitrate which give correct results are as follows:

REAGENTS

1—Salicyl-sulfonic acid: 40 g. salicylic acid are made up to one liter with concentrated sulfuric acid

2—Sodium hyposulfite (hypo): Commercial photographic, pea-size

3—Potassium, or sodium, sulfate, preferably dry powder

4—Metallic mercury (or mercuric oxide)

5—Caustic soda: Dissolve 30 lbs. commercial caustic soda in about 2.5 gal. water, let settle, siphon off the clear solution. This strong caustic soda is practically free from carbonate

6—Sodium sulfide: Dissolve 100 g. fused sodium sulfide[2] in water and dilute to 1,000 cc.

7—Pure granulated, or 20 to 30 mesh zinc: Pure zinc is essential as impure zinc reacts so actively with the NaOH that the rapid evolution of hydrogen carries over by entrainment some free alkali, even when using the Hopkins connecting bulb. This causes a variable blank. The Davisson[1] bulb will prevent this entrainment

8—0.5 N sulfuric acid solution

9—0.25 N or 0.1 N NaOH solution

10—Sodium alizarin sulfonate: 2 g. in 100 cc. water.

METHOD

Transfer 0.8517 g. $NaNO_3$ (one gram may be used if desired) preferably to a 650 cc. Pyrex Kjeldahl flask, add 50 cc. salicyl-sulfonic acid, preferably from a dispensing burette, rinsing down neck of the flask, warm over low heat or in boiling water or steam bath until action begins, shaking frequently until solution is complete, add 5 g. hypo, and heat over low heat until frothing ceases (about 5 min.), then add 10 g. sodium of potassium sulfate, 1 g. mercury, and continue digestion until clear, and for an hour afterwards, boiling briskly; cool, dilute with water to about 400 cc., add a small piece of granulated zinc, or a small pinch of 20 or 30 mesh zinc (0.1 g. is sufficient), 70 to 80 cc. caustic soda in which are dissolved 2 g. of fused sodium sulfide. The ammonia is distilled and collected in 0.5 N sulfuric acid. About 150 to 200 cc. distillate are sufficient, requiring about 45 min. Use in receiving flask 21 cc. 0.5 N sulfuric acid diluted to 125 to 150 cc. with water and three drops of sodium alizarin sulfonate. The number of cc. 0.5 N sulfuric acid neutralized by the ammonia gives per cent of the latter when using 0.8517 g. $NaNO_3$.

TABLE III

REAGENTS: 0.8517 g. sodium nitrate 1 (20.01 per cent NH_3); varying the amount of salicyl-sulfonic acid (containing in each case 2 g. salicylic acid) warming to effect solution; 5 g. hypo; 10 g. potassium sulfate; 1 g. mercury; 2 g. sodium sulfide, etc., digesting at brisk boil for one hour after clear.

No. determinations	3	7	7	15[1]	3
Salicyl-sulfonic acid, cc.	35	40	45	50	55
Maximum result, per cent	19.91	20.00	20.05	20.03	20.03
Minimum result, per cent	19.79	19.92	19.96	19.98	19.99
Average of all, per cent	19.86	19.96	20.00	20.01	20.02

[1] In these 15 determinations only 0.5 g. mercury and 1 g. sodium sulfide were used, but 0.5 g. mercury has been found to give (when using 10 g. potassium sulfate) the same result as 1 g., except the latter amount causes the solution to clear more quickly.

TABLE IV—USING SODIUM NITRATE 1 (20.01 PER CENT NH_3)

1.7034 g. sample; salicyl-sulfonic acid as shown (containing in each case 4 g. salicylic acid); 1 g. sodium sulfide for each 0.5 g. mercury.

	MIXTURE					RESULTS		
Cc. Acid	Hypo G.	K_2SO_4 G.	Mercury G.	DIGESTION	No. of Detns.	Maximum	Minimum	Average
50	6–7	5	0.5	0.5 hr. after clear, gentle boil	4	19.80	19.73	19.78
65	10	10	0.5	4 hrs. after clear, gentle boil	5	19.85	19.79	19.83
	10	10	1.0	1 hr. brisk boil after clear	4	19.95	19.90	19.93
	10	10	1.0	2 hr. brisk boil	5	19.93	19.80	19.86
70	8	3	0.5	4 hrs. after clear, gentle boil	2	20.02	19.97	20.00
	10	5	1.0	1 hr. brisk boil after clear	5	20.02	19.94	20.01
75	10	5	1.0	1 hr. brisk boil after clear	2	19.95	19.95	19.95
80	10	5	1.0	1 hr. brisk boil after clear	3	20.00	19.93	19.96

Run a blank on all reagents and apply the correction thus obtained. With good reagents the blank should not amount to more than 0.10 cc. 0.25 N sulfuric acid, or on basis of 0.8517 g. $NaNO_3$ to 0.05 per cent NH_3.

In the course of this work about 150 blanks were made, always running several at a time, and they

---

[1] *Loc. cit.*

[2] The sodium sulfide solution can be most conveniently made a part of the water used to dissolve the caustic soda. About 75 to 80 cc. of this caustic is required where 50 cc. sulfuric acid have been used. Two grams sodium sulfide should be used for each test.

[1] THIS JOURNAL, 11 (1919), 465.

varied from about 0.25 per cent NH_3 in the early work to 0.05 per cent later when a new lot of sulfuric acid free from nitrogen was secured. Results were the same with the high blank as with the low one, but a low blank is obviously to be desired, and without making the blank allowances results exceeding the theoretical would have been obtained.

COMPARISON OF METHOD

Having developed a method for the accurate determination of nitrate, as ammonia, it was decided to compare it with the West Coast, or Refraction, method. Accordingly, four samples of commercial nitrate, Nos. 36059, 60, 61, and 62, received at our plants at various times, were selected for comparison, and results appear in Tables V and VI. These results only confirm the more elaborate comparison of Mr. W. S. Allen,[1] and show that on one sample the agreement is very close. In another it is very close after making allowance for the potassium nitrate, but in the other two samples correcting for potassium nitrate still leaves a discrepancy of nearly 1.5 per cent $NaNO_3$, indicating that this error is due to other impurities. The West Coast method gives results which are often very deceptive and the buyer may pay for as much as 2 to 3 per cent $NaNO_3$ more than he actually receives.

TABLE V—RESULTS ON SAMPLES BY THE WEST COAST OR REFRACTION METHOD

	36059 Per cent	36060 Per cent	36061 Per cent	36062 Per cent
Insoluble	0.27	0.14	0.16	0.48
NaCl	0.94	2.37	1.69	1.29
Na_2SO_4	0.16	0.22	0.25	0.32
H_2O at 130° C.	0.41	0.55	0.59	0.56
TOTAL	1.78	3.28	2.69	2.65
$NaNO_3$ by difference	98.22	97.62	97.31	97.35
K_2O found	0.34	3.33	3.26	4.26
Equivalent to KNO_3	0.73	7.15	7.00	9.14
Error in $NaNO_3$ due to KNO_3	0.12	1.14	1.12	1.46

TABLE VI—RESULTS ON SAMPLES BY DETERMINATION AS NH_3 BY USING 0.8517 G., 50 CC. SALICYL-SULFONIC ACID, ETC.

	36059 Per cent	36060 Per cent	36061 Per cent	36062 Per cent
	19.69	18.84	18.96	19.22
	19.68	18.89	18.98	19.18
	19.63	18.90	18.95	19.28
	19.69	18.94	18.99	19.22
	19.64	18.86	18.96	19.22
AVERAGE,	19.67	18.89	18.99	19.22
Equivalent to $NaNO_3$	98.17	94.27	94.77	95.92
Error due to West Coast—per cent $NaNO_3$	0.05	2.45	2.54	1.43
Net error as $NaNO_3$ after correcting for per cent KNO_3	0.07	1.31	1.42	0.03

The advantages of the proposed method are: (1) It is convenient, especially fitting the routine of the fertilizer laboratory. A determination may be completed in two and one-half hours; (2) the results are unusually concordant for a material of such high percentage and give theoretical results on pure potassium nitrate.

APPLICATIONS OF METHOD

Following this work and the discovery that a rapid solution of the nitrate in the salicyl-sulfonic mixture by heating gave theoretical results, the question which naturally arises is: Would this same procedure apply to samples of mixed fertilizers containing nitrate? Accordingly a series of samples were prepared as follows:

1—Cured acid phosphate ground to pass 30 mesh.

2—Commercial nitrate of soda, dried, and ground to pass 30 mesh.

3—Tankage ground to pass 30 mesh.

4—Sulfate of potash, dry powder, same as used in ammonia digestions and found free from nitrogen.

The following five mixtures were made up from these samples; the ammonia was not determined on the materials used, but only in the mixture. Mixture 1 serves as a check sample, the amount of acid and tankage remaining the same in all five mixtures. All of these mixtures were made with the utmost care, 200 g. mixtures being made, and all materials weighed on an analytical balance and very carefully mixed.

	(1) Grams	(2) Grams	(3) Grams	(4) Grams	(5) Grams
Acid phosphate	100	100	100	100	100
Tankage	50	50	50	50	50
Nitrate	—	10	20	40	50
Sulfate of potash	50	40	30	10	—

The sample of nitrate of soda was analyzed by the method previously described and found to contain 18.98 per cent ammonia.

The ammonia content of Sample 1 was determined using salicyl-sulfonic acid and reagents as described in Table VII. Various amounts of acid were used but 35 cc. gave the same results as greater amounts, and the ammonia was found to be 2.40 per cent. Samples 2, 3, 4, and 5, therefore, should contain as follows:

(2) 3.35 per cent ammonia (0.95 per cent from nitrate)
(3) 4.30 per cent ammonia (1.90 per cent from nitrate)
(4) 6.20 per cent ammonia (3.80 per cent from nitrate)
(5) 7.15 per cent ammonia (4.75 per cent from nitrate)

From a study of Table VII it will be noted that results are practically theoretical when allowing the sample to remain in the salicyl-sulfonic acid 10 to 15 min. at room temperature with frequent shaking, then adding hypo and proceeding as in the method de-

TABLE VII

1.7034 g. sample; 35 cc. salicyl-sulfonic acid (containing 1 g. salicylic acid); 5 g. hypo; 5 to 6 g. potassium sulfate; 0.5 g. mercury; 1 g. sodium sulfide; etc., digesting 1.5 hrs. after clear at gentle boil

SAMPLE	2	3	4	5
CALCULATED, Per cent	3.35	4.30	6.20	7.15
(1) Standing overnight in salicyl-sulfonic acid at room temperature and shaking several times before adding reagents	3.32	4.30	6.08	6.98
	3.34	4.28	6.07	7.06
	3.37	4.32	6.08	6.96
	3.36	..	6.06	7.07
AVERAGE,	3.36	4.30	6.07	7.02
(2) Standing 10–15 min. after adding salicyl-sulfonic acid at room temperature and shaking several times before adding reagents	3.32	4.28	6.09	7.07
	3.34	4.25	6.17	7.14
	3.34	4.23	6.15	7.09
	3.30	4.23	6.14	6.97
	3.37	4.25	6.15	7.08
	3.32	4.28	6.11	6.97
AVERAGE,	3.33	4.25	6.13	7.05
(3) Same as (2) but stood 1.5 hrs. after adding salicyl-sulfonic acid	3.31	4.27	6.12	7.06
	3.42	4.27	6.13	6.91
AVERAGE,	3.36	4.27	6.13	7.01
Same as Above, but Using 0.8517 g. Sample				
Same as (1) above	3.33	4.27	6.12	..
	3.33	4.34	6.12	..
	3.32	4.36	6.14	..
	3.37	4.24	6.19	..
	..	..	6.13	..
AVERAGE,	3.34	4.30	6.13	..
Same as (2), above	3.33	4.27	6.13	7.14
	3.38	4.27	6.18	7.11
	3.30	4.34	6.18	7.14
	3.32	4.29	6.16	7.18
AVERAGE,	3.33	4.29	6.16	7.14

---

[1] *Loc. cit.*

scribed previously. It will be noted that results for Samples 3, 4, and 5 are slightly better when using 0.8517 g. than when using 1.7034 g. It was also found from results not reported, using Sample 5, that using 1.7034 g. sample with 50 cc. salicyl-sulfonic acid (containing 1.5 g. salicylic acid), 5 g. hypo, 5 to 6 g. potassium sulfate, 1 g. mercury, 2 g. sodium sulfide, etc., that the same results were obtained as when using 0.8517 g. with 35 cc. of acid, etc. Here again the proportions of reagents and sample are found to be important and indicate that for samples containing high nitrate content, say, 4 per cent ammonia or over, more correct results may be obtained by using 0.8517 g. sample, as the use of 50 cc. of acid is hardly to be recommended.

Determinations were also made on Samples 2, 3, 4, and 5 by warming after adding the salicyl-sulfonic acid, as described before in case of nitrate, but for all samples the results were slightly lower, amounting from 0.05 per cent to 0.10 per cent ammonia.

SUMMARY

From the foregoing it appears that the best and really most convenient procedure for the determination of total ammonia in fertilizers containing nitrate is as follows:

Weigh 1.7034 g. sample (0.8517 g. in case of mixtures high in nitrate) preferably into a 650 cc. Pyrex Kjeldahl flask, add 35 cc. salicyl-sulfonic acid, containing 1 g. salicylic acid, and after shaking frequently for 15 min. add 5 g. hypo, heat gently until frothing ceases, add 5 g. potassium or sodium sulfate, 0.5 g. mercury, and digest until clear and for 1.5 hrs. or more afterwards, cool, dilute to about 400 cc. and proceed as in case of nitrate samples, using in this case 1 g. sodium sulfide.

Altogether over 1,000 determinations have been made on nitrate samples and about 200 determinations on the four mixed fertilizer samples and the results summarized in the foregoing tables. All determinations have been corrected for ammonia in reagents, by blank determinations run with nearly every set, measuring apparatus carefully calibrated, standard solutions frequently checked and allowance made for change in temperature of these solutions. The 0.5 *N* sulfuric acid solution was standardized by three methods: (1) By sodium carbonate prepared as per Scott,[1] (2) by acid potassium phthalate prepared and used as described by Dodge,[2] and (3) by determination as barium sulfate, cold precipitation method of Allen and Bishop. The results by the three methods of standardizing agreed closely, and were as follows in order named: 100.20 per cent, 100.13 per cent, and 100.17 per cent 0.2 *N*, average 100.17 per cent.

The writer hopes that this contribution to the literature for the correct valuation of nitrate and the determination of total nitrogen in samples of fertilizer containing nitrate may be of value to others and serve in some measure toward the adoption of more uniform methods of analysis; also that the West Coast method for valuation of commercial nitrate of soda may be relegated to the obsolete.

---

[1] *Loc. cit.*

[2] THIS JOURNAL, 7 (1915), 29.

A DIRECT METHOD FOR THE DETERMINATION OF RUBBER HYDROCARBON IN RAW AND VULCANIZED RUBBER[1]

By W. K. Lewis and W. H. McAdams

DEPARTMENT OF CHEMISTRY AND CHEMICAL ENGINEERING, MASSACHUSETTS INSTITUTE OF TECHNOLOGY, CAMBRIDGE, MASS.

In the fall of 1916 the writers noticed in the literature[2] a number of articles concerning analytical methods for the determination of rubber hydrocarbon in raw and vulcanized rubber, depending upon the bromine absorbed by rubber. A survey of these articles showed that several different types of bromination methods had been proposed, but the results appeared to be very discrepant. The most promising type seemed to be the one involving the addition of a known amount of bromine, dissolved in a suitable organic solvent, such as carbon tetrachloride, to a solution of the rubber in an organic solvent. After the solution had stood for a period of time the unabsorbed or excess bromine was determined by titration, usually by the addition of standard sodium thiosulfate in the presence of KI solution and starch paste.

Inasmuch as this paper will not deal with the structural formula of rubber, the conventional formula $(C_{10}H_{16})_n$ will be used to designate rubber hydrocarbon, with the understanding that this contains $2n$ double bonds. Two types of reaction are possible, involving in the first case, addition, and in the second, substitution, of bromine.

$$(C_{10}H_{16})_n + 2nBr_2 = (C_{10}H_{16}Br_4)_n \qquad (1)$$

$$(C_{10}H_{16})_n + nBr_2 = (C_{10}H_{15}Br)_n + nHBr \qquad (2)$$

In the second reaction it will be noted that *two* atoms of bromine are necessary to substitute *one* atom of hydrogen, thereby producing one formula weight of HBr. Some of the investigators found that substitution occurred, and tried to choose conditions such that substitution would be minimized or entirely prevented.

McIlhiney[3] has shown that in the bromination of unsaturated oils in an organic solvent the bromine not only adds to the double bonds as in Reaction 1 but a considerable amount is substituted as in Reaction 2. Gill[4] states that rosin oil shows a very high substitution.

This paper deals with the application of the McIlhiney method, developed for unsaturated oils, to rubber hydrocarbon. Briefly, it consists in determining by a volumetric method the substitution which does occur under the particular conditions of the analysis in question, and deducting twice the observed substitution from the bromine consumed, which gives a measure of the true bromine addition, from which the rubber hydrocarbon is readily calculated.

EXPERIMENTAL PART—RAW RUBBER

PREPARATION OF PURE RUBBER HYDROCARBON SOLUTION—Plantation pale crêpe was extracted overnight with acetone[5] in the standard extraction apparatus

---

[1] Read before the Rubber Division at the St. Louis Meeting, American Chemical Society, April 12–16, 1920.

[2] See Bibliography, p. 676.

[3] *J. Am. Chem. Soc.*, 21 (1899), 1084.

[4] "Oil Analysis," 9th Ed. (Revised), p. 67.

[5] The acetone was allowed to stand with Na_2CO_3 and $CaCl_2$ and then fractionally distilled.

to remove the resins, then carefully dried, dissolved in pure carbon tetrachloride, and finally filtered to remove the proteins and other insoluble matter. The resulting solution of pure rubber hydrocarbon was analyzed by evaporation to determine the total solids present in a known volume, and this known rubber hydrocarbon content was used as a basis of comparison with the calculated figures found by bromine addition as described below.

PROCEDURE—To a known volume of the above pure rubber hydrocarbon solution, containing approximately 0.2 g. $(C_{10}H_{16})_n$, a measured volume of bromine in pure carbon tetrachloride corresponding to approximately 150 per cent excess bromine above that necessary for addition was added, and the mixture was allowed to stand in glass-stoppered bottles for varying lengths of time in a dark closet at room temperature. After this exposure to bromination, 10 cc. of a 3 per cent KI solution were added to take up the excess bromine, and the resulting iodine was titrated by means of 0.25 *N* standard sodium thiosulfate, using starch paste as an indicator. In order to determine the substitution which had occurred, 10 cc. of 5 per cent KIO_3 were now added to convert the equivalent of the HBr into iodine, which was then titrated to a second end-point. A blank was run under the same conditions as the rubber determination in order to determine the bromine added to the rubber analysis, and to eliminate any error caused by impurities in the reagents used. The sample calculation given below indicates the relation of the various readings.

Rubber taken = 0.2000 G. Thiosulfate Solution = 0.235 *N*

Equivalent Weight of Rubber Hydrocarbon, $(C_{10}H_{16})_n$ = 34

	Cc. Thio.
For blank	70.00
Excess found by first titration	38.00
Consumed	32.00
Twice second titration, (2) (3.5)	7.00
True addition	25.00

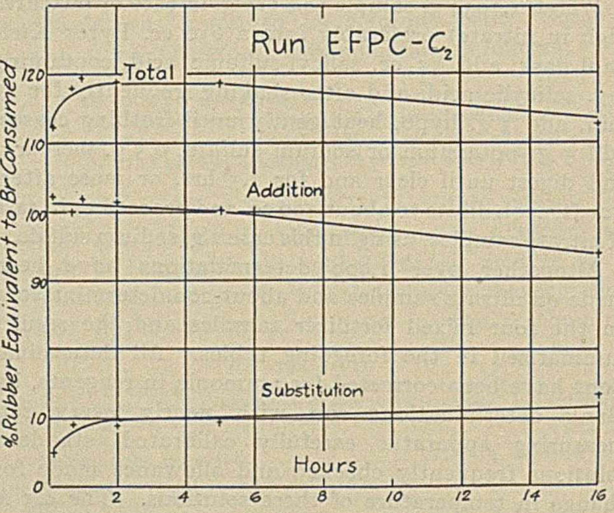
$$\frac{25.00 \times 34 \times 0.235 \times 100}{0.2000 \times 1000} = 100 \text{ per cent theoretical addition}$$

NOTES—In order to avoid loss of bromine vapor upon opening the bottle after the bromination period, the bottle was cooled by immersion for a few minutes in ice water, in the dark, and the KI solution was introduced by means of 1-in. rubber tubing attached to the neck of the bottle and extending up above the stopper.

The carbon tetrachloride was purified by subjecting it to the action of saturated chlorine water for several days in diffused daylight, followed by washing with water and drying with CaO, previous to a distillation in which the fraction boiling within 1° C. of the proper boiling point was taken.

DISCUSSION OF RESULTS—The attached plot shows typical results[1] of runs on extracted, filtered pale crêpe. It will be noticed that the substitution increased with the bromination time, but that the addition followed closely the theoretical 100 per cent line, when the time of exposure was 2 to 4 hrs. The substitution is doubtless a function of the conditions under which the experiment is conducted. For example, it is quite possible that a small amount of water present during the bromination would increase the substitution.

One possible objection to this method is that substitution might continue after the addition of the KI solution. It will be noted that after this time no halogen except iodine is present in the solution, and iodine is known to be much less apt to substitute hydrogen than bromine. Furthermore, if iodine substitution during titration took place before the first end-point no error whatever would be introduced; the effect would be the same as if bromine had been substituted, as the HI so formed would be determined in the second titration and hence an automatic correction would be introduced for this action. However, if iodine substitution took place after the first end-point (by the substitution of iodine produced by the action of KIO_3 on the HI already present) the second titration would be high and the calculated addition would be low, because the addition is obtained by subtracting twice the second titration from the bromine consumed in the first. Furthermore, if at any time HBr splits off from the brominated molecule and this action is not accompanied by further addition, the calculated addition would be low. The results as shown in the attached plot indicate that if such undesirable actions occurred they were negligible in effect.



VULCANIZED RUBBER

The above results have shown the method to be satisfactory for raw rubber; it remained for Sackett and Seltzer[1] to adapt it to vulcanized rubber. Since carbon tetrachloride will not "dissolve" vulcanized rubber, a new "solvent" had to be obtained, and the choice was tetrachlorethane.

In addition to the rubber hydrocarbon, resins, and proteins present in raw rubber, vulcanized rubber may contain fillers and compounding materials, such as mineral oxides or salts, carbon, mineral rubber, organic accelerators, vulcanized oils (factice), free sulfur, and sulfur combined as polyprene disulfide, $(C_{10}H_{16}S_2)_n$.

The acetone extraction of the finely cut sample was made to remove not only resins but also free

---

[1] Brandegee, 1917. Thesis submitted in partial fulfillment of the requirements for the S.B. degree at the Massachusetts Institute of Technology.

[1] Sackett and Seltzer, 1918. Thesis submitted in partial fulfillment of the requirements for the S.B. degree at the Massachusetts Institute of Technology.

sulfur and other acetone-soluble materials. The rubber residue was dissolved by refluxing with tetrachlorethane[1] for several hours, diluted to a definite volume with carbon tetrachloride, and allowed to settle, then an aliquot part was pipetted out for bromination as in Brandegee's method. The finely divided fillers were excluded from the aliquot part so taken by placing a wad of cotton in the tip of the pipette, and applying a gentle suction. By this means the troublesome centrifuging procedure for eliminating solid material was avoided.

During the course of the experimental work Sackett and Seltzer found that substitution could be reduced practically to zero by titrating the brominated sample in very dim daylight, thus eliminating a difficulty previously met by them in an unstable end-point during the second titration. When the per cent excess bromine above that necessary for addition was small (50 per cent) the results were low; 100 to 150 per cent excess bromine gave the best results.

COMBINED SULFUR PROCEDURE—The combined sulfur was found by evaporating to dryness in a porcelain casserole an aliquot part of the tetrachlorethane solution free from insoluble matter, and determining the sulfur by the method of Davies.[2] This consisted in adding 10 cc. of saturated arsenic acid solution, 10 cc. of fuming nitric acid, and 3 cc. of bromine water, and evaporating to a sirupy consistency. (If all the organic matter is not destroyed, more fuming nitric acid is added, and the mixture again evaporated to a sirupy consistency.) After the addition of a few crystals of potassium chlorate, the solution is evaporated to dryness, heated to boiling with 50 cc. of 10 per cent hydrochloric acid solution, filtered through paper, and diluted to 300 cc. with distilled water in a beaker. The sulfuric acid is precipitated as barium sulfate by the addition of barium chloride, and determined gravimetrically in the usual manner.

CALCULATION OF TOTAL RUBBER HYDROCARBON—The rubber hydrocarbon combined with the sulfur thus found is calculated by multiplying the percentage of sulfur by $\frac{C_{10}H_{16}}{S_2}$, or $\frac{136}{64} = 2.13$.

The total rubber hydrocarbon is calculated by adding the rubber hydrocarbon combined with the sulfur and the uncombined rubber hydrocarbon found from the bromine addition.

PROCEDURE FOR VULCANIZED RUBBER—Extract a weighed sample (approximately 1.5 to 2.0 g.) of vulcanized rubber with acetone for 8 hrs. in the standard extraction apparatus, evaporating the acetone to obtain the percentage of acetone-soluble material.[3] Aspirate CO_2 through the rubber to remove the traces of acetone, reflux 4 hrs. with approximately 100 cc. of tetrachlorethane, cool, and make up to mark in a 250 cc. calibrated flask with carbon tetrachloride. Remove a 25 cc. aliquot portion by applying gentle suction to a pipette containing a small piece of cotton in its tip. Place this sample in a glass-stoppered bottle of 250 to 500 cc. capacity, add from a burette a measured amount of bromine in carbon tetrachloride corresponding to at least 100 per cent excess bromine above that necessary for the addition reaction, insert the stopper tightly, and allow to stand for 3 hrs. in a dark closet. At the end of this time, darken the room, add 10 cc. of 10 per cent KI solution, shake, and titrate rapidly with 0.1 *N* standard sodium thiosulfate, using starch paste as an indicator. As soon as the first end-point has been noted, add 10 cc. of 1 per cent KIO_3 solution, and titrate rapidly to the second end-point with thiosulfate. The titration of a blank run under similar conditions gives the thiosulfate equivalent of the bromine added.

The method of calculation of the results is entirely similar to that used in the case of raw rubber, except that to get total rubber the rubber equivalent to combined sulfur is added to that determined by bromination.

RESULTS

TABLE I—RUBBER HYDROCARBON BY ADDITION

SAMPLE	INDIVIDUAL RUNS PER CENT				AVERAGE PER CENT
A	86.2	86.6	86.2	83.8	85.5
B	63.5	66.5	..	..	65.0
C	56.6	52.6	52.7	50.7	53.2
D	54.2	55.0	53.5	55.0	54.4
E	45.4	45.2	..	..	45.3
F	80.5	81.9	74.9	74.7	78.0

The final analyses are summarized in Table II.

TABLE II

Sample	Acetone Extract Per cent	Nature of Compounding Material	Combined Sulfur Per cent	Rubber Equivalent of Combined Sulfur Per cent	Uncombined Rubber (by Bromination) Per cent	Total Rubber (by Analysis) Per cent	Total Rubber Known Composition Per cent	Error in Total Rubber Hydrocarbon Per cent
A	3.46	None	3.4	7.2	85.5	92.7	93.7	—1.05
B	1.90	Litharge	3.1	6.6	65.0	71.6	75	—3.4
C	2.33	Zinc oxide	3.3	7.0	53.2	60.2	60	+0.2
D	2.33	Sublimed lead	1.6	3.5	54.4	57.9	60	—2.1
E	..	Zinc oxide and organic accelerator	1.1	2.4	45.3	47.7	48	—0.4
F	..	Mineral rubber and accelerator	2.16	4.6	78.0	82.6	80.5	+2.1

Table II compares the percentages of total rubber hydrocarbon as found by analysis with the known figures for rubber content supplied by the compounders of the samples. In no case did the analyst have any information as to the composition of the samples. The analytical figures average low, as they should do because of the resin and protein content of the raw rubber. The analytical results are, however, probably high for true rubber hydrocarbon, because any sulfur combining with resin, protein, or accelerator to give a product insoluble in acetone but soluble in tetrachlorethane is figured over to its equivalent of rubber, and, further, any sulfur substituting in rubber hydrocarbon itself will increase the results. These factors are probably negligible, except for sulfur combined with artificial accelerators. Any unsaturated organic material insoluble in acetone but dissolved by tetrachlorethane will also increase the analytical results. This is probably a cause of the high figures in the presence of mineral rubber. Few compounding materials are

---

[1] This solvent was purified in the same manner as the carbon tetrachloride.

[2] *Chemist-Analyst*, **15** (1915), 4.

[3] In case "factice" (vulcanized oil) is present, it should be removed by treatment in the usual manner by extraction with alcoholic potash. This treatment was unnecessary for the samples used.

sufficiently unsaturated, however, to be serious in this regard.

It is believed that this procedure is by far the simplest and most accurate direct estimation of the rubber content of vulcanized articles. It should prove especially useful in the evaluation of shoddies, because it shows the extent to which the unsaturation of the rubber has disappeared, due to previous vulcanizations.

Within the experimental error, the results prove that rubber hydrocarbon is unsaturated to an amount equivalent to four atoms of bromine for each $C_{10}H_{16}$, and further that "combined" sulfur reduces this unsaturation by two bromine atoms for each sulfur combined. These facts seem incompatible with any theory other than that the sulfur taken up by rubber on vulcanization is chemically combined.

SUMMARY

I—Working with a filtered carbon tetrachloride solution of acetone-extracted plantation pale crêpe, it has been shown by a volumetric method involving a double titration that the bromine consumption, corrected for the observed substitution, is a true measure of the actual amount of pure rubber hydrocarbon known to be present. Although the amount of substitution increases with the length of the bromination period, the addition corresponds quantitatively to the actual amount of pure rubber hydrocarbon present, when the bromination time is from 2 to 4 hrs.

II—Experimental data are given to show that the actual per cent of $(C_{10}H_{16})_n$ in vulcanized soft rubber can be determined by a volumetric bromination method herein described, involving a second titration to correct for the substitution which accompanies the particular analysis; by titrating in dim daylight, this substitution correction can be made very small.

III—Tetrachlorethane is a suitable "solvent" for the soft vulcanized rubber goods used.

IV—Experimental data shows that the so-called "combined sulfur" in soft vulcanized rubber is actually combined with the double bonds of the rubber hydrocarbon.

ACKNOWLEDGMENT

The writers desire to express their appreciation of the coöperation of the Chemical Department of the Goodyear Tire & Rubber Company, in preparing samples of known rubber content, and their indebtedness to Messrs. Brandegee, Sackett and Seltzer for developing the analytical procedures and making most of the analyses.

BIBLIOGRAPHY

Gladstone and Hibbert, "The Optical and Chemical Properties of Caoutchouc," *Trans. Chem. Soc.*, **53** (1888), 679.

Th. Budde, "The Valuation of Cold Vulcanized Rubber Ware by the Tetrabromide Method," *Chem. Abs.*, **3** (1909), 3013.

P. Schidrowitz, *Rubber*, London, **1911**.

C. O. Weber, "The Chemistry of India Rubber," London, **1909**.

Hinrichsen, Quensell and Kindscher, "The Chemistry of India Rubber. III—Addition Compounds of Hydrogen Halides and Halogens with Rubber," *Chem. Abs.*, **7** (1913), 2482.

Hinrichsen and Kindscher, "Direct Determination of Caoutchouc as Tetrabromide," *Chem. Abs.*, **7** (1913), 2861.

Caspari, "India Rubber Laboratory Practice," London, **1914**.

W. A Ducca, "Testin; Methods of Rubber Content in Raw and Vulcanized Rubbers," THIS JOURNAL, **4** (1911), 372.

Utz, "The Determination of Rubber as Tetrabromide," *Chem. Abs.*, **6** (1912), 2009.

D. Spence and J. C. Galletly, "The Determination of Caoutchouc as Tetrabromide," *Chem. Abs.*, **6** (1912), 302.

E Kirchhof, "Direct Determination of Rubber by Titration with Bromine," *Chem. Abs.* **7** (1913), 270.

G. Huebner, "Analyses and Analytical Methods for Hard Vulcanized Rubber," *Chem. Abs.*, **3** (1909), 1697.

G. Huebner, "Method for Determining Rubber in Hot Vulcanized, Soft Rubber Goods," *Chem. Abs.*, **3** (1909), 2388.

G. Huebner, "The Direct Determination of Rubber in Vulcanized Rubber Wares," *Chem. Abs.* **5** (1911), 3172.

Caspari, "Bromination of Vulcanized Rubber," *Chem. Abs.*, **6** (1912), 162.

W. Esch, "The Analysis of Vulcanized Rubber by the Bromine Method," *Chem. Abs.*, **6** (1912), 806.

T. Budde, "A New Method of Determining the Combined Sulfur in Vulcanized Rubber," *Chem. Abs.*, **3** (1909), 1936.

W. Vaubel, "Contribution to the Bromide Method for the Determination of Rubber," *Chem. Abs.*, **7** (1913), 275.

W. Schmitz, "The Direct Volumetric Estimation of Rubber by Bromine, *Chem. Abs.*, **7** (1913), 4084.

G. Fendler and O. Kuhn, "New Researches on Rubber," *Chem. Abs.*, **2** (1908), 593.

G. Huebner, "Concerning Hard Vulcanized Rubber," *Chem. Abs.*, **4** (1910), 1550.

B. D. Porritt, "The Chemistry of Rubber," *Chemical Monograph*, No. 111, New York.

(Many articles discussing methods, including minor modifications of the same, proposed by the above writers have been omitted from this list.)

METHOD FOR THE DETERMINATION OF THIOCYANATES IN AMMONIACAL LIQUOR AND WASTE LIQUOR FROM AMMONIA STILLS IN THE BY-PRODUCT COKING INDUSTRY

By Joseph A. Shaw

KOPPERS COMPANY LABORATORIES, PITTSBURGH, PA.

Received January 8, 1920

To 500 cc. of ammoniacal liquor are added 3 to 5 g. of ferrous sulfate in water solution and about 100 cc. of a 10 per cent sodium hydroxide solution. This is stirred thoroughly and allowed to stand overnight. It is then filtered with gentle suction and washed with cold water containing about 30 g. of sodium hydroxide per liter. The long standing in alkaline solution is necessary in order that the insoluble organic matter may be removed thoroughly from the solution. If this is not done it will continue to settle out from time to time and is liable to cause the analyst to be doubtful of the sharpness of some of his reactions.[1]

By this procedure organic and inorganic material insoluble in alkalies and the sulfides have been removed from the sample and the cyanides changed to ferrocyanides. The alkaline solution is heated to 60° C., made slightly acid with dilute sulfuric acid, and the ferrocyanides precipitated with a slight excess of a 10 per cent ferric chloride solution. After standing a short time the Prussian blue, together with a considerable sludge insoluble in acid solution, is filtered off on a filter similar to the one used to filter the sulfide sludge. The residue may be used to determine total cyanogen after having been washed with a 5 per cent solution of sulfuric acid containing 5 per cent by weight of sodium sulfate.

The filtrate is now made to a definite volume and

---

[1] It is necessary to use a special filter for the removal of the sludge from the solution mentioned above. This filter is made by placing a perforated porcelain plate in a funnel attached to a filter flask and covering it with a piece of oversized filter paper. When this is drawn into place by the suction a thin layer of asbestos pulp is poured on the filter and another piece of filter paper cut considerably under size is placed on top. After a little washing and adjustment the filter is ready to use. Or if desired and available a small Büchner funnel may be used instead of the funnel and porcelain plate employed above.

one-tenth aliquot parts taken. These are heated to 70° C., about 15 cc. of 1 : 4 sulfuric acid added, followed by a decided excess of sodium acid sulfite solution, and the thiocyanate precipitated with a hot 10 per cent solution of copper sulfate. After standing 10 min. the CuCNS is filtered off through a Gooch crucible, the bottom of which is covered with a circle of filter paper, followed by a layer of paper pulp which is in turn partly covered by a small piece of filter paper. Too long standing previous to filtering is to be avoided as it increases the tendency of the CuCNS to pass through the filter as a colloid. The impure CuCNS is washed with hot water of about the same temperature as the original solution which was filtered. Since a little CuCNS sometimes passes through the filter in the colloidal form, the filtrate, if clear, should be removed and discarded as soon as the last of the liquid has been poured through the filter. If a little of the precipitate now passes through the filter upon washing, a quantity of paper pulp may be added to it and the wash water refiltered through an ordinary paper filter, washed with hot water, and combined with the major portion of the CuCNS precipitate.

The residue, consisting chiefly of paper pulp and CuCNS, is treated with 25 cc. of *N* sodium hydroxide, a little bromine water is added to oxidize the copper, and after a few minutes the paper pulp is filtered off and thoroughly washed with hot water. A little more bromine water is added to the alkaline solution and it is digested at the boiling point for one hour. It is made slightly acid with hydrochloric acid, the bromine boiled off, and the solution taken to dryness. The dry residue is taken up with hot water containing a little HCl and filtered from silica. The sulfur present originally in the CuCNS is the only sulfur now left in the solution and it is entirely in the form of sulfate. This may now be precipitated with barium chloride and determined in the usual manner and the thiocyanate calculated.

For the purpose of checking the method, the thiocyanate content of the liquor was determined by the above method and also the thiocyanate content of the liquor to which a known amount of potassium thiocyanate had been added. The results indicated that thiocyanate in ammoniacal liquor can be determined by this method to within 0.01 g. per liter.

Aside from the exercise of a little technique in filtering, the analysis proceeds quite nicely.

VOLUMETRIC METHOD FOR THE DETERMINATION OF DIASTATIC CAPACITY

By J. T. Flohil

Cereal Products Company, Manitowoc, Wisconsin

Received January 16, 1920

A 5 per cent malt solution is prepared according to Lintner.[1] One cc. of the extract is introduced into 100 cc. of a 2 per cent soluble starch solution in a 200 cc. flask, left to act for exactly one hour at 20° C., then treated with 10 cc. of 0.1 *N* sodium hydroxide to stop the diastatic action, and diluted with water to the 200 cc. mark.

Twenty-five cc. of this solution are transferred to a 300 cc. Erlenmeyer flask, exactly 10 cc. each of Fehling's Solutions I and II added, and the volume brought up to 50 cc. with 5 cc. water. The mixture is boiled slowly for 2 min., and immediately cooled under the cold water faucet, but not much under 25°. Ten cc. of a 30 per cent potassium iodide solution, or 3 g. potassium iodide and 10 cc. of a 25 per cent sulfuric acid solution, are added successively and the liberated iodine titrated with a 0.1 *N* thiosulfate solution to a creamy yellow color, the soluble starch present serving as an indicator.

CALCULATION

Let p cc. be the amount of thiosulfate used in this titration, n cc. the amount of thiosulfate used in a blank test (25 cc., 5 cc. water, 20 cc. Fehling's solution); then $n - p = m$ is the amount of thiosulfate equivalent to the copper sulfate reduced by the maltose sugar.

According to Lintner, if 1 cc. of a 5 per cent malt infusion in 100 cc. of 2 per cent starch solution produces just enough maltose to reduce 50 cc. of Fehling's solution its strength is 100°. This solution diluted to 200 cc. of 1 per cent starch solution will also reduce 50 cc. of Fehling's solution, and 25 cc. will reduce 6.25 cc. of Fehling's solution. We here assume that the quantity of copper sulfate reduced is proportional to the amount of maltose present, without making an appreciable error.

If 20 cc. of the Fehling's solution is equivalent to n cc. 0.1 *N* thiosulfate, then 1 cc. is equivalent to $n/20$ cc. thiosulfate.

If 25 cc. of the solution mentioned in the beginning of this article reduce Fehling's solution equivalent to $6.25\ n/20$ cc. of thiosulfate, the Lintner value is 100°.

If 25 cc. of the solution reduce Fehling's solution equivalent to m cc. of thiosulfate, its strength is

$$\frac{m}{\frac{6.25\,n}{20}} \times 100 = 320\,\frac{m}{n} \text{ degrees Lintner.}$$

EXAMPLE—In a blank test the amount of iodine set free by the copper sulfate was equivalent to 27.0 cc. 0.1 *N* thiosulfate, or $n = 27.0$.

In the actual test the amount of iodine set free by the copper sulfate was equivalent to 15.0 cc. 0.1 *N* thiosulfate; therefore $m = 27.0 - 15.0 = 12.0$ and the diastatic capacity is 320 12/27 or 142° Lintner.

THE UNIFORM COMPOSITION OF COMMERCIAL "STEARIC" ACIDS, AND ITS EXPLANATION

By Ben H. Nicolet

Mellon Institute of Industrial Research, Pittsburgh, Pa.

Received October 27, 1919

Sometime ago the writer had occasion to make some comparative tests on a number of samples of commercial stearic acids chosen at random. The titers were surprisingly uniform throughout the series and when these were corrected for the small and varying iodine numbers which the different samples showed, the agreement became startling. The maximum variation in the corrected titers for the six samples, coming from five entirely different sources, was less than 0.5°.

---

[1] *Z. prakt. Chem.*, **1386**, 386.

Such a finding appeared to demand an explanation, and one is here offered, based on the known series of solid solutions formed in mixtures of stearic and palmitic acids.

The results of interest in the present connection are given in Table I. The samples examined may be described as follows:

(1) Domestic, source unknown
(2) "Best," Celina Stearic Acid Company
(3) "Standard," Emery Candle Company
(4) "Double pressed," Rub-No-More Company
(5) "Triple pressed," Rub-No-More Company
(6) "Finest imported," Dutch

Technical stearic acid is produced by successive pressings of fatty acid mixtures which originally have a considerable content of unsaturated fatty acids. At each pressing, a lower melting fraction is removed, and after two or three pressings little of the unsaturated acids remain, as shown by the iodine numbers found.

TABLE I—EXAMINATION OF COMMERCIAL "STEARIC" ACIDS

Sample number	1	2	3	4	5	6
Iodine number	1.3	1.6	8.0	4.0	1.9	2.4
Oleic acid (calc. per cent)	1.4	1.8	8.8	4.4	2.1	2.6
Titer[1]	55.2°	55.3°	53.9°	54.3°	55.0°	54.8°
Titer for zero I_2 number (calc.)	55.48°	55.66°	55.66°	55.18°	55.42°	55.32°

[1] The titers calculated for zero iodine number were obtained by adding to the titers found 0.2° for each per cent oleic acid present. This is an approximately correct allowance for small amounts of oleic acid in any stearic-palmitic acid mixture.

The constancy of the titers given in the last line of the table indicates that the ratio of stearic to palmitic acid approaches a definite value as the pressing is continued.

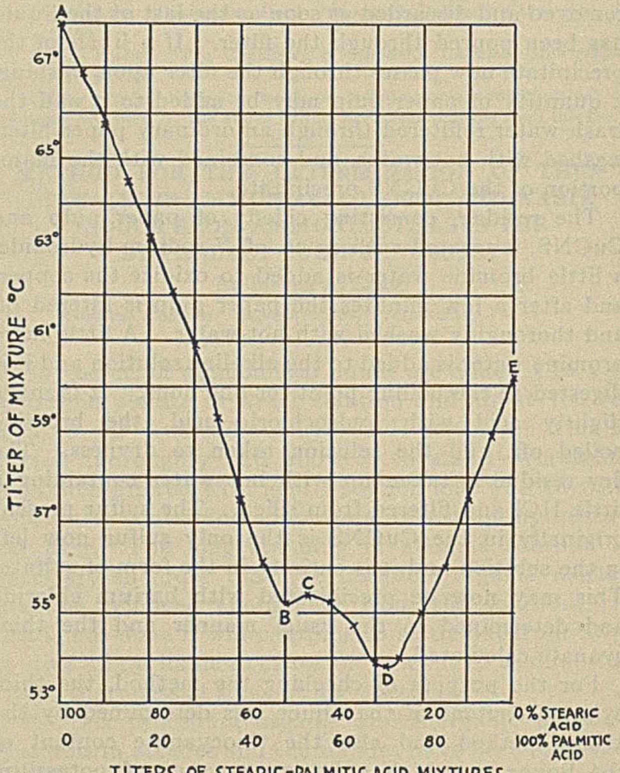
To explain this it is necessary to refer to the curve showing the solidifying points of the system stearic acid–palmitic acid. De Visser[1] and Carlinfanti and Levi-Malvano[2] have drawn such curves and pointed out the relations which they indicate as to the formation of solid solutions. Since titer determinations as ordinarily carried out vary somewhat in principle as well as in practice from the carefully determined solidification points of these authors, a similar curve of titers of mixtures of stearic and palmitic acids is given in the accompanying curve. The values from which this curve is constructed were determined for a different purpose and will be given elsewhere.

From A, the titer of pure stearic acid, the curve falls continuously to B, then with further addition of palmitic acid rises slightly to a maximum at C and then falls to D. From D the curve again rises normally to E, the titer of pure palmitic acid. The curve is thus divided into sections in which the effect of fractional crystallization, either with or without a solvent, will be very different. The pressing to which the acids are subjected is equivalent to a fractional crystallization.

The general laws describing the behavior of solid solutions indicate that a mixture whose composition was represented by any point between B and A would eventually yield pure stearic acid. Similarly, pure palmitic acid would be the end-product from a mixture lying between D and E. On the other hand, a mixture originally represented by a point on the section BD would approach the composition represented by the maximum at C and thereafter show no further separation.

The point C represents a palmitic acid content of 55 per cent, and a titer of 55.4°. This appears to be a rather close description of the "stearic" acids examined. Furthermore, their titers are depressed by successive small additions of palmitic acid. A little stearic acid added lowers the titer, and further additions raise it rather rapidly. The acids have all therefore approximately the composition C, which apparently is represented on the curve with a titer a few tenths of a degree too low.

All the samples examined are thus seen to contain palmitic acid to the extent of 52.5 to 55 per cent of their saturated acids. If the explanation here put forward be accepted, it follows that in the material originally subjected to pressing, the *saturated* acids contained between 50 and 72 per cent palmitic acid.



TITERS OF STEARIC-PALMITIC ACID MIXTURES

Very little is actually known as to the ratio of palmitic to stearic acid in fats and oils. Most of the information on the subject in the literature comes from the work of Hehner and Mitchell[1] on the determination of stearic acid. They report a commercial stearic acid examined by them as having iodine number 2.3, melting point (not titer) 56.2°, and stearic acid 48.7 per cent (the mean of five determinations varying from 47.0 to 50.6 per cent). In view of the uncertainty of their method, these results do not contradict the conclusions here reached.

SUMMARY

Attention is called to the remarkable constancy of the ratio of palmitic to stearic acids in a number of technical "stearic" acids.

---

[1] *Rec. trav. chim.*, **17** (1898), 182, 316.

[2] *Gazz. chim. ital.*, [II] **39** (1909), 368.

[1] *Analyst*, **21** (1896), 316.

An explanation of this constancy is offered based on the solid solutions formed between these two acids. This requires that "stearic" acids showing this effect should have as their source acid mixtures, the saturated acids of which contain between 53 and 72 per cent of palmitic acid.

As the explanation given must stand or fall with the necessity of the fulfillment of this condition, and as the writer is not sufficiently familiar with the manufacture of "stearic" acid to know whether in general it is fulfilled, discussion on this point is particularly invited.

LABORATORY AND PLANT

PHOTOBIBLIOGRAPHIC RESEARCH METHODS—THE COMPILATION OF TECHNICAL DATA WITH THE AID OF PHOTOGRAPHY

By Arthur Worischek

Patent Department, General Electric Co., Washington, D. C.

Received May 11, 1920

The methods employed by professional searchers, compilers and "experts in patent causes," although not yet standardized, have nevertheless been very ably described.[1]

The weakest point in any system of searching and collating technical data is the accurate transcription of tabulated matter and the reproduction of illustrations when these occur in the text. In the author's opinion, it should be possible to copy articles, tables, complicated diagrams and even entire chapters of textbooks by means of some cheap and simple process and without causing annoyance to fellow readers or damage to the books consulted. If a journey to a distant library is necessitated it should be possible to so plan the trip that copies of all the data needed in a given investigation may be brought home in a convenient form. A research worker would then be able to compile his own textbook on any given subject and arrange the subject matter to suit his own particular convenience and needs.

The author having had occasion to undertake extensive research searching during the past few months was struck by the lack of any simple method for copying articles and diagrams. Tedious scribbling appears to be the method exclusively employed, with occasional laborious and time-consuming tracing. A search through the literature revealed a number of attempts along this line, all based upon some adaptation of photography.[2]

Microphotographic copying of printed matter was employed for the transmission of intelligence during the siege of Paris in 1870.[3] Despatches were printed on a large sheet of paper which was then microphotographically copied onto a film of collodion. According to Tissandier[4] each page contained some 5,000 letters, equivalent to 300 despatches, and 16 of these pages were copied onto a collodion film 2 in. long and 1 in. wide, weighing less than one grain. These films were transmitted by carrier pigeons and upon arrival at headquarters were placed in a projecting lantern and thrown upon a screen, while a number of copyists or scribes transcribed the despatches.

Fessenden[1] described a method for photographically compiling his own data collection. His method consisted in copying pages of books onto standard sized plates from which standard sized prints could be made, suitable index numbers being marked upon the film before printing. Other workers have also proposed similar schemes and a method was described in 1909[2] in which the book to be copied was placed upon the floor and copied by means of a camera equipped with a reversing mirror and using bromide paper instead of plates. In the "reflection by contact" or "catatype" method[3] a sheet of sensitized paper is placed with its sensitive side in contact with the print to be copied, the light being allowed to pass through the paper. The method does not, however, appear to have come into practical use.

J. L. Mauch[4] has obtained a patent for a "photogenic copying device." He has combined "means for applying a sensitized strip to a surface to be copied with means for projecting light through said strip upon said surface and means to obtain a uniform exposure of said strip." The apparatus is to be held in the hand and "rolled" over the page of a book, etc., the unexposed sensitive paper being drawn from one spool and the exposed paper wound up on another spool. This, therefore, appears to be a mechanically controlled application of the "reflection by contact" method. A patent by H. H. Curtiss[5] relates to a similar device.

Various commercial methods have been proposed for copying directly upon bromide paper without the intermediary of a negative.

A number of years ago there was introduced a commercial bromide paper copying machine known as the "Record Camera,"[6] which was equipped with a

---

[1] A. B. Eason, "Sources of Technical Information," *Electrician* (*Lon.*), **82** (1919), 326–7; E. D. Greenman, "Technical Literature and How to Use It," *Special Libraries*, **9** (1918), 89; H. Hibbert, "The Art of Searching Chemical Literature," *Chem. & Met. Eng.*, **20** (1919), 578; W. F. Jacob, "The Technical Library and Its Use," *Polytech. Eng.*, **16** (1916), 36; E. N. Jessop, "German Patent Bibliography," THIS JOURNAL, **8** (1916), 1053.

[2] For an extensive bibliography on the subject of "Photography in Library Work," see G. A. Evers, *Maandblad voor Bibliotheekwezen* (*'sGravenhage*), **1** (1913), 10, 225.

[3] Dagron, "The Preparation of Microphotographic Despatches on Film," *J. Phot. Soc. London*, **15** (1871), 102; *Brit. J. Phot.*, **18** (1871), 616.

[4] "History and Handbook of Photography," London. Translated by J. Thomson. Low, Marston, Low and Searle, 1876. "Microscopic Despatches during the Siege of Paris," Chap. 6, pp. 234–48.

[1] "Use of Photography in Data Collections," *Elec. World*, **28** (1896), 222.

[2] "Photographing Rare Books and Manuscripts in Libraries," *Bull. de la Soc. Franc. de Phot.*, [2] **25** (1909), 396.

[3] E. E. Fournier d'Albe, "On Photography by Reflection under Contact," *Sci. Proc. Roy. Dublin Soc.*, (New Series) **12** (1909), 97; G. de Fontenay, "Cataphotography; Reproduction of Documents by Reflection," *Compt. rend.*, **152** (1911), 1055, 1298.

[4] "Photogenic Copying Apparatus," U. S. Patent 1,251,076 (Dec. 25, 1917); *Offi. Gaz. U. S. Pat. Office*, **245** (1917), 894.

[5] "Method and Apparatus for Photography," U. S. Patent 1,007,120 (May 19, 1914); *Offi. Gaz. U. S. Pat. Office*, **202** (1914), 745.

[6] "Making Photographic Copies without a Negative," *Am. Machinist*, [1] **31** (1908), 781; Eastman's "Record Camera."

reversing prism and a magazine containing a roll of bromide paper. Upon development a reversed negative (white on black) was obtained. This machine was followed by the well-known "photostat"[1] which embodies the basic principles of the earlier machine but is equipped with means for immediately developing and fixing the print. The cost of a photostat print ranges from 25 to 35 cents per page. Positives (black on white) are secured by copying a negative print, and enlargements and reductions within the capacity of the machine are readily made. S. C. Williams[2] has discussed the cost of photostat work from a scientific viewpoint, and gives formulas for computing the various elements of expense involved. A comparison of the relative costs of brown prints, blue prints, and photostat copies (on a square foot basis) has been made by the Ordnance Department, U. S. Army.[3] The "rectigraph"[4] and the Powers "cameragraphs" (simplex and duplex)[5] are similar machines, the last mentioned being designed to photograph on both sides of a sheet.

In addition to methods intended purely for the copying of printed matter there has been developed a portable camera for use in reading electric and gas meter indices. This is termed the "factograph" camera[6] and is arranged to carry its own source of illumination in the form of several small battery-operated lamps which are lighted by means of a lever which latter also automatically opens the shutter for any desired length of time. A modification of this camera is termed the "finger-print camera" and is used for keeping uniform-sized finger-print records.

EXPERIMENTAL

The author modified Dagron's microphotographic process by substituting motion picture film for the wet collodion pellicle and an *f* 3.5 anastigmatic lens for the slow doublet of 1870. Textbook and periodical pages were photographed, the process used being essentially the same as that used by the cinema companies in copying titles, telegrams, and letters for incorporation into film plays. It was found possible to enlarge the negative film onto bromide paper up to a certain limit in size, *e. g.*, about 5 by 7 in., and although the image was slightly blurred the outlines of the diagrams could nevertheless be distinguished and could be strengthened by inking in. When the film (both negative and positive) was projected onto a screen the print and diagrams were distinctly readable. Oswald,[7] however, has little faith in the use of cinema film for purposes of direct enlargement.

Reasoning from Simpson's and Dagron's postulates, cited above, there appears to be no reason why entire works like Beilstein could not be photographed onto a continuous film[1] (larger in size than ordinary cinema film if necessary)[2] and projected with a suitable apparatus.

A second series of experiments involved the construction of a portable machine of the "photostat" or "rectigraph" type.

This was accomplished, as shown in Fig. 1, by using an Eastman double-extension bellows, revolving back, 3.25 in. by 4.25 in. Auto-Graflex camera. The advantage of the reflex camera resides in the ease with which focusing may be effected, although any ground-glass focus camera may, of course, be used. The lens was equipped with a photoengraver's reversing prism and a simple type of studio shutter, although the camera's own focal plane shutter could also be used. Eastman "Insurance Bromide" paper, Grade B, was used instead of film, being wound on the regular film spools. An exposure (ordinary daylight) of 15 to 20 sec. at *f* 4.5 to *f* 6.3 produced a perfect miniature "photostat" print. Exposure under one 16 candle-power incandescent lamp, representing ordinary New York library lighting conditions, required about 10 sec. longer. Experiments were made using a 5 by 7 view camera, equally good results being obtained, the detail and sharpness of the prints being remarkable. A whole page of Beilstein when reduced to 3.25 by 4.25 in. in size was as distinctly readable as the original, and when such a small print was recopied and enlarged several times by a regular "photostat" machine its size was restored to that of the original page without any material sacrifice in sharpness of definition.

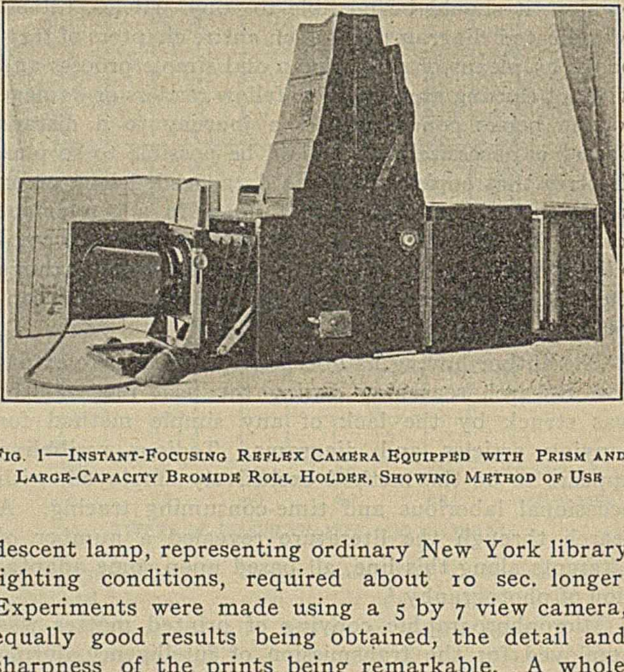


FIG. 1—INSTANT-FOCUSING REFLEX CAMERA EQUIPPED WITH PRISM AND LARGE-CAPACITY BROMIDE ROLL HOLDER, SHOWING METHOD OF USE

Such spools of bromide paper when tightly wound so as to fill completely the regular film spools in the case of the camera mentioned above represent 15 to

---

[1] L. D. Burlingame, "Photostat and Its Uses," *Machinery*, **21** (1915), 951. J. X. Cohen, "Methods and Cost of Reproducing and Reducing Engineering Drawings," *Eng. & Contr.*, **46** (1916), 198; "New Reproducing Machine of the Commercial Camera Co.," *Iron Trade Rev.*, **56** (1915), 1106.

[2] "Photostatic Reproduction Cost," *Am. Machinist*, **44** (1916), 487.

[3] U. S. Army, Ordnance Dept., "Cost of Photostats, van Dykes, Blue Prints, Compared," *Eng. News-Record*, **82** (1919), 1065.

[4] *Lumière élec.*, **25** (1914), 217.

[5] *Eng. Rec.*, **72** (1915), 32; *Iron Age*, **95** (1915), 1122.

[6] "Camera for Reading Meters," *Am. Gas Lt. J.*, **103** (1915), 141; *Machinery*, **22** (1915), 75. Folmer, U. S. Patents 1,139,022, 1,139,023 (May 11, 1915); *Offi. Gaz. U. S. Pat. Office*, **214** (1915), 497.

[7] "The Movie and the Still Picture," *Literary Digest*, July 5, 1919.

[1] Otlet, in 1918, exhibited examples of such work before various American library gatherings.

[2] "Projecting Larger Pictures with the Standard Motion Picture Film," *Phot. J. Am.*, **56** (1919), 180. Standard size of film retained. Film is moved horizontally and picture is as high as the ordinary image is wide and twice as wide as the regular image is high. The greater size of the film permits greater enlargement.

20 exposures, and a dozen or two of such spools are sufficient for copying a book of respectable size.

The development of a roll of 20 exposures is of course an extremely simple matter, requiring but a few minutes, while the developed paper may be accorded much rougher handling than would be withstood by film. It may, for example, be cut and trimmed while still wet, pasted onto sheets and reports while still damp and portions of illustrations may be inked in with waterproof India ink, the rest of the image being bleached out by some suitable means[1] and the "line cuts" so obtained used for reproduction. After the first print is made it may be recopied to produce black-on-white copies or "positives."

It should, of course, be noted that a suitable "head" or starting piece and "tail" or ending piece of non-actinic or "post office" paper must be pasted onto each end of the sensitive strip for the purpose of starting and ending the spool without spoiling the first and last exposures made. It was discovered, however, that simple blackening of both sides of the bromide paper with India ink for a distance of about one foot from each end of the roll served the same purpose. Inasmuch as no black "duplex" backing paper is used with the bromide paper as in the case of film, the number of the exposure must be indicated in some manner. This is readily accomplished by marking the number of the exposure on the back of the paper with the aid of a cardboard template; a rachet-controlled winding jig is also a great aid in rolling up such homemade spools. The numbering and marking of 10 to 100 ft. of bromide paper proved to be somewhat tedious and a solution of the difficulty was found in the use of the magazine film holder corresponding to the Bannister patent.[1]

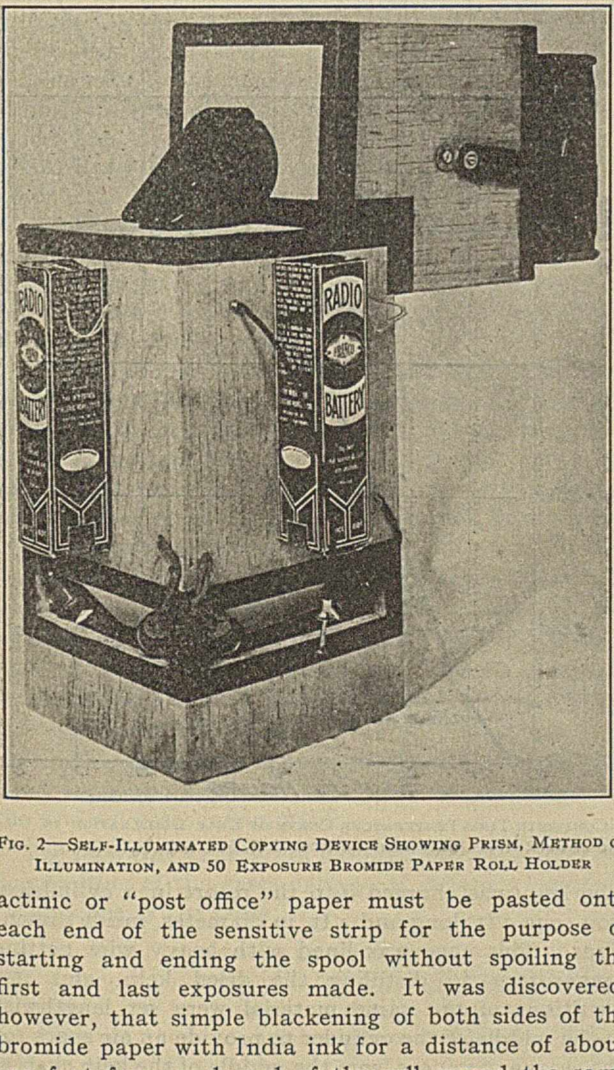


FIG. 2—SELF-ILLUMINATED COPYING DEVICE SHOWING PRISM, METHOD OF ILLUMINATION, AND 50 EXPOSURE BROMIDE PAPER ROLL HOLDER

This type of holder was intended for the use of unbacked, un-numbered film, contained on a wooden spool. The film was caused to pass over a large measuring roller carrying two needle points which punched the film at regular intervals corresponding to the size of the picture desired, and the film was prevented from progressing until a suitable release lever had been depressed, or else some external indicating device signaled readiness for a fresh exposure.

Although the apparatus described above is an extremely convenient device for copying books in a distant reference library, it nevertheless appeared possible to render the process of copying more automatic in operation and less dependent upon the operator's judgment regarding sharpness of focus and conditions of illumination. The author, therefore, utilized the principle of the self-illuminated meter reading and finger-print camera mentioned above and built the experimental apparatus depicted in Fig. 2. It was of course necessary to employ the reversing prism used with the reflex camera previously described. This feature makes the apparatus rather awkward and cumbersome, one portion being at right angles to the other, and therefore slightly "top-heavy," but if such a device were to be produced on a commercial scale, a more convenient form, possibly involving the use of a suitable system of reversing mirrors, would undoubtedly be evolved. Pressing the antinous release opens the shutter leading to the lens and prism and simultaneously lights the battery-operated lamps which illuminate the object to be copied. A suitable clockwork or other timing device may, if necessary, be used to actuate the shutter and light-controlling levers so as to insure uniform length of exposure. We would thus have the following practically ideal operating conditions: fixed focus lens, constant intensity of light, uniform time of exposure, and fixed size of object to be copied with a fixed ratio of original to the copy. The apparatus may be built in various forms and sizes, *i. e.*, "page-copier," "diagram-copier," "equation-copier," etc., depending upon the size and use to which the device is to be put. The device might also be so designed as to operate somewhat like a numbering stamp, a simple downward push sufficing to open the shutter and illuminate the object for the required length of time, the mechanism being arranged to automatically supply and wind up the sensitive paper used. H. S. Leach[2] has suggested the advisability of sending expeditions of research workers abroad equipped with photostat machines to copy and bring back material for scholarly study, and the present author would modify the suggestion to the effect that a more portable machine of this type

---

[1] E. Senior, "Converting a Photograph into a Line Drawing," *Knowledge*, **11** (1914), 71.

[1] Bannister, "Roll Film Holder," U. S. Patent 316,933 (May 5, 1885); *Offi. Gaz. U. S. Pat. Office*, [1] **31** (1885), 523.

[2] "Photostat as an Aid to Research," *Sci. Am.*, **122** (1920), 276, 291.

would be a valuable aid to anybody having occasion to make use of a reference library, and the above-proposed small portable machines carried by investigators and translators may very well be used as "feeders" for the large machine in the home plant. The cost of the bromide paper is about $1.75 per 100 ft., and a 100-ft. roll of paper measures only 4.375 in. wide by 4 in. diameter and has a capacity of about 300 exposures. The entire apparatus weighs but a few pounds and does not occupy more than the table space allotted to any one reader or the space occupied by a large dictionary or several volumes of some periodical, and no reasonable objection to its use can be interposed by any librarian, the argument, if any, being on the side of the reader, inasmuch as the book to be copied may be released for issue to another reader in an incredibly short time after "pushing the button."

CONCLUSION

There is apparently a need for a portable and cheaply operated photographic device for copying illustrations and articles on file in reference libraries—for the purpose of compiling the technical data needed in an investigation. The experiments described indicate that the use and manufacture of such a device are both within the range of commercial possibility. It is believed that a device combining the operating principles of the existing bromide paper copying machines and the self-illuminated meter-reading cameras would prove to be a valuable aid in data compilation, and simple enough in operation for use by non-technical clerical assistants. There appears to be no reason why it should not be possible to simply "push the button—and read the book at home."

POTASH FROM KELP. II—THE EXPERIMENTAL DISTILLATION OF KELP AT LOW TEMPERATURES[1]

By G. C. Spencer

U. S. Kelp Potash Plant, Summerland, California

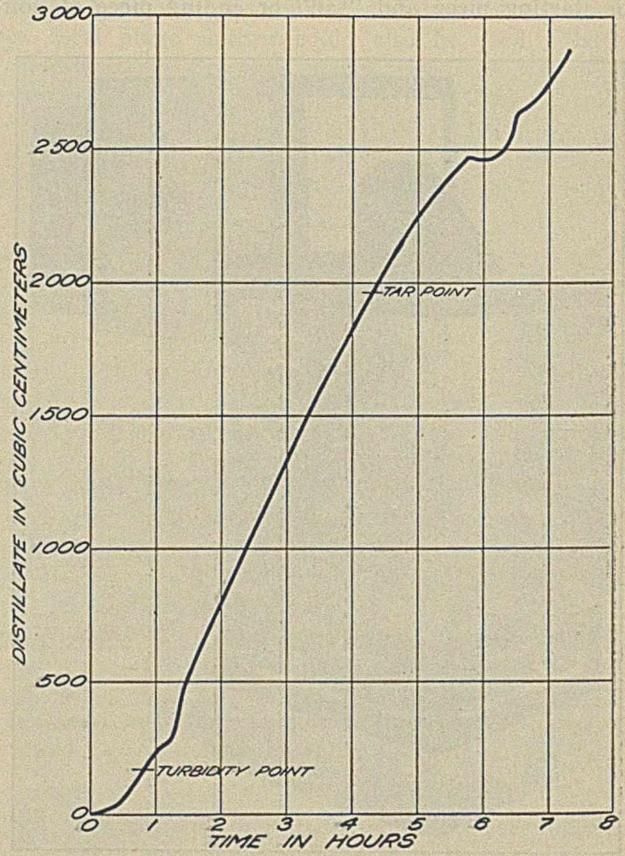
Received March 9, 1920

A study of the experimental, destructive distillation of dried kelp was undertaken to determine whether or not under carefully controlled conditions any relationship could be established between the temperature at which distillation was taking place and the nature of distillation products. The work was done in the Forest Products Laboratory of the U. S. Department of Agriculture, Madison, Wis., in order that the apparatus and technique developed there in the destructive distillation of wood might be available for the research in hand.[2]

Kelp was distilled in an oil-jacketed retort which was built for experimental wood distillation, which has been fully described and illustrated by L. F. Hawley and R. C. Palmer.[3] While this retort was adapted for wood distillation, experience showed that it was not suitable for kelp. However, an interesting collection of data was obtained which is summarized in the paragraphs which follow.

Reference to the specimen log of a distillation run which is here reproduced, as well as a glance at the composite time-distillate curve, will show how the high moisture content of the dried kelp prevents a rapid rise of temperature when the kelp starts to distil. This gradual expulsion of water vapor with its accompanying organic matter undoubtedly tells a story of the volatile kelp constituents that the more rapid heating in a much hotter retort would fail to do.

The retort was preheated by lighting the gas flames of the retort the evening before the run was to be made.



Composite Time-Temperature Curve of Kelp Distillation in Oil-Jacketed Retort Representing Runs 2, 3, 5, 6, and 7

The kelp was charged into the retort in a cylindrical iron cage 3 ft. long and 1 ft. in diameter, inside dimensions. This cage was lined with heavy wire netting preparatory to distilling the dried kelp, and two partitions of the same material were set lengthwise in the middle of the cylinder to provide an air space for the escape of vapors from the middle of the kelp charge.

Sixteen distillations of kelp (average weight of charge 30.2 lbs.) were made in this manner. The first runnings of distillate were always clear and of a bright green color. This condition continued with only a slight increase in density until a temperature, usually above 150° C., was reached, when a turbidity became noticeable in the liquid, which also changed from green to amber. This turbidity indicated a change

---

[1] The first paper of this series, entitled "The Experimental Plant of the U. S. Department of Agriculture," by J. W. Turrentine and Paul S. Shoaff, appeared in This Journal, 11 (1919), 864.

[2] Acknowledgments are due to Director Winslow, to Drs. Hawley, Mahood, and others at the Forest Products Laboratory for assistance and courtesies extended, and to Prof. O. L. Kowalke of the University of Wisconsin for the use of laboratory space and apparatus for supplementary work.

[3] "Yields from the Destructive Distillation of Certain Hardwoods," U. S. Dept. of Agr., *Bulletin* **129**.

in the nature of the distillate and the first distillation cut was accordingly made at this point. As the distillation progressed the turbidity increased until a light brown flocculent precipitate separated, which gradually settled out.

Experience showed that the next cut should logically be made at the temperature where the tar commenced to come over, but for convenience two and sometimes three fractions were taken before this point was reached. The density of the liquids increased from approximately 1.01 to 1.065, and the amounts of dissolved substances also increased as long as the aqueous distillate continued to flow. It can be seen that the liquor driven off up to the point indicated as turbidity is mostly water. The amount of dissolved material in this fraction is necessarily low, and in all probability this turbidity point represents the first stage of the cellular decomposition. There was at no time any evidence of an exothermic reaction.

Reference to the time-distillate curve will show that, from the point where the first turbidity appears to that where the tar begins to flow, the distillation rate is pictured by a nearly straight line. Above the "tar point" the curve begins to bend and the volume of distillate decreases. The nearly straight line may indicate the passing over of some substance of nearly constant composition, and this belief is substantiated by the isolation of the glycerol-like liquid which will be described in another paper.[1]

The tarry substances were emulsified with an approximately equal quantity of the aqueous distillate and consequently always floated on the watery layer. A sharp separation was impossible, however, since the tar showed a strong tendency to adhere to the glass of the cylinder or of the separatory funnel and to conceal the actual amounts of water and tar.

When kelp is distilled at high temperatures the liberated gas is always combustible. This gas has never been analyzed, but its calorific power has been determined and found to be sufficient for use as a fuel.[2] Such gas, if properly applied, would go far toward reducing fuel costs in a kelp plant.

Samples of gas from the low temperature distillations were analyzed and tested but in no case were the evolved gases inflammable.

The following analyses of gas fractions show that the composition of the gases evolved at different temperatures is nearly the same:

ANALYSES OF GAS FRACTIONS

	131°–135° Per cent	163°–167° Per cent	226°–229° Per cent	271°–275° Per cent
CO_2	80.5	85.5	85.0	82.8
O_2	0.0	0.0	0.0	0.6
CO	11.3	10.3	11.4	10.2

The residual gases were in each case mixed with an excess of air in an explosion pipette but in no case was there a contraction of volume.

The last distillation of kelp that was made in the oil-jacketed retort was modified by mixing 24.1 lbs. of kelp with 5 lbs. of quicklime and carrying out the distillation like those preceding. The aqueous distillate differed from those of the former runs by being alkaline, although at no stage was there any odor of ammonia.

Two gas fractions were taken at 158° to 173° and 255° to 260°, respectively. They differed in composition from those described above in containing less carbon dioxide, which was kept back by the lime, and more carbon monoxide, which would naturally be higher in percentage under such circumstances. No advantage was apparent from mixing lime with the kelp charge.

IGNITION OF CHAR 15 IN A CLOSED RETORT

Inasmuch as none of the charcoal produced in the oil-jacketed retort had been heated sufficiently to remove all of its volatile matter, a portion of the charcoal from Run 15 was heated in an iron retort to as high a temperature as could be attained by a flat burner and a Meker burner together. The retort was set in a holder that prevented air currents from interfering with the flames, but even with this advantage the retort did not appear to be heated to redness. Three and three-tenths pounds (1.5 kg.) of No. 15 char were heated for an hour and a quarter. The retort was not entirely tight and a little of the gas was lost by leakage. The first drop of distillate appeared when the off-take temperature was 98° C. At the same time white vapors began to come over through the condenser. After 40 min. the temperature of the distillate had risen to 101° C. At this temperature the aqueous distillate was brown in color and a dark-colored oil floated thereon. A flocculent precipitate appeared in the watery distillate. When the char had been heated an hour the white vapors stopped coming over, and the temperature rose to 109° C. After this the off-take temperature dropped to 80° C. and no more distillate came. The watery distillate contained ammonium salts.

Weight of char after igniting	3.01 lbs.	(1.36 kg.)
Total weight of distillate	54.2 g.	(3.6 per cent)
Weight of tar	11.5 g.	
Weight of water	42.7 g.	

This experiment shows that the charcoal produced in the oil-jacketed retort still contains about 9 per cent of volatile matter, which renders it unfit for many purposes. For instance, its aqueous extract is brown in color when it should be water-white, and it is furthermore not sufficiently ignited to be a good filtering medium or absorbent. The residual charcoal from this ignition, on the other hand, is jet-black and its water extract is free from any color whatsoever.

In June 1918 about 10 lbs. of unleached kelp char from Run 6 were forwarded to Dr. F. W. Zerban, of the Louisiana State Sugar Experiment Station at New Orleans, La. Dr. Zerban reignited this char in a closed iron container and leached the residue with water and acid, then made tests on the resulting charcoal to determine its value as a decolorizing agent for sugar solution.[1]

---

[1] "The Preliminary Examination of Kelp Distillates."

[2] J. W. Turrentine, "Note on the Distillation of Kelp," *Proc. 8th Intern. Congr. Appl. Chem.*, **15**, 313.

[1] F. W. Zerban and E. C. Freeland, THIS JOURNAL, **10** (1918), 812.

The kelp char made in the oil-jacketed retort did not prove as satisfactory as those originally prepared at higher temperatures.

In the specimen distillation log that follows, the column named "middle temperature," indicates the temperature at the center of the kelp charge, while "maximum temperature" refers to the oil bath surrounding the retort.

DISTILLATION OF KELP

Log of Run 7, May 17, 1918

CONDITIONS: Retort previously heated to 220° C. Strongly heated at first

Time A.M.	Middle Temp. ° C.	Max. Temp. ° C.	Total Distillate Cc.	REMARKS
8:20	140	182	...	Charged retort
8:35	104	175	...	Distillation commences but soon stops
8:45	102	187	...	Minimum temperature
9:00	114	211	...	
9:02	118	215	...	Distillation recommences
9:15	134	231	36.5	
9:30	148	244	97	Clear, green
9:45	158	260	177.5	
10:00	170	273	190.5	Becoming turbid. Fraction 104°–170°, 497 g.
10:15	180	285	191	Flaky precipitate
10:30	191	296	184.5	
10:45	201	305	183	
11:00	213	319	188	Fraction 170°–213°, 745 g.
11:15	225	333	185	
11:30	231	340	173	
11:45	247	345	166	Reduced flame[1]
12:00	256	350	144	Tar coming over. Fraction 213°–256°, 668 g.
P.M.				
12:15	267	352	162	
12:30	273	353	119	Tar, 19 g.
12:45	281	354	136	
1:00	289	354	115	
1:15	295	355	109	Water fraction 256°–295°,
1:30	300	355	87	530 g. Tar, 50 g.
1:45	304	352	73	
2:00	308	350	59	Tar, 55 g.
2:15	310	346	48	
2:30	311	345	43	Tar, 29 g.
2:45	313	350	43.5	Shut off gas. Water fraction, including drip next morning, 373 g.
3:00	317	352	34	
3:30	320	...	...	Tar, 71 g. Total weight of tar, 224 g. (1.4 per cent)

[1] There are two kinds of tar oil: the first that comes over is dark brown with a penetrating though faint odor resembling an essential oil or certain ketones; the second is blacker and has a more unpleasant odor.

RESULTS OF RUN 7

Weight of kelp distilled	27.5 lbs.	
Weight of basket and kelp after heating	107.6 lbs.	
Tare	90.0 lbs	
Residual kelp (non-volatile)	17.6 lbs.	(64%)
Loss by heating (total volatile)	9.9 lbs.	(36%)
Weight of charge	12,500 g.	
Total weight of liquid distillate	3,042 g.	(24.3%)
Gaseous distillate	....	(11.7%)

ANALYSES OF WATER CUTS LOG 7

Fraction Degrees	Specific Gravity	Solids in 10 Cc. Grams
104–170	1.007	0.1699
170–213	1.013	0.2687
213–256	1.032	0.9159
256–295	1.050	1.5961
295–320	1.063	2.4833

SUMMARY

Sixteen distillations of dried kelp were made in an oil-jacketed wood retort at temperatures not exceeding 320° C. These yielded aqueous liquor, tar, and a noninflammable gas. The residual charcoal was insufficiently heated either for a good extraction of potash or for use as a filtering medium. This work has demonstrated the necessity for distilling kelp at a much higher temperature.

TAR-STILL OPERATION IN HARDWOOD DISTILLATION PLANTS

By L. F. Hawley and H. N. Calderwood, Jr.

FOREST PRODUCTS LABORATORY, U. S. FOREST SERVICE, MADISON, WISCONSIN

Received February 21, 1920

Sometime ago the authors carried on efficiency studies at several wood-distillation plants with the purpose of preparing a standard set of directions for the use of the refinery foremen and stillmen. The results of the work on the operation of the tar still were of special interest. Since these results are directly applicable to the tar-still operations of many other wood-distillation plants, it was decided to publish them.

The operation carried out in the tar still is for the purpose of recovering the acetic acid that remains in the settled tar after the pyroligneous acid has been removed. In general, the method consists of heating the tar by means of steam in closed coils until the water is driven off, and then, while keeping the closed coils still in use, blowing live steam through the charge.[1] It is obvious that the most desirable conditions for running a tar still are those under which the most acetic acid may be produced in the shortest time and with the greatest degree of concentration. The best conditions for this distillation with respect to speed, length of time, and pressure of steam in the closed coils, have never been determined, and the commercial practice varies greatly.

EFFECT OF SPEED OF DISTILLATION

Two test runs were made with a charge of 510 gal. of tar in a depth of 40 in. The conditions were the same in both runs, except that the speed at which the live steam was blown through the tar was different. The results are shown in Tables I and II. The tables show that the speed has a slight effect on the concentration, that is, that a certain number of gallons of water in the form of steam going through the charge of tar will remove more acetic acid when the steam

TABLE I—SLOW SPEED

Time Start 12 : 50	Speed Gal. per Hr.	Total Distillate Gallons	Acid Per cent	In Fraction Pounds	Total Pounds	Pressure in Coils
1 : 05	40	10	20.6	17.1	17.1	45
2 : 05	30	40	19.35	48.1	65.2	43
3 : 05	25	65	13.31	27.6	92.8	46
4 : 05	24	89	9.68	19.2	112.0	41
5 : 05	23	112	6.25	12.0	124.0	43
6 : 05	26	138	4.62	10.0	134.0	43
7 : 05	24	162	3.00	5.9	139.9	35
8 : 05	23	185	2.12	4.1	144.0	42
8 : 35	22	196	2.08	1.8	145.8	40
9 : 05	22	207	1.87	1.6	147.4	40
9 : 35	24	219	1.69	1.6	149.0	41
10 : 05	12	225	1.61	0.8	149.8	45
10 : 35	12	231	1.52	0.8	150.6	45

Residual tar 0.28 per cent acid

---

[1] There is no mysterious effect of "steam distillation" in this separation of acetic acid from tar. The steam is superheated all the time it is in contact with the tar and acts like any neutral gas in its effect on the distillation. In fact, as far as the *distillation* is concerned, the same fractionation of acid and oil would be obtained by direct fire heat in a distillation without steam. The separation of acid from oil actually takes place after the condensation of the vapors when the acid is divided between the water and oil according to its relative solubility in these two substances. This indicates the desirability of washing certain fractions of the oil with water to recover a further quantity of acetic acid.

TABLE II—FAST SPEED

Time Start	Speed Gal. per Hr.	Total Distillate Gallons	Acid Per cent	In Fraction Pounds	Total Pounds	Pressure in Coils
2 : 25	75	...	...	...	...	45
2 : 40	50	20	23.1	35.8	35.8	44
3 : 20	51	54	15.0	42.3	78.1	47
3 : 40	60	70	9.4	12.5	90.6	47
4 : 10	50	95	6.5	13.4	104.0	49
4 : 40	46	118	4.7	9.0	113.0	43
5 : 10	44	140	3.1	5.6	118.6	40
5 : 40	46	163	2.6	5.0	123.6	43
6 : 10	44	185	2.0	3.6	127.2	38
6 : 40	50	210	1.8	3.6	130.8	46
7 : 10	50	235	1.6	3.3	134.1	44
7 : 40	48	259	1.5	3.0	137.1	47
8 : 10	48	273	1.8	2.1	139.2	46
8 : 40	24	285	1.9	1.8	141.0	46
9 : 10	24	297	1.7	1.6	142.6	46

Residual tar 0.82 per cent acid

goes through slowly than when the steam goes through quickly.

A slow speed of distillation is, therefore, desirable from the standpoint of high concentration of distillate, and the speed should be kept as low as is consistent with the capacity of the tar stills. Sometimes it might even be desirable to build an additional still in order to make slower distillation possible.

EFFECT OF STEAM PRESSURE

The effect of different pressures of steam in the closed coils was not determined in the commercial still, but a laboratory experiment was carried out that showed this effect. Two charges of the same tar were distilled with steam. The tar was kept at a temperature corresponding in one case to a 10-lb. steam pressure, in the other case to a 50-lb. steam pressure. The results of the two distillations are shown in Table III.

TABLE III—EFFECT OF STEAM PRESSURE

	15 Lbs. Pressure				50 Lbs. Pressure			
Fraction No.	Cc.	Acid Per cent	Acid in Fraction Grams	Total Acid	Cc.	Acid Per cent	Acid in Fraction Grams	Total Acid
1......	66	3.5	2.3	2.3	58	6.8	3.9	3.9
2......	71	2.8	2.0	4.3	74	5.1	3.8	7.7
3......	78	2.0	1.6	5.9	69	2.5	1.7	9.4
4......	73	1.6	1.2	7.1	71	1.3	0.9	10.3
5......	77	1.1	0.8	7.9	73	0.45	0.3	10.6
6......	81	0.8	0.7	8.6	70	0.25	0.2	10.8

It is plainly to be seen from these results that more acetic acid can be obtained with less steam when the tar is kept at a higher temperature, that is, when the pressure in the closed coils is kept higher. For instance, at a 50-lb. pressure four fractions, with a total volume of 272 cc., contain 10.3 g. of acid, but, at a 15-lb. pressure six fractions, with a total volume of 446 cc., contain only 8.6 g. of acid.

END-POINT OF DISTILLATION

After it is known that a slow speed and high steam pressure are desirable conditions for tar distillation, to insure an efficient operation one other matter must be determined—the point at which to stop the operation. It may be seen from all three tables that the concentration of acid in the distillate decreases as the distillation progresses;[1] it finally reaches a point at which the concentration is so small that it is no longer profitable to recover acetic acid from the distillate. The proper degree of concentration at which to stop the distillation is determined, on the one hand, by the price of acetate, and, on the other, by the cost of the necessary fuel, labor, and apparatus. This computation must be made for each plant according to local costs for these items, the main cost being for the fuel required to make the steam for removing the acid from the tar and for evaporating the acetate of lime solution.

After the proper end-point for stopping the distillation has been decided upon, it is necessary to find a method for determining when this point has been reached. Probably most tar stills are operated on a time schedule. If the proper lengths of time are determined for reaching the end-point under certain constant conditions of distillation, and if those constant conditions could be maintained in subsequent runs, this method of operating the still would be satisfactory. However, variations in the original tar are unavoidable, and accurate regulation of speed and pressure are very difficult to maintain, so that the end-point cannot readily be ascertained by the length of time of distillation. The end-point is most accurately determined by an actual chemical analysis by titration of the acid contained in the distillate. This analysis may readily be made by the stillman if he is furnished with standard alkali and a graduated cylinder or pipette.

In some plants the dissolved tar left in the copper still after the distillation of the crude pyroligneous acid is mixed with the settled tar and distilled in the tar still; in other plants the copper still has sufficient capacity so that the dissolved tar may be distilled by steam without removal for the recovery of the acetic acid. In the latter case the same suggestions as to speed, pressure, and end-point apply to the dissolved tar in the copper still as to the settled tar in the tar still.

DIRECTIONS FOR TAR-STILL OPERATION

The following set of directions for running the tar still at one plant is given as an example:

(1) Distil with closed coils at steam pressure of 10 lbs. at the start, increasing to 50 lbs., as rapidly as possible without boiling over, until very little distillate is coming over.

(2) With steam in closed coils remaining at 50 lbs. or higher, if readily possible, blow in live steam at a speed of 30 to 35 gal. per hour when the charge is 40 in. deep (equivalent to 510 gal.), or at a correspondingly different speed when the charge is different. Continue this speed until 9.1 cc. of distillate are required to neutralize 10 cc. of 0.5 *N* alkali.

(3) Reduce the flow of live steam to at least one-half, and continue the distillation as long as not more than 9.4 cc. of distillate are required.

Such directions will, of course, vary with different plants and with varying conditions at the same plant. With the information offered in this paper, combined with a knowledge of local conditions, it should not be difficult to prepare a similar set of directions that would help to prevent loss of product on the one hand and loss of efficiency on the other.

---

[1] There are two reasons for this: First, the proportion of acetic acid in the oil distilling over decreases as the distillation progresses, since the boiling point of the acid is lower than that of the oil; and second, the proportion of oil to water decreases, since at a constant temperature the vapor pressure of the residual tar decreases as the lower boiling oils are removed and more water vapor is required to carry over the same amount of oil.

THE HESS-IVES TINT-PHOTOMETER AND ITS USE WITH RAW SUGARS[1]

By George P. Meade and Joseph B. Harris

Central Control Laboratory, Cuban-American Sugar Co., Cardenas, Cuba

The Hess-Ives tint-photometer is described by the makers as "an instrument for comparing different shades and hues of light colored materials or liquids with each other and with fixed standards, in order to differentiate and classify them and give them numerical expression." Light from an artificial daylight lamp placed at the back of the instrument shines on two blocks of magnesia from which it is reflected by means of a mirror through two adjustable light openings or slits into the instrument itself. The light from each slit is distributed over the corresponding half of a circular field by means of a patent device called an optical mixing wheel, which is rotated rapidly by a small motor. The left-hand slit may be adjusted from full open to tight closed by means of a shutter operated by a lever. The lever moves over a scale which reads zero when the slit is closed and 100 when it is open. The right-hand slit is adjustable through narrower limits, the adjustment being used to bring the two halves of the field to equal intensity when the lever actuating the left-hand slit is brought to 100 on the scale.

When a sample is placed on the magnesia block before the right-hand opening the light entering this aperture is reduced, and the corresponding half of the field appears darker. To measure the amount of the reduction the lever is moved down the scale until the field is matched again, and the amount the left-hand aperture has been closed is read on the scale. If the sample is of a gray shade without any specific color the field can be matched without the use of a color screen, but for colored materials readings are made by matching the field through each of the three color screens, red, green and blue-violet, provided with the instrument.

When working with liquids the solution to be examined is placed in a small glass cell with clear glass bottom on a shelf interposed between the right-hand magnesia block and the right-hand light opening, so that the light passes through the column of liquid. Provision is made so that no light enters the liquid except through the bottom. It is essential to place a cell containing distilled water to the same depth as the solution under examination on the shelf before the left-hand light opening, to compensate for the light cut off by the glass and the water of the solution. If this is done the same reading will be obtained irrespective of the depth of the column of liquid, provided the same amount of color is maintained. Our work has been done entirely with liquids, and no study has been made by us of the use of the instrument with solid materials.

The pamphlet accompanying the tintometer is vague, particularly as regards the arrangement of the attachment for liquids, and considerable study is necessary to set the instrument up properly. The position of the scale makes it awkward to read, but otherwise the tintometer as it comes from the manufacturers is well made and mechanically easy to operate.

SCALE READINGS

The problem which presents itself when the instrument is put into practical use is the meaning of the scale readings. The readings are in "per cent luminosity" or, subtractions of the readings from "one hundred per cent darkness." But how can these percentages be interpreted in terms of color concentration? Journal articles dealing with the tint-photometer which have come to our attention since this study was taken up also recognize this difficulty. Kress and McNaughton[1] add the three readings through the three color screens together and subtract from 300, calling the results "parts black." As will be seen later this method will serve only for readings in one part of the scale, and will then be only approximate.

Zerban[2] says, "since the readings obtained in degrees of the instrument bear no direct relation to the color concentration, it was first necessary to standardize the instrument for the purpose of translating the readings into concentrations." This he did by making an arbitrary standard solution of dark molasses, 30 g. per liter, and making up a series of dilutions containing 1 per cent, 2 per cent, 3 per cent, etc., up to 20 per cent, then by 2 per cent intervals up to 30 per cent, and by 5 per cent intervals to 100 per cent. Each of these solutions was then read in the tintometer through the three color screens and the readings recorded. He called the color of his standard molasses solution 100, and by comparing readings of other solutions with those obtained on the various concentrations of his standard he was able to express any scale reading in terms of the standard.

For reading solutions other than sugar solutions (*e. g.*, colorimetric determinations of iron and of the polyphenols) he made similar series of readings on standard solutions of the substance to be examined, but here he encountered the difficulty that "the color readings of the solution to be analyzed did not always agree in each color region with the readings obtained on those of known concentration."

INTERPRETATION OF SCALE READINGS

The tint-photometer was purchased by this laboratory for determining the color of raw sugars, and practically all of our study has been done on solutions of raw sugars. A weighed amount of sugar was dissolved in a given quantity of water, a liberal amount of kieselguhr added, filtered through paper, pouring back until the filtrate was brilliant.

A series of solutions of a certain raw sugar were made up containing, respectively, 1 g., 2 g., 3 g., in 25 cc. and readings were made on each of the solutions through each of the color screens. The tabulation shows the readings, which are presented graphically in Fig. 1.

---

[1] Presented at the 59th Meeting of the American Chemical Society, St. Louis, Mo., April 15, 1920.

[1] "A Numerical Expression for Color as Given by the Ives Tint-Photometer," This Journal, **8** (1916), 711.

[2] "The Color of Sugar Cane Products," *Louisiana Bulletin* **165**, March 1919.

The instrument is accurate to one degree of the scale, the blue field being somewhat more difficult to match than the others.

TABLE I—READINGS ON VARIOUS STRENGTH SOLUTIONS OF THE SAME RAW SUGAR, SHOWING THE VARIATION OF THE SCALE READINGS FOR CONCENTRATION

Sugar in 25 cc. G.	Red Screen	Green Screen	Blue Screen
1	87	74	47
2	75	55	24
3	65	40	12
4	57	30	7
5	50	22	Too
6	43	16	dark
7	37	12	to
8	32	8	read
9	27	6	accu-
10	24	4	rately

It is seen from these figures that neither the scale readings themselves nor the scale readings subtracted from 100 are direct expressions of the relative amounts of color, but that as the amount of sugar is increased the scale readings decrease by a constantly decreasing amount.

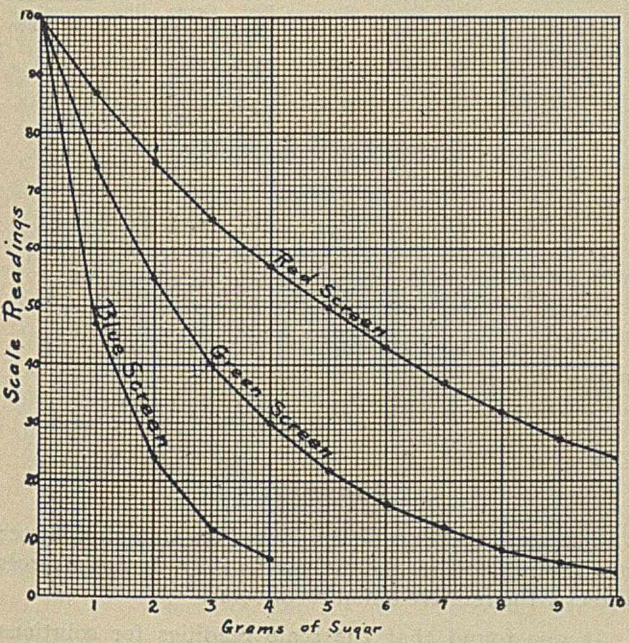


FIG. 1

A study of these curves and figures and of other similar series brought out the fact that the readings in any series run in powers of the first reading of the series, *i. e.*, the reading for one unit of material, no matter which color screen or what class of material was used. (For mathematical purposes, the scale readings must be considered as decimal fractions.) For example in the first series given in Table I: $.87 \times .87 = .757 \times .87 = .658 \times .87 = .574$, etc. The scale does not admit of the reading of fractions.

The reason why the readings follow this rule lies in the make-up of the instrument itself. Consider the two halves of the field evenly matched with the scale set at 100, *i. e.*, with the same amount of light entering both apertures. Now if a solution containing one gram of the sugar used in Table I is placed before the right-hand opening, it will permit only 74 per cent of the light to pass when viewed through the green screen. To match the field again, the left-hand opening must be reduced until it is 74 per cent of its size, or, in other words, the lever must be moved down the scale to 74. The conditions are now the same as when the instrument was in adjustment at 100; the same amount of light is entering both apertures and the field is evenly matched.

The first solution is now replaced by one containing 2 g. of the sugar. This second gram of sugar will cut off the same *percentage* of light that the first gram did, that is, it will permit only 74 per cent of the light now entering to pass, and the left-hand opening must be closed to 74 per cent of its present width in order to match the field again. ($.74 \times .74 = .548$ or 55 in practice.) A third gram of sugar will again reduce the light 74 per cent of the amount entering after the field is matched with the 2 g., and the scale must be moved to 74 per cent of .548, or .406, a reading of 40 in practice.

This relationship between the scale readings and the amount of material taken can be expressed algebraically by the equation $y = K^x$,

where y = any scale reading
K = the scale reading for one unit of the material
x = the number of units of material which will give the scale reading y.

With this equation, given the scale reading for a unit amount of material, it is possible to calculate what reading would be obtained with any other amount of the same material; or, given the scale readings of unit quantities of two materials, the amount of color in the one as compared to the other may be computed.

For example: To find what 5 g. of sugar would read through the red screen, given that one gram reads 87,

$$y = .87^5 = .498 \text{ or } 49.8 \text{ against } 50 \text{ in practice (Table I)}$$

Five grams of Raw Sugar A read 58 through the green screen; the same quantity of Raw Sugar B reads 41. Problem: To find how much more color B has than A or how much of A would be required to give the reading of 5 g. of B.

Solving the equation for x:

$$x = \frac{\log y}{\log K} = \frac{\log .41}{\log .58} = \frac{.3873}{.2366} = 1.636$$

That is, through the green screen, B has 1.636 times as much color as A, or it would require 1.636 times as much of A to give the reading of any given quantity of B.

To prove this in practice, a solution containing 8.2 g. of A (1.64×5 g.) was read through the green screen. One observer read 41, the other 42, as against a reading of 41 for 5 g. of B.

Through the red screen, 5 g. of A read 78, and 8.2 g. read 67. What should the 8.2 g. have read according to the equation?

$$y = .78^{1.64} = .665 \text{ or } 66.5 \text{ on the scale}$$

TABLE FOR TRANSLATING SCALE READINGS TO UNITS OF COLOR

It is also possible by means of the equation to compare all scale readings with some fixed number as a standard. This has the advantage that K becomes a constant in the logarithmic calculation.

To avoid repeating the logarithmic calculation, we adopted K = 99 as a standard, and calculated a table by means of the equation for each scale reading from 100 to 1 (Table II). By this table all solutions are compared to a hypothetical solution which would give a reading of 99 for all three color screens. The number 99 was selected because it represents the smallest amount of color which can be read in the instrument, and therefore the color corresponding thereto can be taken as unity. The table gives opposite each scale reading (y) the number of units (x) of the hypothetical solution (K = 99) which it would take to give the scale reading; or, taking the color of the hypothetical solution as unity, the number of color units corresponding to that scale reading.

TABLE II—FOR TRANSLATING HESS-IVES TINT-PHOTOMETER SCALE READINGS TO UNITS OF COLOR

$y = K^x$, where y = scale reading; x = units of color; K = 99 (constant)

Scale Reading y	Units of Color x	Scale Reading y	Units of Color x	Scale Reading y	Units of Color x	Scale Reading y	Units of Color x	Scale Reading y	Units of Color x
100	0.0	80	22.2	60	50.8	40	91.1	20	160.0
99	1.0	79	23.4	59	52.5	39	93.6	19	165.0
98	2.0	78	24.7	58	54.2	38	96.2	18	170.5
97	3.0	77	26.0	57	55.9	37	98.9	17	176.0
96	4.0	76	27.3	56	57.7	36	101.6	16	182.0
95	5.1	75	28.6	55	59.4	35	104.4	15	189.0
94	6.1	74	29.9	54	61.3	34	107.3	14	196.0
93	7.2	73	31.2	53	63.1	33	110.3	13	203.0
92	8.3	72	32.6	52	65.0	32	113.3	12	211.0
91	9.4	71	34.0	51	67.0	31	116.5	11	219.0
90	10.5	70	35.5	50	68.9	30	119.7	10	229.0
89	11.6	69	37.0	49	70.9	29	123.1	9	239.5
88	12.7	68	38.4	48	73.0	28	126.6	8	251.0
87	13.8	67	39.9	47	75.1	27	130.2	7	264.0
86	15.0	66	41.3	46	77.2	26	134.0	6	280.0
85	16.2	65	42.8	45	79.4	25	137.8	5	298.0
84	17.4	64	44.4	44	81.6	24	141.9	4	320.0
83	18.6	63	46.0	43	83.9	23	146.2	3	349.0
82	19.8	62	47.6	42	86.2	22	150.6	2	389.0
81	21.0	61	49.2	41	88.7	21	155.0	1	458.0

Any of the illustrations used to show the workings of the equation can now be worked out much more simply by the table.

For example: To find what 5 g. of sugar would read through the red screen, given that 1 g. reads 87.

From the table, 87 = 13.8 color units

$$13.8 \times 5 = 69.0$$

From the table, 69 corresponds to a scale reading of 49.8 (50)

Sugar A reads 58 through the green screen

Sugar B reads 41 through the green screen

From the table, 58 = 54.2 color units; 41 = 88.7 color units

Then B has $\frac{88.7}{54.2} = 1.64$ times as much color as A

By means of the table, then, any scale reading for any color screen can be immediately translated into units of color, irrespective of the class of material under examination.

DETERMINATION OF COLOR OF RAW SUGARS

The following method for determining the color in raw sugars has been adopted: Dissolve 20 g. of sugar in distilled water and make up to 100 cc. Filter through paper in which are placed two or three teaspoonsful of kieselguhr, pouring back until the filtrate is brilliant.

Transfer 25 cc. of this solution to the observation cell on the right side of the instrument, and place a cell containing 25 cc. of distilled water on the left-hand side. Read the solution through each of the three screens, record the readings, and note the color units for these readings as taken from the table. Add the color units thus obtained and divide by three, recording this as the color of the sugar. Whole numbers only are used.

In case the sugar is so dark that any of the readings fall below 20 it is best to take a smaller amount of the solution (diluting it to 25 cc. if desired). The result can be calculated to 5 g. The instrument is rather hard to read accurately in the lower part of the scale, and one degree error with the lower numbers of the scale represents a much greater amount of color than with the higher scale readings.

The use of 5 g. and the division of the sum of the color units by three were adopted as they were found to give convenient sized numbers for our work. The time required to make a color determination in this way is about the same as that required for a direct polarization.

TABLE III—TYPICAL RESULTS OF COLOR DETERMINATIONS ON RAW SUGARS

Class of Sugar	SCALE READINGS			UNITS FROM TABLE			$\frac{\text{Total Units}}{3}$ or Color
	Red Screen	Green Screen	Blue Screen	Red Screen	Green Screen	Blue Screen	
Cuban Raws							
1920 Crop......	64	40	12	44.4	91.1	211.0	116
	65	41	16	42.8	88.7	182.0	105
	66	47	25	41.3	75.1	137.8	95
	70	50	21	35.5	68.9	155.0	87
	72	52	25	32.6	65.0	137.8	78
	71	53	31	34.0	63.1	116.5	71
	70	55	37	35.5	59.4	98.9	64
	80	62	35	22.2	47.6	104.4	58
Cuban Raws							
1919 Crop......	59	29	10	52.5	123.0	229.0	135
	69	42	16	37.0	86.0	182.0	102
	72	53	41	32.6	63.1	88.7	62
Washed Raw							
1919 Crop......	84	76	53	17.4	27.3	63.1	36
1920 Crop......	99	97	85	1.0	3.0	16.2	7
Refinery							
High Remelt...	95	89	73	5.1	11.6	31.2	16
Refinery Molasses Sugar							
One Gram....	73	51	23	31.2	63.1	150.6	82
				(Calculated to 5 g.) 82 × 5 = 410			

SUMMARY

The scale readings of the Hess-Ives tint-photometer are meaningless in themselves as they do not express directly the relative amounts of color.

It was found that the scale readings for solutions containing 1, 2, 3, 4, etc., units of material run in powers of the reading for one unit, considering the scale readings as decimal fractions. This is due to the mechanical make-up of the instrument, and is true no matter which color screen or what class of material is used.

Expressed algebraically, this relationship between the scale readings and the amounts of color becomes $y = K^x$, where y is any scale reading, K is the reading for one unit of material, and x is the number of units of material required to give the scale reading y.

By means of this equation solved for x the color of two materials may be compared, given scale readings for equal quantities; or all scale readings may be compared to a standard.

To avoid the repeated calculation, a table has been calculated which gives the units of color corresponding to each scale reading from 100 to 1. A convenient method for determining the color of raw sugars, using the table, is given, together with results for various sugars.

PRESERVATIVES FOR FERMENTABLE BEVERAGE SAMPLES

By J. S. McCune and A. N. Thurston
Division of Laboratories, Ohio State Department of Health, Columbus, Ohio

Received October 27, 1919

Before prohibition went into effect, the Liquor Licensing Board in Ohio was collecting many samples of alcoholic beverages in connection with the licensing laws. These were analyzed at the Department of Health Laboratory, where all state analytical work is now done. Some of the samples collected in territory which was dry because of local option were near beers and ciders.

Argument arose about these samples as to the possibility of fermentation before delivery to the laboratory, but after the sale of the liquid. With near beers and particularly with ciders there was no doubt as to this having occurred in some instances where delivery was unavoidably delayed. For the purpose of testing out the necessity of preservatives, and the chemicals best suited for this use, coöperation between the two departments was arranged. However, when prohibition became effective, the Liquor Licensing Board ceased to exist, which has handicapped the work to some extent. Sweet cider could not be obtained, and so the preliminary experiments have been made on near beer. The tests are of interest, however, and will be of value in court cases for the enforcement of dry laws in the state.

The experiments made are listed below, and since lack of samples has prevented any extensive study, the conclusions are open to revision. Information from any bureaus or departments engaged in control work would be welcome.

EXPERIMENT 1—"Bruin," a near beer manufactured in Columbus, Ohio, and sold in bottles, was allowed to stand open at room temperature.

ALCOHOL CONTENT: PER CENT BY VOLUME

Original	None
After 1 day	None
After 2 days	None
After 5 days	0.19 (heavy mold growth)

Conclusion: The bottled beer would not ordinarily ferment, even when open.

EXPERIMENT 2—Same, with 1 cc. of a suspension of Fleischman's yeast.

ALCOHOL CONTENT: PER CENT BY VOLUME

Original	None
After 1 day	1.47
After 5 days	2.05

Conclusion: With yeast 2.00 per cent alcohol were obtained, the large portion in 24 hrs.

EXPERIMENT 3—Bruin and 1 cc. yeast as above, but with varying preservatives, each added as 0.20 per cent.

ALCOHOL CONTENT: PER CENT BY VOLUME

	Mercuric Chloride	Salicylic Acid
After 1 day	0.20	0.20
After 4 days	0.13	0.10
	Sodium Benzoate	Sodium Bisulfite
After 1 day	0.16	0.73
After 4 days	0.10	2.20

Conclusion: Two-tenths per cent of mercuric chloride, salicylic acid, or sodium benzoate are satisfactory to prevent fermentation. The same percentage of sodium bisulfite is unsatisfactory.

EXPERIMENT 4—Same as Expt. 3, but beer was allowed to ferment one day before adding preservative, to determine whether started fermentation would be checked.

ALCOHOL CONTENT: PER CENT BY VOLUME

	Mercuric Chloride	Salicylic Acid
After 1 day, without preservative	1.08	1.04
Same, 3 days, after preservative added	1.04	1.02
	Sodium Benzoate	Sodium Bisulfie
After 1 day, without preservative	1.04	1.04
Same, 3 days, after preservative added	1.95	1.89

Conclusion: Two-tenths per cent of mercuric chloride or of salicylic acid are satisfactory to check started fermentation, but the same amount of sodium benzoate or sodium bisulfite is unsatisfactory.

EXPERIMENT 5—Same as Expt. 4, but with sodium benzoate only, to determine whether the sodium benzoate result could be confirmed.

ALCOHOL CONTENT: PER CENT BY VOLUME

	Sodium Benzoate	
	No. 1	No. 2
One day's fermentation before adding benzoate	0.47	0.48
After 3 days additional with 0.20 per cent benzoate	1.94	1.95

Conclusion: Sodium benzoate (0.20 per cent) is unsatisfactory to check fermentation.

EXPERIMENT 6—Draft near beer, purchased from keg, with 0.20 per cent sodium benzoate and without, to determine whether draft beer would ferment without added yeast and whether benzoate would prevent the fermentation.

ALCOHOL CONTENT: PER CENT BY VOLUME

	Without Preservative	With 0.20 Per cent Benzoate
As purchased	0.43	0.43
After 1 day	1.18	0.47
After 3 days	1.43	0.43
After 8 days	..	0.43 (heavy mold growth)

Conclusion: (*a*) Draft near beer would ferment rapidly as purchased; (*b*) sodium benzoate, 0.20 per cent, prevented this fermentation.

EXPERIMENT 7—Draft near beer, without yeast, preservatives, 0.20 per cent, added on day of purchase.

ALCOHOL CONTENT: PER CENT BY VOLUME

	Mercuric Chloride	Salicylic Acid
As purchased	0.63	0.63
After 3 days with preservative	0.63	0.60
	Sodium Benzoate	Boric Acid
As purchased	0.63	0.63
After 3 days with preservative	0.75	1:53

Conclusion: Mercuric chloride, or salicylic acid (0.20 per cent), satisfactory to prevent fermentation; sodium benzoate (0.20 per cent), doubtful; boric acid (0.20 per cent), unsatisfactory.

EXPERIMENT 8—Draft near beer as in Expt. 7, but preservatives added after one day's standing.

ALCOHOL CONTENT: PER CENT BY VOLUME

	Mercuric Chloride	Salicylic Acid
As purchased	0.63	0.63
After 1 day without preservative	1.35	1.35
After 3 days additional with preservative	1.30	1.36
	Sodium Benzoate	Boric Acid
As purchased	0.63	0.63
After 1 day without preservative	1.35	1.35
After 3 days additional with preservative	1.50	1.56

Conclusion: Fermentation nearly complete in one day, but results in general checked conclusions of Expt. 7.

GENERAL CONCLUSIONS

These experiments show the efficiency of the above preservatives used in untreated draft beer and bottled beer treated with Fleischman's yeast.

Sodium benzoate, 0.20 per cent, is unreliable as a preservative.

Sodium bisulfite and boric acid, 0.20 per cent, are worthless.

Mercuric chloride and salicylic acid, 0.20 per cent, are satisfactory preservatives.

Due to the poisonous nature and higher cost of mercuric chloride, salicylic acid, 0.20 per cent, is recommended as the preservative to be used when fermentable samples are to be tested for alcohol content and when immediate delivery to the laboratory is not possible.

A SIMPLE ALCOHOL-TESTING DEVICE[1]

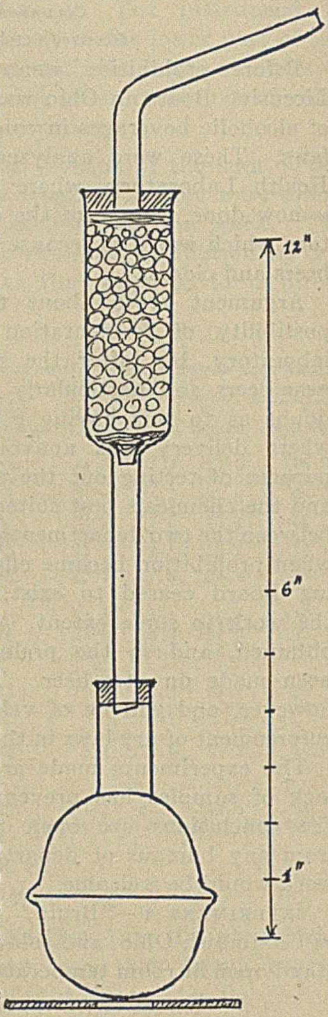
By Sigmund Waldbott

THE LLOYD CHEMICAL RESEARCH LABORATORY, CINCINNATI, OHIO

Received December 16, 1919

A simple device has been developed which readily differentiates between a beverage of low alcohol content, *e. g.*, 0.5 per cent, and one containing more, *e. g.*, 3 per cent alcohol. It is not intended to replace the exact methods of alcohol determination, but rather to aid them, by giving preliminary information as to alcoholic contents. Its principle is to boil out the alcohol, separate it from the water vapor by means of a simple fractionating device, and demonstrate the alcohol vapor by ignition.

The apparatus consists of a copper flask and a fractionating attachment as shown in the sketch, made of glass or metal. The fractionating medium consists of glass beads which fill the receptacle of the attachment. The bend in the exit tube has some advantage in the demonstration. In heating the flask by a Bunsen burner, or by an alcohol lamp, use an asbestos plate with a 1-in. opening in the center.



Fifty cc. of the fluid, *e. g.*, beer, are put into the flask, and heated over a medium-sized Bunsen flame to boiling, regulating the flame according to apparent needs. When using a glass attachment, the progress of condensation is easily observed. In the case of metal, the sense of touch will tell. When the alcohol vapor appears at the orifice, ignite it with a burning match or candle.

If the beer contains 3 per cent alcohol, a semi-luminous, blue flame about 3 in. long will continue to burn for 120 to 150 sec. depending on the size of the Bunsen flame. When the beer contains only 0.5 per cent alcohol, the flame of the same length lasts about 20 to 25 sec. under the same conditions of heating the bulb. Thus the duration of the flame is approximately proportional to the alcohol contents. To this extent, the apparatus permits even approximate quantitative estimations.

The apparatus may serve as a lecture experiment, and may prove useful as a legal instrument in the hands of government officials charged with the enforcement of laws relating to alcoholic contents of beverages.

---

[1] Presented at a meeting of the Cincinnati Section, American Chemical Society, October 15, 1919.

ADDRESSES AND CONTRIBUTED ARTICLES

A PLAN FOR INCENTIVE TO RESEARCH IN PURE AND APPLIED CHEMISTRY[1]

By William J. Hale

DOW CHEMICAL CO., MIDLAND, MICHIGAN

In these post-bellum days of industrial reconstruction we cannot fail to note the prominence gained by American chemists. Those with adequate training and experience meet with little difficulty in their forward progress, but far too many realize more and more that lack of broader training in their early academic career. Only those young men thoroughly grounded in the fundamentals of their science and well versed in engineering and mathematics can ever expect to gain the prominence they seek. Colleges everywhere must recognize this situation and strengthen decidedly their prescribed chemical training.

For the university man who is soon to enter actively the chemical profession it has thus appeared as most reasonable to recommend the further pursuit of the science for a year or more on some problem of research, and under competent guidance; to this end a number of industries have endeavored to lend encouragement through the offer of material aid. This aid usually follows some plan more or less in keeping with precedent at the particular university and beyond this much is left to the good will of all concerned. Though the best of motives may have prompted this action on the part of the industries, I feel that they have not sufficiently weighed the outcome of their well-intended efforts. In other words, I believe that a far better plan for the furtherance of chemical research lies open to us. You may characterize my plan as daring, but I shall choose to call it the plan of incentive, and in order to draw sup-

---

[1] Read at the 59th Meeting of the American Chemical Society, Division of Industrial and Engineering Chemistry, St. Louis, Mo., April 12 to 16, 1920. A committee has been appointed by this Division to study the questions herein presented and to draw up a definite plan for actiod.

port for my propositions, I wish now to analyze the present-day conditions.

Introductorily speaking, I found myself only a few years ago firm in the conviction, unreasonable to the point of intolerance, that any research save that in pure science was merely guesswork. Its degrading effect was apparent to all who really studied the question and hence I constituted myself a disciple of pure unadulterated chemistry. Do not think me alone in this category. Too many, yes, far too many of our chemists thought alike with me. We all entertained a horror of perverting the science to mundane ends, and thus, in contrary fashion, sought steadfastly to elevate it at least into terrestrial temples. The march of progress has made steady inroads upon chemical knowledge; the simpler points readily yielding to industrial development, as noted so well by the applications of inorganic and physical chemistry. Applications of the more complex, the physiological and organic, have only recently come into prominence. In this latter field I am obliged to characterize myself as having held aloof and thus ensconced its isolation from a world of activities. Whether or not I have erred I leave to you to judge.

TYPES OF RESEARCH

Let us consider briefly three general types of research in operation. First, the purely industrial research carried on in industrial laboratories either in connection with specific industries or under independent organization. Second, university research which, as its name implies, embraces those researches conducted at a university either by the university staff or its advanced students. And third, the endowed research or that type supported by permanent endowments and placed under the direction of our institutes of research.

This latter class is indeed ideal in its organization and deserves our heartiest support. The highest class of work may here be accomplished through the employment of well-known scientists. It is possible also that a number of these purely research men may be attracted by universities to research professorships, the highest possible bestowal of honor by any university, and there exert a marked influence upon scientists yet in the making. An admirable outline of this plan with its purpose and scope is to be found in an address by Dr. R. A. Millikan, entitled "The New Opportunity in Science," and published in *Science*, September 26, 1919. In one sentence, "Our greatest need is not for facilities, but for the selection and development of men of outstanding ability in science," Dr. Millikan has summed up the situation of scientific endeavor in America, and upon this theme rests the *raison d'être* of my discussion.

The first or purely industrial type of research will receive due consideration throughout my discussion. It must be recognized as broad in scope and broadening more day by day. The results, however, for commercial reasons do not find immediate publicity save in patent literature, and this literature, I regret to state, is little fathomed by American chemists. It is coming rapidly into prominence and bids fair to rank as high here as it does in other countries. In many cases, the applications for a patent far outrank some of the most noted articles appearing in our purely scientific journals.

It is the second or university type of research that I would now set before you for analysis. It will be necessary for us to consider it in detail and for this purpose I choose to mention the two distinct policies in its prosecution. The first policy comprehends the operation of those special departments, usually at state universities, where competent men are employed to investigate questions affecting the health and welfare of the public. The men thus engaged are subject to call for assistance on the part of various public organizations, but they are able also to devote considerable time to problems of research, and much good results therefrom. As an outgrowth of this plan we may view the possible establishment, already under discussion at several universities, of Research Bureaus, at the request of various manufacturers' associations. These bureaus are to be organized to handle problems submitted to them by the manufacturers. The reports no doubt are to be made public, but at the same time to be utilized by whatsoever manufacturer directly interested. Though good results may be attained, the researches themselves will rarely embrace any more than passing fancies. In this day any vital question necessary for a growing manufacturing establishment will have its best investigators closely connected with the establishment—otherwise the manufacturers might well seek some more elementary pursuit. The scheme, however, should work to advantage in promulgating new enterprises and offering new possibilities to old ones. Further, it will acquaint the university man with elementary industrial problems and thus serve to widen his all-too-narrow vision to the point of making him more decidedly human—an attribute most desirable in those who would teach others.

The second policy comprehends the operation of the graduate schools and corresponding faculties in research for itself alone. Students enrolled in these graduate schools receive a thorough training as ground work for their chosen subject and find it possible to devote about half of their time to special research. Within a few years they are enabled thus to attain the degree of Doctor of Philosophy, and the results of these researches immediately find publicity in our scientific publications. It is this form of research that appeals strongly to those who have received just such training. Naturally it may be contended that this is only the early stage in research work. True, of course, but much of value I believe may be accomplished by the student in this prescribed course, especially so if he is of mature and original mind.

In order that students may be encouraged to spend these few extra years at the university, various kinds of assistantships and fellowships are open to award. The assistantships offer only small recompense but are of inestimable value where not more than a quarter of the students' available time is required. The fellowships carry with them a not inconsiderable stipend or honorarium without the adjunct of teaching responsibilities and when awarded upon the basis of merit and ability alone there is no higher honor in student life. Both assistantships and fellowships, therefore, concern us directly as research opportunities.

INDUSTRIAL FELLOWSHIPS

I come now to the particular point at issue. In recent years we note the introduction of a new class of fellowship—possibly falling under the supervision of graduate schools but more or less removed from such beneficent influence and left to various departments as distinctly technical and pertaining more directly to a super-graduate school; at least the holder of such fellowship will preferably be chosen from among those who have attained their doctorates. I refer to the so-called industrial fellowship established by such and such company at this or that university—a living stipend for the Fellow, reservation of publications by the university, but nevertheless certain prior claims in honor due as reports to the company. Though a number of such fellowships have been maintained throughout American universities in former days, this number has been more than trebled during the past year or two; a direct result of the war, as you may infer, and yet more directly a result of growth and advertising on the part of the specific industry. At bottom, however, the industries will eventually benefit by the further training of our young chemists, and if industral fellowships alone were to make this possible, then industrial fellowships there must be.

It is my purpose to criticize this purely industrial fellowship and offer a new plan which I believe will accomplish all that the founders of fellowships could ever hope for and at the same time give to each and every chemist, no matter where or how situated, an equal chance with every other chemist to do and to make or the betterment of himself and his profession. Possibly I ought first to characterize what I shall call the scientific stupidity of any, if such there are, who believe that a young chemist, as holder of a fellowship, is suddenly endowed with such powers as will enable him to assist any particular industry in solving some all-important problem. Were the holder of a fellowship of equal rank to a Doctor of Philosophy, as the average instructor, I still could not be persuaded that he had anything at all in his favor unless perchance he had first-hand and direct knowledge of the industrial side of the question before him. If, on the other hand, the fellowship is awarded to a mere graduate in chemistry or chemical engineering, he has no chance whatsoever of accomplishing anything worth while in shorter time than a less well-trained chemist could accomplish under proper guidance at the chemical plant itself.

TECHNICAL AND PHILOSOPHIC PROFESSORS

This is not an arraignment of our college graduates in chemistry. Latently they possess much ability and knowledge and will develop in good order when once introduced to chemical practice. They display, however, little evidence of chemical common sense; their minds function but not in unison with the reactions before them. Only practical experience with abundant responsibility will develop them effectively for serious work. This same adverse criticism is not alone applicable to the young chemist graduate. It is even more applicable to the average professor. Certain professors, the technical professors, who are indeed scientific artisans, have become well informed in matters of chemical industry and realize the importance of industrial procedure over recognized methods. The artless, however, the philosophic professor, is that class of man who never pictures to himself the conditions necessary for worldly success. It is extraneous in his make-up. To him processes in plant operation are merely large-scale laboratory experiments. I have yet to learn of a process developed in the laboratory which remained identical with its original set-up when once the process had been placed in operation in a plant. There may be one on record, but none has been reported to me from numerous inquiries of industrial leaders. How less likely then are the students of these philosophic professors ever to be able to cope with industrial problems. The technical professor naturally will be abundantly able to direct the students in proper style if he himself continues to keep abreast of the times, but this type of professor is so rare that we can hardly expect the result of his teaching to produce more than a ripple on the sea of chemical training for yet a few years. It is not my intention to belittle the work of the philosophic professor. I intend to show that it should be ideal. He ought not, however, to concern himself with industrial research. It is beyond his ken. How few, however, of these very philosophic professors will thank me for my words. They know; they comprehend; their vision is full; all is as glass. Bear with me in this invective, for remember I was once as such. It is this type of man one enjoys showing about a chemical plant. They converse learnedly, admire swiftly, condemn idiotically, and depart thinking they have learned something of the various steps in our processes. The technicist arrives and is aware of his ignorance. He departs and realizes he has yet much to learn. He is the kind though who knows that it is absolutely impossible for the greatest chemist on earth to learn much at a plant beyond what is scheduled for him to learn. There are some reported as still extant who think they can learn by observation alone.

ANALYSIS OF PRESENT STATUS OF CHEMISTRY IN AMERICA

As my theme concerns itself with the development of men into able chemists, it is manifest that this second type of university research policy—the graduate school—affords the greatest opportunity for broad and higher training and hence for betterment of chemistry in America. I shall confine myself therefore to this phase of the subject, and aim to show the actual interdependence of research in both scholastic and applied domains. In order that I may draw support for my constructive plan of research, I wish first to typify the present status of chemistry in America as visualized in both university and industry. I make bold, therefore, to introduce four premises and after their cursory examination we shall pass directly to my conclusions. Inwardly I delight in such procedure, for by these very premises I wish, as it were, to offer my critics their choice of weapons.

My first premise, as follows, will probably incite greatest furore: *There is scarcely any possibility of direct advance in industrial chemical enterprise through industrial research undertaken in a chemical laboratory of a university.* Five years ago there was every possibility of such an advance. Since then the transition is complete and the suddenness of it simply baffles our imagination. During this period of transition a number of men actually developed processes which stood ready for semi-plant operation. This eventuality may occur again sporadically but it will more than likely be the result of some enthusiast coming upon new ideas which he is particularly well suited to develop alone. The enormous strides recently made by the industries have reacted in fact upon these organizations and they have come into a full realization of the importance of constant and continuous research as a *sine qua non* for healthy and vigorous growth. The industrial laboratories are equipped for every phase of work attending their plant processes, and are fast approaching the most ideal conditions necessary for those new features which stand in contemplation. Every modern appliance is placed at the chemist's disposal; whatever he desires he has but to mention and the most costly contrivance is provided without a moment's hesitation. The thought of farming out, as it were, any new project of an industrial plant to a university laboratory is ridiculous in the extreme and the horrible thought of asking some outside party to conduct researches, to assist the plant with difficulties underlying any one of its valued processes, is utter nonsense—almost sacrilegious. The only possibility thus left to an outsider will be the investigation of minor points which in themselves are valueless but which, through more detailed and extended study, may appear in new garb and become of value when they again are turned over to the industrial specialists. A little outside investigation at little cost may save time and energy on the part of the really productive men; quite so, but I must soon demonstrate the criminal nature of this procedure.

Granted, however, that industrial fellowships are to be founded only for altruistic purposes and that they are to carry with them no ties whatsoever to influence the recipient to take up industrial problems, there remain yet two most necessary adjuncts—the fellowships must be awarded through some manner of examination and the holder must be subject to the rulings of the graduate schools. I do not hesitate to recommend a Board of Examiners who shall prepare questions and submit the same to possible candidates at certain stipulated times and places. The successful candidates will then select the university and type of work they hope to pursue. Any method of establishing such ideal industrial fellowships at this or that university because of some family ties or of some favored influence is not to be encouraged in this modern era. What chemistry needs is able men, what able men need are energy and enthusiasm. These cannot come into being through continuation of old, befogged ideas. The young men must seek out the real enthusiasts in the chemical world and together they will make for advancement. Thus, I believe, the young fellowship men should be given *carte blanche* to go to whatsoever university they would and seek the guidance of those men most highly esteemed in the scientific world. University instructors everywhere soon would picture to themselves that goal of goals: wherein they, through the publication of excellent researches, might be able to draw brilliant young chemists to their laboratories. With industrial fellowships thus constituted as a type of higher university fellowship I doubt if any can deny the highly beneficial effect this plan may exert, but this particular ideal industrial fellowship I have outlined does not seem to be the type recently launched. Indeed that form most frequently brought to our attention is an industrial fellowship founded for industrial ends, and, if perchance the industrial purpose is dormant, the fellowship is apparently allotted to a university with power to act. The baneful effect of this latter step may be gathered from my earlier remarks.

THE UNIVERSITY EVIL

In further corroboration of my first premise and distinctly as basis of my second, I must express myself on the evils that obtain at our universities and are operating in such manner as to retard the very advancement of chemistry in America. Do not think for a moment that I wish to enumerate all of the defects that may be apparent either here or there in the university world, but let me state my second premise gathered from many observers and fast becoming known to all: *The science of chemistry in the American university to-day is not keeping pace with its advancement in the industries.* Many of my hearers will scorn this remark, many will question my veracity, but none can disprove it. Of course, there are some outstanding advances in the realms of the pure science itself, but for every one of these I can cite two or three remarkable discoveries made outside the academic walls. That I may escape the brunt of possible criticism, allow me to read at once a recent statement by John Hayes Hammond: "Our universities do not lead in scientific thought, but usually follow. In mining, in engineering, and in nearly all of the professions excepting possibly medicine, where the union between teaching and practice is admirable, the universities are as a rule well behind instead of well ahead of current practice. It cannot be otherwise if the teaching staffs are in touch with current practice only by hearsay."[1]

This statement I assure you will be substantiated by the great majority of America's best chemists. But the cause is what I seek, and herein lies a university evil that escapes our earnest efforts to combat. This evil is briefly expressed. The university authorities do not recognize the absolute subservience of ability to enthusiasm and energy on the part of their teaching staff. The industries employ chemists of just as much and even more ability than the universities, but the industries prize those men who possess greatest energy and enthusiasm. These are the men who master almost superhuman difficulties and accomplish ends almost unbelievable. I will grant that this same type of men would play havoc within moss-covered walls. My critics, of course, will insist that my reference must chiefly concern mechanical questions, but can you cite for me any chemical process that is not in the main mechanical? I can reverse hundreds of processes which run apparently in one way in your laboratory because of your peculiarly small volume reactions within narrow ranges of temperature, pressure, and catalyst. The man who has taught chemistry for a long time and accustomed himself to think in certain time-worn channels and to operate in certain erroneous textbook methods of procedure is almost useless in a plant. His advice as to how the industries should handle questions is worse than useless. All respect him but his day is done.

DEGRADATION OF UNIVERSITY INSTRUCTOR

There is, however, at the university a type of man whose interests I have at heart. This is the young instructor, the newly fledged Doctor, but a man who has in him all that American chemistry needs for the future. He has ability, of course, and his energy and enthusiasm are growing in leaps and bounds. His reputation is all before him, but he is unfortunately human. The salary he earns is meager and it really cannot be otherwise, for you would not advise a sumptuous salary till he has shown his worth. Above all, he has highest hopes of achieving something and thus he should be given all the aid possible. Amid the difficulties that encompass this young instructor, the lack of competent student assistants to aid his researches, and the continual encroachments on his free time weigh more and more heavily. His advancement is found to depend upon so many factors that he soon comes to realize that the least important is his productivity in research; the most important, unfortunately, is his subservience to those higher up. It may be that the young man will seek to gain recognition through general service in university activities, or he will seek outside work more often in the capacity of analyst for industrial corporations. In both events his ability to accomplish the bigger things in chemistry is likely to atrophy and that is the end of him. What the young chemist needs is actual help and guidance on the part of the older men, and all the assistance possible heaped upon him. Encouragement possibly, in a word, will express my meaning, if the financial side is not overlooked, and where there is a likelihood of university trivialities making an inroad upon his time, the older men should guard him against the evil thereof. The young man's energy and enthusiasm is bound to carry him forward, but unless he is guarded on every hand his ability in the service of chemistry will soon be lost sight of amid the daily routine roundabout. Think of the constant interruption at a university in one's plans for investigation. Think of the childish meetings for discussions which may be far better handled, classified, and solved by an average clerk. Will the universities ever wake up to the importance of advancement in their several sciences and humanities to the utter banishment to Augean stables of all the silly nonsense that encumbers the many departments? Why permit this perversion of enthusiastic young chemists, with marked ability to accomplish something worth while, to the role of clerk and committee-man just because he has energy and a goodly nature withal. So soon as a university chemist is drawn into clerical work and begins his lifelong service on a committee for this or a committee for that, that moment his future possibilities in advanced chemical research will vanish. From a man of ability he has sunk into the hardly able class in the eyes of the chemical world.

Lest some one surmise that these statements arise from an overdose of committee work during my university service, let me set his conjectures at rest; during fourteen years' residence at one university I served on only one committee. I recall it well, it was a military committee of some sort. After a few meetings, at tea and crackers, I observed that one professor had a penchant for conversation and debate which eventuated in the elimination of all other views but his own. These same analytical results were obtained by a few of my friends on the committee, but out of respect for the president, board of regents, governor of the state, and others, they did not refrain as I did from attending subsequent meetings. Now, of course, I had just as much respect for all these dignitaries but I felt they had just as much for me and would not mind if I chose in preference the stinks and smells of my natural habitat. I did, though, sign the voluminous document—a report, I think, they called it—and actually did keep my promise to attend the otherwise soporific faculty meeting where it was read. I recall further that I was invited to serve on one other committee, but the only hour I could get free was 11 P. M., and that was too late for tea and crackers, which was over at nine.

No one can deny this frightful waste of valuable time among our professors at a university. There are exceptions, of course, but the general condition is exactly as I have portrayed it. I

---

[1] *Collier's*, January 10 (1920), p. 36

am forced to characterize this condition as a crime, but more often it is styled as the university evil. Many university men will tell you it is a part and function of university service. ' Those who really know will tell you it is a part and function of university non-productiveness. The condition naturally did have its inception in that period where a few men alone constituted the entire working mechanism of a university. But to-day when every university is organized more or less upon a rational basis, why are we cursed with this primeval practice? If we accept it as academic custom, then away henceforth with such obsolete custom. The cause of this evil is not far to seek, simply this: The non-recognition of ability as subservient to energy and enthusiasm.

THE TEACHER PAR EXCELLENCE

I must not, however, overlook the teacher par excellence. In the college and high school, his position is paramount and rightly so. Teaching for itself alone is his aim in life. Through close association with productive minds, he may reflect in classroom the best spirit of the day. His service in colleges and college departments of universities will ever command the greatest respect and appreciation, but in the graduate and technical schools of a university, as the seats of highest learning, he can play but minor part. The greatest inspiration to wide-awake students will ever come from the productive professor no matter whether he lectures or rambles in the classroom. In the ideal university, therefore, the non-productive professor, usually of the leisure class, will best devote himself to service in clerical and curule capacity outside of the four to eight actual hours per week required of him in university teaching. The ideal professor thus freed from detail and retarding influence of his non-productive colleague could then march steadily forward to the glory of himself and the university. Though these ideal conditions are far from realization, there are many professors who actually accomplish much of value in the realms of advanced thought. You will ever find them among those who shun this routine side of university life. Imagine, for example, the title of the clerical professor suddenly changed to read Chief Clerk of Committee on Attendance. How quickly he would resent this correct appellation, and yet this is his true category. He masquerades only under the title of professor and, due to his inactivity in what he professes, chemistry to-day at the university is no longer keeping pace with its advance in the industries.

IDEAL CONDITIONS FOR RESEARCH

If this deplorable condition is what we desire in our universities, then there is no point in my discussion. I am convinced, however, that the university is the proper place for research in pure chemistry. The conditions at the university which should make for quiet, undisturbed study, and rich and abundant association with minds alert in chemical and allied sciences, offer the very best opportunities for the development of the true chemist. The young man develops thus in the broadest manner and his outlook is characteristically broad. His subjects for investigation should be purely scientific and not necessarily commercial. The industrial chemical problems ought not to be overly emphasized in the earlier years. The closer the young man keeps to the pure science itself the better for him, the better for the universities, and the better for the industries. When, later, he may choose to enter industrial work he will find his training none amiss and his breadth of view most highly serviceable. It is here that I must firmly maintain that those young men who jump into industrial problems too soon after graduation have little or no chance of broadening their general knowledge of chemistry. The tendency to specialization is too well marked even in the industrial world. Everywhere you will meet with young men who realize this all-important fact. Their opportunity for broader development is past. It is not likely to return. Discouragement is easily discernible in their features. Had they but known of some plan upon graduation which would have offered a chance for pure research with a real live hope of remuneration, that chance would have been seized forthwith. Whatever the result, the better self of that future chemist never could deprecate his early ventures, and corresponding content would characterize now his daily endeavors.

Through the planning of researches and publication of results the science as a whole will advance. Chemical self-confidence is obtained no more readily and decisively than by publications, for these stand out as theses which the author is ready to sustain before a world of chemists. Indeed, this side of research may after all appeal so strongly to the young man that he will choose to continue it in the university setting. His gradual rise to a professorship with research ever his goal will inaugurate an era of greatest chemical activity in America. We have at present all too few of this type, so few that they are almost lost amid the multitude. I have never been able to see any other form of investigation in chemistry than that of pure unadulterated research, for itself alone, with no thought of service or utility, as anywhere near so broadening in its influence upon the student's development. The university is, of course, the proper place for such studies and I deduce from the considerations above my third premise: *There is the greatest possibility for advancement in pure chemistry in the investigations undertaken in a university or endowed laboratory.*

It should be clearly emphasized that I am not criticizing the research at special institutions and at industrial research laboratories. The young chemist graduate, however, when situated in these positions has not the freedom to browse about to his liking and strike out where his fancy leads. The atmosphere of the university almost overstimulates this freedom, but the results cannot be other than encouraging for the future of the science.

Upon the three premises already discussed my fourth premise finds support and may be stated as follows: *The industries will profit most in a general way from the published investigations in pure chemistry issued from the university and endowed laboratories.* Naturally, the industries will profit by their own advanced researches. The scientists though who conduct these researches must ever keep in touch with advanced thought. New ideas and new methods as elucidated from various investigators are seized with greatest zeal and thus almost a direct good results to the industrial organization through the advances made in the pure science. So firmly am I convinced of this that I feel it my duty to draw attention of chemists everywhere to the golden opportunities that manifest themselves for furthering this devotion to pure chemistry. They will not develop by themselves. The road is too difficult and encumbered with many hazards. My conclusion, therefore, to the four premises I have drawn is simply this: *The universities, the industries, and American chemists must encourage the further study of pure chemistry by the young graduates of our colleges.*

MONETARY REMUNERATION FOR SCIENTIFIC CONTRIBUTIONS

I have consumed so much time in the statement of basic troubles that I shall merely outline the plan which I believe is adequate in all respects. I would have the royalties paid to authors for books and magazine articles paralleled in some form for those who publish original chemical contributions in our representative chemical journals. These contributions need not be confined to any one branch of chemistry. They should be all-comprehensive. The only criterion for consideration is their mark of originality.

In detail I should suggest that the three most interested parties contribute in every way possible to encourage chemical research. Thus, a group of universities, a group of industries, and the AMERICAN CHEMICAL SOCIETY might share in this contribution. It cannot be assumed that these three interested groups will contribute equally, but each in itself may assist directly in the remuneration which I believe the contributors should receive. First and foremost, the several Boards of Editors of the AMERICAN CHEMICAL SOCIETY should be willing to serve as judges upon contributions received. When an article is accepted, the author or authors should receive at once an immediate award or honorarium. The amount possible must of course depend upon the resources at hand and, more directly, upon the size of the endowment which we hope can be established in the SOCIETY for this purpose. Naturally without such endowment, the SOCIETY could not encourage expenditures in this direction. From the university in whose honor the contribution was made, the author cannot expect to receive any monetary remuneration other than the continued supply of needed resources for actual work. Public recognition, however, of each contribution should freely be accorded but the university will best honor itself and the young authors by establishing a title of honor directly attainable upon publication of some ten to twelve researches; thus such title as "Assistant Director of Research" would amply gratify the young man who was working for the advancement of science. Their former title might still stand as a "Fellow," "Assistant," or "Instructor," but when it is actually possible to earn a title by real work, you can appreciate how much more valuable the entire framework of a university career would loom up in the eyes of thinking men. After another ten to twelve researches, the contributor may well be given the title of "Director of Research." The overabundance of non-productive chemists in our universities would, I assure you, most clearly be demonstrated by such a procedure. Naturally, our chemical drones will object to these suggestions, but I doubt if any university will weigh their objections seriously. They have so little influence in the chemical world that they must sooner or later lose all influence in the academic world.

INDUSTRIAL ORGANIZATIONS WILL FINANCE PROPOSITION

The real financial remuneration for the articles accepted must come, undoubtedly, from some industrial endowment. It would seem best to make only a portion of this subject to award on the part of the AMERICAN CHEMICAL SOCIETY. The larger portion should remain intact and should be subject to award by a committee properly elected by the industries concerned. Upon notification by the Boards of Editors of the AMERICAN CHEMICAL SOCIETY of the award of an honorarium to some author for a contribution of original research, this industrial endowment committee should award the author a proportionately larger sum and thus the findings of our editors may be made the criterion for the second allotment. For example, let us suppose the editors voted an honorarium of one hundred dollars from their share of the industrial endowment. The industrial endowment committee might then be able to quintuple the award and thus six hundred dollars could be sent to the author at once. This is, I assure you, none too small a recompense when we consider the annual royalties paid by publishers to authors o our second and third rate textbooks.

RESEARCH MEN DESERVE BEST REWARDS

The critics of my plan will naturally jump at the opening I leave; namely, monetary award for scientific endeavor. "The honor of contributing to our JOURNAL should suffice alone as recompense." To this I can answer now but briefly: You speak of by-gone days. Here and there a young man will turn his fancy into research with little thought of to-morrow; the best men, the live men, the men who have the mental grasp for bigger things, will not tarry too long where life is difficult and honor shallow. A world of endeavor surrounds us all and we must join in the quest or remain as useless adjuncts of a forgotten past. No matter how our philosophers may wail, they can no more change the trend of endeavor to-day than a firefly can change the temperature of a mighty iceberg.

Young chemists everywhere may here find a really practical way in which to devote themselves to research. They must have some incentive else only the weak will be left to carry on the university work. The strong may just as well develop in one direction as in another and the plan I offer is simply one to induce the best types of minds to try their fortune at pure investigation for itself alone. Our journals accordingly should overflow with good material. But I can well imagine the thunderous protests that will resound from pseudo-chemists who will fear gravely that a mass of worthless material may find its way to publication. Our editors, however, are chosen for their ability and are representative of the best American chemists. I should not lose confidence in them. Just let the pseudo-chemist contribute a trifle now and then himself. It is usually his type who will find the most worthless data. What we wish is everything that savors of originality and I assure you the mass cannot be too voluminous to bother for a moment our industrial specialists. If we must wade through oceans of data, the mere chance of finding an isolated and much-valued pebble on some far-off beach will well repay us. According to this plan, you will note how both instructor and professor, how both college graduate and assistant come into the same consideration. The title of the chemist is supernumerary, the results only will count. Possibly the older professor may be awakened to the opportunities that confront him and ideas almost dormant may be brought into the light of day. Certainly the young graduates will find herein an excellent means for further development. The assistantships at a university will suddenly be enhanced in value, for these positions can well be held by men intent upon research. The small livelihood which an assistantship affords will be amply sufficient to start the young men on their course. In fact, it is to be hoped that a greater number of assistants in our laboratories can be honored by an opportunity to work under the direction of productive men. There is nothing so demoralizing about a chemical laboratory and nothing so detrimental to the spirit of research therein as the presence of an aimlessly drifting assistant. If all assistantships could be offered primarily to young investigators, I feel confident there soon would be need of devising some scheme for receiving and weighing applications from young men intent upon pursuing advanced work at the various universities. With increased competition in selection of graduate assistants who actually are more effective than most professors in laboratory teaching, the overabundance of dead professorial timber may finally be eliminated, to the good of the university and of the science.

Just how far this plan ought to be extended, if ever put in practice, would be a matter left to the endowment committee. Though I have had only the young assistants and instructors in mind, I see no reason why it could not be equally applicable to the full professors. Certainly they should receive a monetary recompense for their services—no matter whether or not the old, moss-grown ideas of honor alone as recompense still obtain in their circles. The entire plan appears, upon closer analysis, to be the counterpart of that ideal industrial fellowship heretofore described as a fellowship nowhere existent but one that might be granted to young chemists through competitive examinations and then placed in operation at whatsoever university the fellowship holder selects. Such ideal industrial fellowships naturally entail the services of an examining board for the applicants and this board indeed might serve for many industries. My plan simply eliminates all examinations and accepts the findings of the several Boards of Editors as a final criterion of merit. The recompense may thus be construed in the same sense as that for an ideal industrial fellowship—but with a distinction that is distinct; namely, the money is paid only upon the accomplishment of something worth while and not thrown around helter-skelter among doubtful candidates in pleasantly located laboratories, hoping for something to happen.

This plan of reward for meritorious research comes now as a further bestowal of honor upon those fellowship holders who would offer original contributions to science. Throughout my discussion you will find this plan—the plan of incentive—ever adds to the good things that are and detracts from the bad that persist. The system certainly offers possibilities far beyond what I have pictured.

I see in this plan no adverse turn. It should accomplish what universities have long desired. It should instill in the young men that desire for research which teaching alone is slow to impart. It should open to industrial scientists the possibility of further study in special fields under the direction of some prominent university man. Through this advance in the study of pure and applied chemistry, the entire chemical activities of America will march forward in steady progress.

In substantiation of my selection of the three most interested parties, I scarcely need explain the interests which representative universities will ever take in problems of research; technical schools and institutes of research naturally fall into the same group. Industrial organizations already realize the importance of scientific advance as directly stimulating their activities and instilling new spirit into their investigations. In a far broader sense and with correspondingly greater responsibility do I view the interests of the AMERICAN CHEMICAL SOCIETY in the advancement of research in America. Having grown with prodigious strides into the greatest society of its kind in the world, is it not fitting that we launch some project that will return to port laden with scientific production—the most valued treasures in this world of progress? To this end, I earnestly hope our several Boards of Editors will consider my proposal in its several aspects—more poignantly from the standpoint of increased duty in judicial capacity. If our Directors and members of the Council will then approve the expenditures of money to this purpose, I assure you the industries will gladly espouse a cause so heartily endorsed by these several groups of scientific men. Many universities, I believe, will aid in every way possible a step that will make for the enhancement of all those lesser positions now so poorly filled. Any reward of honor naturally will come slowly, but I believe it will come surely.

The universities should lead in scientific thought and endeavor. The difficulties in chemical art should best find their solution in university circles. Thus the universities may be dependent upon the industries for the revelation of reactions so little understood, and again the industries should ever be dependent upon universities for leadership both in scientific development and in the broadest intellectual development of the young college graduates. Both university and industry must benefit through mutual progress, and as thus interdependent may they ever be of greatest assistance the one to the other.

THE RELATION OF THE CHEMIST TO THE UNITED STATES PHARMACOPOEIA[1]

By Charles H. LaWall

CHAIRMAN, REVISION COMMITTEE, U. S. PHARMACOPOEIA

The United States Pharmacopoeia this year celebrated its centenary, the first issue of the work having been published in 1820. It is the second oldest national Pharmacopoeia in the world, the only older one being the French Codex, as the corresponding work is called in France. It was originated by physicians, and the statement of the reasons for its beginning made in the preface of the original 1820 edition is essentially true to-day, as will be seen by the following quotation:

> It is the object of a Pharmacopoeia to select from among substances which possess medicinal power, the utility of which is most fully established and best understood, and to form from them preparations and compositions in which their powers may be exerted to the greatest advantage. It should likewise distinguish those articles by convenient and definite names, such as may prevent trouble or uncertainty in the intercourse of physicians and apothecaries.

It was planned at the beginning to revise it every ten years, and this has continued up to the present time.

In the 1830 edition the help of apothecaries was acknowledged, and in 1840 the pharmacists were requested officially to aid in the revision of the work. When the first Pharmacopoeia was issued in 1820, there were no separate schools of chemistry or pharmacy and few chemical manufacturers. Most pharmacists were chemists and some of them like Scheele, Vauquelin, Pelletier, Caventou, Serturner, Derosne, and others, had made discoveries of moment.

The earlier editions of the Pharmacopoeia, therefore, contained a large number of methods for preparing chemical substances now manufactured only on a large scale and never at present prepared in the small way for commercial purposes. Among these may be mentioned:

Citric acid—from lemon juice.
Ether (then called sulfuric ether)—from alcohol and sulfuric acid.
Water of ammonia—from ammonium chloride, lime, and water.
Ammonium carbonate—from ammonium chloride and calcium carbonate.
Silver nitrate—from silver, nitric acid, and water.
Bismuth subnitrate—from bismuth, nitric acid, and water.
Mercuric chloride (called oxymuriate)—from mercury, sulfuric acid, and sodium chloride.
Mercurous chloride—from mercuric chloride and mercury.
Sodium bicarbonate—from sodium carbonate and carbon dioxide.
Zinc oxide—from metallic zinc by oxidation in a red-hot crucible.
Processes for manufacturing morphine and quinine were also included in some of the early editions.

These will indicate that the pharmacist of those early days had to possess chemical knowledge and manipulative technique of no mean order. There was little or no attempt to standardize substances except in such a crude way as is shown under hydrochloric acid, then called *muriatic acid*, in which it is stated that "a fluid ounce should dissolve 220 grains of carbonate of lime."

The usefulness of pharmacists upon the Revision Committee of the Pharmacopoeia was so apparent that by 1880 they had begun to predominate, a condition which has continued down to the present time.

The United States Pharmacopoeia of 1880 may be said to be a turning point in its history, for by this time manufacturers supplying chemicals of all kinds were common enough to enable some of the processes for manufacturing chemicals to be dropped and to require more thorough consideration of scientific standardization. The change then begun has developed until at the present time the Pharmacopoeia is largely a book containing the approved nomenclature, descriptions, and standards of substances which are used in medicine and the processes are limited to those mainly for galenical preparations, *i. e.*, the liquid preparations of vegetable drugs, such as fluidextracts, tinctures, etc., and also for the simpler chemical solutions.

The personnel of the Committee of Revision has likewise changed—originally composed of physicians only, later of pharmacists and physicians, the committee of fifty who revised the tenth edition (ninth revision) in 1910, which is official until the work of the convention meeting in May 1920 is completed, included physicians, pharmacists, chemists, biologists, pharmacognocists, and other workers in specialized lines. Inasmuch as nearly one-half of the 782 official substances may be classified as chemical substances, there is a comparatively large group of chemists on the present Committee of Revision. I believe that over 20 per cent of the fifty members are members of the AMERICAN CHEMICAL SOCIETY and several of these have been prominently identified with that organization for years. The United States Pharmacopoeia is not a work of interest solely to physicians and pharmacists. In the Federal Food and Drugs Act and in the corresponding laws of many states, its standards are recognized as having legal authority.

---

[1] Presented at the April Meeting of the Philadelphia Section of the American Chemical Society.

The business management of the Pharmacopoeia is vested in a Board of Trustees of seven members who, with the Committee of Revision of fifty members, are elected decennially from the delegates who assemble in Washington on the second Tuesday of May in the years whose figure ends in a cipher. These delegates constitute a convention in whom the ownership of the Pharmacopoeia is vested and which is now a chartered body. The delegates mainly come from medical and pharmaceutical colleges and societies, but the AMERICAN CHEMICAL SOCIETY is one of the organizations regularly authorized to participate, as are also the various departments of the United States Government whose work bears any relevancy to medicine or pharmacy.

WILLARD GIBBS MEDAL AWARD

Brief mention was made in the June issue of THIS JOURNAL of the presentation of the Willard Gibbs Medal to Dr. Frederick G. Cottrell, recently appointed Director of the U. S. Bureau of Mines. It gives us pleasure to reproduce below the admirable address of the recipient of the medal, and Dr. Whitney's remarks in presentation.—[EDITOR.]

PRESENTATION ADDRESS

By W. R. Whitney

Dr. Cottrell, in presenting to you this medal on behalf of the Chicago Section of the AMERICAN CHEMICAL SOCIETY, in memory of the great Gibbs, I am not going to recite a list of your well-known achievements. This is not merely because these achievements are so well known, but because I prefer to preach a brief sermon.

I choose to call attention to your *process* rather than your product. I believe that the *way* you have done things is even more important than the actual things themselves. Our country might get along without the particular products of your effort, but it could not get along without men personifying your procedure. We are not so much interested in recovery of fumes, the abatement of the smoke nuisance, in the saving of silver, zinc, or sulfuric acid, in the production of helium, or any other of the definite undertakings which are attributed to you, though these are in themselves very valuable, but rather we exalt that human *quality* which insures advanced undertakings of public welfare and interest. You are one of those who intelligently decline to proceed along entirely old paths but map out and push along new ones. You are one who, no matter how wisely you may act, must withstand many rebuffs and disappointments, because, in blazing new trails, countless obstructions must be encountered and surmounted. Herein you have always retained your momentum and your good nature. All our improved ways of living, our broadened views, our greater activities, and our increased pleasures are due to new and difficult steps, often seemingly absurd when judged by us who are impelled by habit only. In tilling new ground and in opening unbeaten paths you have needed your well-advanced scientific education for keeping high your coefficient of success; a splendid personal quality of open-heartedness and a carefully controlled physical constitution. All of these you have clearly and unselfishly devoted to the public good. You have not been satisfied to accept things as they were, and we chemists highly appreciate ideals and those new ideas which, while not at first acceptable to the average man, can be made so by human effort. Your support of one universal language may be looked at as only a few years too early, but it is a type of the thing for the realization of which people will have to depend on someone like yourself, if it is ever to be realized. Your work in the West and your radical undertaking in the East, in establishing the Research Corporation, together constitute a trail blazer which calls attention to the possibilities of coöperative research and to the position of our Government in relation to ideas and inventions of its employees. You have started something which may well grow far beyond your anticipation. You have set an example not only in accepting the responsibility of such public work, but your patient persistence in carrying out your own plans with support which only your well-known integrity, industry, and enthusiasm could insure, forms a new high plane of activity for the emulation of the American chemist.

We are glad that you have been added to that group of chiefs of the Bureau of Mines, Holmes and Manning, which our Government has so fortunately possessed. We will be willing to forgive in you many possible errors or mistaken undertakings in the future, if your ideals and your will to be right and the intuition to be constructively dissatisfied with imperfect existing conditions, persist as they have hitherto existed.

MEDAL ADDRESS
INTERNATIONAL SCIENTIFIC RELATIONS

By F. G. Cottrell

The experiences of the war have served to illustrate as never before the importance of the position held by the scientist both pure and applied, not only to our daily life at home, but also in international affairs, and during the war perhaps no branch of science carried a more important load or gave a better general account of itself than did chemistry in all its various branches. The details of chemical war work have filled a good part of our journals for so long that to dwell on them here would be bringing coals to Newcastle; but what the chemist's part is to be in the reconstruction period upon which the world is entering still lies before us and has a most legitimate and insistent call on our serious attention.

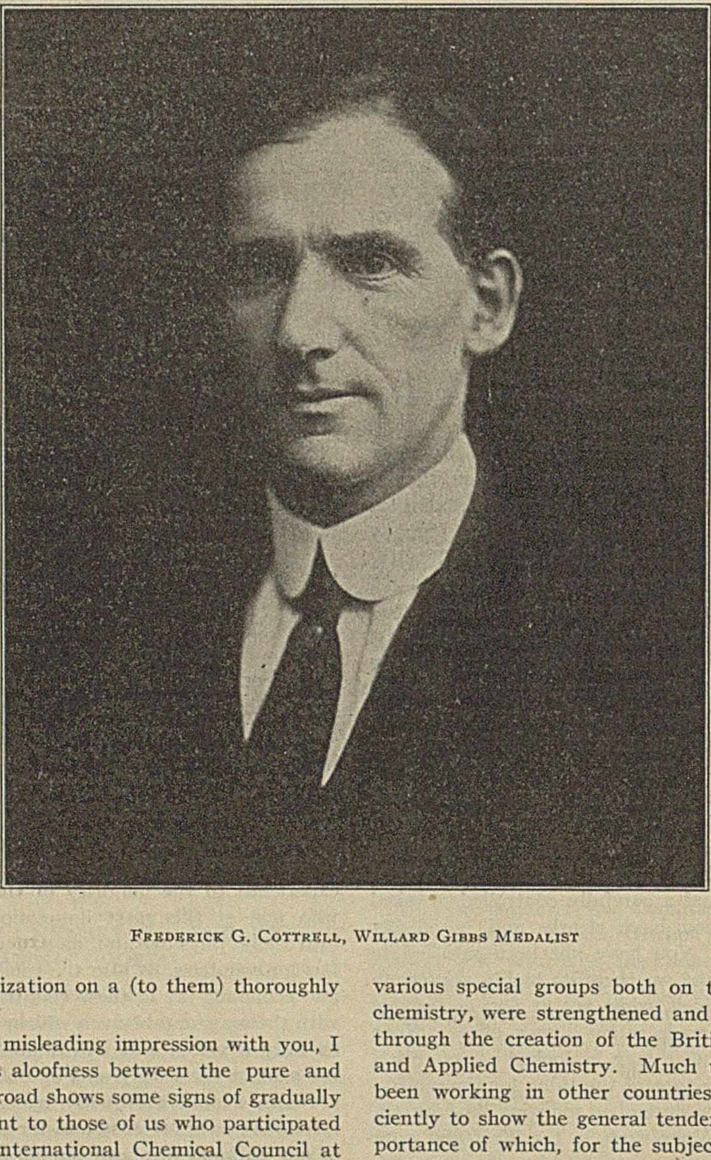
The war certainly furnished a great stimulus to discovery and invention, but from the standpoint of technical achievement it bids fair to have served only as the prelude to a vaster industrial development of truly international character which may reasonably be expected to follow in its wake. The stress and strain of the period we have passed through has awakened the imagination of the rising generation to a world consciousness as nothing less far-reaching could possibly have done, and we in this country are destined perhaps even more than those in Europe itself to feel the full effect of this swelling pulse, because to the European nations international contacts have long been matters of course and from a physical aspect almost as natural as intercourse among the states of our own Union. But this was a new experience to the majority of the 4,000,000 young Americans who just at this most impressionable age have been thrown into daily physical and mental contact with their fellows of foreign countries, as also the conditions and traditions of home and economic life in these countries. They have brought back with them a mental leaven which, though it may take some time to fully develop, and will undoubtedly require careful guarding against the wild yeasts of Bolshevism and other extraneous growths, should eventually make itself clearly felt as a tremendous power for good and for the broadening of the concepts of our national life both human and economic.

INTERNATIONAL RELATIONS FOSTERED

Chemists in this country, as a class, have recognized and fostered international relations probably as much, if not more, than have most of the technical and engineering professions. This is perhaps due in no small part to the very close and cordial coöperation which has existed from the first between pure and applied chemistry in this country, for it is usually easier to get perfectly free international coöperation in the pure sciences than in applied branches where questions of business expediency at once intrude themselves. But the chemists in the past have been remarkably successful with their undertakings in both fields; as, for instance, the International Committee on Atomic Weights on the one hand and the International Congresses of Applied Chemistry on the other. Even the primarily social side has found its expression in the systematic exchange of courtesies between such organizations as the Chemists' Club in New York, which has come to be really a national rather than local institution, and the more recently established Chemical Industry Club in London, while beyond there is already the foreshadowing of a similar club among the French chemists.

The fact that the Chemists' Club in New York avowedly embraces in its membership all fields of chemistry, both pure and applied, while even in its name the London club emphasized the distinction between the academic and the industrial phases, emphasizes a rather clearly marked difference in point of view between chemists in this country and abroad.

In this country we have come to accept so tacitly as almost axiomatic, the close coöperation of these two divisions of the profession, that we may easily fail to appreciate how questions of this kind appear to our friends across the water, and the difference in historical background underlying this. Yet if we are to work most effectively with our foreign colleagues we must acquire a sympathetic understanding of their points of view, whether we entirely agree with them or not, and where we do not agree let us hope we will restrain ourselves sufficiently to avoid unduly adding to the partly justifiable and now rather widespread impression among Europeans that so many Americans feel themselves thoroughly competent both from experience and native ability to come over and quite *de novo* reorganize European civilization on a (to them) thoroughly satisfactory basis.



Frederick G. Cottrell, Willard Gibbs Medalist

Lest I leave an unfair and misleading impression with you, I should perhaps add that this aloofness between the pure and applied sides of chemistry abroad shows some signs of gradually abating, as was clearly evident to those of us who participated in the organization of the International Chemical Council at the meetings in Paris, London, and Brussels last year.

A significant illustration of this is also to be found in the history of the Société de Chimie Industrielle de France organized during the war. At the outset the newcomer was looked upon with a good deal of anxiety by the old established Chemical Society of France, which naturally and historically represented primarily the academic side of the subject and was controlled largely by chemists in academic positions. Their chief fear was apparently that the creation of the new society and journal would only serve to scatter and dilute over too wide a field the meager funds available to the chemists as a whole for organization and publication purposes, and thus weaken the entire structure. As a matter of fact, however, the new society and its journal "*Chimie & Industrie*" have, under the able and broad-minded leadership of Paul Kestner, the society's first president, prospered from the very start, and, instead of dividing and weakening the support of the older society, have brought in such a wide range of new interests and given such stimulus to French chemical industry in general that both societies are profiting by it.

Those responsible for the guidance of the new society have definitely and officially taken the constructive and liberal stand that since pure science is fundamental to industrial science, but the latter is naturally more closely associated with the material returns and with the interest of the public generally, it has a moral responsibility to help the academic organizations to maintain their high standards of scientific work and independence of industrial considerations.

Due to entirely different traditions and circumstances existing in France, it would not be easy or probably desirable, at any rate for the present, to merge the two branches in one large society and single group of journals as we have them in this country, but the French are rapidly working out their own methods of close and cordial coöperation which will undoubtedly fit their needs as well as our form of organization meets our own.

Great Britain, as you well know, has had her strong Society of Chemical Industry for many years and there has long been a fair degree of coöperation between this and the Chemical Society of London as well as other academic groups, but as a result of war emergencies these, as well as other bonds between various special groups both on the pure and applied side of chemistry, were strengthened and to a large extent centralized through the creation of the British Federal Council for Pure and Applied Chemistry. Much the same processes have also been working in other countries, but the above serve sufficiently to show the general tendency of the day, the great importance of which, for the subject in hand, lies in the greater facility which these broadly comprehensive national organizations furnish for effective international coöperation.

PROTECTION AGAINST MISUNDERSTANDINGS

If international coöperation in science can be made effective in both the pure and applied branches it will unquestionably furnish one of the most powerful forces possible against the growth of national misunderstandings. It is, of course, easier for the devotees of pure science to work freely together than for those more closely associated with the business relations of their subjects.

Secrecy, however, even in business relations, if not disappearing, is at least rapidly coming to be viewed generally from a very different standpoint than heretofore, and it is interesting to note how this change began and first received official recognition on the distinctively scientific and technical side of business, *viz.*, in the matter of patents of invention, the government offering the inventor certain definite legal regulation and protection in lieu of his secrets if he would make these latter frankly and fully known to the public. At the time our patent system was created, few, if any, would probably have been found who could have clearly seen foreshadowed in this even as a distant possibility, the necessity or the justification for the publicity of purely business details of railroad operation, with which the Interstate Commerce Commission has since been charged. This step, again, was recognized in the special case of the transportation companies and other public utilities corporations long before people were ready to consider the extension of similar ideas to other lines of business. Thus the creation of the Federal Trade Commission came many years after that of the Interstate Commerce Commission, though in the broad economic sense the greater part of the work of the Interstate Commerce Commission, as well as many questions of patent administration now left chiefly to the courts, logically constitute only special branches of the general subject for whose study and guidance the Federal Trade Commission was established, *viz.*, the maintenance and safeguarding of fair trade practices, including the regulation of monopolies in the public's interest.

The fact is that as the civilized world progresses and we accept the benefits of that developing civilization, we are less and less able to live to ourselves alone. What was so nearly a man's purely private affair when his business was small, becomes with the growth of that business itself so much more largely a public concern that we may very properly recognize the need eventually for a certain readjustment in the distribution between the individual and the public both of interest in, and responsibility for, the conduct of that business.

These same problems must eventually enter in principle at least into the consideration of the international questions which are rapidly being forced upon our attention. Fortunately, however, very few of these latter are apt soon to reach a stage where detailed legislation must be undertaken, but in working out the corresponding national phases of the same problems we should at least have in mind the desirability of building up an intelligent public interest in the international aspects of the subject.

RESPONSIBILITIES OF THE SCIENTIST

The order of approach in the international phase is apt to be much the same as in the national, which again inevitably places special responsibilities upon scientific and technical men. Thus, at the meeting of the Engineering Section of the International Research Council in Brussels last July, the chief subject of discussion was the possibility of working out some form of international patent which would gradually tend to unify the present wide divergence in patent procedure among the different countries and also to lighten the burden both of detail and expense upon the patentee. The opinion was pretty clearly brought out at the meeting that it would probably not be practicable even if desirable to secure any essential changes in the patent laws of the individual countries as an immediate result of such a program, but that the effort should be made rather to secure a central international office where the specifications and claims might be gotten into a standard form acceptable to all of the countries and the novelty and patentability of the invention or discovery determined as far as possible by an examination somewhat similar to that made in our own patent office, but of wider scope with reference to foreign laws, publication, and practice. This would then be filed in the countries selected and would be subject in each to the laws governing patents therein. It was hoped, however, that all the countries would coöperate in working out the procedure of the central office and that this in turn would have a tendency to react upon the development of their own patent system, at least in so far as gradually adopting reciprocal relations in regard to the treatment of these international patents.

It was generally felt that coöperative international effort of this kind would have a very stimulating and salutory effect on the patent system of the individual countries involved. Here again, this should be looked upon as a matter of interest, not merely to the technical man and the inventor, but one in which the academic scientist should also take a part. The spirit of our patent laws clearly holds that a patent to be valid must be a true and full disclosure of the novel discovery or invention claimed, but it is too often the evident intent of the patentee or his attorney to give only as little real information as he feels is absolutely necessary to fulfill the letter of the law. If more genuinely scientific work were published by scientists of standing and ability in the form of patents, even if also published elsewhere, it would have a very much to be desired effect in raising the general tone and standard required in this respect in patent specifications; for patent examiners and courts are after all human, like the rest of us, and we are all influenced, if not even compelled, when placed in a judicial position, to set our standards with some reference to the average of the material available.

SPREAD OF THE METRIC SYSTEM

Turning for a moment to the matter of standardization of weights and measures: This is, of course, one of the oldest and best recognized fields for international coöperation, and here again the scientists have led the way. The use of the metric system is now essentially universal in pure science, and is making good headway in the newer branches of technology; such as chemical and electrical engineering, and to a considerable extent in mining and metallurgy, but in mechanical and civil engineering, especially where these touch, respectively, land surveys and diversified manufacturing, the greatest opposition to change is naturally to be found. While my whole training as a chemist naturally gives me the strongest possible prejudice in favor of the metric system, and, viewed from the international side, I see nothing else in the field having any chance of final world-wide adoption, it yet seems to me that in this country at least for the present, reform should still be sought more through education and example than by resort to mandatory legislation. We already have legislation permitting and encouraging the widest possible use of the metric system in all its details throughout the whole country. It must be remembered that mandatory legislation touching as broad a field as this, and so ramifying into every detail of the daily life of every individual, can only be really successful in attaining its object if its enactment is preceded by a thorough awakening of interest in the public mind and a real appreciation of the question at issue. This statement should not be construed to mean that the present rather widespread agitation for mandatory legislation in Congress on the use of the metric system is producing no useful result. On the contrary, I am inclined to think that entirely aside from whether any of the legislation sought is eventually secured, the effort expended will be beneficial by maintaining a focus of public interest and discussion upon the whole problem of international units and standardization of technical equipment generally.

The metric system goes a long ways toward international standardization, so far in fact for the purposes of the chemist that he is apt to overestimate its relative significance in, say, the field of mechanical engineering, where to-day probably the greatest inertia, not to say, opposition, exists with respect to its general adoption. The chemist is dealing for the most part merely with quantities of matter in bulk. He could change his weights and measures and go over in almost any chemical factory from any other system to the metric, and *vice versa*, almost overnight, without a single change in his machinery except for the scales and measuring appliances themselves. Whereas, if his master mechanic were forced to do the same thing with regard to all his tools and repair parts, it would mean practically a complete rebuilding of the factory. Nor would the mere general adoption of the metric system completely solve the mechanical engineer's problem of international standardization. Take as fundamental a matter as screw threads. Each country, and one might almost say, each industry, has adopted its own shape and standard pitch. With this, and many other similar discrepancies staring him in the face, is it any wonder that the manufacturer of mechanical equipment has a tendency to view the metric system in its present stage of development as only one bite at the cherry, and to ask himself whether, in view of the changing of all his patterns, and jigs, and machine equipment in this one respect, when there are others just as important awaiting later consideration, it is not like cutting off the dog's tail by degrees?

GENERAL INTERNATIONAL STANDARDIZATION

Do not misunderstand me as arguing against the adoption of the metric system, but rather for a wider and more comprehensive application of its fundamental conception of general international standardization, even if in so doing, our progress for the moment may seem to slacken somewhat.

What is needed most is a wider, stronger, and more closely interwoven network of international, scientific, and technical organizations, working earnestly for better understanding and coöperation between all concerned for the purposes of the common needs and common good of humanity as a whole, and fortunately, the spirit of the times gives good promise of real and rapid progress in this direction.

DEVELOPMENT OF AN AUXILIARY LANGUAGE

In his introductory remarks Dr. Whitney made reference to my interest in the problem of an international auxiliary language. Even here we have an interesting illustration of how prominent a part the physical scientist has played in international projects even so far removed from his own direct field as the problem of language would seem to be; for closely linked with the history of the Esperanto and Ido movements we find such well-known names as Lorenz, Bourlet, Ostwald, Ramsey, Donnan and Morse, while last year in London and Brussels it was among the members of the chemical group that the project first took shape to secure the appointment by the International Research Council of a committee "to investigate and report to it the present status and possible outlook of the general problem of an international auxiliary language" and "to coöperate in its studies with other organizations engaged in the same work."

This has already led to the authorization of similar committees by the British and the American Associations for the Advancement of Science, as well as a definite movement in a number of our leading universities to take up the problem not only from the purely linguistic aspect but even more particularly from the side of sociology, psychology, and pedagogy, since as a matter of research these latter departments find a more fertile and interesting field for study than do even the linguists. In fact, if we are to have synthetic linguistics as a seriously recognized subject we must probably develop a new type of linguist for it, just as the coming of physical chemistry necessitated the development of a new kind of chemist. Why, it may be asked, should the scientist take more interest in this problem than the linguist? For answer, one may point to the development of stenography and ask where it would be to-day if it had been left to the linguist, for the fact is that the present type of professional linguist is interested almost exclusively in either the philological or the literary aspect of his subject and looks upon its purely utilitarian developments from much the same point of view as the extreme type of academic research scientist does upon the routine of applied technology.

What the scientist, and for that matter the business man and the man in the street, wants for international purposes is primarily not a literary language but a simple and serviceable code, bearing somewhat the same relation to the ordinary ideas of every-day life that the formulas of the chemist or the equations of the mathematician do to these specialists' own domains of thought. As a matter of fact, scientific terms are already practically international and so all that remains to be done is to bring the simpler and non-technical half of our concepts up to the same international standard of expression.

Here, too, we must again emphasize the auxiliary character of such a proposed international code language, since it is in no wise intended to compete with any national language except for purely international purposes, where the only desiderata are simplicity, clearness, and entire neutrality. Herein as a purely auxiliary tool it escapes completely from the type of opposition already discussed in connection with the compulsory adoption of the metric system.

Few realize how much real progress has been made and how much tangible material as the basis for a critical study of the general problem has already been accumulated in this field. Thus at the outbreak of the war there were, for example, about a hundred journals being published in Esperanto alone and over half this number survived the cataclysm and are still appearing, with new ones being constantly added, especially from the newly created states, and suspended journals gradually resuming publication.

However it is not my purpose to go into details here, but merely to use this subject as a suggestive illustration of how wide and fundamental are even the apparently incidental contacts of the scientist with these larger world problems.

SOUND INTERNATIONAL POLICY

In the above I have confined myself chiefly to what might be termed the intangibles, because in casually thinking of the subject we are perhaps more apt to overlook these. However, from the material side the chemist's responsibilities are, if anything, even greater and more specific. Whatever the political excuses given, in reality more wars have probably centered about the control of raw materials than any other question; and who is there better equipped than the chemist to look into the future with regard to the world's needs for raw materials? He it must be who will ultimately advise our statesmen as to these basic facts upon which alone a sound and just national as well as international policy can be built. In this regard we are certainly at the parting of the ways, as discussions and negotiations all over the world clearly indicate. The petroleum situation perhaps occupies for the moment the center of the stage and well illustrates how a single raw material may become the pivot on which the whole international situation turns.

Petroleum and natural gas in this connection immediately suggest helium, which, though almost the antithesis of petroleum as regards its scarcity, is commanding nearly equal international interest on this very account, coupled with its unique military importance. Thus far we have in this country the only developed large natural supplies and the only other known source which as yet promises to be significant lies to the north of us in the hands of our friends, the Canadians. The newness of this whole subject and the intricacy of its relations to both war- and peace-time aviation present problems of national and international policy in whose decision the advice of the chemist and engineer must necessarily enter with great weight.

Petroleum and helium are only two examples picked almost at random from the multitude of available material already familiar to you, but they serve well to illustrate the point I set out to emphasize; namely, how responsible and important a part the chemist must take in the guidance of new international relationships now rapidly pressing upon us.

CHEMICAL READING COURSES[1]

Prepared by the Committee on the Preparation of a Recommended List of Chemical Texts for Libraries
W. A. Hamor, *Chairman*, L. C. Newell, A. M. Patterson, W. Segerblom

ELEMENTARY OR INTRODUCTORY CHEMISTRY[2]

The facts and principles of chemistry are available for all who will take time to read. But do not conclude that the essential facts and leading principles of chemistry are buried deep in technical journals and heavy textbooks. There are many interesting and reliable books on chemistry written solely for those who have not made a special study of this science. Hendrick's "Everyman's Chemistry" (Harper, 1917; 374 pp.) is a lively account of modern chemistry. This book is "designed chiefly for those who declare that they do not understand anything about the subject." Another book written for the general reader is Philip's "Romance of Modern Chemistry" (Lippincott, 1910; 348 pp.). It contains an absorbing discussion of flame, fuels, explosives, extremes of temperature, crystals, and the by-products of chemical processes. Still another is Slosson's "Creative Chemistry" (Century Co., 1920; 311 pp.), which describes in plain language such timely topics as cellulose, rubber, plastics, sugars, fats, and electric furnace products. Chemistry is so vast that you can read several similar books without duplicating topics. Hence you will enjoy two books by Martin: "Modern Chemistry and Its Wonders" (Van Nostrand, 1915; 358 pp.), and "Triumphs and Wonders of Modern Chemistry" (Van Nostrand, 1911; 358 pp.). Both books describe in non-technical language some of the recent striking advances in chemistry. Special topics are treated in Bird's "Modern Science Reader" (Macmillan, 1911; 217 pp.). This book consists of reprints of original articles by experts on such topics as the oxy-hydrogen process of cutting metals, combustion of coal, coal-tar dye industry, electrochemistry, and artificial silk.

CHEMISTRY HAS ITS HUMAN INTERESTS

Three books by Duncan should be read by everyone who wishes to know something about modern chemistry and its relation to human interests. "The New Knowledge" (A. S. Barnes and Co., 1905; 263 pp.) is a lucid account of the new physics and new chemistry in their varied applications. "The Chemistry of Commerce" (Harper, 1907; 263 pp.) is a fascinating interpretation of various phases of modern industrial chemistry. "Some Chemical Problems of To-day" (Harper, 1911; 254 pp.) recites recent contributions to theoretical and industrial chemistry. These books are written in beautiful language, which can be understood by the general reader.

Chemistry is a progressive science. Many commonplace facts are deeply rooted in chemistry. Going back many years we can find their origin and trace their growth. Tilden's "Chemical Discovery and Invention in the Twentieth Century" (Dutton, 1917; 487 pp.) is a vivid narration of the growth and progress of chemistry from 1800 to the present time. Sometimes its development is so rapid we can hardly keep pace with the movement. We need a survey of the field, especially in the application of what is sometimes called "pure chemistry." Findlay's "Chemistry in the Service of Man" (Longmans, 1916; 280 pp.) is a review of the world's indebtedness to the chemist, including applications of modern principles to chemical industries. Alexander's "Colloid Chemistry" (Van Nostrand, 1919; 90 pp.) is a summary of this interesting field of chemistry.

HEALTH AND SANITATION

The relation of chemistry to personal and public health is a vital topic. Bailey's "Sanitary and Applied Chemistry" (Macmillan, 1917; 394 pp.) treats of the atmosphere, fuels, heating, ventilation, lighting, purification of water supplies, disposal of sewage and household wastes, cleaning, and disinfectants. The book also contains many experiments. A more elaborate book is Baskerville's "Municipal Chemistry" (McGraw-Hill Co., 1910; 526 pp.). This is a compilation of a series of lectures by experts treating of such important topics as city sanitation, drinking water and disease, purification of water, milk, food adulteration, food inspection, street cleaning, smoke, textiles, paints, corrosion of iron, and cement.

Everyone has heard of radium, but not all realize the comprehensive inquiries conducted by chemists to find out the many secrets of this marvelous substance. A brief, accurate, and entertaining book on radium is Venable's "Radio-Activity" (Heath, 1917; 54 pp.). A larger work is Soddy's "The Interpretation of Radium" (Putnam, 1912; 284 pp.). This book is the substance of several popular lectures and is written in non-technical language.

Chemistry does not stand alone. It overlaps other sciences, especially physics. To understand part of the language of chemistry we need to read border-line topics. In Stewart's "Chemistry and Its Borderland" (Longmans, 1914; 327 pp.) physico-chemical topics are discussed. Talbot and Blanchard's "Electrolytic Dissociation Theory" (Macmillan, 1907; 85 pp.) treats of the theory of electrolytic dissociation, ions, and ionization in a compact, informative way. A summary of the work of the leaders in chemistry and physics during the last quarter of a century may be found in Jones's "New Era in Chemistry" (Van Nostrand, 1913; 328 pp.).

Chemistry grows so fast it is hard to keep up with it; hence sometimes we must resort to periodic reports. One of the best of these is "Annual Reports on the Progress of Chemistry" (Van Nostrand). These are epitomes of the progress of pure chemistry compiled by specialists selected by the Chemical Society of London and are indispensable for those who wish the latest data.

HISTORY AND BIOGRAPHY

Chemistry is essentially a human science. Its important discoveries are linked with the names of men whose lives are often like a romance. Some knowledge of the history of chemistry, especially of the great discoveries, is needed for an intelligent interpretation of the chemistry of to-day. There are scores of books telling about famous chemists and their work. One of the best of the shorter books is Thorpe's "History of Chemistry" (Putnam, 1910; Vol. I, 195 pp.; Vol. II, 202 pp.). It begins with the chemistry of the ancients and ends with contemporary chemistry. Many portraits add to its interest. Venable's "Short History of Chemistry" (Heath, 1894; 163 pp.) is a compact, brief book. Two other books covering the same ground, though in a different way, are Hilditch's "History of Modern Chemistry" (Van Nostrand, 1911; 273 pp.) and Armitage's "History of Chemistry" (Longmans, 1907; 286 pp.). The former contains a biographical index of chemistry and a chronology of chemical events since the time of Boyle.

We must not forget that chemistry is largely the product of experiments and that these experiments were performed by diligent men who worked much as chemists work to-day. Hence, there are many books devoted to the biographical side of chemistry. Roberts' "Famous Chemists" (Macmillan, 1911; 247 pp.) describes briefly the chief work of famous chemists and the part played by each in the development of chemistry. Muir's "Heroes of Science—Chemists" (Young, 1883; 338 pp.) recounts the progress of chemistry by incorporating selected biographies into a popular history of chemistry, while Smith's "Chemistry in America" (Appleton, 1915; 356 pp.) is a vivid story of the chemical pioneers in our own country; this book contains valuable and interesting information not readily found elsewhere. Thorpe's "Essays in Historical Chemistry" (Macmillan, 1902; 582 pp.) is a collection of biographies of about a dozen famous chemists from Boyle to Mendeléeff; considerable space is given to the French chemist Lavoisier, who laid the broader foundations of the science of chemistry. Ramsay's "Gases of the Atmosphere" (Macmillan, 1916; 314 pp.) is a narrative of early experiments and speculations on gases and of the author's discovery of argon, helium, and other rare gases in the atmosphere. Portraits enhance the value of the book.

Among the larger books devoted to the history of chemistry, which may be consulted or read in part, are "Chemical Society Memorial Lectures, 1893–1900" (Gurney and Jackson, London, 1901), Von Meyer's "History of Chemistry" (Macmillan), Muir's "History of Chemical Theories and Laws" (Wiley, 1907; 555 pp.), and Freund's "Study of Chemical Composition" (Macmillan, 1904; 650 pp.).

PERSONALITIES

There are many books about the life or the special work of a single chemist. The "Century Science Series" (Macmillan) includes in separate volumes biographies of Dalton, Davy, Faraday, and Liebig. No one should fail to read Tyndall's "Faraday as a Discoverer" (Appleton, 1894; 171 pp.)—a charming biography of a famous chemist written by a noted scientist. A discoverer's own account of his work brings us close to the man. Fortunately there are reprints in English of many original papers. Several are included in the "Alembic Club Reprints" (University of Chicago Press, Chicago, Ill.). In these little books Priestley tells how he discovered oxygen, Cavendish recounts his work on air, etc. Nos. 1, 2, 3, 4, 6, 7, 8, 9, 12, 13, 18 are the best.

---

[1] Presented to the Council at the 58th Meeting of the American Chemical Society, Philadelphia, Pa., September 2, 1919.

[2] By Dr. Lyman C. Newell.

OTHER BOOKS

This reading list is short. There are many other interesting books on chemistry, especially on single topics. Here is a supplementary list of a few more books you may wish to read:

Benson, "Industrial Chemistry." A condensed treatment of important industries.

Derr, "Photography for Students of Physics and Chemistry." Treats of the general principles and processes of photography.

Greene, "Coal and Coal Mines." An account of personal experiences.

Martin, "Story of a Piece of Coal." Condensed.

Meade, "Story of Gold." Non-technical.

Smith, "Story of Iron." Non-technical.

Snyder, "Chemistry of Plant and Animal Life." Popular.

Surface, "The Story of Iron." Non-technical.

Tower, "Story of Oil." A non-technical account of the industry.

Woolman and McGowan, "Textiles." A comprehensive account of the chemical nature of textiles.

HOUSEHOLD CHEMISTRY[1]

The home is the center of our national life. Chemistry plays a large part in the activities of the home, and some knowledge of chemistry is essential for its successful management.

ECONOMY IN THE HOME

Let us begin with the household itself. The United States Government recently issued a series of bulletins containing indispensable information on household chemistry and allied subjects (*Circulars* **55, 70,** and **75,** Bureau of Standards, Washington, D. C.). The first tells about measurements in the home. The second describes the most common materials, other than food and drugs, used in the home. The third discusses the problem of safety. However, the main task in the home is to feed the family. You may find out how to do it in Rose's "Feeding the Family" (Macmillan, 1917; 449 pp.). The author tells in a clear and concise way how to provide proper food for different members of the family, young, old, thin, fat, sick, and well. Even if you yourself do not have to feed a family, you will find the book an entertaining and accurate account of the application of the science of nutrition to daily living. Household management is also a task in the home. A suggestive, practical book is Kinne and Cooley's "Foods and Household Management" (Macmillan, 1914; 401 pp.). This book treats of kitchen furnishings, preparation of food, cost and purchase of food, menus and dietaries, the family budget, how to buy, clean, and launder. Cook books are not usually regarded as books to read, but perhaps a cook book may be needed to supplement the two books just described. Farmer's "Boston Cooking-School Cook Book" (Little, Brown and Co., Boston, 1918; 656 pp.) is said to be "The best general cook book on the market." It contains considerable information about food besides the recipes. More description and fewer recipes will be found in Wellman's "Food Study" (Little, Brown and Co., Boston; 324 pp.); besides the selected recipes and the accurate descriptive material, there are practical tables of food values; a supplementary pamphlet on "Economy in Food" (36 pp.) contains helpful suggestions about saving money in buying, serving, storing, and cooking food.

FOOD AND NUTRITION

If you wish to read about the whole field of food and nutrition in its intimate relation to yourself and others, you will find reliable information in "Food and the War" (Houghton Mifflin Co., 1918; 379 pp.). Don't be misled by the title. Although this book was prepared under the direction of the United States Food Administration as a war book, it has become a peace book. It is full of all kinds of up-to-date information about food, nutrition, and diet. Incidentally, you will find in this book a great deal about feeding the Belgians and other European peoples. After all, eating is the large problem. We have many old-fashioned notions on food and eating. Now if you wish to start on the right track in improving your food habits, read Hawk's "What We Eat" (Harper, 1919; 232 pp.). This book is based on simple experiments which were performed to find out "what the human stomach actually thinks of all the common foods which enter into the diet of man." It answers in a simple way questions about drinking water with meals, drinking milk, coffee, and tea, oleomargarine versus butter, and eating ice cream, pastry, and candy. Parents who wish to feed their children properly ought to read this book. Sherman's "Chemistry of Food and Nutrition" (Macmillan, 1918; 454 pp.) is a more scientific book on food requirements and nutrition. While it is a textbook and perhaps intended for those who have some knowledge of chemistry, much of the book is suited to the needs of readers who wish to have reliable information about digestion, metabolism, food requirements, dietary standards, and inorganic food material. Special emphasis is laid on the food requirements of man. The best of the brief books on food values is Sy's "Food Values" (Bulletin No. 2, University of Buffalo, Buffalo, N. Y., 1917; 40 pp.; not available for free distribution). This compact account of the value of food is illustrated by colored charts depicting the proportions of the food principles in common foods.

If you desire general information about food products, read Bailey's "Source, Chemistry, and Use of Food Products" (Blakiston, 1917; 539 pp.). The book contains practical rather than specialized information about the sources, history, manufacture, preparation, composition, and uses of the important foods and beverages. One of the simpler books dealing with food, its nutritive value, and cost, is Olsen's "Pure Foods" (Ginn and Co., 1911; 210 pp.). Numerous experiments of varying degrees of difficulty are grouped at the ends of the chapters.

DEFEAT FOR THE HIGH COST OF LIVING

The relation of the composition of food to its cost may be found in two books. In Rose's "Laboratory Handbook for Dietetics" (Macmillan, 1917) there is an explanation of the problems involved in calculating food values, food requirements, and the construction of dietaries; while in Gephart and Lusk's "Analysis and Cost of Ready-to-Serve Foods" (American Medical Association, Chicago, 1915; 71 pp.) you will find both text and tables showing the composition, fuel value, and cost of well-known kinds of food served in a chain of New York restaurants. This is a unique book, because it shows the close relation of money to food actually prepared and served to thousands of patrons.

A book devoted entirely to household chemistry is Snell's "Elementary Household Chemistry" (Macmillan, 1914; 307 pp.). It contains text and experiments. The first half is devoted to general chemistry; the second half to household chemistry, including the study of foods, soap, bleaching, dyeing, and many other chemical topics.

Besides the books described in this reading course, there are many others you may wish to read. The following might be mentioned:

Belcher, "Clean Milk" (Orange Judd Co., N. Y.). Describes practical methods for excluding bacteria from milk.

Conn, "Bacteria, Yeasts, and Molds in the Home." Well-tested book on the relation of microörganisms to household affairs.

Grant, "Chemistry of Bread-Making." Treats of the process on a large scale.

Hart, "Leavening Agents." Brief discussion of baking powders and yeast.

Halligan, "Fundamentals of Agriculture." Answers many questions about the farm.

Jordan, "Principles of Human Nutrition." Non-technical; helpful tables.

Kellogg and Taylor, "The Food Problem." A war book on food and digestion.

Sherman, "Food Products." A comprehensive book (594 pages); contains an appendix on the Food and Drugs Act.

Vulté and Venderbilt, "Food Industries." Covers the production and manufacture of staple foods.

Wood, "Story of a Loaf of Bread." Starts with wheat growing and ends with the finished loaf.

GENERAL AND PHYSICAL CHEMISTRY[1]

The Great War raised American chemistry to its rightful place. First came the shutting-off of imports, then the demands of the Allies, and finally the needs of Uncle Sam in action. The story is so recent that, for the most part, it has yet to be told, but all men know that the American chemist acquitted himself well, both in research and in engineering. Is he quitting his efforts with the ending of the emergency? Of course not! Stimulated by great opportunities and by the knowledge that the nation now realizes much more clearly its need of him, the chemist returns to the arts of peace with a new spirit and with new recruits.

War called for quick action and the use of knowledge and skill already attained; now comes "the long pull," in which the successful will be those who best learn and keep up with their subject—who saturate themselves with it. And this means reading, both review and new.

UP OUT OF THE DARKNESS

To turn, once in a while, and look down over the way we have come is an inspiration; and what a fascinating past chemistry has! Our present-day commonplaces take on new interest

---

[1] By Dr. Lyman C. Newell.

[1] By Dr. Austin M. Patterson.

when we reflect how they were once fought over. Moreover, yesterday's solution may contribute a hint to to-day's problem. "A History of Chemistry" by J. C. Brown (Blakiston, 1913; $3.50[1]) is not only one of the latest of such histories, but is a valuable and eminently readable book. Those who are especially interested in recent progress (which, as is well known, has been very rapid) will find real enjoyment in reading "Chemical Discovery and Invention in the Twentieth Century" by W. A. Tilden (Dutton, 1917; $3.50). It recounts the advances in pure and applied chemical science and describes several famous laboratories in different countries.

"WHAT OTHERS HAVE DONE...."

One of the greatest stimuli one can have is acquaintance with the struggles and triumphs of great men through their biographies. "Famous Chemists" by E. Roberts (Macmillan; $0.80) consists of short sketches of the life and work of such men as Boyle, Lavoisier, Liebig and Pasteur. A fascinating biography of Ramsay, the discoverer of the inert gases, has recently been written by an East Indian: "Sir William Ramsay," by T. C. Chaudhuri (Butterworth & Co., 1918; 66 pp.).

SOME AMERICAN ACHIEVEMENTS

American chemical history has been somewhat neglected by textbook writers, but Dr. Edgar F. Smith, chemist and also provost of the University of Pennsylvania, is a delightful writer and lecturer, and his fellow scientists are correspondingly indebted to him for two recent books about important early achievements here: "The Life of Robert Hare" (Lippincott, 1917; $5.00) and "Chemistry in America" (Appleton, 1914; $2.50).

FOR THE BEGINNER

Chemistry is one of the most fascinating of studies. The mysterious transformations which matter undergoes never fail to excite interest, which can be held if the subject is well presented. No one will be content with merely reading about chemical experiments or even with seeing them performed; he should *make them himself* to whatever extent is possible. Fortunately many elementary books are now available which contain directions for experiments or are accompanied by a laboratory manual. Laboratories are also numerous in these days, but the real future chemist will not be deterred by lack of access to one. Indeed, he will get some helpful experience in "rigging up" his own apparatus while finding his way to regular equipment. Among the best of the beginning books are "General Chemistry" by L. C. Newell (Heath, 1914; $1.20, complete in two parts; or Part I, $1.00, and Part II (experiments), $0.60), which gives very full laboratory directions, and "A First Course in Chemistry" by McPherson and Henderson (Ginn; $1.25), which is full of practical applications. Persons who have never studied chemistry but are mature enough to use a somewhat more advanced text may prefer "An Elementary Study of Chemistry" by McPherson and Henderson (Ginn, 1917; $1.60).

COLLEGE COURSES

A well-known chemist once said that he made it a practice to read a good college chemistry every so often because, being immersed in his specialty, he could best realize the advance of the science in that way. There are many who could profit by doing this and who would enjoy the review. There are others who have already studied chemistry in an elementary way and are ready for something harder, or even beginners who are not afraid to tackle a college text. Among the books of proved value for these purposes are "General Chemistry for Colleges" by Alexander Smith (Century Co.; revised edition, 1916) and "A Textbook of Chemistry" by W. A. Noyes (Holt, 1913; $2.25).

MORE INTERESTING THAN IT LOOKS

To some minds the words "theoretical chemistry" may not presage thrilling interest, yet after all man never observes facts that he does not immediately start to inventing theories about them. Even as a child he wants to know "why." Both facts and theory are indispensable to the growth of science and the aspiring chemist will lay a good theoretical foundation. "Elementary Chemical Theory" by J. M. Wadmore (Van Nostrand, 1912; $1.50) is a simple treatment; a larger book, presupposing some knowledge of both physics and chemistry, is "Outlines of Theoretical Chemistry," by F. H. Getman (Wiley, 1913; $3.50).

THE DAY OF PHYSICAL CHEMISTRY

Physical chemistry is a development of the last generation, but it will play a large part in the new era, commensurate with its importance to-day. Physics and chemistry are at bottom one, and physical chemistry is not a mere borderland province, but a central area of immense strategic importance. An excellent book for a first course is "Practical Physical Chemistry" by F. W. Gray (Macmillan; $1.25). Somewhat more advanced are "Theoretical and Physical Chemistry" by S. L. Bigelow (Century Co., 1912; $3.00) and "An Introduction to the Principles of Physical Chemistry" by E. W. Washburn (McGraw-Hill, 1915; $3.50). The latter brings the discoveries in radioactivity and atomic structure up to a recent date.

AND COLLOIDS!

It is a long time since Graham first distinguished between "colloids" and "crystalloids," yet colloid chemistry is one of the most active fields of physical chemistry. An unusually readable little book which gives a very comprehensive idea of the subject is "An Introduction to the Physics and Chemistry of Colloids" by Emil Hatschek (Blakiston; $1.00). One of the most satisfactory books on colloids in English is "The Chemistry of Colloids, and Some Technical Applications" by W. W. Taylor (Longmans; $2.00).

SOME OTHER BOOKS

Historical

T. P. Hilditch, "A Concise History of Chemistry." Van Nostrand, 1911; $1.05. A brief sketch, valuable as an aid in reviewing the subject; not recommended as an introduction.

A. Ladenburg, "Lectures on the History of the Development of Chemistry since the Time of Lavoisier." Translation; Van Nostrand, 1900. An interesting lecture course published originally in 1869 and brought down nearly to the end of the century by added lectures.

E. von Meyer, "A History of Chemistry." Third English edition; Macmillan, 1906. Long a standard book on this subject.

E. Thorpe, "History of Chemistry." 2 vols., illustrated. Putnam, 1910; $2.00.

Beginning Chemistry

R. P. Brownlee, and others, "The Chemistry of Common Things." Allyn & Bacon, 1914; $1.50. Rather superficial and unsystematic but contains a great deal of information about applied chemistry; presented in an elementary way.

J. C. Hessler and A. L. Smith, "Essentials of Chemistry." Sanborn & Co., New York, 1912; $1.25 (with manual, $1.45). A revised edition of a good textbook of the "assemblage of facts" type.

L. Kahlenberg and E. B. Hart, "Chemistry and Its Relations to Daily Life." Macmillan, 1913; $1.25. A helpful, practical book by experienced teachers.

Wilhelm Ostwald, "Conversations in Chemistry." Wiley, 1906; Part I, $1.50; Part II, $2.00. General chemistry in simple language in the form of conversations between teacher and pupil.

Alexander Smith, "A Textbook of Elementary Chemistry." Century Co.; $1.25. A very valuable and interesting book by a well-known author. Not as simple as some elementary works.

M. S. H. Unger, "Review Questions and Problems in Chemistry." Ginn, $0.50. A complete and satisfactory compilation useful for those who have finished a beginners' course.

Advanced General Chemistry

C. L. Bloxam, "Chemistry, Inorganic and Organic." Tenth edition; Blakiston, $5.50. A standard reference book probably containing more information in one volume than any other in English. It has some faults common to descriptive books which have passed through many editions.

W. McPherson and W. E. Henderson, "A Course in General Chemistry." Ginn, 1913; $2.25. A college text in which modern theory and descriptive material are well balanced.

Wilhelm Ostwald, "Outlines of General Chemistry." Translation; third edition, 1912; $4.50. A well-known book by a leading German chemist, in which chemistry is treated especially from the standpoint of energetics, the atomic theory being avoided.

Laboratory Manuals

W. S. Hendrixson, "A Laboratory Manual of General Chemistry." Fourth edition; Grinnell, Iowa, 1918. An elementary book which will give a good understanding of the relation of qualitative analysis to general chemistry.

A. B. Lamb, "Laboratory Manual of General Chemistry." Harvard Press, 1916. Contains advanced experiments, many of which are new, and is notable for its clear discussions. The last third of the book is devoted to inorganic preparations.

H. B. North, "Laboratory Experiments in General Chemistry." Van Nostrand, 1913; $1.00. Contains 500 well-selected experiments.

Theoretical and Physical

S. Arrhenius, "Theories of Chemistry." Longmans, Green, 1907; $1.75. A critical examination of chemical theories by a recognized authority.

S. Arrhenius, "Theories of Solutions." Yale Press, $2.25. A course of lectures delivered in 1911 by a scientist of first rank and broad viewpoint. Valuable for those who are somewhat versed in physical chemistry.

---

[1] Prices quoted throughout are as of the date of preparation of this report and are printed simply to serve as a guide to the general trend of prices.

W. H. Bragg and W. L. Bragg, "X-Rays and Crystal Structures." Macmillan, 1915; $2.25.

A. Findlay, "Osmotic Pressure." Longmans, Green, 1913; $1.00. An excellent exposition of a subject, the literature on which is somewhat confused.

A. Findlay, "Practical Physical Chemistry." Third edition; Longmans, Green, $1.20. A standard laboratory manual.

A. Findlay, "The Phase Rule and Its Applications." Longmans, Green, 1917; $1.75. A book of recognized merit which first appeared in 1904.

J. B. Firth, "Practical Physical Chemistry." Van Nostrand, 1916; $1.00. A laboratory manual not differing greatly from other books of its kind, but of recent date and including some experiments on colloids.

W. McC. Lewis, "A System of Physical Chemistry." Longmans, Green, 1916; 2 vols., $2.50 each. Very good as an advanced text.

J. W. Mellor, "Chemical Statics and Dynamics." London, 1914; $2.00.

R. A. Millikan, "The Electron." Chicago Press, 1917; $1.50. Based on the work of the author and his students.

J. L. R. Morgan, "The Elements of Physical Chemistry." Fifth edition; Wiley, $3.00. A well-known book which is not now altogether up to date in general viewpoint.

W. Nernst, "Theoretical Chemistry." Macmillan, 15s.

Wilhelm Ostwald, "Fundamental Principles of Chemistry." Translation; Longmans, Green, 1909; $2.25. A clear, attractive presentation by a foremost German chemist.

Wolfgang Ostwald, "An Introduction to Theoretical and Applied Colloidal Chemistry." Translation; Wiley, 1917; $2.50. A broad survey of the whole subject, taken from lectures delivered in 1913–14.

S. E. Sheppard, "Photo-Chemistry." Longmans, Green, 1914; $3.50. A most excellent resumé containing a surprising amount of material. Not to be confused with works on photographic chemistry.

S. Smiles, "The Relations between Chemical Constitution and Some Physical Properties." Longmans, Green, 1910; 14s. A most valuable collection and review of otherwise scattered data.

James Walker, "Introduction to Physical Chemistry." Macmillan. A good non-mathematical treatment.

R. Zsigmondy, "Colloids and the Ultramicroscope." Translation; New York; $3.00. A well-known text.

MISCELLANEOUS

R. M. Bird, "Modern Science Reader." Macmillan, 1911. A collection of interesting articles on pure and applied chemistry for collateral reading.

M. L. Dolt, "Chemical French." Chemical Publishing Co., $3.00. A companion volume to "Chemical German."

J. W. Mellor, "Higher Mathematics for Students of Chemistry and Physics." Fourth edition; London, 1913; $4.50. Enlarged edition of a highly successful work showing how the calculus and other mathematics are applied to practical problems.

J. C. Olsen, editor, "Van Nostrand's Chemical Annual." 1918; $3.00. A handbook of useful data.

J. R. Partington, "Higher Mathematics for Chemical Students." Methuen & Co., London, 1911; 3s. 6d. A valuable book for students who have had some mathematics but need help in applying it to their chemical problems.

F. C. Phillips, "Chemical German." Second edition; Chemical Publishing Co., $2.00. Includes exercises in chemical German, extracts from the writings of German authors, and a German-English vocabulary.

INORGANIC AND ANALYTICAL CHEMISTRY[1]

American chemists made a substantial contribution to winning the war. They now face a great opportunity and a great responsibility. Most laymen now have a greatly increased interest in chemical developments as an important factor in our future national life. It is up to the chemist. He must broaden his outlook; he must add to his mental equipment, however good. The science of chemistry makes rapid progress. To pause or be satisfied with one's present training is to lose ground.

Inorganic chemistry has made noteworthy advances during the past twenty-five years, among which may be mentioned the discovery of the inert gases and of the radioactive elements, the further investigation of the rare earths, and the study of the complex compounds of cobalt, platinum, and other metals. Industrial processes based on inorganic chemistry have been of great importance in a military way and will be even more so in times of peace. An unconventional, racily written "Introduction to Modern Inorganic Chemistry" (Longmans, Green, 1914; $1.30) is furnished by J. W. Mellor. The same author has written another stimulating book called "Modern Inorganic Chemistry" (Longmans, Green, 1912; $2.20), which is prized by teachers and as a small reference book.

AN UP-TO-DATE SURVEY

Any one who has had a year or more of chemistry and who wishes to take up the special study of the inorganic department of the science, as well as the chemist who wishes—and it is a splendid idea—to review the field, will find an excellent and up-to-date survey in Alexander Smith's "Introduction to Inorganic Chemistry" (third edition, rewritten; Century Co., 1917; $2.60). Another standard book suitable for those who have already had a good preparation in chemistry, and some calculus, is "A Textbook of Inorganic Chemistry" by A. F. Holleman (fifth English edition; Wiley, 1916; $2.20).

THE PHYSICO-CHEMICAL VIEWPOINT

Inorganic chemistry and physical chemistry are, of course, not two separate and mutually exclusive fields. The first is part of a classification based on the kind of substances with which it has to deal, while the second is a particular way of looking at all substances. The study of inorganic chemistry from the physico-chemical standpoint is most instructive. "Inorganic Chemistry" by H. P. Cady (McGraw-Hill, 1912; $2.50) comes well recommended for this purpose.

LINKING UP WITH INDUSTRY

It is not often that one finds a treatment combining pure science with industrial applications, yet such an arrangement is entirely rational, for the two go hand-in-hand. E. Molinari's "Treatise on General and Industrial Inorganic Chemistry" (Blakiston, 1912; $6.50) is a well-known Italian work translated into English. It is provided with many illustrations.

METALS AND NON-METALS

This list would not be complete without a mention of Roscoe and Schorlemmer's comprehensive "Treatise on Chemistry" (Macmillan, Vol. I, 1911; Non-metals, $5.00; Vol. II, 1913; Metals, $7.50), which serves as a small reference work and is good reading besides.

FOR BALLOONS—HELIUM

One of the latest illustrations of the close relation of pure and applied science is the use in balloons of helium, a gas which only yesterday was considered rare and which day before yesterday (or so it seems) was known only by its spectrum in the sun! "The Gases of the Atmosphere" by William Ramsay (Macmillan, 1915; $2.00) presents an account of the discovery of helium and the other gases of the same family.

UNRAVELING THE COMPLEX

Years ago Werner proposed a novel theory for explaining the complex inorganic compounds. Many orthodox chemists looked askance at him, but he and his pupils have calmly gone their way accumulating evidence till no one can afford not to know something about it. "New Ideas on Inorganic Chemistry" by A. Werner (Translation; Longmans, Green, 1911; $2.50) records the Wernerian theories.

THE PLACE OF ANALYSIS

It is true that the present-day tendency is away from the job of the routine analyst, a title formerly thought of as almost synonymous with "chemist." Nevertheless, chemistry, being an "exact science," is and always will be dependent on analytical methods. The intelligent use of existing methods and the development of improved ones call for qualities of a high order. A good introduction to qualitative analysis may be had through "Qualitative Chemical Analysis" by A. A. Noyes (Macmillan; $1.50); "The Elements of Qualitative Analysis" by W. A. Noyes and G. McP. Smith (Holt); or "Qualitative Analysis" by F. P. Treadwell (fourth edition; Wiley; $3.00).

AVOIDING "RULE-OF-THUMB" METHODS

The really good analyst must be resourceful and hence must have a good understanding of the principles on which analysis is based. A great advance in the presentation of these principles is made in "The Elements of Qualitative Analysis" by J. Stieglitz (Century Co., 1911; Vol. I, $1.40; Vol. II (laboratory manual), $1.20). A well-selected course presented throughout from the standpoint of electrolytic dissociation and mass action is Bailey and Cady's "A Laboratory Guide to the Study of Qualitative Analysis" (eighth edition; Blakiston; $1.50).

NOT ONLY "WHAT?" BUT "HOW MUCH?"

A leading biological chemist, who died recently, used to state that he wished that all specialists in his line had had the good old-fashioned drill in quantitative analysis. Certainly the training which it gives is valuable to any chemist. A selected course with a good introduction is given in the "Quantitative Analysis" of H. P. Talbot (Macmillan; $1.50). "Quantitative Chemical Analysis" by Clowes and Coleman (tenth edition; Blakiston, 1914; $3.50) is a carefully revised form of an excellent standard text, and J. C. Olsen's "Textbook of Quantitative Chemical Analysis" (fifth edition; Van Nostrand, 1916; $3.50) is also a standard work.

---

[1] By Dr. Austin M. Patterson.

THE USE OF THE BLOW PIPE

In practiced hands this little instrument, familiar to every chemistry freshman, may be made to perform wonders, not only in qualitative analysis, but also for quantitative work. Its use is a real art, fascinating to those who specialize in it. Plattner's "Manual of Qualitative and Quantitative Analysis with the Blow Pipe" (Translation; eighth edition revised; New York, 1912; $4.00) is one of the best-known books on this subject. A small treatise of recent date is G. M. Butler's "Pocket Handbook of Blow Pipe Analysis" Chapman and Hall, 1916; $0.75).

SOME OTHER BOOKS

INORGANIC CHEMISTRY

P. E. Browning, "Introduction to the Rarer Elements." Third edition; Wiley, 1912; $1.50. The latest edition of a book of recognized value.

J. N. Friend, *et al.*, "A Textbook of Inorganic Chemistry." In nine vols.; Vol. I, Parts 1 and 2; Lippincott, $3.00. Part I is an introduction, and Part 2 treats of the inert gases. The beginning of what promises to be a standard reference work.

E. S. Kipping and W. H. Perkin, "Inorganic Chemistry." Part I (non-metals); Chambers, 1917; $2.00.

D. Mendeléeff, "The Principles of Chemistry." Third English edition; 2 vols.; Longmans, 1905; $10.00. This book by the famous Russian chemist was given its original form in 1868–70. It has become a classic and is still a fertile source of ideas for research.

L. C. Newell, "Laboratory Manual of Inorganic Chemistry for Colleges." Heath, 1909; $2.00. A book noted for its clear and full directions and the comparative simplicity of its apparatus.

ANALYTICAL CHEMISTRY

W. C. Blasdale, "Principles of Quantitative Analysis." Van Nostrand, 1914; $2.50. A well-arranged introduction to quantitative analysis, with a good discussion of the "reasons why."

E. G. Mahin, "Quantitative Analysis." McGraw-Hill, 1914; $3.00. Combines the principles and standard methods with some industrial applications. Elaborate discussion is avoided.

(To be concluded in the August issue)

SCIENTIFIC SOCIETIES

IN THE ALEMBIC OF PUBLICITY

By John Walker Harrington

A. C. S. News Service, New York, N. Y.

As a sign of what may be done to bring the progress of chemistry before the public at the fall meeting of the American Chemical Society, to be held in Chicago next September, stands the record of our publicity for the St. Louis sessions.

The notice which that 59th gathering received from the press was obtained, too, in days of a famine of paper and of a feast of general news.

That the volume of publicity was nearly twice that of the Philadelphia meeting indicates, however, that there is a constantly increasing interest in chemistry throughout the United States. This recognition was due entirely to chemistry itself, for unlike the conclave of the knights of the retort in the City of Brotherly Love, the meeting at St. Louis had as speakers no Cabinet members or men who were figures in national politics. The assemblage at St. Louis, therefore, probably furnished as thorough a test of the chemical appeal to the people as could be had.

As there has been time to organize the publicity of the Society in every direction, the A. C. S. News Service enlisted the enthusiastic coöperation of the Associated Press, the United Press, the International News Service, and other large news gathering organizations.

The distribution of advance material was made from three points. By consulting the accompanying map it will be seen that the newspapers interested are mostly within circles described with a 500 mile radius from the cities of New York, St. Louis and San Francisco. In the strip of territory stretching downward from North Dakota and including South Dakota and the western portions of Nebraska, Kansas, Oklahoma, and Texas, publications are far from numerous, and there are also comparatively few in eastern Montana and in New Mexico.

No state, however, could have failed to have been informed by the press concerning the activities of the American Chemical Society, as newspapers circulate far beyond the borders of the state in which they are published. At El Paso, for instance, close to the boundary line between Texas and New Mexico, are several important newspapers which circulate to the north and into a considerable territory of New Mexico. Many papers printed in the United States serve Canada, in which country also four of the leading journals carried reports of the St. Louis meeting.

On the map printed in this issue has been indicated by shaded areas the hundred mile radius of the circulation of the various newspapers, following a rule in vogue among newspaper publishers.

The publicity returns, as shown, are visualized according to their geographical as well as their journalistic trend.

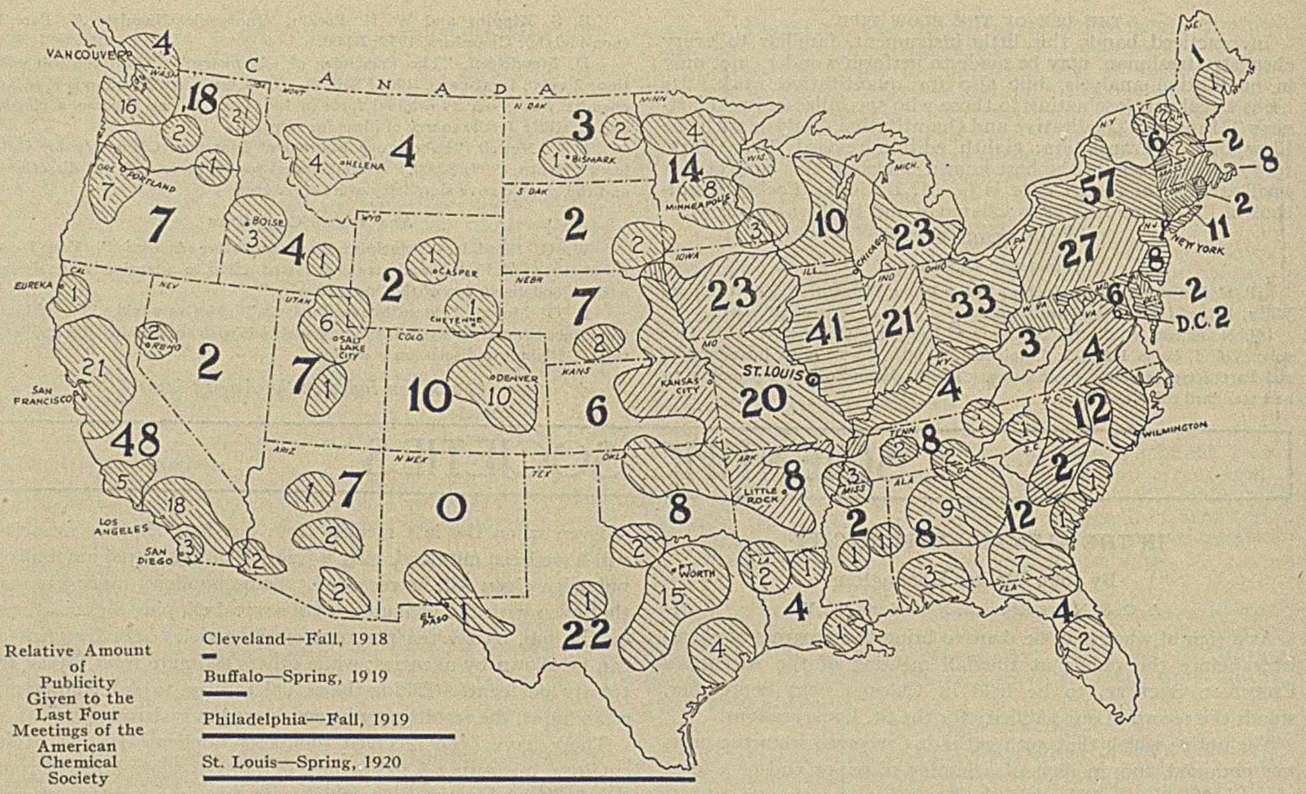
Even when the last returns from our four clipping agencies will have been obtained, only a fraction of the actual amount of publicity given will be recorded. Some publicity managers say that even with the vigilant eyes of several clipping organizations on the job, barely ten per cent of the articles actually printed can be shown by agencies; while others estimate from fifteen to twenty per cent. Taking the actual returns to date, however, as an index, the results may be considered as satisfactory.

There have been received (omitting duplicates) 1,180 press cuttings, measuring 8,460 inches, or 118,440 agate lines. From the Philadelphia meeting, which was the first meeting of the Society at which a nation-wide publicity program was installed, 700 press notices were collected. As far as can be learned, the meeting at Cleveland in the fall of 1918 was practically ignored by the papers of that city and received about 15 notices from the fourth estate. The spring meeting of 1919, held in Buffalo, received 67 notices in all from the Buffalo papers, and 35 from the press at large, as indicated by the reports of the clipping agencies. In the year 1919 there were distributed, exclusive of the two general meetings of the Society, 33 bulletins, which resulted in 894 clippings, measuring 7,963 inches. This shows the value of the news interest in disseminating chemical information, as the returns from St. Louis are larger than those from the bulletins issued for all of last year.

In the various states the press was influenced in the amount of space given to the St. Louis meeting to a large extent by the number of members of the Society in the respective commonwealths, as well as by the presence of chemical industries or of enterprises under chemical control.

New York, a state close to the chemical center of the United States, as shown by the membership of the Society, leads the accompanying list with 57 papers giving more or less full reports of the St. Louis meeting, while neighboring Pennsylvania, pioneer state of industrial chemistry, has 27. California, in which the already large chemical roster is rapidly increasing, is second, with 48 newspapers. From the main distributing center, St. Louis, which to quote the slogan of that enterprising city is "surrounded by the United States," the 500 mile radius has full play. Twenty leading newspapers in Missouri; 41 in Illinois; 33 in Ohio; 21 in Indiana; 14 in Minnesota; 10 in Wisconsin, and as many in Arkansas, evinced a lively interest in the proceedings of the Society. In this connection, the A. C. S. News Service wishes to acknowledge the able assistance of the St. Louis Convention and Publicity Bureau, the coöperation of which was obtained by the Local Section.

In every state, with the exception of New Mexico, in which there is no section of the Society and where manufacturing chemistry has not as yet made a place, the papers had accounts of the St. Louis meeting. A list of the states, prepared in



Relative Amount of Publicity Given to the Last Four Meetings of the American Chemical Society

Cleveland—Fall, 1918

Buffalo—Spring, 1919

Philadelphia—Fall, 1919

St. Louis—Spring, 1920

accordance with their quotas of the 545 newspapers which are known to have reported the spring meeting of 1920, is submitted for consideration.

Number of Newspapers in Each State Which Printed Reports of the St. Louis Meeting of The American Chemical Society[1]

	Newspapers		Newspapers
New York	57	Oregon	7
California	48	Utah	7
Illinois	41	Kansas	6
Ohio	33	Maryland	6
Pennsylvania	27	Vermont	6
Iowa	23	Florida	4
Michigan	23	Idaho	4
Texas	22	Kentucky	4
Indiana	21	Louisiana	4
Missouri	20	Montana	4
Washington	18	Virginia	4
Minnesota	14	North Dakota	3
Georgia	12	West Virginia	3
North Carolina	12	Delaware	2
Connecticut	11	District of Columbia	2
Colorado	10	Mississippi	2
Wisconsin	10	Nevada	2
Arkansas	8	New Hampshire	2
Alabama	8	Rhode Island	2
Massachusetts	8	South Carolina	2
New Jersey	8	South Dakota	2
Oklahoma	8	Wyoming	2
Tennessee	8	Maine	1
Arizona	7	New Mexico	0
Nebraska	7		
		Total	545

[1] These reports, based on the returns of 4 clipping agencies, necessarily represent only a fraction of the publicity obtained. The common rule would be to multiply these results by 7 to estimate the volume of publicity.

There is food for thought in the character of the subjects which received the most attention from the lay press, as shown by an analysis of clippings received.

Probably the last thing in the world in which a newspaper editor might be considered as having an interest several years ago would be a "colloid." The greatest number of clippings, however, came from Mr. Jerome Alexander's paper, "Some Practical Applications of Colloid Chemistry." The description of the colloidal fuel evolved by Lindon W. Bates was the cause of the wide popular interest evinced in what ordinarily would have seemed an abstraction.

The papers of the new Leather Section, bearing as they did upon the all-important subject of shoes, were also favored at the copy desks. The proceedings of the Rubber Section, especially the paper on "The Microscopic Examination of Rubber Products," by Dr. H. J. Masson and Miss Irene Diner, bounded and rebounded through the press because they had a bearing on the durability of rubber tires. The editors know that every American, in fact or thought, is an automobile owner. The potash papers of Dr. Turrentine, concerning American agriculture, and also two papers which indicated the making of dyes from the humble corn cob, were likewise favorites.

A brief interview obtained from Dr. Ralph H. McKee, in which he spoke of the use of cheap alcohol for automobile fuel, was reprinted in scores of papers throughout the United States, and stood fourth in popularity.

One factor in the success of the St. Louis publicity was the coöperation of various members of the Society, who at the request of the technical manager gave time to explain details to inquiring newspaper representatives, and to assist in the preparations of such bulletins as had to be issued from the press room at the last minute.

The results obtained in publicity work for the Society, by the way, depend largely upon the issuing of abstracts and news synopses of papers well in advance of meetings, and subject to release. In this way, with the coöperation of the press associations, matter can be distributed over the United States and put in type, ready for printing at the touch of the telegrapher's keys. The A. C. S. News Service, in preparing for the Chicago meeting, would, therefore, appreciate the prompt forwarding of abstracts or condensations of technical papers, so that in September it may stand ready to serve the American press.

CONVENTION OF CANADIAN CHEMISTS

The convention of Canadian Chemists, held at Toronto on May 27 and 28, 1920, was a great step towards the consolidation of chemical interests in Canada for purposes most beneficial to the welfare of the profession, the industries they represent, and the country as a whole.

For those not perfectly familiar with the rapid developments among chemical organizations in Canada in recent years, it may be news that there are two quite active groups of organized chemists in the Dominion. The Society of Chemical Industry

Photo by British and Colonial Press, Toronto

Left to Right, Front Row: L. J. Rogers, Lash Miller, W. L. Goodwin, R. F. Ruttan, W. H. Ellis, Chas. H. Herty, H. van der Linde, T. H. Wardleworth. Second Row: H. E. Bigelow, J. T. Burt-Gerrans, A. Lehman, F. T. Shutt, G. D. MacIntyre, H. J. Roast, J. B. Bell, A. R. McLean, H. M. Lairash. Third Row: L. E. Westman, F. C. Collier, G. A. Evans, L. F. Goodwin. Back Row: F. J. Hambly, H. M. Lancaster, W. H. Thom, C. F. Thurlow, Alfred Burton, L. T. Acton, E. G. R. Ardagh, J. Watson Bain, S. J. Cook

has had a Canadian Section for eighteen years. Branches have been established in Toronto, Montreal, and Ottawa, and a Canadian Pacific Section established in Vancouver to develop western interests. In Manitoba, the Manitoba Chemical Society, centered in Winnipeg, attends to the chemical interests of that province, while the eastern provinces of New Brunswick and Nova Scotia have organized under the Maritime Chemists' Association. All these bodies are broad in their interests and aim to include in their membership all those who are interested in chemistry.

The Canadian Institute of Chemistry is a dominion-wide organization of chemists, restricted to those whose academic or industrial training has been sufficient to qualify them as fully trained and competent professional men. The recent gathering was a joint meeting of these several organizations.

The University of Toronto offered rather ideal facilities to the visiting chemists. Space for an industrial exhibit was secured in the Mining Building for some twelve companies whose displays went to show that the idea of such an exhibition of apparatus and chemicals had a strong appeal to the representatives of manufacturers and laboratories who were present. This will probably become a growing feature of future conventions. The favor of using Hart House, which is the latest addition to the buildings of the University, and in many ways one of the most remarkable and beautiful buildings on the continent devoted to the use of university students and professors, was fully appreciated. The meetings held there were most impressive and perhaps revealed best the spirit of the convention.

The message of Dr. Chas. H. Herty, who was the honored guest of the chemists of Canada, was delivered here. The problems of "Chemistry under a Constitutional Government" are alike both in the United States and Canada, and for that matter throughout the Anglo-Saxon world. A resumé of what was being done by a proper utilization of the public press of the United States in matters relating to the building up of a national series of chemical industries, free from harmful connections and with the exalted aim that the proper function of industrial chemistry was to facilitate happiness and peace, and not to promote destruction and war, was an ideal which voiced fully the sentiments of every Canadian chemist.

The Canadian Institute of Chemistry, under the guidance of Professor J. W. Bain, president, and H. J. Roast, secretary, reported a successful year. The membership has grown rapidly and a permanent constitution is being developed. The great distances separating members makes the working out of details in such an organization a slow process, but the end in view, namely, a Canadian national chemical organization, designed to represent the chemists of the Dominion in matters relating to both internal and external affairs, is something much to be desired and sure to receive support.

TORONTO, CANADA L. E. WESTMAN

REPORT OF THE COMMITTEE ON COTTONSEED PRODUCTS, DIVISION OF INDUSTRIAL AND ENGINEERING CHEMISTRY, AMERICAN CHEMICAL SOCIETY[1]

This report covers cottonseed, cottonseed oils, cake and meal, crude cottonseed oil, crude peanut oil, crude coconut oil, crude soy-bean oil, refined oils, soap stock and acidulated soap stock, titer tests, and an appendix specifying oil grades, both crude and refined. It has received the unanimous endorsement of the committee: Thomas C. Law, *Chairman*, Frank N. Smalley, Archibald Campbell.

I—COTTONSEED

1—The sample to be used should weigh not less than 10 g. (approximately 100 seed).

---

[1] Approved by the Supervisory Committee on Standard Methods of Analysis, American Chemical Society.

2—Cut the seed by hand, using a sharp knife or similar instrument; remove the kernels and determine the percentage of the kernels and the hulls by weight.

3—Determine the percentage of moisture in the hulls.

4—Determine the percentage of moisture, oil, and nitrogen in the kernels.

(*a*) Determine moisture and nitrogen strictly in accordance with the methods given below under Meal.

(*b*) Determine the percentage of oil in the same manner as given below for Meal, but extract the sample for 2 hrs., then regrind it and extract again for 2 hrs. Remove the last trace of petroleum ether by heating the flask over a flame until the contents just begin to smoke.

5—Calculate moisture, oil, and ammonia in the whole seed.

6—Determine the yield of meal by multiplying the percentage of ammonia in the whole seed by 1900 and divide by the percentage of ammonia desired in the meal, expressing the results thus obtained in "Available pounds of meal per ton of seed."

7—Determine the yield of oil by multiplying the percentage of oil in the whole seed by 1900 and divide by 7.5, the result being total gallons. Deduct 60 lbs. or 8 gal. to allow for loss in milling and express the results as "Available gallons of oil per ton of seed."

8—In the case of damaged seed make an additional deduction in allowing for the milling loss on a basis of one additional gallon for each 10 per cent damaged from 5 to 25 per cent and an additional gallon for each 5 per cent of damaged seed above 25 per cent.

Air-tight friction-top tin containers must be used for forwarding samples of cottonseed.

II—COTTONSEED HULLS

1. WHOLE SEED AND UNCUT MEATS—Pick out the whole seed and uncut meats in 100 g. of the sample and report the percentages determined by weight.

2. OIL—Place 5 g. of the hulls, free from meats and seeds, in a suitable extraction apparatus and extract for 3 hrs. with petroleum ether, boiling below 65° C. Evaporate the extract at the temperature of boiling water to constant weight and report as percentage of oil.

3. FORM OF REPORT—The following form of report is recommended as the standard, employing the following conversion factors:

(*a*) To convert whole seed to oil, use the factor 0.2.

(*b*) To convert uncut meats to oil, use the factor 0.33.

Oil in whole seed and uncut meats, per cent.
Oil in hulls, per cent.
Total oil in hulls, per cent.

4. LINT DETERMINATION—Dry 5 g. of hulls, free from meats and seed, to constant weight in a moisture dish. Transfer to a beaker containing 40 cc. of sulfuric acid (sp. gr. 1.84), and stir with a glass rod for exactly 30 sec. Pour immediately into a beaker containing 200 cc. of water and wash all particles of hulls from the acid beaker with an additional 50 cc. of water. Filter at once through linen cloth, using suction. Thoroughly washed tracing cloth, placed in a hard rubber straining funnel, is recommended for this purpose. Wash until free from acid, drain the residue thoroughly and transfer to the original moisture dish. Dry at 110° C. to constant weight. Three determinations should be made and the average of those agreeing within 1 per cent reported. Calculate the percentage of lint on the original moisture and report as available hull fiber or lint. The average cellulose content of hull fiber is 70 per cent, which factor takes into consideration the solvent action of the acid on hulls.

III—CAKE AND MEAL

1. PREPARATION OF SAMPLE—Grind the sample to uniform fineness and then mix thoroughly, but without sieving. These operations must be performed as rapidly as possible to avoid loss or gain of moisture.

2. MOISTURE—Heat from 2 to 5 g. of the sample for 3 hrs. in an oven at a temperature of 100° C., using an aluminum dish with cover. As soon as the dish is removed from the oven, cover, and cool in a desiccator. Report the loss in weight as moisture. For comparative purposes moisture tests are required on all analytical reports.

3. OIL—Extract 2 to 5 g. of the sample for 3 hrs. in any suitable fat extraction apparatus, using petroleum ether, boiling below 65° C., as the solvent. Evaporate off the ether until no trace remains. Weigh the residue and report it as oil.

4. FAT—Whenever cottonseed meal is sold as a feedstuff a sample must be analyzed for fat according to the official method of the Association of Official Agricultural Chemists.

5. NITROGEN—AMMONIA—PROTEIN—(*a*) Digest 1.7034 g. of the sample with approximately 0.5 g. of metallic mercury or 0.7 g. of mercuric oxide, 10 g. of sodium or potassium sulfate, and 25 cc. of sulfuric acid (sp. gr. 1.84). Place the flask in an inclined position and heat below the boiling point of the acid from 5 to 15 min., or until frothing has ceased. Now increase the temperature and continue digestion until the liquid becomes colorless or until complete digestion has been obtained. From this point on employ the regular Kjeldahl method except that no potassium permanganate is to be added.

(*b*) DISTILLATION—After cooling the digestion flask and contents, add about 300 cc. of distilled water, a few pieces of zinc to prevent bumping, and 25 cc. of a 4 per cent solution of potassium or sodium sulfide, or a sufficient amount to precipitate all the mercury. After mixing thoroughly, add 60 cc. of a caustic soda solution, sp. gr. 1.50, or sufficient to make the solution strongly alkaline, pouring it down the side of the flask, so that it does not mix at once with the acid solution. Connect the flask with a condenser of block tin, mix the contents of the flask by shaking, and distil into an accurately measured quantity of standard sulfuric acid solution, preferably 0.5 *N*, to which have been added 50 cc. of distilled water. Continue the distillation until at least 200 cc. of distillate are obtained, taking care that the delivery tube reaches below the level of the standard acid. Now titrate the distillate with a standard fixed alkali solution, preferably 0.25 *N* sodium hydroxide.

(*c*) A blank should be run on all the agents employed.

(*d*) The conversion factor used is nitrogen × 6.25 = protein. The atomic weights used are nitrogen 14.01, and hydrogen 1.008.

IV—CRUDE COTTONSEED OIL[1]

1. SHAKING THE SAMPLE—Samples submitted for analysis must be thoroughly shaken with the container in an inverted position before drawing a portion for analysis.

2. MOISTURE OR VOLATILE MATTER—Place 10 g. of the well-mixed sample in a tared metal or porcelain dish and heat over a direct flame with a rotating and rocking motion until the oil barely smokes. Cool and report the loss in weight as moisture. Set aside the residue for meal determination.

3. MEAL OR IMPURITIES—Wash the residue from the moisture determination into a beaker, using kerosene, and warm to dissolve the oil. Now filter on a tared Gooch crucible and wash the residue with petroleum ether until free from oil, dry to constant weight, divide by 0.8, and report as percentage of meal or impurities.

4. FREE FATTY ACIDS—Weigh or measure with a pipette 7.05 g. of the well-mixed oil sample into a 4-oz. oil bottle. Add 50 cc. of neutral saturated, chemically pure sodium chloride solution and 2 cc. of phenolphthalein indicator. Titrate with standard 0.25 *N* sodium hydroxide solution until, upon the addition of one drop and violent shaking, a permanent pink color results. Report as percentage of free fatty acid. The reading of the burette in cubic centimeters is the percentage of free fatty acid calculated as oleic acid. (A free fatty acid determination is required on all oils.)

---

[1] See appendix for definition of grades.

5. REFINING LOSS—(A) *Prime Oil*—(*a*) The quantity of dry sodium hydroxide to be used in refining prime oil must not exceed 1.097 per cent by weight of the oil for hydraulic pressed oils, or 1.437 per cent by weight of the oil for cold pressed oils, and must be employed in solutions of 10, 12, 14, 16, 18, and 20° Baumé at 20° C. No oil shall be rejected unless treated with three or more sodium hydroxide solutions of the above strengths; nor shall any claim for excessive refining loss be established if it can be proven that the color can be made with a smaller loss than is produced with the maximum amount of sodium hydroxide allowed; nor shall any claim for deficiency in color be established if it can be proved that the color can be obtained without exceeding 9 per cent refining loss with the maximum amount of sodium hydroxide allowed.

(*b*) APPARATUS—*Scales*, of 1,000 g. capacity and sensitive to 0.5 g.

Weights, 500 g. to 0.5 g.

Refining cups, seamless or enameled cups 4.5 in. in diameter and 4 in. in depth.

Refining apparatus, a mechanical stirrer the speed of which may be controlled. Water bath fitted with thermometer and suitably arranged to hold cups used in the tests.

(*c*) REAGENTS—Sodium hydroxide solutions of 10, 12, 14, 16, 18, and 20° Baumé at 20° C., made from a saturated solution of pure material, containing 77.5 per cent sodium oxide.

SODIUM HYDROXIDE TABLE

Degrees Baumé	NaOH Contents Per cent	Amount Per cent
10	6.55	16.8
12	8.00	13.7
14	9.42	11.6
16	10.97	10.0
18	12.62	8.7
20	14.37	7.6

(*d*) REFINING PROCESS—Place 500 g. of a thoroughly mixed sample of crude oil in a tared refining cup and bring it to a temperature of from 24° to 27° C. Add the necessary amount of sodium hydroxide solution and stir for at least 5 min. without heating, and in the case of cold pressed oil, not less than 30 min. Apply heat and raise the temperature gradually until at the end of 15 min. a temperature of 40° C. has been reached. Stir constantly and continue stirring until a complete separation of the oil and soap stock results. At times, to effect this separation, it will be necessary to attain a temperature of 65° C. When a separation has been secured, place the cup in a water bath at 45° C. for 3 hrs. Allow the cup to stand overnight or cool thoroughly, so that the soap stock will become hard and firm. Now decant the refined oil into a tared refining cup and drain the soap stock for 20 min. If necessary, melt the soap stock on the steam bath, to complete the separation of the oil. Weigh both oil and soap stock cups and contents.

(*e*) CALCULATIONS—The refined loss should be determined by two methods of calculations, the results of which should check within 0.25 per cent.

Method 1—Weight of crude oil minus the weight of refined oil.

Method 2—Weight of soap stock, plus loss in evaporation, minus the weight of sodium hydroxide solution used.

(B) *Off Oil*—(*a*) Any oil with less than 3 per cent of free fatty acid may be refined with any amount of sodium hydroxide solution necessary to neutralize the free fatty acid plus a reasonable excess, provided that the total sodium hydroxide does not exceed 1.097 per cent of the oil for hydraulic pressed oils, or 1.437 per cent for cold pressed oils. On any oil with over 3 per cent free fatty acid three refinings shall be made, using different strengths of sodium hydroxide solution, provided that the maximum amount of sodium hydroxide allowed in the table below is not exceeded, and that one of the tests be made with this maximum amount.

(*b*) APPARATUS—The same as noted under Prime Oil.

(*c*) REAGENTS—Sodium hydroxide solutions of 10, 12, 14, 16, 18, 20, 22, and 24° Baumé at 20° C. made from a saturated solution of the pure material (77.5 per cent sodium oxide).

(*d*) SODIUM HYDROXIDE TABLE FOR USE ON OFF OILS

Weight of Caustic Necessary to Neutralize Free Fatty Acids
Plus 0.6716 Per cent Excess NaOH

Free Fatty Acids	NaOH	Excess	Degrees Baumé							
			10	12	14	16	18	20	22	24
3.0	0.4245	1.0970	16.8	13.7	11.5	10.0	8.7	..	..	..
3.5	0.4963	1.1679	17.8	14.6	12.4	10.7	9.3	..	..	..
4.0	0.5672	1.2388	18.9	15.5	13.1	11.3	9.8	..	..	..
4.5	0.6381	1.3097	..	16.4	13.9	11.9	10.4	9.1	..	..
5.0	0.7090	1.3806	..	17.3	14.7	12.6	10.9	9.6	..	..
Plus 0.7500 Per cent Excess NaOH										
5.5	0.7799	1.5299	..	19.1	16.2	13.9	12.1	10.6	..	..
6.0	0.8508	1.6008	..	20.0	17.0	14.6	12.7	11.1	..	..
6.5	0.9217	1.6717	..	..	17.8	15.2	13.2	11.6	..	..
7.0	0.9926	1.7426	..	..	18.5	15.9	13.8	12.1	..	..
7.5	1.0635	1.8135	..	..	19.2	16.5	14.3	12.6	..	..
Plus 0.8750 Per cent Excess NaOH										
8.0	1.1344	2.0094	..	..	21.3	18.2	15.8	13.9	..	..
8.5	1.2053	2.0803	..	..	22.1	19.0	16.5	14.5	..	..
9.0	1.2762	2.1512	..	..	22.8	19.6	17.0	15.0	..	..
9.5	1.3471	2.2221	..	..	23.6	20.3	17.6	15.5	..	..
10.0	1.4180	2.2930	..	..	24.3	20.9	18.1	16.0	..	..
Plus 1.0 Per cent Excess NaOH										
10.5	1.4889	2.4889	..	..	..	22.7	19.7	17.3	15.6	14.1
11.0	1.5598	2.5598	..	..	..	23.3	20.3	17.8	16.1	14.5
11.5	1.6307	2.6307	..	..	..	24.0	20.9	18.3	16.5	14.9
12.0	1.7016	2.7016	..	..	..	24.6	21.4	18.8	17.0	15.3
12.5	1.7725	2.7725	..	..	..	25.3	22.0	19.3	17.4	15.7
Plus 1.125 Per cent Excess NaOH										
13.0	1.8434	2.9684	..	..	..	27.0	23.5	20.6	18.6	16.8
13.5	1.9143	3.0393	..	..	..	27.7	24.1	21.1	19.1	17.2
14.0	1.9852	3.1102	..	..	..	28.4	24.7	21.7	19.5	17.6
14.5	2.0561	3.1811	..	..	..	29.0	25.2	22.1	20.0	18.0
15.0	2.1270	3.2520	..	..	..	29.7	25.8	22.6	20.4	18.4
Plus 1.25 Per cent Excess NaOH										
15.5	2.1979	3.4479	..	..	..	31.4	27.3	24.0	21.7	19.5
16.0	2.2688	3.5188	..	..	..	32.1	27.9	24.5	22.1	19.9
16.5	2.3399	3.5899	..	..	..	32.7	28.4	25.0	22.5	20.3
17.0	2.4106	3.6606	..	..	..	33.4	29.0	25.5	23.0	20.7
17.5	2.4815	3.7315	..	..	..	34.0	29.6	26.0	23.5	21.1
Plus 1.375 Per cent Excess NaOH										
18.0	2.5524	3.9274	..	..	..	35.8	31.1	27.3	24.7	22.3
18.5	2.6233	3.9983	..	..	..	36.4	31.7	27.8	25.1	22.6
19.0	2.6942	4.0692	..	..	..	37.1	32.3	28.3	25.6	23.0
19.5	2.7651	4.1401	..	..	..	37.8	32.8	28.8	26.0	23.5
20.0	2.8360	4.2110	..	..	..	38.4	33.4	29.3	26.5	23.9

(*e*) REFINING PROCESS—The same method is used as in the case of Prime Oil, with the following variations: After adding the sodium hydroxide solution, stirring is continued for 15 min. without heating; then the oil is heated to 60° C., which temperature is to be reached during an interval of 15 min., and a maximum temperature of 65° C. is allowed.

6. COLOR—(*a*) The color of refined oil is to be reported in terms of Lovibond's equivalent color scale, using yellow and red glasses.

(*b*) The color scale examinations shall be made as follows: Place the oil in a white polished-bottom bottle or cylinder of such depth that a column of oil of 5.25 in. can be accurately measured therein. Such a bottle or cylinder is to be not less than 0.75 in. in internal diameter. The bottle shall be placed in a tintometer which is to be protected from any light except reflected white light, and the reading made at a temperature of 21° C. or by such method as may be recommended by the U. S. Bureau of Standards, provided the same be approved by the Cottonseed Products Committee of the AMERICAN CHEMICAL SOCIETY and provided that the color determined shall be expressed in Lovibond terms. If the oil is of a deeper color than the glass standard 35 yellow, 7.6 red, it shall not be classed as prime.

7. FLAVOR—Oil which is not rancid, musty or sour, and does not have a flavor foreign to cottonseed oil, shall be graded as prime flavor.

V—CRUDE PEANUT OIL

1. REFINING PROCESS—The method of refining shall follow the same general procedure as in the case of Crude Cottonseed Oil (p. 708); but in all cases the stirring shall be continued at least 30 min. before applying heat; and when the free fatty acid does not exceed 3 per cent, one test shall be made using 10° Baumé

sodium hydroxide solution with 0.1 per cent sodium hydroxide in excess of the amount necessary to neutralize the free fatty acids.

VI—CRUDE COCONUT OIL

1. REFINING PROCESS—Follow the same general procedure as in the case of Crude Cottonseed Oil. In the case of oils containing less than 5 per cent free fatty acids the amount of sodium hydroxide used shall not be more than 0.2 per cent in excess of the amount necessary to neutralize the free fatty acids.

VII—CRUDE SOY-BEAN OIL

1. REFINING PROCESS—Follow the same general method as in the case of Crude Cottonseed Oil. In the case of oils containing less than 3 per cent free fatty acids the amount of sodium hydroxide used shall not be more than 0.2 per cent sodium hydroxide in excess of the amount necessary to neutralize the free fatty acids.

VIII—REFINED OILS

1. BLEACHING—Place 300 g. of refined oil in a refining cup, heat to 120° C. and add 6 per cent of standard fuller's earth. Stir thoroughly for 5 min., taking care that the temperature does not fall below 105° C. Filter through filter paper and after sufficient oil has passed the filter to insure clearness, collect a sample for color reading. Cool to 21° C. and determine the color reading on a column 5.25 in. in depth, according to the method under IV, *6a*.

2. COLD TEST—(Applicable to winter oils.) Fill a 4-oz. sample bottle with the oil to be tested, insert a thermometer, and seal hermetically. Warm the oil slowly to a temperature of 26° C. and allow it to remain for 15 min. Chill and hold at a temperature of 0° C. for 5 hrs. If the oil has been properly treated in the winterizing process it will remain clear, brilliant, and limpid at the conclusion of the test.

IX—SOAP STOCK AND ACIDULATED SOAP STOCK

1. PRESERVATION OF SAMPLES—To prevent fermentation and subsequent chemical decomposition in samples of soap stock and acidulated soap stock which are not intended for immediate analysis, 0.1 per cent of oil of cassia must be added and thoroughly mixed with such samples when drawn.

2. TOTAL FATTY ACID—(Not applicable to copra and palm kernel soap stock or acidulated soap stock of same, for which see 3.)

From a weighing bottle weigh out 8 or 10 g. of a well-mixed sample of cottonseed foots, or 4 to 5 g. of acidulated soap stock, and transfer the same to a 400 cc. beaker. Saponify with an excess of alcoholic sodium or potassium hydroxide and after saponification is complete, heat in a steam bath, with stirring, until all the alcohol is driven out. Add 200 to 250 cc. of distilled water and heat until the soap is completely dissolved. Acidify with dilute hydrochloric acid (1 : 1) using only a small excess, cover with a watch glass and continue heating until the fatty acid layer is clear. Cool the fatty acid layer until solid, filter off the liquor and wash thoroughly with cold water. Allow the washed fatty acids to dry, most conveniently overnight. Now dissolve the fatty acids in warm petroleum ether, which has a boiling point below 65° C., and make a total volume of approximately 125 cc. with the solvent. Filter through a dry filter paper into a tared Soxhlet flask. Wash the filter with warm petroleum ether. Finally, evaporate off the petroleum ether, heat in an oven at 100° C. to constant weight, and report as percentage of total fatty acids.

3. METHOD FOR THE DETERMINATION OF THE TOTAL FATTY ACIDS AND UNSAPONIFIABLE MATTER OF COCONUT OILS AND PALM KERNEL OILS—From a weighing bottle weigh out a sample which will furnish approximately 5 g. of fatty acids. Saponify in a 250 cc. beaker, using 50 cc. of 95 per cent alcohol and 2 to 3 g. of stick potassium hydroxide, or an equivalent of a stock solution of potassium or sodium hydroxide. Heat on a steam bath, with occasional stirring, until saponification is complete; then evaporate off the alcohol. This evaporation is facilitated by a current of filtered air. When the alcohol is evaporated, add 100 cc. of water and heat until the soap is dissolved. Wash the contents of the beaker into an extraction cylinder with hot water, taking care to use no more than 130 cc. The glass cylinder must be glass stoppered, graduated at 40 cc., 80 cc., and 130 cc., have a diameter of approximately 1.375 in. and a height of about 12 in. Acidify with dilute hydrochloric acid (1 : 1), being careful to avoid too large an excess. Mix by gently rotating the cylinder and when it has cooled to about 50° C. add 50 cc. of petroleum ether. It is not necessary for the fatty acids to have cleared thoroughly. Stopper the cylinder and shake; then allow it to stand until the petroleum ether layer has separated. Syphon off the petroleum ether through a 9 cm. filter paper into a 600 cc. beaker. Make at least four more extractions, using from 25 to 30 cc. of petroleum ether each time. Allow the filter to drain well; then wash with a spray of petroleum ether from a wash bottle until all the fatty acids are extracted. Add 50 cc. of 95 per cent ethyl alcohol, redistilled from caustic, to which a few drops of phenolphthalein have been added. Titrate with N sodium hydroxide, evaporate off the petroleum ether on a steam bath, then concentrate until the volume is reduced to about 25 to 30 cc., and transfer with hot 95 per cent alcohol redistilled from caustic to a 250 cc. beaker, tared, including a stirring rod. Finally, evaporate and dry in an oven at 105° to 110° C. to constant weight. In order to calculate the soda soap to fatty acids, a correction must first be made for the neutral salts in the caustic solution. To do this, neutralize 20 cc. of normal caustic soda with 0.5 N hydrochloric acid, using a small amount of phenolphthalein as indicator. Evaporate to dryness. Heat to constant weight at 105° to 110° C. and from the weight of the residue found subtract the weight of salt calculated. The difference divided by 200 gives the correction per cubic centimeter for neutral salts. From the weight of the soda soap subtract the product of the titration of the fatty acids, multiply by the factor (0.022 plus correction for neutral salts), divide the result by the weight of sample used, and multiply by 100, which gives the result to be reported as percentages of total fatty acids and unsaponifiable matter.

X—TITER

The method to be employed is that for fat given in THIS JOURNAL, 11 (1919), 1163, under "Standard Methods for the Sampling and Analysis of Commercial Fats and Oils."

XI—ALL OTHER TESTS

In the case of tests not covered by these standard methods, the methods of the AMERICAN CHEMICAL SOCIETY are to apply, and in cases where such methods have not been provided by the SOCIETY, those of the Association of Official Agricultural Chemists[1] shall be considered satisfactory methods.

APPENDIX—OIL GRADES[2]

CRUDE OIL

CHOICE CRUDE cottonseed oil must be made from sound decorticated seed; must be sweet in flavor and odor, free from water and settlings, and shall produce, when properly refined, choice summer yellow oil at a loss in weight not exceeding six per cent.

PRIME CRUDE cottonseed oil must be made from sound decorticated seed; must be sweet in flavor and odor, free from water and settlings and must produce prime summer yellow oil with the use of caustic soda by the official methods adopted by the Chemists Committee, with a loss in weight not exceeding 9 per cent; provided, that any oil that refines with a greater loss than 9 per cent, but still makes prime summer yellow oil, shall not be rejected, but shall be reduced in price by a corresponding percentage of the contract price of the oil.

---

[1] Published by the Association of Official Agricultural Chemists, Washington, D. C.

[2] Adopted by the Interstate Cottonseed Crushers' Association.

OFF-CRUDE cottonseed oil, if neither choice nor prime, shall be called "Off Oil."

REFINED OIL

CHOICE SUMMER YELLOW cottonseed oil must be sweet in flavor and odor, prime in color, clear and brilliant in appearance and free from moisture.

PRIME SUMMER YELLOW cottonseed oil must be clear, sweet in flavor and odor, free from water and settlings, and of no deeper color than 35 yellow and 7.6 red on Lovibond's equivalent color scale.

PRIME WINTER YELLOW cottonseed oil must be brilliant, free from water and settlings, sweet in flavor and odor, and of prime summer yellow color as described above, and must stand limpid at a temperature of 32° F. for 5 hrs.

GOOD OFF SUMMER YELLOW cottonseed oil may be off in flavor and odor, must be prime in color and free from water and settlings, and shall not contain more than 0.25 per cent of free fatty acid.

OFF SUMMER YELLOW cottonseed oil shall be free from water and settlings, off in flavor or odor, but of no deeper color than 35 yellow and 12 red on Lovibond's color scale and shall not contain more than 0.5 per cent of free fatty acid.

REDDISH OFF SUMMER cottonseed oil, designated as such, may be of inferior flavor and odor and of no deeper color than 35 yellow and 20 red on Lovibond's equivalent color scale, shall be free from water and settlings and shall not contain more than 0.75 per cent free from fatty acid.

BLEACHABLE PRIME SUMMER YELLOW cottonseed oil must be clear, sweet in flavor and odor, free from water and settlings, and when bleached shall be of no deeper color than 20 yellow and 2.5 red on Lovibond's equivalent color scale.

PRIME SUMMER WHITE cottonseed oil must be clear, free from water and settlings, sweet in flavor and odor, and the color of the oil shall not be darker than the combined standard glasses, 20 yellow and 2.5 red, Lovibond's color scale.

PRIME WINTER WHITE cottonseed oil must be brilliant, sweet in flavor and odor, free from water and settlings, and the color of the oil shall not be darker than the combined standard glasses, 20 yellow and 2.5 red of Lovibond's color scale, and must stand the cold test as prescribed.

ANNUAL MEETING OF THE DIVISION OF CHEMISTRY AND CHEMICAL TECHNOLOGY, NATIONAL RESEARCH COUNCIL

The annual meeting of the Division of Chemistry and Chemical Technology, National Research Council, was held in Washington, D. C., May 7, 1920. There were present Messrs. Alsberg, Bancroft, Bleininger, Derick, Fink, Francis, Johnston, Lamb, Moore, Noyes, Stieglitz, Washburn; and by invitation Messrs. Angell, Christian, Cottrell, Kellogg, Mendenhall, Munroe, and Yerkes.

The following officers were elected for the ensuing year: Vice Chairman, Julius Stieglitz; Members-at-Large, A. A. Noyes, E. W. Washburn. The members of the Executive Committee will be the chairman and vice chairman, C. L. Alsberg, A. B. Lamb, John Johnston, and W. D. Bancroft, *ex officio*, retiring chairman. The American Chemical Society nominated C. L. Alsberg, W. D. Bancroft, and C. G. Derick as members of the division, and the American Institute of Chemical Engineers nominated H. K. Moore.

In connection with the meeting of the International Chemical Union to be held in Rome in June, Dr. Charles L. Parsons was appointed delegate. The admission to the Union of Poland and Czecho-Slovakia was favored, the division expressing the unanimous opinion that any neutral nation, eligible from the point of view of its scientific activities, that might apply for admission should be admitted.

In presenting the report of the Committee on Synthetic Drugs, Julius Stieglitz, chairman, pointed out the valuable work done by this committee in furnishing information and advice to manufacturers. The committee on request had acted in advisory capacity to the Chemical Foundation, which controls most of the important formerly foreign-owned patents. The committee was convinced that real service could be rendered, particularly to the organic chemical industries, through the existence of such a committee of unbiased men of scientific training and of experience in public affairs without any trade interests. The committee was continued for another year.

The report of the Committee on Explosives Investigations was presented by the chairman, Professor Charles E. Munroe. The following excerpts from the report indicate the various lines of activity of the committee:

> The committee has been engaged in arranging for, coöperating with or supervising investigations on the corrosion of rifle barrels; the development of Neumann Bands in metals subjected to exposure to the detonation of explosives of known rates or to blows from impact machines with a view to ascertaining if these bands may be used as a means of determining whether a fracture in metal is due to mechanical stress or strain or to the effects of an explosive; the utilization of surplus military explosives (1) by developing their adaptabilities for employment as explosives in industries; (2) by determining their availability for use as or conversion into fertilizers; products of combustion and detonation of TNT; heats of combustion and detonation of TNT isomers; development of insensitiveness in TNT during storage; nitration of corn-cob cellulose; the fudging of ammonium nitrate and the effect of this material on the rate of detonation of gelatin dynamite; coefficient of expansion of nitroglycerin; determination of nitro-sucrose in the presence of nitroglycerin; production of flashless powder; coloring matter for special shell; water storage of explosives; premature explosion of nitroglycerin in oil well shooting; development of modified TNT; catalytic nitrations.
>
> The committee has served as an intermediary in effecting coöperative agreements for investigations and developments between the Bureau of Mines and the College of Agriculture of the University of Wisconsin; the Ordnance Department, U. S. A., and several of our universities; the Bureau of Mines and E. I. du Pont de Nemours & Co. It has served as a channel of communication and for transmission of documents and publications between our people and specialists in England, France, Italy, Canada, Australia, and India; also as a general bureau of information, it having been called upon for service by a large number of the divisions of the State, Treasury, War, Navy, Interior and Agricultural Departments, as well as by officials in the industries and by students preparing theses. The information card catalog has been continued and now contains about 4,000 cards.
>
> Following the expressed desire of the Council, Munroe's "Index to the Literature of Explosives" is now being brought up to 1907, at which date the publication of *Chemical Abstracts* was begun, and the completed manuscript has now been placed in the vault of the Council for the following journals: *Philosophical Transactions Royal Society*, 1665–1906; *American Journal of Science*, 1819–1906; *Proceedings U. S. Naval Institute*, 1878–1906; *Journal Society of Chemical Industry*, 1882–1906.

As a result of this coöperation, various articles have appeared in scientific publications.

The Committee on the Thermal Properties of Explosive Materials was not continued, the work being transferred to the Committee on Explosives Investigations. This latter committee was requested to associate with itself W. P. White and others interested in the study of the thermal properties of explosives.

In the absence of H. N. Holmes, chairman of the Committee on Colloids, the report of the committee was presented by W. D. Bancroft. This report gave an account of the courses of lectures given at universities, industrial plants, government bureaus, and local sections of the American Chemical Society by W. D. Bancroft, H. N. Holmes, W. A. Patrick, I. N. Kugelmass, and Martin Fisher. The need of suitable texts and laboratory manuals for the teaching of colloid chemistry was emphasized and the announcement made that W. D. Bancroft now has in preparation a textbook soon to be published, while a laboratory manual is being prepared by H. N. Holmes. A detailed classified bibliography on colloid literature is being compiled which it is hoped to publish later. A small manual for the use of the ultramicroscope is being prepared by Jerome Alexander.

Following remarks by John M. Francis, the Committee on Pharmaceutical Research was authorized and requested to formulate a program of research. The report of the Committee on Ceramic Research was read and the committee continued for another year.

Owing to delays in getting organized, the Committee on Sewage Disposal, of which Edward Bartow is chairman, had no report, but as definite plans for future activity of the committee were being formulated, the committee was continued for another year.

Upon the suggestion of C. G. Derick, a Committee on Methods of Organic Analysis was appointed.

The need for coöperation between the various laboratories and individuals working on contact catalysis was pointed out by Chairman Bancroft, and as a result a Committee on Contact Catalysis was appointed, with W. D. Bancroft as chairman.

The report of the Board of Trustees for the Publication of Critical Tables of Physical and Chemical Constants was presented by H. K. Moore. The Council approved the suggestion that a special agent be employed to devote his entire time to the solicitation of funds for this publication. Thereupon H. E. Howe was appointed a fourth member of the Board of Trustees; W. D. Bancroft and C. E. Mendenhall were authorized to pay for the drawing up of a preliminary plan for the scientific organization of the Tables, as a concrete basis for obtaining subscriptions. C. G. Derick called attention to the need of constants in the dye industry to determine the purity of materials.

Dr. Cottrell was appointed to draw up a resolution in support of the Patent bill, with instructions to forward the resolution to the Patent Office Committee of the National Research Council for such action as it saw fit to take.

PRESENTATION OF HONORARY MEMBERSHIP CERTIFICATE TO M. ERNEST SOLVAY

On May 27, 1920, in the city of Brussels, the United States Ambassador, Hon. Brand Whitlock, in company with Dr. Charles L. Parsons, Secretary of the American Chemical Society, presented to M. Ernest Solvay, on behalf of the Society, an engrossed certificate of honorary membership, carrying the Society's seal and colors. M. Solvay, though 83 years of age, is in vigorous health, as is evidenced by the fact that he walks five miles each day from his country place, reaching his office at 8 A. M. M. Solvay was deeply appreciative of the honor of honorary membership and the method of its presentation.

U. S. PHARMACOPOEIAL CONVENTION, CENTENNIAL MEETING

The eleventh decennial United States Pharmacopoeial Convention met at the New Willard Hotel, Washington, D. C., on May 11 to 13, 1920, celebrating the centennial of our national pharmacopoeia and arranging for the new revision of that standard of drugs and medicines.

An outstanding feature of the convention was the presidential address of Harvey W. Wiley, in which he described the first convention (that of 1820) and cited the sacrifices made by the pharmacopoeia makers of the intervening century; and outlined the ideals that should govern the coming revision.

Other important business transacted was the election of officers, trustees, and members of the Revision Committee of fifty, and the adoption of general principles upon which the forthcoming revision will be based.

The newly elected president of the convention is Dr. Reid Hunt, of Harvard Medical School, while Dr. James H. Beal, of Urbana, Illinois, was re-elected chairman of the trustees.

The committee of fifty elected to carry on the revision consists of seventeen members chosen by the medical delegates and thirty-three chosen by the pharmaceutical delegates. The following members of the American Chemical Society were among those chosen by the pharmaceutical delegates:

C. L. Alsberg
H. V. Arny
G. M. Beringer
T. J. Bradley
C. E. Caspari
A. H. Clark
E. F. Cook
A. R. L. Dohme
A. G. DuMez
F. R. Eldred
J. M. Francis
L. B. Havenhill
C. B. Jordan
C. W. Johnson
Henry Kraemer
C. H. LaWall
B. L. Murray
E. L. Newcomb
F. W. Nitardy
W. O. Richtman
G. D. Rosengarten
W. L. Scoville

Two of the three delegates to the convention from the American Chemical Society were selected as members of the Revision Committee, while the chairman of the five sub-committees most concerned in the chemicals of the pharmacopoeia are likewise members of the Society. These are H. V. Arny (inorganic chemicals), G. D. Rosengarten (organic chemicals), C. H. LaWall (reagents and test solutions), W. O. Richtman (volatile oils), and C. E. Caspari (drug assays). E. Fullerton Cook, of Philadelphia, was elected chairman of the Revision Committee.

The convention adjourned on May 13 to meet next in May 1930.

MANUFACTURING CHEMISTS' ASSOCIATION

At the annual meeting of the Manufacturing Chemists' Association of the United States, held June 9, 1920, the following officers were elected: President, Dr. Charles L. Reese, of E. I. du Pont de Nemours & Co.; vice presidents, H. H. S. Handy, of the Semet-Solvay Company, and C. Wilbur Miller, of the Davison Chemical Company; treasurer, Lindsley Loring, vice president of the Merrimac Chemical Company; secretary, Arthur H. Weed, 84 State St., Boston; executive committee, Henry Howard, of the Grasselli Chemical Company, Lancaster Morgan, of the General Chemical Company, H. H. Dow, of the Dow Chemical Company, Adolph Rosengarten, of the Powers-Weightman-Rosengarten Company, Mr. Miller, and D. W. Jayne, of The Barrett Company. The chief business concerned the annual report of the executive committee, which emphasized the success of the committee in coöperating with the Chemical Foundation, and gave particular attention to the progress of dye legislation. In spite of the discouraging delays, the Association is confident of the ultimate adoption by Congress of adequate protective legislation. The work of the National Industrial Conference Board, particularly in the campaign for the revision of the tax laws, was commended.

CALENDAR OF MEETINGS

Insecticide and Disinfectant Manufacturers' Association—Midsummer Meeting, Boston, Mass., July 15 and 16, 1920.

Forest Products Laboratory Decennial Celebration—Madison, Wis., July 22 and 23, 1920.

American Chemical Society—Sixtieth (Annual) Meeting, Chicago, Ill., September 7 to 10, 1920.

Sixth National Exposition of Chemical Industries—Grand Central Palace, New York, N. Y., September 20 to 25, 1920.

NOTES AND CORRESPONDENCE

UNSAPONIFIABLE MATTERS

Editor of the Journal of Industrial and Engineering Chemistry:

In THIS JOURNAL, 11 (1919), 1161, is given the final report of the Committee on Analysis of Commercial Fats and Oils of the Division of Industrial and Engineering Chemistry of the AMERICAN CHEMICAL SOCIETY, as adopted April 14, 1919, by unanimous vote.

Anyone who has served on such a committee knows the great amount of work required of its members and the difficulty of harmonizing varying ideas, with the result that the final conclusions are apt, to some extent, to be compromises. Owing to the difficulties of getting members together and actually illustrating certain points by tests, all points cannot be satisfactorily worked out, without unduly prolonging the time required to make a report.

We wish here to call particular attention to the method adopted for the determination of unsaponifiable matters. Although this method is better than any other published commercial method with which we are acquainted, in that it is easy to carry out and gives fairly concordant results, it contains a source of error which may not be constant, and which is very easily eliminated with practically no additional work. We refer to the presence of free fatty acids in the final residue recorded as "unsaponifiable matter." By the method of extraction and washing employed this final residue always contains more or less free fatty acids, yet the committee made no mention of the fact nor suggested any method of correction. Although this point was called to the attention of the committee and illustrated by examples, they did not see fit to add to their method the operation which would eliminate the error in question.

During the petroleum ether extractions the amount of alcohol in the solution are not over 30 per cent by volume. Fifty per cent of alcohol are required to prevent hydrolysis of most soap solutions. Therefore, unless there is a sufficient excess of alkali at this point, the soap is bound to undergo hydrolysis to some extent, with the result that the petroleum ether extracts will contain some free fatty acids. They will also, probably, contain some soaps. The combined extracts are washed with 10 per cent alcohol washes, with the result that any soaps present in the petroleum ether extracts may undergo some hydrolysis. The final residue is, therefore, bound to contain free fatty acids, and not necessarily in constant proportional amounts. Actual tests show this to be the case.

Twitchell's method,[1] which is much the same as the committee method but employs ethyl ether for extraction, takes into account the free fatty acids left with the final residue and deducts their amount, calculated as oleic acid. The objection to this method is that these free fatty acids are not oleic acid, and in many cases a serious error is introduced by so considering them.

On trying the committee method we have always found the final residue to contain titratable amounts of free fatty acids, and it is obvious that any free fatty acids in this residue should be deducted from unsaponifiable matter. It might be claimed that the amount of free fatty acids would be practically constant and that they would make little difference in a commercial method. But the amount of free fatty acids left is not constant and the residue may contain more free fatty acids than it does true unsaponifiable matter.

Using the committee method as a basis, we modified the method of washing the petroleum ether extracts, by introducing a wash with dilute caustic solution, whereby free fatty acids are removed from the petroleum ether and hydrolysis of any soap prevented, at least to a great extent. Our residues contain either no titratable amounts of free fatty acids, or such small amounts that considering them as oleic acid will introduce no appreciable error.

Our method is as follows: Five g. of the fat or oil are saponified according to the committee method, which is followed in all details up to obtaining the petroleum ether extracts. The extracts are washed with 25 cc. portions of 10 per cent (10 cc. diluted to 100 cc.) alcohol as directed, then with 0.5 N sodium or potassium hydroxide solution, followed by 10 per cent alcohol again, and then with water until all alkali has been washed out. Determinations by the committee method and our modification are given below:

UNSAPONIFIABLE MATTER IN SOY-BEAN OIL BY COMMITTEE METHOD

Five petroleum ether extractions were made and the extracts combined before washing. After three washes with 10 per cent alcohol (25 cc. each time), the extracts were transferred to a weighed flask, the ether evaporated off, and the residue dried in an air oven at 105° C. for about half an hour, cooled, and weighed.

	Gram	Per cent
Residue recovered..................	0.0422	0.844
Free fatty acid titrated with 0.1 N NaOH (0.29 cc.), combining weight of 279 (found for pure soy-bean oil fatty acids)......................	0.0081	0.162
Net unsaponifiable matter........		0.682

In a second test the method was modified to include a wash with dilute hydrochloric acid, as in Twitchell's method.

	Gram	Per cent
Residue recovered..................	0.0532	1.064
Free fatty acid titrated as before (0.72 cc.)............................	0.0201	0.402
Net unsaponifiable matter........		0.662

MODIFICATION OF THE COMMITTEE METHOD

Five grams of oil were saponified as directed by the committee, and their method followed until after the first set of alcohol washes. The extract was then washed with 25 cc. of 0.5 N sodium hydroxide solution, followed by alcohol washes and water until alkali was all washed out.

	Gram	Per cent
Residue recovered (No 0.1 N NaOH required for free fatty acids)......	0.0337	0.6740

A second test followed, modified to include a wash with dilute hydrochloric acid after the first set of alcohol washes, followed by our regular method, including an alkaline wash.

	Gram	Per cent
Residue obtained (No 0.1 N NaOH required for any free fatty acids left in residue)......................	0.0325	0.6500

These illustrations are sufficient to show that free fatty acids are left in the residue obtained by the committee method, while by our modification no titratable amount of free fatty acids is left in most cases, but sometimes there are very small amounts. When we heat the residues with neutral alcohol, add phenolphthalein and one drop, or a fraction of a drop, of 0.1 N sodium hydroxide and obtain a strong permanent red coloration, we consider that there is no titratable amount of free fatty acids present.

The above matter illustrates our point, but it may be of interest to compare the above methods with a similar method, using ethyl ether instead of petroleum ether for extractions.

It is to be noted that ether always gives higher results than petroleum ether. This is to be expected, as petroleum ether is a poorer solvent than ether for oxidized and hydroxy fatty matters. Ether probably gives more accurate results, but for a commercial method petroleum ether appears more satisfactory.

---

[1] THIS JOURNAL, 7 (1915), 217.

Test 1—Five grams were saponified as usual, transferred to an extraction cylinder, made up to 40 cc. with alcohol, treated with 120 cc. of water and 50 cc. of purified (water- and alcohol-free) ether, shaken, and separated. After five extractions the ether extracts were joined and washed with dilute hydrochloric acid as in Twitchell's method, then with *N* KOH, and then with water until free from alkali.

	Gram	Per cent
Residue obtained	0.0445	0.8900
Free fatty acid titrated with 0.1 *N* NaOH	0.0056	0.1116
Net unsaponifiable matter		0.7784

Test II—This was a duplicate of Test I.

	Gram	Per cent
Residue obtained	0.0450	0.90
Free fatty acids	0.0056	0.11
Net unsaponifiable matter		0.79

Test III—This was the same as Tests I and II, except that 75 cc. of ether were used the first time and three additional 50 cc. extractions were made.

	Gram	Per cent
Residue obtained	0.0498	0.9960
Free fatty acid	0.0073	0.1460
Net unsaponifiable matter		0.850

Test IV—This was the same as Test III, except that a wash with hydrochloric acid was included.

	Per cent
Corrected unsaponifiable matter	0.8361

DRY EXTRACTION METHOD

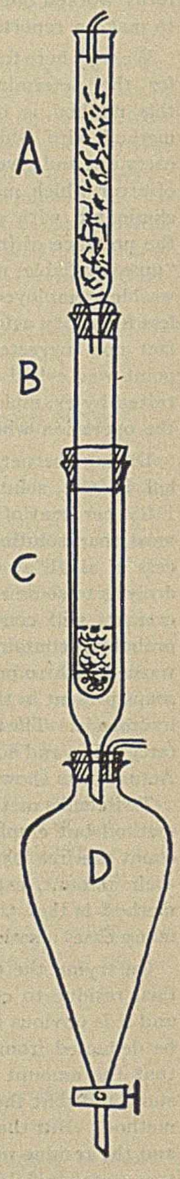
Comparison with a dry extraction method may be of interest, particularly in connection with another extraction of soy-bean oil of which we will speak later. Our method of making a dry extraction uses a special arrangement of apparatus and ethyl ether purified from water and alcohol. This is obtained by adding a good excess of dry granular calcium chloride, shaking well at intervals, and allowing to stand overnight. Filter off the ether (protecting from circulation of air) and to each liter of ether add 50 g. of dry rosin and distil off the ether. The apparatus is illustrated.

GENERAL METHOD OF OPERATION—The fat or oil is saponified by boiling with excess of alcoholic alkali, the alcohol evaporated off, and drying finished in a vacuum oven at as high a heat as the material will stand. When perfectly dry and free from alcohol (usually within a short time) the material is mixed with at least twice its weight of dry soda ash and finely ground in a glass mortar and placed in the extraction tube. About 75 cc. of ether, purified from water and alcohol, are run in on top of the material to be extracted and allowed to percolate through it. The percolate will generally be so strong in unsaponifiable matter by the time it reaches the end of this tube that it is ready to crystallize. After one or two percolations the material is removed from the tube, dried for a short time, and repulverized to expose new surfaces. This is the only drawback to the dry extraction method, but is not as much trouble as it would appear. There is generally no trouble with the percolation, if the ether is properly purified, but suction may be used, if necessary.

APPLICATION TO SOY-BEAN OIL—Five grams of the oil were saponified as usual, the alcohol driven off, and the resulting mass dried and mixed with 20 g. of anhydrous sodium carbonate, ground finely in a mortar and put in the tube. One hundred and fifty cc. of purified ether were used for the first extraction. Percolation was steady and became faster as extraction proceeded. Each ether extract was washed first with neutral water to see if there was any indication of alkali in solution, as shown by phenolphthalein. A little was shown each time, but with other stocks these extracts are often free from any soap or other alkali. The water wash was then followed by a wash with 0.5 *N* NaOH solution to remove any soaps and prevent hydrolysis as much as possible. Finally all sodium hydroxide was washed out with pure water and the matter remaining in solution recovered.

	Gram	Per cent
First extract with 150 cc. dry ether	0.0541	1.082
Free fatty acids, 0.03 cc. 0.1 *N* NaOH	0.0008	0.016
Net unsaponifiable	0.0533	1.066
Reground mixture and extracted again:		
Second extract with 150 cc. dry ether	0.0035	0.070
No free fatty acid indicated.		
Reground again:		
Third extract with 150 cc. dry ether	0.0018	
Free fatty acid, 0.05 cc., 0.1 *N* NaOH	0.0014	
Net unsaponifiable	0.0004	0.008
Reground again:		
Fourth extract with 150 cc. dry ether	0.0004	0.008
Free fatty acid too small to titrate		
Total unsaponifiable matter		1.150

To all appearances the unsaponifiable matter obtained by the dry method was the same as that obtained by the wet method referred to below. It is noticeable how much higher the unsaponifiable matter is than is shown by either the committee method or by our modification.



A—Large tube filled with granular calcium chloride, to keep moisture of air from entering apparatus

B—Extraction tube with several small holes through bottom. Into this tube is put the powder to be extracted. There is a plug of cotton wool in the bottom and a perforated lead plate just above it to hold it in place and make a seat for the substance to be extracted. It also facilitates removal of the substance for regrinding

C—Adapter to allow extraction tube to be connected with 500 cc. (or other size) separatory funnel

D—Squibb's form of separatory funnel. Proportions are about as shown, diameters of tubes varying from an inch to an inch and a half

WET EXTRACTION METHOD

WET EXTRACTION OF UNSAPONIFIABLE MATTER FROM SOY-BEAN OIL—Soy-bean oil was filtered through paper and 50 g. of the filtered oil used. Without going into details, the general method was to saponify with excess of alcoholic potash solution, and dilute with water so that the resulting solution would contain about one-fourth of its volume of alcohol. After making about three ether extractions at this dilution, more water was added to reduce the alcohol content to about one-sixth of the total volume, and further extractions with ethyl ether were made. The various residues obtained were reboiled with excess of alcoholic potash and the unsaponifiable matter reëxtracted. Unfortunately the first and main residue was not tested for free fatty acids, and so probably appears higher than it should, and the spread, after resaponification and extraction, is greater than it would be had the fatty acids been deducted.

SUMMARY OF OPERATIONS

The residue (uncorrected for free fatty acids) amounted to 1.16 per cent unsaponifiable matter, which checks with that found by the dry method, which, however, was corrected for any free fatty acids. This residue was resaponified with the following results:

	Per cent from 1/4 alcohol	Per cent from 1/6 alcohol	Per cent Total
	1.1500	0.0100	1.1600
Recovered upon first resaponification.........	0.8420	0.0036	0.8456
Recovered upon second resaponification.......	0.7840	....	0.7840
Recovered upon third resaponification.......	9.7183	0.0240	0.7423
Recovered upon fourth resaponification.......	0.6770	trace	0.6770

It is evident that we have continually lost weight and it would seem that this loss cannot be satisfactorily explained by failure to extract each time the full amount of unsaponifiable matter left, but rather points to the action of alcoholic alkali on the residues, either in decomposing some of the true unsaponifiable matter or in completing the saponification of some very difficultly saponifiable matter, which would then be present in all ordinary residues and reckoned as unsaponifiable matter. The resaponifications and reëxtractions were stopped at the above point because the amount of free fatty acids to be corrected for had apparently reached a constant figure in the last two cases. Whether this was the true end we doubt. The point happens to give the same final result as the determination by the committee method, but this may be considered only a coincidence.

THE CUDAHY PACKING COMPANY
OMAHA, NEBRASKA
December 15, 1919

WILSON H. LOW

....

Editor of the Journal of Industrial and Engineering Chemistry:

Mr. Low's remarks in the early part of his paper, entitled "Unsaponifiable Matters," on the difficulties which confront committees working on analytical methods are interesting but do not apply to the committee of which I am chairman. This committee made a practice of meeting regularly once a month during its period of greatest activity, and thereafter meetings were held whenever a sufficient number of results from coöperative work had accumulated to warrant calling a meeting. Conferences in various laboratories were held from time to time for the purpose of coöperating on one or another moot point in the various methods, several members of the committee meeting in this way first in one laboratory and then in another until the point was settled and unanimity reached. Such coöperative work was done on the unsaponifiable method.

It is difficult for any committee to adopt a method of analysis satisfactory to all workers and this is perhaps particularly true of the unsaponifiable determination. In my opinion, however, the method finally adopted by the committee in point of accuracy, convenience, simplicity, speed, and reliability in various hands comes as near perfection as we can hope for in the present state of our knowledge.

The point which Mr. Low discusses was brought to the committee's attention by him. After careful consideration the committee decided that it was not necessary to add the manipulation advocated by Mr. Low. Mr. Low states that "By the method of extraction and washing employed this final residue always contains more or less free fatty acids, yet the committee made no mention of the fact nor suggested any method of correction." The answer to this statement is that the amount retained is very small; so small, in fact, as to make no appreciable difference in the results, particularly for commercial purposes. In the majority of samples of oils, fats and greases the amount of free fatty acids at this point usually varies from 0.02 to 0.07 per cent, rarely it amounts to as much as 0.1 per cent; and the high figures are generally found in the case of an oil whose general peculiarities are well known—soy-bean oil. Considered as an absolute error on the positive side, this amount is not great and not worth taking into consideration in the commercial analysis of oils and fats. On the other hand, from the point of view that it may be a counter-balancing error working against incomplete extraction, it is of still less moment. Good manipulation reduces the amount, poor manipulation increases it. Judging by his own figures in comparison with the results by members of the Fat Analysis Committee, Mr. Low's manipulation is open to criticism.

Mr. H. J. Morrison, of the Procter and Gamble Company, writes as follows in regards to Mr. Low's argument:

> I have read over carefully Mr. Low's paper in which he criticizes the method for determining the unsaponifiable matter in the Standard Methods for the Analysis of Commercial Fats and Oils.
>
> Mr. Low's paper is worded unfortunately in that one gets the impression that the committee did not accept criticisms and did nothing to investigate them. This, of course, is quite wide of the mark.
>
> On that particular point raised by Mr. Low I believe we did more work than on any other point raised by outside criticism. The committee was quite alive to the danger of high results due to hydrolysis. One point which we discussed was the amount of caustic alkali to be used for the saponification, and the amount was fixed at a very large excess over the theoretical for the purpose of rapid and complete saponification, on the one hand, and to have an excess present to prevent hydrolysis when diluted for extraction, on the other hand. Mr. Low mentions an excess as a preventive of hydrolysis but either has not calculated the amount of excess the method calls for or must consider a very excessive amount necessary to prevent hydrolysis.
>
> Doubtless Mr. Low has many more than the one analysis by the committee method on which he bases his contention that a serious error is introduced by this hydrolysis. From the experience of this laboratory I cannot help but feel that the source of Mr. Low's error is due to faulty technique. In order to protect our results from carelessness we have practiced having the unsaponifiable titrated and in 445 such titrations on the great variety of fats, oils and greases which pass through our laboratory the average titration was 0.076 cc. 0.1 *N* NaOH, equal on 5 g. of fat to 0.04 per cent. This is quite within the limit of error of this particular operation, and I may point out that these 445 results are of course the daily routine analyses, by routine men such as are employed in such laboratories.
>
> Mr. Low seems to explain the error and its necessary correction in the ethyl ether extraction method by the hydrolysis theory. This is not at all the case as I think we demonstrated. It is due to actual solution of soap, owing to the solubility of both water and alcohol in the ethyl ether phase. Mr. Low's error when using the committee method, as stated above, is due, I believe, to technique, possibly by not allowing sufficient time for a thorough separation or by drawing off the petroleum ether extract too close.
>
> I am not going to enter a discussion here concerning the other methods Mr. Low speaks of, as I think it patent to anyone conversant with fats and their analysis wherein they cannot give correct results. Our committee, I believe, cannot be accused of not trying out any suggestions and I feel are willing now at any time to try out anything which promises an improvement, but I feel that this should be done by direct appeal to the committee and not by the method of rushing into print, unless the author has irrefutable proof.

The amount of free fatty acids left in the final residue is usually neutralized by one drop of 0.1 *N* alkali. The one drop of 0.1 *N* alkali may or may not be an excess but frequently does amount to an excess. This point is indicated in Mr. Morrison's letter and is considered more at length in a letter received from Mr. Rudnick, of Armour and Company's laboratory, which follows:

> The assumptions made in the first paragraph of the paper by Wilson H. Low on unsaponifiable matters are not warranted by the facts. The methods adopted by the Fat and Oil Committee were not based merely on ideas, but on the outcome of actual tests, so that the final conclusions are not compromises of varying ideas. Indeed the outcome of the coöperative work done by the committee more than once upset long-held views

and while these views were sometimes abandoned with reluctance, in the end the votes of the members were always based on the facts developed.

This committee had no particular difficulty in point of attendance of its members. Occasionally differences in results became apparently irreconcilable by the usual coöperative procedure and in such cases special subcommittees were appointed to carry out the required tests jointly in the several laboratories in order to develop under personal observation the facts necessary to cover the disputed points.

None of the members of the committee reported any unusual difficulties in determining the unsaponifiable matter in soy-bean oil, to which the entire discussion in Low's paper is devoted, although it is well understood that some samples of soy-bean oil show an erratic behavior in other respects, notably in the determination of the titer.

It is difficult to see how any sound criticism of the committee method for determining unsaponifiable matter can be based on the data presented in Mr. Low's paper. The total amount of unsaponifiable matter obtained by him in accordance with the committee method and without applying his corrections for fatty acids, is well within the normal limits for natural unsaponifiable matter in soy-bean oil.

In order to determine, however, whether such corrections as described by him were true of other samples of soy-bean oil than the one on which he reports, a sample of crude oil was tested in this laboratory with the following results:

Weight of Sample	Unsaponifiable Matter Gram	Per cent
5.015 grams	0.050	1.00
5.012 grams	0.056	1.11
Mean		1.06

Fifty milligrams of unsaponifiable matter is a very small amount to titrate for free fatty acids and it was therefore not surprising that titration with 0.1 N alkali, observing the precautions directed in the committee method, should amount to 0.4 cc., equivalent to 0.22 per cent of free fatty acids, calculated as oleic, on the basis of the weight of the first sample.

On repeating the titration with 0.02 N alkali and carrying along a blank titration, matching as closely as possible the final color of the phenolphthalein, the net titration amounted to 0.72 cc. of 0.02 N alkali, equivalent to 0.08 per cent of free fatty acids, calculated as oleic, on the basis of the weight of the second sample.

It would seem, therefore, that if the corrections developed in Low's paper had been determined with a standard alkali of more appropriate concentration relative to the weight of material titrated, he might have obtained results on his sample of soy-bean oil of about the same order as those which the members of the committee obtained in their original coöperative work on which the method adopted was based.

There is nothing new in the work described in Low's paper on the Twitchell and the dry extraction methods. Both these methods were thoroughly tested by the committee and were rejected on the basis of the results obtained with them in coöperative work.

Summing up, there seems to be no good ground for criticism of the committee method for determining unsaponifiable matter, either on the basis of the data presented in this paper or the method by which they were obtained.

Mr. Low speaks of Twitchell's method "which is much the same as the committee method." Anyone who is acquainted with the two methods must realize that the methods are distinctly and decidedly different in principle.

As to the dry extraction method mentioned by Mr. Low, this has been tried out in this laboratory off and on for the past fifteen years and has been found on the whole to be inaccurate and unreliable. The Committee on the Analysis of Commercial Fats and Oils also tried the method out and arrived at the conclusion that the method was wholly impossible and had no place among modern methods of analysis.

Since the adoption of the committee method, agreements on unsaponifiable determinations which were impossible before, have been reached between various laboratories and the method has proved a great advantage to the oil and fat trade.

The committee does not consider the additional alkali wash advocated by Mr. Low as either necessary or desirable if the committee method is followed and a moderate degree of skill acquired in manipulation.

Chemical Laboratory of
Swift & Company
Chicago, Illinois
March 31, 1920

W. D. Richardson,
Chairman, Committee on the Analysis of Commercial Fats and Oils

AWARDS FOR THESES ON FOODS AND BEVERAGES

In order to stimulate more general research along the lines of better preparation and packing of foods and beverages, and to increase our knowledge of changes induced by preparation or storage of food products, Dr. A. W. Bitting, Director of Research of the Glass Container Association of America, Chicago, Ill., has announced that awards will be made for theses on these subjects.

A thesis may cover any phase of the subject of foods or beverages—technological, bacteriological, or chemical. It may treat of any legitimate method of preparation, as sterilization by heat, pasteurization, salting, drying, smoking, pickling, sugaring, etc., the product to be packed in glass. The thesis may be bibliographical with abstracts, or may be a translation from work along the lines indicated. It must be submitted prior to June 10, 1921.

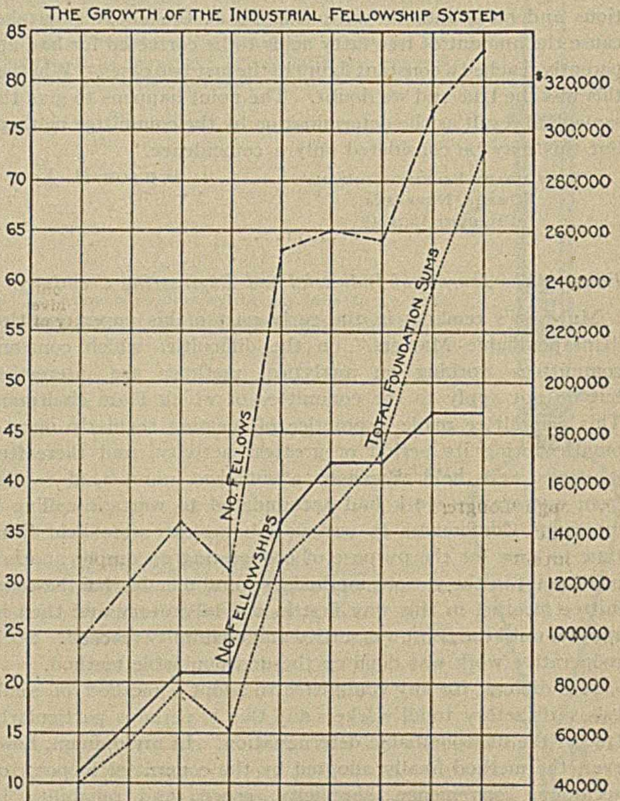
The awards will be as follows:

One	$150
One	125
One	100
Two	75
Two	50

Any student or graduate student working for a degree in any college or university is eligible to compete. The awards will be made by a competent committee and as soon after June 10, 1921, as practicable.

THE INDUSTRIAL FELLOWSHIPS OF THE MELLON INSTITUTE

Under the above title there has been issued recently by the Director of the Mellon Institute for Industrial Research, Dr. Raymond F. Bacon, an attractive pamphlet covering the progress of the industrial fellowship system during the past fiscal year. The growth of the Institute's system is described in detail and shown graphically in the accompanying figure, which has been reproduced from the booklet. The new form of contract, which replaces the one reprinted in This Journal, 11 (1919), 371, is included, also a list of the fellowships in operation on March 1, 1920. The following fellowships have been established since the issuance of the 1919 report:



No.	Names of Industrial Fellowships	Industrial Fellows, Names and Degrees	Foundation Sums and Dates of Expiration
192	Enameling	R. D. Cooke (M.S., University of Wisconsin)	$3,000 a year. April 1, 1920
193	Leather Belting	P. G. McVetty (M.E., Cornell University), Senior Fellow M. J. Pooley (B.S., Dakota Wesleyan University)	$5,650 a year. April 1, 1920
195	Protected Metals	J. H. Young (Ph.D., Ohio State University)	$4,500 a year. June 1, 1920
196	Collar	H. D Clayton (B.A., Ohio State University)	$3,300 a year. May 1, 1920
197	Sulfur	H. S. Davis (Ph.D., Harvard University) Mary D. Davis, assistant (B.A., Dalhousie University) C. N. Wenrich, assistant (Ph.D., University of Pennsylvania)	$7,200 a year. June 1, 1920
198	Dental Products	C. C. Vogt (Ph.D., Ohio State University)	$2,400 a year. July 1, 1920. Bonus royalty on sales
199	Copper	C. L. Perkins (B.S., New Hampshire College) R. E. Sayre (M.S., University of Wisconsin)	$6,500 a year. July 1, 1920
200	Physiological Research	K. K. Jones (M.S., Kansas State Agricultural College)	$5,500 a year. August 1, 1920
201	Quartz	William Stericker (B.S., University of Wisconsin)	$2,100 a year. September 8, 1920
202	Oil	W. F. Faragher (Ph.D., University of Kansas), Senior Fellow W. A. Gruse (Ph.D., University of Wisconsin) F. H. Garner (M.S., Birmingham University)	$10,000 a year. September 1, 1920. Bonus, $10,000
203	Glass	R. R. Shively (Ph.D., University of Pittsburgh)	$4,100 a year. September 1, 1920
204	Gas	J. B Garner (Ph.D., University of Chicago), Senior Fellow R. W. Miller (Ph.D., University of Pittsburgh)	$7,500 a year. September 15, 1920
205	Yeast	F. M. Hildebrandt (Ph.D., Johns Hopkins University) G. S. Bratton (B.A., University of Tennessee) Madalyne S. Schairer, assistant (B.A., Vassar College) H. C. Hoover, assistant (B.A., Ursinus College)	$15,000 a year. November 1, 1920
206	Illuminating Glass	A. H. Stewart (B.A., Washington & Jefferson College)	$3,000 a year. October 1, 1921
207	Glycerol	Melvin DeGroote (B.Ch.E., Ohio State University)	$4,000 a year. November 15, 1920
208	Fiber	J. D. Malcolmson (B.S., University of Kansas)	$4,500 a year. November 15, 1920
209	Household Utilities	F. F. Rupert (Ph.D., Massachusetts Institute of Technology) E. R. Edson (B.A., Clark College)	$6,000 a year. November 1, 1920
210	Zirconium	J. H. Becque (M.S., Rose Polytechnic Institute)	$4,000 a year. November 15, 1920
211	Fish Products	D. K. Tressler (Ph.D., Cornell University)	$4,000 a year. April 1, 1921
212	Copper	G. A. Bragg (B.S., University of Kansas)	$6,000 a year. November 1, 1920
213	Silicate	J. L. Crawford (B.S., University of Illinois)	$3,000 a year. December 1, 1920
214	Fuel	J. G. Davidson (Ph.D., Columbia University)	$5,000 a year. January 1, 1921
215	Silverware	H. E. Peck (B.S., Clarkson Memorial College of Technology)	$3,000 a year. December 11, 1920
216	Magnesia	T. S. Taylor (Ph.D., Yale University) R. H. Heilman, assistant (University of Pittsburgh)	$10,000 a year. January 1, 1921
217	Insecticides	O. F. Hedenburg (Ph.D., University of Chicago)	$4,200 a year. January 1, 1921
218	Organic Solvents	(Senior Fellow to be appointed) H. W. Greider (M.S., University of Kansas)	$7,500 a year. January 1, 1921
219	Coke	F. W. Sperr, Jr. (B.A., Ohio State University), Advisory Senior Fellow O. O. Malleis (M.S., University of Kansas) R. L. Brown (Ph.D., University of Chicago) (A third Fellow to be appointed)	$5,580 a year. January 1, 1921
220	Organic Synthesis	G. O. Curme, Jr. (Ph.D., University of Chicago), Senior Fellow C. J. Herrly (B.S., Pennsylvania State College) J. N. Compton (Ch.E., Rose Polytechnic Institute) E. W. Reid (N.S., University of Pittsburgh) C. O. Young (Ph.D., University of Pittsburgh) F. W. Hightower (B.A., University of Texas)	$41,000 a year. January 1, 1921
221	By-products	Walther Riddle (Ph.D., University of Heidelberg) H. E Gill, assistant (B.Chem., University of Pittsburgh)	$3,000 a year. January 1, 1921
222	Fertilizer	H. H. Meyers (B.S., University of Pennsylvania), Senior Fellow W. E. Vawter (B.S., University of Kansas)	$8,000 a year. January 5, 1921 Bonus, $5,000
223	Glue	R. H. Bogue (Ph.D., University of Pittsburgh)	$4,000 a year. January 5, 1921
224	Metalware	W. G. Imhoff (B.A., University of Wisconsin)	$5,000 a year. February 16, 1921
225	Distillation	David Drogin (M.S., University of Pittsburgh) Isaac Drogin (Ph.D., University of Pittsburgh)	$5,000 a year. January 18, 1921
226	Fruit Beverages	H. A. Noyes (M.S., Massachusetts Agricultural College)	$4,000 a year. March 1, 1921
227	Plastics	(Fellows to be appointed)	$7,000 a year.
228	Soap	O. H. Schunk (B.S., University of Wisconsin)	$2,500 a year. March 1, 1921
229	Laundry	H. G. Elledge (M.S., University of Pittsburgh), Senior Fellow Alice L. Wakefield (B.S., Carnegie Institute of Technology)	$6,400 a year. February 15, 1921
230	Refractories	R. M. Howe (M.A., University of Pittsburgh), Senior Fellow S. M. Phelps (University of Toronto) Mark Sheppard (B.S., Alfred University) R. F. Ferguson (B.S., University of Pittsburgh)	$12,000 a year. March 1, 1921. Bonus, $2,000
231	Keratin	(Fellow to be appointed)	$4,000 a year. Bonus, $5,000
232	Alloy	(Fellow to be appointed)	$3,500 a year.

CONSERVATION OF STANDARD SAMPLES OF THE BUREAU OF STANDARDS

Although Congress has failed to continue the appropriation for the work during the coming fiscal year, the Bureau of Standards will continue to prepare and issue standard samples. The money received from the sales of standard samples has always reverted directly to the Treasury, and is not available to the Bureau for any purpose whatsoever, therefore the cost of procuring, preparing, standardizing, and distributing the samples will necessitate the curtailment of other lines of established Bureau activities.

It is earnestly requested that all users of standard samples refrain from any unnecessary waste of the material, and that educational institutions limit their use strictly to primary standardization and to advanced research.

A list of the standard samples issued by the Bureau, together with charges and ordering and shipping regulations, is given in the Bureau of Standards *Supplement to Circular* 25, which will be sent upon application. In the future, standard samples will be issued only direct to users. This policy has been adopted to avoid, as far as possible, any chance of contamination or mixing of samples. Such chemical supply houses as have in the past carried a limited supply of certain standard samples for the accommodation of their customers can still render them service by remitting their orders with the proper fees to the Bureau for shipment.

RECENT RENEWALS—Sample 6*c*, a renewal of Iron D. Sample 25*a*, a renewal of the Manganese Ore 25, and Sample 42*a*, Melting Point Tin, have recently been renewed.

SAMPLES IN PREPARATION—Sample 45*a*, Melting Point Copper, and Sample 13*b*, a renewal of the 0.6 C Basic Open Hearth Steel, will be issued on or before June 15. Sample 50, a new chrome (3.6 per cent)-tungsten (17.5 per cent)-vanadium (0.75 per cent) steel sample, will be issued on or before July 1. Sample No. 32*a*, a renewal of the chrome-nickel-steel, will be issued on or before August 1. Samples 23*a* 0.8C Bessemer Steel, and 51, a new 1.2C Electric Furnace Steel, will be issued on or before September 1. Sample No. 10*c*, a renewal of 0.4C Bessemer Steel, has been ordered and should be ready by October 1.

OBITUARY

GEORGE GILBERT POND

In the death of Dr. George Gilbert Pond, Professor of Chemistry and Dean of the School of Natural Science at the Pennsylvania State College, the chemical profession loses one of its most able and productive teachers. Although apparently in the best of health, he contracted pneumonia while on a business trip, and after an illness of but five days died in New Haven, Conn., on May 20.

Dean Pond was born at Holliston, Mass., March 29, 1861, and came from fine old New England stock. His father, Abel Pond, was a merchant of high standing in the community, and his mother was Amelia Robinson. After attending the Holliston High School he went to Amherst, from which college he graduated in 1881; the remainder of that year and the following he spent at the University of Göttingen. On his return from Germany Dr. Pond became instructor in chemistry at Amherst, where he remained until the summer of 1888, when he accepted the professorship of chemistry at the Pennsylvania State College. In 1896 he became dean of the newly created School of Natural Science.


George Gilbert Pond

Dean Pond found the Pennsylvania State College a struggling institution almost lost in the mountains of Pennsylvania. He left it a powerful university of three thousand men with a loyal group of alumni of great strength doing yeoman work throughout the country. While many factors contributed to the growth of the institution, none was more potent than the high standard of scholarship which it was soon obvious was to be maintained there. Of this moral and intellectual standard Dean Pond was the accepted exponent—a rock of rectitude which no emergency, however real, could move. His moral courage and high principles of action not only kept him from faltering when confronted with difficult situations, but were a positive force which upheld the hands and hearts of his colleagues.

As a teacher of great strength Dean Pond will be long remembered by his many students. A clear and exact expositor, he led the novice through the difficult paths of chemical philosophy into the glories of a great science. A constant inspiration and source of encouragement to those who worked, he was without consideration for the indolent or the wayward. He formed a worthy ideal of scientific accuracy and conscientious endeavor which was reflected throughout each one of his many lines of activity. Indeed the standard he set for himself was so high and he held himself to it with such rigorous exactness that he was often judged severe; and no doubt it was frequently difficult for him to appreciate the shortcomings and struggles of characters less lofty than his own. But under what sometimes appeared an austere and uncompromising exterior there beat a noble and generous heart; there existed an impelling desire to help anyone and everyone who exhibited the least inclination to help himself; there was present a sympathy, deep and broad and strong, that constantly flowed underneath his every action.

Dean Pond was a man of great physical and mental vigor, and worked with increased efficiency as the load became greater. During the recent protracted illness of the president of the College, Dean Pond carried his own teaching work, conducted the School of Natural Science, and, as acting president, performed the manifold duties of the executive office. But in addition to serving the college, he also served the community. The industries of the state are stronger for his having lived. Many a litigation has been expedited by the clarity and impartiality of his testimony. He held nothing in common with those "experts" who strive for triumph rather than for truth.

Dean Pond is gone; but his spirit lives on in those whom he has prepared by precept and by practice to "fear God, and take your own part." W. H. W.

WASHINGTON LETTER

By J. B. McDonnell, Union Trust Building, Washington, D. C.

The second session of the Sixty-sixth Congress came to an end on June 5 without taking final action on many measures of close interest to the chemical world.

As was forecast, the session ended with the peculiar deadlock over the dye bill in the Senate unbroken. Indications at the present time are that the dye people intend to stand by their guns and hold out for the full protection given by the committee bill.

A survey of the situation leads to the belief that either the fight will have to be resumed in December for the committee bill, or the dye men will have to wait until after March 4, next, when the new Congress will be convened and the country has given one party or the other greater leeway in the number of votes and, consequently, a stronger control of legislation.

The Democrats of the Senate will not accept the Moses amendment. They have already emphatically refused to do so and the same personnel will sit at the December session. That the House Democrats will refuse to accept such a tariff proposal, also is true. Adoption of the Moses amendment as the mainstay of the dye protective bill would simply mean that the support offered heretofore by the Democrats would be lost almost in its entirety.

Washington has seethed with politics recently. Some of those who have felt the dye bill did not receive the hearty support it should have from the Republican leaders are asking if there is possibly any connection between politics and the generalship employed by Senator Watson, of Indiana, in charge of the bill. Senator Moses, of New Hampshire, was one of the campaign managers of General Wood for the Republican nomination. Senator Watson, reports here say, is not having any too easy a time in his own state and is anxious to mend every one of his fences possible. The Indiana delegation declared for Wood. The Wood forces at the convention threw their strength to him and named him chairman of one of the most important committees of the convention.

Senator Wolcott, of Delaware, in a speech on the floor of the Senate, just before adjournment, took issue with statements made during the speech of Senator Thomas, of Colorado, with reference to the agreement entered into between the du Pont Company and Levinstein's, of England. The charges made by the Colorado senator, that the agreement was designed to create a monopoly and was an agreement between the two concerns not to compete with each other, Senator Wolcott declared were entirely unjustified by the facts. The agreement, Senator Wolcott continued, dealt solely with patents and secret processes owned and controlled by the concerns themselves and did not attempt to deal with any other dyestuffs, processes, or possible markets.

Following a very determined fight on the part of Senator Norris, of Nebraska, the Nolan-Norris bill providing for reorganization of the Patent Office here was sent to conference between the House and Senate during the last three days of the session. As passed by the Senate, the increased salaries provided for in the bill passed by the House were somewhat reduced. The measure will retain its parliamentary status and be ready for action by a conference committee when the Congress reconvenes in its next session.

A very important amendment attached to the bill was the Nolan-Norris bill providing for the acceptance by the Federal Trade Commission of patents and licensing manufacture under them. This bill, added as an amendment to the Patent Office reorganization measure, authorizes the Federal Trade Commission "to accept assignment of, or license or other rights or powers under, to develop, to issue or refuse to issue licenses under, to encourage the industrial use and application of" inventions, patents and patent rights on behalf of the United States, which may be tendered from time to time by government employees. Provisions which would permit anyone to assign patents to the Commission were changed before passage by the Senate. Use of patents assigned to the Government under the bill would be licensed for a fee to be paid into the Treasury of the United States. The inventor would be paid such portion of this fee as the President deemed just.

Creation of some machinery under which inventions, processes and patents developed by experts and others in the government employ could be turned over to the Government and controlled by it has been urged for some time. Hearings held on this bill before the House Patents Committee, however, developed some opposition to its form, Dr. Bernhard Hesse, representing the Manufacturing Chemists' Association and the General Chemical Company, appearing before the committee in opposition to the measure as drawn. E. C. Prindle, New York patent attorney, representing the association, also appeared.

This hearing was held prior to the attachment of the bill to the Patent Office reorganization measure in the Senate. Director of the Bureau of Mines Cottrell and Drs. Alsberg and Stewart, of the Bureau of Chemistry of the Department of Agriculture, together with Dr. Hesse and Mr. Prindle, were named informally as a committee to work out an agreement upon the points at issue by Representative Nolan, of California, author of the measure. Dr. Stewart has said that following conferences the differences between the two sides did not appear to be radical and he thought that a compromise could be reached without great difficulty.

Representative Nolan tried to get the measure up on the floor of the House the last of May but an objection made by Representative Gard, of Ohio, defeated the request. The bill now has passed the Senate in the form of the amendment as above outlined and goes to conference between the two Houses. It must wait, of course, until Congress reconvenes before any further action can be taken.

Referring to the bill during debate in the Senate, Senator Norris declared that it had "brought forth some objection not from inventors but from great corporations like the National Chemical Co., the great fertilizer trust, and others like that."

"To my mind," he continued, "it is of as much importance as the reorganization of the Patent Office itself. We have in the Government employ a large number of scientific men, in the Bureau of Standards, in the Bureau of Chemistry, in the Bureau of Mines, and perhaps some other bureaus. They are high-class men, wedded to their profession, particularly in the Bureau of Chemistry, which has excited so much interest during the war and since all over the world. They are continually making inventions and improvements. Scientific men all over the country are unanimously behind that."

Strong resentment is expressed against the report of the Graham committee of the House which investigated the Muscle Shoals nitrate plant in publicity matter which has been printed and distributed by the Air Nitrates Corporation. "It will take more mud than this to hide the real achievements at Muscle Shoals," it is declared in a statement sent out.

Dye investigations carried on by the color laboratory of the Bureau of Chemistry of the Department of Agriculture will be considerably reduced as a result of the cut in appropriations given by Congress, Secretary of Agriculture Meredith has pointed out in a public statement. "The appropriation for color investigations in the Bureau of Chemistry has been reduced by $30,020, or more than 30 per cent below the funds available this year," Secretary Meredith said. Work of the laboratory already has resulted in the granting of sixteen patents, he continued, and cited development of a process by which phthalic anhydride could be manufactured for 45 cents a pound as an example of the work done by the laboratory.

Discovery that the entire content of corn-cobs can be converted into useful products was cited by the Secretary as another example of the work of Department chemists. Furfural has been discovered as one of the by-products of corn-cobs, while other by-products include acetate of lime, from which acetic acid is made, cellulose, and an adhesive of exceptionally high quality.

An amendment to the third deficiency appropriation bill proposed by Senator Lodge, of Massachusetts, provides an appropriation of $2,500 for two officers of the Public Health Service to represent the United States at the Sixth International Sanitary Conference which is to be held at Montevideo, Uruguay, from December 12 to 20, 1920.

Suit against the Alien Property Custodian has been filed in the Supreme Court of the District of Columbia by Merck & Company, of New York City, a drug and chemical manufacturing concern. The suit, filed against the Alien Property Custodian and the Treasurer of the United States, is to recover $538,000, of which amount $490,000 represents a debt owed by the firm of E. Merck, drug and chemical manufacturers, Darmstadt, Germany. The Alien Property Custodian refused to make payment in the absence of the representatives of the concern in Germany.

The second questionnaire dealing with consumers' needs for German dyestuffs will be sent to consumers throughout the country in the near future. Some time ago the War Trade Board Section sent out a questionnaire to dye manufacturers relative to their needs for German intermediates.

Relief from the situation of having no funds to carry on its work after the end of the present fiscal year was granted the War Trade Board Section by the insertion in the sundry civil appropriation bill of an appropriation of $25,000 to carry on the work of the Board. With two representatives of the Board in Europe at the present time, the belief has been expressed that this sum is not sufficient to meet the needs for the next year.

Establishment of a chemical research laboratory was announced recently by George Washington University.

Along with the dye measure, bills which failed to pass through the legislative mill of Congress and which were pending when the session ended, included the laboratory glassware and scientific instrument tariff bill, bills dealing with fertilizers, government production of atmospheric nitrogen, stream pollution by chemicals, and a measure prohibiting exportation of helium.

June 14, 1920

INDUSTRIAL NOTES

The suit of the Dow Chemical Company *vs.* American Bromine Company and A. E. Schaefer, chemist of the American Company and formerly a chemist of the Dow Company, to restrain the American Bromine Company from operating its Midland plant in the recovery of bromine and the manufacture of bromides, and involving charges of using the trade secrets of the Dow Company, has been decided by the Michigan Supreme Court in favor of the American Bromine Company. Previous to the war, the Dow Company had a practical monopoly in Michigan and controlled the bromide market, but when the price of bromides rose enormously after the beginning of the war, the Emerson Drug Company of Baltimore and the Dr. Miles Remedy Company of Cincinnati, both large users of bromides, organized the American Bromine Company and began the manufacture of bromides at Midland. The charges of the Dow Chemical Company, brought more than two years ago, were sustained at that time in the Midland County Court, and an appeal was taken to the State Supreme Court, whose decision in the matter is final. That body has now reversed the decision of the County Court. The court's opinion is in part as follows:

> We are impressed that the claim of secrecy on the part of the plaintiff is an afterthought, indulged in, possibly, by reason of the fact that Mr. Dow felt that relief through injunction process from the Federal Courts for infringement was doubtful. We are of the opinion that the Schaefer patent, under which the corporate defendant is now operating, was, as claimed by him, based upon investigation and invention subsequent to the termination of his employment by plaintiff. It is, therefore, not subject to the terms of the contract existing between him and the plaintiff during the latter years of his employment. If said patent is an infringement on any patent owned or controlled by plaintiff (as originally claimed by Mr. Dow), plaintiff has its remedy in the Federal courts for such infringement. Upon broad, equitable principles and in consideration of public policy, plaintiff should be denied relief unless its claim thereto is established beyond peradventure. After a careful consideration of this very voluminous record, we have no hesitation in reaching the conclusion that plaintiff has failed to so establish its right to relief. The bill is dismissed, with costs in both courts.

A committee has been appointed in France to take charge of the establishment in Paris of laboratories for chemical research, under the name "Institut de la Victoire." The committee is composed of representatives of French science, politics, and industry, and will work in close coöperation with government officials.

The Steel Company of Canada, Ltd., affiliated with the United States Steel Corporation, is installing a benzol plant in connection with its coke ovens. It is expected that 100,000 gal. of motor fuel per month will be turned out. Production is expected to begin in September.

Total exports of tanning materials from the United States during the month of April 1920 were valued at $278,461, according to the official figures of the Bureau of Foreign and Domestic Commerce. The countries receiving the largest amounts are as follows:

Belgium	$4,500
France	11,228
Italy	6,986
England	120,668
Canada	107,529
Cuba	4,960

Rosin and glycerol valued at $30,000 were destroyed by a recent fire at the storage yards of Procter and Gamble at Ivorydale, Ohio. The fire started from a spark from a locomotive and burned so fiercely that the firemen could do practically nothing to stop it. Trenches were dug around the fire, and all movable property carried from the danger zone.

The fertilizing plant of Armour and Company at Chicago was destroyed on May 24 by a fire which originated in an explosion of an ammonia tank. Damage estimated at $150,000 was caused and for a time a large section of the stock yards was threatened.

The United States Civil Service Commission has announced an examination for Research Engineer at $3,000 to $3,600 a year, to fill a vacancy at Watertown Arsenal, Watertown, Mass., and positions requiring similar qualifications. Requirements include graduation in metallurgical, mining or chemical engineering from a college of recognized standing, and at least eight years of experience in mining, metallurgy or allied work, at least four years of which must have involved experience in the handling of men. A reading knowledge of scientific French and German is also required. Theses, reports, and publications must be filed with application. Applications must be filed with Civil Service Commission, Washington, D. C., by July 20, 1920.

The Commission has also announced an examination for organic chemist and inorganic chemist (open to men only) to fill vacancies in the Philippine Service and similar positions, at $3,000 a year. Applicants must be college graduates with training and experience in organic and in inorganic chemistry. Applications must be filed with the Civil Service Commission, Washington, D. C., by July 6, 1920.

The Monsanto Chemical Works have recently announced their intention to begin the manufacture of synthetic camphor on a large scale. The plant is expected to be producing in quantity by the end of 1920. With the decreasing supply of Japanese camphor and the increasing demand, especially from the celluloid industry, the future of synthetic camphor looks very promising.

The Graesser Monsanto Chemical Works, Ltd., has been established in Great Britain, by an amalgamation of the British interests of Monsanto Chemical Works of St. Louis with R. Graesser, Ltd., of Ruabon, N. Wales. The new firm will begin manufacture in Ruabon of the full line of chemicals manufactured by the Monsanto Chemical Works in America, and will take over the London office for the handling of all European sales.

A Canadian Bureau of Scientific Research is to be established at an initial cost of $600,000 for site, building, and equipment, and $50,000 for the first year's salaries and upkeep. Standardization of all measures of length, volume, weight, etc., of all forms of energy and of scientific apparatus used in industry and the public service will be one of its main functions.

$250,000 damage was caused by a fire at the Frankford Works of The Barrett Company on May 19, 1920. One of the boiler houses was destroyed and the power plant temporarily crippled, but it is not expected to interfere seriously with the production of naphthalene.

Construction of the plant of the Atmospheric Nitrogen Company near Syracuse, N. Y., is about to begin. According to present plans, the first unit, which is to be completed in about a year at a cost of $2,000,000, will be only the beginning of the new industry. The first buildings to be erected will include process building, conversion building, gas producer, two large gas holders, service building and boiler house, all of the industrial type.

At a conference recently held in Manchester, England, it was decided to establish a British Chemical and Dyestuffs Traders' Association, to protect the interests of the merchants and to see that the distributing side of the chemical and dyestuffs industry receives proper recognition in departmental regulations.

The Dominion Bureau of Statistics of Canada has completed a preliminary report on the cocoa and chocolate industry in Canada during 1918. The total capital invested was $3,694,339, the total cost value of materials used was $3,835,479, and the total production value was $6,306,379.

During the three months ending March 31, 1920, the factory production of fats and oils in the United States (exclusive of refined oil and derivatives) was as follows:

	Pounds
Vegetable oils	605,285,917
Fish oils	4,211,336
Animal fats	455,897,306
Grease	89,767,212
TOTAL	1,155,161,771

PRODUCTION OF REFINED OILS

	Pounds
Cottonseed	357,572,013
Coconut	60,736,738
Peanut	12,748,195
Soy bean	38,393,120
Corn	24,745,843

The dye and chemical properties of the Ault & Wiborg Company have been sold to a syndicate representing the three principal dye works of Basle, Switzerland. An American corporation is to be formed to take over the operation of the works and the management will be in the hands of the Swiss interests. The Ault & Wiborg Company will retain a large interest in the new corporation, and Mr. Ault will be a director. New dye and chemical plants are planned, as well as enlargements of those already in operation. It is stipulated in the contract that no German money, directly or indirectly, shall ever be used in this industry. The Swiss companies interested are the Society of Chemical Industry, the Sandoz Chemical Works, and J. R. Geigy, S. A.

Trustees of Dartmouth College have announced the construction of a new $350,000 chemical laboratory.

Rules and regulations for the leasing of phosphate deposits in the public lands have just been approved by Secretary of the Interior Payne. About 2,500,000 acres of land in Wyoming, Idaho, Utah, and Montana for a number of years have been awaiting legislation recently enacted by Congress. They will now be open to lease in areas not exceeding 2,560 acres each, and should add largely to the fertilizer supply of the United States. Applications for leases are to be filed with the local land offices for transmission to the Secretary of the Interior.

The Federal Trade Commission has cited the United Indigo & Chemical Co., Ltd., of Boston, engaged in the manufacture and sale of dyestuffs, chemicals, etc., in formal complaint of unfair competition. The company is charged with secretly giving cash commissions and other rewards to employees of its customers and its competitors' customers to induce the purchase of its products.

The progress of the French chemical industry due to the effort made during the war is shown by the following table of production:

	1913 Tons	1920 Tons
Sulfuric acid	850,000	1,700,000
Nitric acid	20,000	300,000
Oleum	6,000	200,000
Liquid chlorine	none	18,000
Bromine	none	500
Synthetic nitrates	none	250,000
Phosphorus	300	3,000

The zinc oxide plant of the Empire Zinc Co., Canon City, Col., is nearing completion and ores are now being roasted, preparatory to actual production of the zinc oxide. The plant is modeled along the lines of the plants of the New Jersey Zinc Co., of which the Empire Zinc Company is a subsidiary, and the same grade of zinc oxide will be produced. The plant is expected to become an important unit in the New Jersey Zinc Company's operations, and has been built with a view to future extensive additions.

To protect aluminum and aluminum alloys from corrosion, L. von Grotthuss has tried browning the metal electrolytically. The aluminum is suspended in an electrolyte consisting of a sulfur compound of molybdenum with a zinc anode at a temperature of 60° to 65° C. The aluminum is soon covered with a dark brown coating and may be bent or rolled without cracking the coating. Aluminum thus treated was immersed in a salt solution for two months without showing the slightest trace of corrosion

PERSONAL NOTES

The council of the Society of Chemical Industry awarded its medal for the year 1920 to **Monsieur Paul Kestner**, first president of the Société de Chimie Industrielle of France, in recognition of his distinguished services to the chemical industry.

Mr. L. W. Haskell, vice president of the Southern Cotton Oil Co., died at his home in Savannah, Ga., on June 4, 1920.

The honorary degree of doctor of laws was conferred upon **Dr. William H. Nichols** at the recent commencement exercises of New York University.

Mr. Henry H. Mayers has left E. I. du Pont de Nemours & Co., Paulsboro, N. J., where he was employed as supervisor of the strontium nitrate department, to accept a position in the research department of Rohm & Haas Co., Bristol, Pa.

Mr. Leroy M. Law, formerly chief chemist of the Interocean Oil Co., New York and Baltimore, announces his association with the New Orleans Refining Co., New York City.

Mr. R. W. Ruprecht, formerly chemist and superintendent of the fertilizer factory of F. W. Tunnell & Co., Inc., of Philadelphia, has been appointed chemist of the Florida Experiment Station, Gainesville, Fla.

Mr. E. Wurgler, for many years assayer and chemist at various plants of the American Smelting and Refining Company, and recently chief chemist at their Durango Plant, resigned his position with that firm to enter business with Oscar J. Frost, assayer, Denver, Colo.

Mr. Robert V. Townend resigned his position as chemist in charge of the chlorinated toluene products with the Semet-Solvay Co., Syracuse, N. Y., to accept a position with the Victor Talking Machine Co., Camden, N. J., where he will organize and direct their department of chemical research. Up until the time Mr. Townend left Syracuse, he served as secretary of the Syracuse Section of the American Chemical Society.

Mr. W. N. Pritchard, Jr., is now doing research work on dyes for the Calco Chemical Co., Bound Brook, N. J., having formerly been with Procter and Gamble at Augusta, Ga., as superintendent of a pulp mill.

Mr. Howard Stoertz, formerly analytical and experimental chemist at the Harrison Works of E. I. du Pont de Nemours & Company, is now research chemist for the Crown Cork and Seal Co., Baltimore, Md.

Mr. Allen H. Lloyd resigned his position in the St. Louis Meat Inspection Laboratory of the U. S. Department of Agriculture to accept a position in the Commercial Laboratories at Indianapolis, Ind.

Mr. Peter J. Waldstein, formerly chief chemist for John Lucas and Co., Inc., Philadelphia, Pa., is employed at present in a similar capacity with the Continental Varnish Co., Brooklyn, N. Y.

Mr. Kenneth E. Bell has resigned his position as research associate with the research laboratory of applied chemistry, Massachusetts Institute of Technology, to accept one as chemical engineer with the Lewis Recovery Corporation, Boston, Mass.

Dr. Marston T. Bogert, professor of chemistry at Columbia University, New York City, was recently named a member of the Tariff Commission by President Wilson. Dr. Bogert was elected president of the American Section of the Société de Chimie Industrielle of France, at its recent meeting.

Mr. G. W. Morey, of the Geophysical Laboratory, Carnegie Institution of Washington, who has been on leave of absence and in charge of the optical glass plant of the Spencer Lens Company of Buffalo, N. Y., since November 1918, recently returned to resume his research work at the Laboratory.

The University of North Carolina chapter of Sigma Xi was installed May 26 by **Prof. C. E. McClung**, of the University of Pennsylvania, president of Sigma Xi. The charter members of the North Carolina chapter are **Drs. James M. Bell** and **Joseph Hyde Pratt**, initiated at Cornell and Yale, respectively, and **Drs. F. P. Venable, H. V. Wilson, W. D. MacNider, A. S. Wheeler, W. C. Coker** and **William Cain**, all members of the faculty.

Mr. Clarence W. Holmes has left the employ of the Weirton Steel Co., Wierton, W. Va., to become laboratory assistant at the Universal Steel Co., Bridgeville, Pa.

Mr. R. G. Gutelius has left the experimental laboratory of the Atlas Powder Co., Stamford, Conn., and is in the experimental laboratory of the Aetna Explosives Co., at Emporium, Pa.

Dr. F. H. Rhodes has resigned as director of research for the chemical department of The Barrett Company, Frankford, Pa., in order to accept the appointment as professor of industrial chemistry in Cornell University.

Mr. H. D. Grigsby has resigned from the provisions and clothing department of the Navy where he was in charge of the Inspection Laboratory, and is taking a position as research chemist with the Ward Baking Company at their Bronx Plant, New York City.

Mr. C. E. Herring was named First Assistant Director of the Bureau of Foreign and Domestic Commerce, and **Mr. O. P. Hopkins**, Second Assistant Director of the Bureau, in nominations sent to the Senate May 27 by President Wilson.

The National Research Council has elected the following officers for the year beginning July 1, 1920: Chairman, **H. A. Bumstead**, professor of physics and director of the Sloane Physical Laboratory, Yale University; First Vice Chairman, **C. D. Walcott**, president of the National Academy of Sciences and Secretary of the Smithsonian Institution; Second Vice Chairman, **Gano Dunn**, president of the J. G. White Engineering Corporation, New York; Third Vice Chairman, **R. A. Millikan**, professor of physics, University of Chicago; permanent secretary, **Vernon Kellogg**, professor of biology, Stanford University; Treasurer, **F. L. Ransome**, treasurer of the National Academy of Sciences.

Mr. C. H. McClure has severed his connections with the Commonwealth Edison Co., where he was employed as chief chemist, and has accepted a position as chemist with the Menasha Printing and Carton Co., Menasha, Wis.

Mr. Kenneth P. Monroe has resigned as chemist in the color laboratory, U. S. Bureau of Chemistry, Washington, D. C., to accept a research position in the Jackson Laboratory of E. I. du Pont de Nemours & Co., Wilmington, Del.

Dr. George D. Van Epps, formerly connected with the Citro Chemical Co., Maywood, N. J., has returned to the Continental Motors Corp., Muskegon, Mich., in his former capacity as analytical and research chemist.

Mr. George K. Senior has left the American Textile Soap Co., Springfield, Mass., where he was employed as chemist, to take a similar position with the Warren Soap Manufacturing Co., Boston, Mass.

Mr. Henry Sohn, formerly with the Sanitary District of Chicago, as assistant chemist, has accepted the position of assistant bacteriologist at the Cincinnati Water Works, California, Ohio.

Mr. Asher F. Shupp recently resigned his research fellowship at the Mellon Institute of Industrial Research to become chief chemist to the Faultless Rubber Co., Ashland, Ohio.

Mr. Shreve Clark, formerly chemist and assistant testing engineer to the State Highway Department of Ohio, Ohio State University, Columbus, O., recently became testing engineer to the State Highway Department of Virginia, Virginia Polytechnic Institute, Blacksburg, Va. In addition to his regular duties in the Highway Department, Mr. Clark will instruct a class of senior civil engineers on highway materials.

Mr. Frank E. Glassman has severed his relations with the Specials Chemical Co., Highland Park, Ill., and has organized the Vulcan Chemical Co., Chicago, Ill., which firm will manufacture a complete line of soldering compounds for the electrical and automobile industries.

Mr. E. F. Berger, formerly assistant chemist at the Michigan Agricultural College Experiment Station, is at present connected with the Patton Paint Co., Milwaukee, Wis., as chemist in the insecticide plant.

Mr. Robert G. O'Kane, previously engaged with the Brown Company on certain special electrochemical investigations, has become an executive on the operating staff of the Carrollville, Wis., plant of the Newport Company.

Mr. Raymond E. Whitney has left the employ of the Monsanto Chemical Works, E. St. Louis plant, to accept a position as chemist of the ammonia leaching and allied processes plant of the Calumet and Hecla Mining Co., Lake Linden, Mich.

Dr. H. R. Kraybill has left the Bureau of Plant Industry, U. S. Department of Agriculture, where he was assistant physiologist, and has been appointed professor of agricultural chemistry and head of the department of chemistry in the Experiment Station of New Hampshire State College, Durham, N. H.

GOVERNMENT PUBLICATIONS

By Nellie A. Parkinson, Bureau of Chemistry, Washington, D. C.

NOTICE—Publications for which price is indicated can be purchased from the Superintendent of Documents, Government Printing Office, Washington, D. C. Other publications can usually be supplied from the Bureau or Department from which they originate. Commerce Reports are received by all large libraries and may be consulted there, or single numbers can be secured by application to the Bureau of Foreign and Domestic Commerce, Department of Commerce, Washington. The regular subscription rate for these Commerce Reports mailed daily is $2.50 per year, payable in advance, to the Superintendent of Documents.

CONGRESSIONAL COMMITTEES

Nitrogen. Production of atmospheric nitrogen, hearing on S. 3390, to provide further for national defense, to establish self-sustaining federal agency for manufacture, production, and development of products of atmospheric nitrogen for military, experimental, and other purposes, to provide research laboratories and experimental plants for the development of fixed-nitrogen production, and for other purposes. 104 pp. 1920.

Zinc. Senate Report 511. 3 pp. April 9, 1920. Production of zinc ores, report to accompany H. R. 6238 (to provide revenue for the Government and to establish and maintain production of zinc ores and manufactures thereof in the United States); submitted by Mr. Watson.

GEOLOGICAL SURVEY

Lead in 1917. General Report. C. E. Siebenthal. Separate from Mineral Resources of the United States, 1917, Part I. 17 pp. Issued May 8, 1920. The output of refined lead in the United States from domestic ores in 1917 was 548,450 short tons, valued at $94,333,000, at the average sales price of refined lead, compared with 552,228 tons, valued at $76,207,000 based on the average "outside" spot quotation at New York in 1916—a loss in quantity of 3,778 tons, or nearly 1 per cent, and a gain in value of $18,126,000, or nearly 24 per cent.

Anticlines near Maverick Springs, Fremont County, Wyoming. A. J. Collier. Contributions to Economic Geology, 1919, Part II. 18 pp. Issued April 26, 1920. The oil produced in this district is a heavy asphaltic dark brown oil, having an odor of hydrogen sulfide.

Geology of Alamosa Creek Valley, Socorro County, New Mexico, with Special Reference to the Occurrence of Oil and Gas. D. E. Winchester. Contributions to Economic Geology, 1920, Part II. 15 pp. Bulletin 716-A. Issued May 12, 1920.

The Upton-Thornton Oil Field, Wyoming. E. T. Hancock. Contributions to Economic Geology, 1920, Part II. 18 pp. Bulletin 716-B. Issued April 29, 1920.

Mineral Resources of the United States 1917. Part II. Non-metals. G. F. Loughlin. 1293 pp. Cloth bound. 1920. The chapters covered by this report are: fuel briquetting; strontium; phosphate rock; sulfur, pyrites, and sulfuric acid; magnesite; talc and soapstone; gypsum; graphite; slate; feldspar; gems and precious stones; salt, bromine, and calcium chloride; mica; asbestos; sand-lime brick; silica; abrasive materials; asphalt, related bitumens, and bituminous rock; fuller's earth; peat; barytes and barium products; fluorspar and cryolite; sodium salts; cement; sand and gravel; potash; mineral waters; clay-working industries; lime; stone; petroleum; coal (Part A), production; natural gas; gasoline from natural gas; coke and by-products in 1916 and 1917; coal (Part B), distribution and consumption. These separate chapters have previously been reviewed.

Chromite in 1918. J. S. Diller. With Papers on Foreign Deposits of Chromite by E. F. Bliss and H. R. Aldrich, and on Chromite and Chromiferous Iron Ore in Cuba by E. F. Burchard. Separate from Mineral Resources of the United States, 1918, Part I. 69 pp. Published May 15, 1920. The year 1918 was the most eventful in the history of chromite. The domestic production was by far the largest in the history of the country and demonstrated that when necessary the United States can, at least for a few years, supply its own chromite. The total quantity of chromite of all grades mined and shipped in the United States in 1918 was 82,430 long tons (92,322 short tons), valued at $3,955,567.

BUREAU OF MINES

Monthly Statement of Coal-Mine Fatalities in the United States, February 1920. W. W. Adams. 10 pp. Paper, 5 cents. April 1920.

Procedure for Establishing a List of Permissible Gas Masks; Fees, Character of Tests, and Conditions under Which Gas Masks Will Be Tested. Supplement to Schedule 14. 4 pp. 1920.

Approved Explosion-Proof Coal-Cutting Equipment. L. C. Ilsley and E. J. Gleim. Bulletin 78. 53 pp. Paper, 25 cents. 1920. The first part of the bulletin deals with the general theory of protection from fire damp, gives the schedule of the Bureau of Mines, and shows its application to the testing of commercial apparatus. The second part covers a detailed description of the apparatus that has been tested and approved under this schedule, together with a resumé of the tests on which the approvals were based.

Coal-Mine Fatalities in the United States, 1919, and Coal-Mine Statistics Supplementing Those Published in Bulletin 115. List of Permissible Explosives, Lamps, and Motors Tested prior to January 31, 1920. A. H. Fay. Bulletin 196. 86 pp. Paper, 15 cents.

The Determination of Mercury. C. M. Bouton and L. H. Duschak. Technical Paper 227. 44 pp. Paper, 10 cents. 1920. The method described in this report was developed by the Bureau of Mines for use in a study of mercury condenser losses. Illustrations are given, showing the apparatus employed. A bibliography on analytical methods for the determination of mercury is also included.

Accident Prevention in the Mines of Butte, Montana. Technical Paper 229. Covers safety organization and work of Butte mining companies, etc. Paper, 10 cents. Issued June 1920.

Development of Liquid Oxygen Explosives during the War. Technical Paper 243. Paper, 10 cents. Issued June 1920. Covers historical review, Bureau of Mines experiments, recent German development of liquid oxygen explosives, European observations of bureau representatives, summary and conclusions regarding liquid oxygen explosives, including blasting, equipment, safety precautions, etc.

Quarry Accidents in the United States during the Calendar Year 1918. A. H. Fay. Technical Paper 245. 52 pp. Paper, 10 cents. April 1920.

Perforated Casing and Screen Pipe in Oil Wells. E. W. Wagy. Technical Paper 247. Petroleum Technology 55. 48 pp. Paper, 20 cents. Issued April 1920.

Effect of Gasoline Removal on the Heating Value of Natural Gas. Technical Paper 253. Paper, 5 cents. Issued June 1920. Covers a review of the situation, character of natural gas examined, advantages of gasoline plants, and experimental results in laboratories.

BUREAU OF STANDARDS

Recommended Specification for Iron-Oxide and Iron-Hydroxide Paints. Circular 93. 8 pp. Issued April 21, 1920. Prepared and Recommended by the United States Interdepartmental Committee on Paint Specification and Standardization, March 22, 1920. Outlines general specifications for the pigment, liquid, semi-paste, and ready-mixed paint, methods of sampling and analysis, and describes the reagents to be used in examining these products.

Recommended Specification for Black Paint, Semi-paste and Ready-mixed. Circular 94. 8 pp. Issued April 21, 1920. Prepared and recommended by the United States Interdepartmental Committee on Paint Specification and Standardization, March 22, 1920. Outlines general specifications for the pigment, liquid, semi-paste, and ready-mixed paint, methods of sampling and analysis, and describes the reagents to be used in examining these products.

Weights and Measures. Miscellaneous Publication 41. 226 pp. Paper, 20 cents. 1920. This is a report of the twelfth annual conference on weights and measures of the United States held at the Bureau of Standards, Washington, D. C., May 21 to 24, 1919.

DEPARTMENT OF AGRICULTURE

Saponified Cresol Solutions. J. M. Schaeffer. Department Bulletin 855. This paper describes a series of experiments undertaken with the object of preparing a saponified cresol solution which would be cheaper and at the same time no less effective as a disinfectant than those at present in use.

Insect Powder. C. C. McDonnell, R. C. Roark and G. L. Keenan. Department Bulletin 824. 100 pp. Paper, 20 cents. Issued June 3, 1920.

Articles from the Journal of Agricultural Research

Effect of Calcium Sulfate on the Solubility of Soils. M. M. McCool and C. E. Millar. 19 (April 15, 1920), 47–54.

Effect of Reaction of Solution on Germination of Seeds and on Growth of Seedlings. 19 (April 15, 1920), 73–95.

COMMERCE DEPARTMENT

Commerce. Schedule governing statistical classification of imports into the United States, with rates of duty, and regulations governing the preparation of monthly and quarterly statements of imports and monthly statement of exports of foreign commodities; approved June 10, 1918. Edition of 1920. Foreign and Domestic Commerce Bureau. Supersedes and combines Schedules A and E. 98 pp. Paper, 10 cents.

UNITED STATES TARIFF COMMISSION

Incandescent Gas-Mantle Industry. Its raw materials and by-products, monazite sand, thorium and cerium nitrates, pyrophoric alloys, and mesothorium. Tariff Information Series 14. 32 pp. Paper, 5 cents. 1920.

FEDERAL BOARD FOR VOCATIONAL EDUCATION

Industrial Accidents and Their Prevention. R. R. Ray. Bulletin 47. Employment Management Series 7. 66 pp. 1920.

NAVY DEPARTMENT

Lubricating Oils as Purchased by Bureau of Steam Engineering. General information for refiners of petroleum regarding tests of lubricating oils at Engineering Experiment Station, Annapolis, Md., also information concerning tests of greases and soluble oils at laboratory of Machine Division, Navy Yard, New York. Revised March 1, 1920. 32 pp.

TREASURY DEPARTMENT

Denatured Alcohol. Regulation 61 relative to production, tax payment, etc., of industrial alcohol, and to manufacture, sale, and use of denatured alcohol under Title 3 of national prohibition act of October 28, 1919. Treasury Decision 2986 (supplement to Treasury Decision 38, No. 10). 107 pp. Paper, 10 cents. 1920.

UNITED STATES PUBLIC HEALTH SERVICE

A Further Study of the Excess Oxygen Method for the Determination of the Biochemical Oxygen Demand of Sewage and Industrial Wastes. E. J. Theriault. 35, 1087–97.

COMMERCE REPORTS—MAY 1920

A report has been prepared showing the development of the **sandalwood oil industry** in India, and the distribution of this trade in previous years. (P. 651)

England has received her first **reparation dyestuffs** from Germany. (P. 677)

The successful cultivation of **camphor** within the British Empire is no longer doubtful. (Pp. 692–3)

There is a steady demand in Japan for **soda ash, caustic soda, bicarbonate of soda, carbolic acid (phenol), boric acid, borate of soda, bichromate of potash,** and **rosin,** for most of which the United States is the principal source of supply. (P. 694)

The effectiveness of the Japanese **camphor** monopoly for increased production is shown by the production in 1919 being almost double the amount produced in 1918. (P. 694)

A description is given of the **extraction of tannin** from the Indian myrobalans. (Pp. 713–5)

Chicle-producing trees have been discovered in British Guiana. (P. 716)

The suggestion that the forestial riches of Argentina be utilized for the manufacture of **paper pulp** is meeting with favor. (Pp. 716–7)

Statistics are given showing the production of the leading Burma **rubber** plantations and the exportation of rubber for the past five years. (P. 727)

Final returns covering the exportation of **tin** from Hongkong in 1919 indicate a disastrous year. (Pp. 743)

Statistics are given showing the imports of **aniline** and **alizarin** into Madras by countries for five years. (P. 745)

The total French imports of **crude rubber** for 1919 were 32,455 metric tons against 19,927 tons in 1918. (P. 745)

Great progress has been made in the revival of the Indian **indigo** industry. (Pp. 746–7)

The imports of **chemicals** into Burma during the year ended March 31, 1918, showed a considerable increase over the four years immediately preceding. (P. 758)

There is a considerable movement of **paraffin** into and out of Hongkong. In 1918 the United States furnished wax to the extent of 59 per cent of the total, but in 1919 furnished but 7 per cent, and that only in the last quarter of the year. (P. 767)

The **artificial silk** establishments of Belgium are employing 90 per cent of their 1914 personnel, but the production is slightly below 32 per cent of the pre-war figure. (P. 798)

The British Government has made arrangements to deliver to the Belgian zinc industry 240,000 tons of **zinc ore** within the maximum limit of two years. (P. 798)

The **mineral resources** of Armenia have been hardly explored or are impossible to exploit on account of the scarcity of available roads. The deposits of copper are innumerable. (P. 807)

Hull is now without doubt the largest **vegetable oil** center in Europe. During January and February 596,939 gal. of linseed oil, valued at $999,770, were invoiced through Hull for shipment to the United States. The outlook of the industry for 1920 is very promising. (Pp. 818–23)

A more or less acute shortage of **paper** is reported in Hongkong, Shanghai, and North China ports. (P. 823)

The utilization of Irish **mineral resources** is suggested. Accurate explorations will have to be made, however, before the practicability of reopening old mines and exploiting new fields is determined. (Pp. 828–30)

The export of **sulfur** from Sicily in 1919 amounted to 147,286 tons, as compared with 231,390 tons in 1918, but is still below the pre-war figures. There were no sulfur exports to the United States during 1919. (P. 835)

German war-time experiments showed that the distillation of **lignite** at a high temperature gave a liquid coal tar which contained certain ingredients suitable as a substitute for gasoline, kerosene, and lubricating oils. (P. 835)

The manufacture of **paper pulp from seaweed** is proving to be a profitable undertaking in Japan. (P. 836)

In spite of the heavy duty imposed on **toilet soaps** imported into Japan the importations of perfumed soaps increased from 105,000 lbs., valued at $39,000, in 1917 to 125,000 lbs., valued at $61,000, in 1918 and jumped to 387,000 lbs., valued at $184,000, in 1919. The demand for laundry soap is largely met by local production. (Pp. 836–7)

Norway in 1916 mined 29,398 short tons of **copper ore** and in 1917, 43,318 tons. During the same period the production of copper pyrites was 325,571 and 362,323 short tons, respectively. (P. 837)

Germany is to pay Switzerland 11,000,000 Swiss francs in order to cancel her war-time contract for the purchase of **aluminum** in Switzerland. (P. 854)

The German delivery of **chemicals** is so uncertain that Switzerland finds it practically impossible to carry on any satisfactory business with Germany. Chemical products from the United States can be purchased in Switzerland at a lower price in many instances than German houses are quoting. (P. 881)

The duties on **coal-tar dyes** imported into Spain have been fixed for a period of ten years at greatly increased rates. (P. 891)

The French **iron and steel** industry is reviewed. (Pp. 902–17)

A new **petroleum** deposit was recently discovered at Pechelbronn. New refineries are to be installed capable of distilling 73,000 tons of crude petroleum per year. The distillation is to be by the "continuous" process, with perfect vacuum. (P. 918)

The effect of the war on the Spanish **iron** and **steel** industry, one of the most important Spanish industries, is noted. (Pp. 934–44)

There is a great demand in Norway for **cellulose,** as well as **wood pulp** and **paper.** Since the armistice the sale of bleached sulfite has been greater than that of paper and sulfate cellulose. (P. 978)

The total shipments of **crude rubber** from the ports of Para, Manaos, and Itacoatiara, Brazil, and Iquitos, Peru, during the month of January 1920 amounted to 6,273,039 lbs. as compared with 6,624,915 lbs. in the corresponding month of last year. (P. 981)

Work on the erection of a **paper** and **pulp mill** in Newfoundland is to be started this month. The pulp and paper produced will be exported to England and the United States. (P. 991)

Previous to 1916 no **manganese** was mined in Costa Rica. Since that time production has steadily increased until in 1919 7,851.5 tons were exported. (P. 998)

American interests recently acquired the rights to three Bolivian **tin mines.** (Pp. 998–9)

The importation of **soda** and **soda ash** into Japan has greatly increased, and from 70 to 80 per cent of these imports come from the United States. (P. 1002)

German **shale** and **peat** have been suggested as substitutes for coal. (P. 1013)

It is reported that work is about to be resumed in the old Orijarvi **copper** and **zinc** mines in Finland. (Pp. 1018–9)

The French Minister of Finance has given notice that the price of **alcohol** distilled from **sugar beets** during 1920–1 will be based on the average price of No. 3 white sugar during November and December 1919. (P. 1043)

The manufacture of by-products from second-grade **coal** in South Africa is about to be commenced. A carbide and by-products plant is now in the course of construction. The seams of this second-grade coal are very thick, and consequently the raw material is easily obtainable. Tests show that the coal is high in nitrogen, and yields over 120 lbs. of sulfate of ammonia, 2.5 gal. of benzol, and 16 gal. of tar oil per ton of coal. (P. 1048)

An enterprising American has secured from the Italian government a concession for the utilization of 800 horse power of the waterfalls at Terni, and maintains that he can turn out nitrogenous plant food for the Italian farmer at a cost as low as 1 lira per kilo. The process has passed beyond the experimental stage and ammonia from this plant has already been put upon the market on a paying basis. (Pp. 1058–9)

The new import duties on **drugs** and **chemicals** in Uruguay affect 1,244 items and, in many cases, greatly increase the duties. (P. 1073)

Mexican **potassium nitrate,** in lots of from 30 to 100 tons per month, may be obtained. (P. 1076)

To ameliorate the serious situation caused by the shortage in France of **superphosphates** for agricultural purposes, the Under Secretary for the Merchant Marine has placed at the disposition of a group of manufacturers of this vitally necessary fertilizing material 35,000 tons of shipping, in addition to 6,000 tons already furnished, to be employed in the transportation of phosphate rock from Northern Africa. (P. 1087)

The Australian restrictions on the importation and exportation of **copra** have been removed. (P. 1089)

The **nitrate industry** of Chile is reviewed. (Pp. 1093–1103)

The **lead and zinc industry** in Japan is reviewed. (P. 1108)

There has been a fall in the prices of **tallow** in Great Britain. (P. 1112)

An acute **scarcity** of **manganese ore** is reported in Great Britain, which greatly hinders the furnaces making ferromanganese. (Pp. 1112–3)

The Australian import prohibition on **dyes** is still effective. (P. 1121)

The British Water Power Resources Committee has a scheme for the utilization of the **water power** of the United Kingdom in place of coal and oil. (P. 1157)

An increase in the exportation of nearly all kinds of **vegetable oils** from the Mukden consular district of China is reported. (P. 1178)

The Spanish **olive oil** yield in 1919 amounted to 336,393 metric tons, compared with 255,202 tons in 1918, and 427,838 tons in 1917. (P. 1189)

The trade in **chemicals** at Hongkong in 1919 showed little life and very little increase over that of 1918, when it was difficult to secure supplies in any line. Importers found it difficult to secure supplies from the United States and most of them returned to their old British connections as rapidly as conditions permitted. (P. 1197)

Rich veins of **mica** have been explored and are now being exploited in the district of Mehedintz, Rumania. (P. 1227)

Special Supplements Issued

United Kingdom—*22c*
Scotland—*22d*
Argentina—*41a*
Brazil—*43a*
Colombia—*45a*
Aden—*52a*
Dutch East Indies—*56a*

Statistics of Exports to the United States

Burma—(P. 733)
Paraffin wax

Chile—(P. 1099)
Nitrate

Sicily—(P. 771)
Essence of bergamot

BOOK REVIEWS

The Condensed Chemical Dictionary. Second Printing, Corrected. The Chemical Catalog Co., Inc. 1 Madison Ave., New York. Price, $5.00.

A number of the errors to which attention was called in the review of the first printing [THIS JOURNAL, 12 (1920), 102] have now been corrected. In particular the editors state that the physical constants have been checked and are believed to be correct with the exception of the following typographical errors: (1) the boiling point of methyl chloride should be —23.73° instead of 23.73°, (2) the melting point of dinitrobenzene (para-) should be 172–173° instead of 372–373°.

AUSTIN M. PATTERSON

Manual of Industrial Chemistry. Edited by ALLEN ROGERS in collaboration with others. 3rd Edition thoroughly revised and enlarged. D. Van Nostrand Co., New York, 1920. Price, $7.50, net.

The third edition of this Manual of Industrial Chemistry comes from the press as a volume of over 1200 pages, an increase of more than 200 pages over the second edition issued in 1915. The book is however no more bulky than its predecessor. The success of the earlier editions is attested by the statement that this is the twelfth thousand printed. Almost every chapter shows revision and in many cases expansion. The first chapter on General Processes, by Professor Rogers himself, is much improved by expansion from 32 to 48 pages. The second chapter on Water for Industrial Purposes, written in the early editions by Mr. R. B. Dole, now deceased, has been rewritten by Messrs. Stabler and Chambers of the Geological Survey and expanded to cover 54 instead of 31 pages, presenting a very excellent treatment of the subject. The excellent chapter on Illuminating Gas by Mr. W. H. Fulweiler is increased from 63 to 82 pages. Other chapters show less expansion. Four new chapters are added, one of 12 pages on Turpentine and Rosin by Dr. Chas. H. Herty; one of 18 pages by Mr. Jerome Alexander on Colloids; one of 15 pages by Mr. Clarence V. Ekroth on Dehydrated, Dried, and Evaporated Foods, Condensed Foods; one of 27 pages on Baking by Mr. Arnold Wahl.

This book has the advantages and disadvantages which are inherent in such a collaborative effort. The writers are specialists in their subjects and errors of fact are few, but there is some duplication and considerable unevenness of treatment. The Industrial Chemistry of Carbon Compounds takes nearly two-thirds of the book and yet there is practically nothing on the manufacture of intermediates or dyes. Paper manufacture is compressed to 15 pages and rubber to 24 pages, but the subjects of paint, varnish, and their raw materials are given nine chapters with a total of 105 pages. In contrast to this we find in the Inorganic Industry the metallurgy of iron and steel, treated, it is true, by a master, Bradley Stoughton, compressed to 17 pages; the industry of clay, bricks, and pottery cut to 7 pages, and the metallurgy of the non-ferrous metals touched on only casually in the chapter on Elements and Compounds.

The Electrolytic Decomposition of Sodium Chloride is given a separate chapter but the industries of evaporation and separation of salts in solution are given inadequate treatment. Common salt and the evaporation of brine are, it is true, given 5 pages, but the treatment is indiscriminating and statistics used in the 1915 edition and dating from 1912 have been carried forward without revision. Potash salts and the manufacture of soda ash receive only scanty and casual treatment in the chapter on Elements and Compounds.

The treatment of the various chapters is mainly descriptive. The increase in our theoretical knowledge is making it increasingly possible to show the reasons for technical success or failure and the limitations of processes. It is hoped that more emphasis may be placed on this aspect in future editions.

Those of us who recall how books like Lunge's "Sulphuric Acid and Alkali," and Allen's "Commercial Organic Analyses" have grown with successive editions may hope that the demand for this work will soon cause it to be issued in several volumes where the inorganic subjects and the theoretical treatment may have more generous recognition. However, in its present form, the book may be rated as the best single volume treatment of industrial chemistry in the English language.

ALFRED H. WHITE

From Newton to Einstein. By BENJAMIN HARROW, PH.D. 74 pp. D. Van Nostrand Co. New York: 1920. Price, $1.00.

This is a small but popular brochure rather than a book and has no doubt sprung out of the opportunism brought about by the popular furore over Einstein and his postulates. The first portion of the book is taken up with a general resumé concerning Newton and his law of gravitation and laws of motion together with his theory of light. Another small chapter deals with the ordinarily accepted, modern theory of light, and a final chapter treats of Einstein's theories and conceptions of the nature of light, and a statement of the observations and results of the astronomical experiments to watch the solar eclipse of May 1919, as far as its bearings on the Einstein theory are concerned. The author is thoroughly convinced of the naïveté of his readers, for in discussing the theory of light, when the term "ether" is mentioned, he assures us in a footnote that this is in no way connected with the well-known anesthetic.

The treatment of the subject is very similar to that in the various "special articles" which have from time to time appeared in the daily newspapers or in the magazine sections of the Sunday editions, concerning Einstein and his theories. Great stress is laid in this review of the work of Einstein in respect to its connection with the conception of light, and the popular sensationalism is catered to by claiming that the startling result of Einstein's discoveries is that light does not travel in a straight line as formerly supposed, but in curves, and consequently, notwithstanding the postulates of Euclid, two parallel lines probably meet long before finity is reached. Of course it is a sensational bit of news to announce to the trusting public that by the wonderful discovery of Einstein the whole conception of geometrical propositions has been proved false, but, as a matter of fact, the physical behavior or the physical nature of a ray of light has nothing whatever to do with the propositions and conceptions of pure mathematics or of geometry. But then perhaps, unless this type of newspaper sensationalism is resorted to, very little popular interest would be aroused in a scientific discussion which really involves very abstruse mathematical propositions.

The author also dabbles a little in the fourth dimension but has the good sense to state that the layman cannot grasp the meaning of the fourth dimension, although the mathematician can play with it in mathematical terms. The same might also be stated as being true of such mathematical toys as imaginary quantities. The square root of minus one, for example, is a plaything with which the mathematician can amuse himself in a variety of different ways, although the layman cannot have the slightest notion of its meaning, nor, in fact, can the mathematician himself, although some time he can use it with very good and practical results as a term in a mathematical series.

This little book of Dr. Harrow's no doubt will prove instructive and entertaining to the unscientific layman who has to be warned that the ether of the outside space cannot be used as an anesthetic, but there does not seem to be anything of interest in this publication for the scientist or the scholar.

J. MERRITT MATTHEWS

NEW PUBLICATIONS

By Clara M. Guppy, Librarian, Mellon Institute of Industrial Research, Pittsburgh

Aluminum: L'Aluminium et ses Alliages. C. Grard. 251 pp. Price, 25 fr. Berger-Levrault, Paris.

Chemical Theory: Elementary Chemical Theory and Calculations. Joseph Knox. 2d Ed. 8vo. 109 pp. Gurney & Jackson, London.

Chemistry: Applied Chemistry; A Practical Handbook for Students of Household Science and Public Health. C. K. Tinkler and Helen Masters. Vol. 1. 8vo. Price, 12s. 6d. C. Lockwood & Son, London.

Chemistry: A Course of Practical Chemistry for Agricultural Students. L. F. Newman and H. A. D. Neville. Vol. 1. 235 pp. Price, 10s. 6d. University Press, Cambridge.

Chemistry: A Foundation Course in Chemistry for Students of Agriculture and Technology. J. W. Dodgson and J. A. Murray. 2d Ed. 8vo. 253 pp. Price, 6s. 6d. Hodder & Stoughton, London.

Coal-tars and Their Derivatives. G. Malatesta. Translated from the First Italian Edition, with Revisions, Corrections and Additions by the Author. 8vo. 530 pp. Price, 21s. E. & F. N. Spon, Ltd., London.

Colloids: The Use of Colloids in Health and Disease. A. B. Searle. 120 pp. Price, 8s. Constable & Co., Ltd., London.

Cosmetics: A Handbook of the Manufacture, Employment, and Testing of All Cosmetic Materials and Cosmetic Specialties with Numerous Recipes. Theodor Koller. Translated from the German. 3d English Edition. 8vo. 264 pp. Price, 8s. 6d. Scott, Greenwood & Son, London.

Foods: The Microbiology and Microanalysis of Foods. Albert Schneider. 8vo. Price, $3.50. P. Blakiston's Son & Co., Philadelphia.

Fuels: Use of Low-Grade and Waste Fuels for Power Generation. J. B. C. Kershaw. 202 pp. Price, 17s. Constable & Co., Ltd., London.

Legal Chemistry and Scientific Criminal Investigation. A. Lucas. 8vo. 181 pp. Price, $3.40. Longmans, Green & Co., New York.

Metallography. S. L. Hoyt. Vol. 1. 8vo. 256 pp. Price, $3.00. McGraw-Hill Book Co., New York.

Nitrates, Phosphates, Potash. R. De Bonard. 200 pp. Price, 18 fr. Ch. Béranger, Paris.

Oils, Industrial: Les Huiles Industrielles et leurs Dérivés; Fabrication, Tranformations; Applications. J. Fritsch. 357 pp. Price, 30 fr. A. Legrand, Paris.

Ore Dressing: Handbook of Ore Dressing, Equipment and Practice. A. W. Allen. 12mo. 239 pp. Price, $3.00. McGraw-Hill Book Co., New York.

Organization as Applied to Industrial Problems. H. T. Wright. 21s. C. Griffin & Co., Ltd., London.

Paper: Modern Pulp and Paper Making; A Practical Treatise. G. S. Witham. Price, $6.00. The Chemical Catalog Co., Inc., New York.

Phosphorus: Phosphore, Arsenic, Antimoine. Bibliothèque de Chimie de L'Encyclopédie Scientifique. A. Boutaric and A. Raynaud. 420 pp. Price, 9 fr. 50. G. Doin, Paris.

Soap: Manual of Toilet Soap-Making Comprising Toilet Soaps, Medicated Soaps and other Specialties. C. Deite. 2d Revised English Edition. 8vo. 356 pp. Price, 21s. 9d. Scott, Greenwood & Son, London.

Steel: Hardening, Tempering, Annealing and Forging of Steel, Including Heat Treatment of Modern Alloy Steels. J. V. Woodworth. 5th Ed. 8vo. 321 pp. Price, $3.00. N. W. Henley Pub. Co., New York.

Volatile Oils. E. Gildemeister. Translated by Edward Kremers. 2 Vols. 2d Ed. 8vo. Price, $7.50 each. John Wiley & Sons, Inc., New York.

Volumetric Analysis. G. S. V. Wills. 8vo. 96 pp. Price, 5s. Wills and Woodnoth, London.

Water Purification Plants and Their Operation. M. F. Stein. 2d Ed. Revised. 8vo. 258 pp. Price, $3.00. John Wiley & Sons, Inc., New York.

RECENT JOURNAL ARTICLES

Aluminum: Increasing Use of Alloyed Aluminum as an Engineering Material. G. M. Rollason. *Industrial Management*, Vol. 59 (1920), No. 6, pp. 456–461.

Aluminum: Rate of Corrosion of Aluminum. G. H. Bailey. *Journal of the Society of Chemical Industry*, Vol. 39 (1920), No. 9, pp. 118*t*–120*t*.

By-Product and Producer Gas Plant: The Lay-out and Operation of a By-Product Producer Gas Plant. W. H. Patchell. *Engineering and Industrial Management*, Vol. 3 (1920), No. 20, pp. 619–622.

By-Product Coke and By-Products of Coal. T. E. Pierce. *Blast Furnace and Steel Plant*, Vol. 8 (1920), No. 5, pp. 283–287.

Catalysis from an Industrial Standpoint. E. B. Maxted. *Journal of the Society of Chemical Industry*, Vol. 39 (1920), No. 8, pp. 95*t*–98*t*.

Chemical Patents. A. E. Macrae. *Canadian Chemical Journal*, Vol. 4 (1920), No. 5, pp. 126–129.

Cyanide: Canadian Cyanide: Its Manufacture and Utilization. W. S. Landis. *Canadian Chemical Journal*, Vol. 4 (1920), No. 5, pp. 130–132.

Cyanogen Chloride: The Preparation of Cyanogen Chloride on a Large Laboratory Scale. T. S. Price and S. J. Green. *Journal of the Society of Chemical Industry*, Vol. 39 (1920), No. 8, pp. 98*t*–101*t*.

Distillation et Rectification. Théorie Thermique de la Distillation, de la Rectification et des Reactions Chimiques Effectuées dans les Colonnes ou les Tours a marche continue. L. Gay. *Chimie et Industrie*, Vol. 3 (1920), No. 2, pp. 157–166.

Dyeing: Para Red Dyeing. A. J. Hall. *Color Trade Journal*, Vol. 6 (1920), No. 6, pp. 177–179.

Dyestuff Industry: National Foreign Policies Important for Development of American Dyestuff Industry. L. W. Alwyn-Schmidt. *Color Trade Journal*, Vol 6 (1920), No. 6, pp. 159–62.

Dyestuffs: Chemical and Physical Properties of Dyestuffs. Acid and Basic Dyes in Gelatin Solutions. J. Traube and F. Koehler. *Scientific American Monthly*, Vol. 1 (1920), No. 5, pp. 408–411.

Dyestuffs: Foundations for Dyestuff Industry. T. H. Norton. *Textile World Journal*, Vol. 57 (1920), No. 23, pp. 132–7.

Experimental Laboratory: How to Equip and Operate an Experimental Laboratory. O. Kress, S. D. Wells and V. P. Edwards. *Paper*, Vol. 26 (1920), No. 13, pp. 11–14, 28; No. 14, pp. 24–27.

Fertilizer Situation: Statement of the Present Tendencies of the Fertilizer Industry. Milton Whitney. *Chemical and Metallurgical Engineering*, Vol. 22 (1920), No. 22, pp. 1021–23.

Laundry Chemist and the "Pure Fabric Law." H. G. Elledge. *Chemical Age*, Vol. 28 (1920), No. 5, pp. 145–146.

Magnesite Refractories. J. S. McDowell and R. M. Howe. *Journal of the American Ceramic Society*, Vol. 3 (1920), No. 3, pp. 185–246.

Maple Products: The Chemistry of Maple Products; The Chemical Analysis of Maple Sap and Sugars, with Methods of Determining Adulteration. J. F. Snell. *Canadian Chemical Journal*, Vol. 4 (1920), No. 5, pp. 122–125.

Meat Packing Industry: Development of the Laboratory in the Meat Packing Industry. L. M. Tolman. *Chemical Age*, Vol. 28 (1920), No. 5, pp. 165–168.

Natural Gas: Conservation of Our Natural Gas. J. B. Garner. *Chemical Age*, Vol. 28 (1920), No. 5, p. 168.

Nitrogen: Determining Nitrogen in Sodium Nitrate. C. A. Butt. *Sugar*, Vol. 22 (1920), No. 6, pp. 341–2.

Potash Recovery in New Jersey. R. N. Shreve. *Chemical Age*, Vol. 28 (1920), No. 5, pp. 149–151.

Potassium Permanganate: Manufacture of Potassium Permanganate. R. B. Stringfield. *Chemical and Metallurgical Engineering*, Vol. 22 (1920), No. 22, pp. 1027–1030.

Producer Gas as Fuel for the Glass Industry. F. J. Denk. *Journal of the American Ceramic Society*, Vol. 3 (1920), No. 2, pp. 94–113.

Rubber: Electricity in the Rubber Industry. C. W. Drake. *Canadian Chemical Journal*, Vol. 4 (1920), No. 5, pp. 133–135.

Salts: The Caking of Salts. T. M. Lowry and F. C. Hemmings. *Journal of the Society of Chemical Industry*, Vol. 39 (1920), No. 8, pp. 101*t*–110*t*.

Starch: Influence of Fermentation on the Starch Content of Experimental Silage. A. W. Dox and Lester Yoder. *Journal of Agricultural Research*, Vol. 19 (1920), No. 4, pp. 173–79.

Steel, Its Defects from User's Viewpoint. F. C. A. H. Lantsbury. *The American Drop Forger*, Vol. 6 (1920), No. 5, pp. 238–242.

Sulfuric Acid: La Fabrication de L'Acide Sulfurique par le Procédé des Chambres. M. Kaltenbach. *Chimie et Industrie*, Vol. 3 (1920), No. 4, pp. 407–421.

Turbine Steels. A Research into Their Mechanical Properties. W. H. Hatfield. *Engineering and Industrial Management*, Vol. 3 (1920), No. 14, pp. 425–426.

Vulcanization: The Acceleration of Vulcanization. D. F. Twiss and S. A. Brazier. *Journal of the Society of Chemical Industry*, Vol. 39 (1920), No. 9, pp. 125*t*–132*t*.

Yeast: Debittered Dried Yeast. L. J. Riley. *The Chemical Age*, Vol. 2 (1920), No. 47, p. 497.

Zinc Industry of the Tri-State (Oklahoma, Missouri and Kansas) Field. F. J. Tietsort. *Compressed Air Magazine*, Vol. 25 (1920), No. 5, pp. 9635–9643.

MARKET REPORT—JUNE, 1920

FIRST-HAND PRICES FOR GOODS IN ORIGINAL PACKAGES PREVAILING IN THE NEW YORK MARKET

INORGANIC CHEMICALS

		June 1	June 15
Acid, Boric, cryst., bbls.	lb.	.18	.18
Hydrochloric, com'l, 22°	lb.	.03½	.03½
Hydriodic	oz.	.19	.19
Nitric, 42°	lb.	.08½	.08½
Phosphoric, 50% tech.	lb.	.25	.25
Sulfuric, C. P.	lb.	.07	.07
Chamber, 66°	ton	22.00	22.00
Oleum	ton	28.00	28.00
Alum, ammonia, lump	lb.	.04	.04
Aluminum Sulfate (iron-free)	lb.	.04	.04
Ammonium Carbonate, pwd.	lb.	.14½	.16
Ammonium Chloride, gran.	lb.	.18	.15
Ammonia Water, carboys, 26°	lb.	.10	.10
Arsenic, white	lb.	.14½	.15
Barium Chloride	ton	160.00	175.00
Nitrate	lb.	.12	.11
Barytes, white	ton	30.00	30.00
Bleaching Powd., 35%, Works,	100 lbs.	5.50	5.50
Borax, cryst., bbls.	lb.	.09½	.09½
Bromine, tech., bulk	lb.	.85	.85
Calcium Chloride, fused	ton	27.50	27.50
Chalk, precipitated, light	lb.	.05	.05
China Clay, imported	ton	18.00	18.00
Copper Sulfate	100 lbs.	8.25	8.25
Feldspar	ton	8.00	8.00
Fuller's Earth	100 lbs.	1.25	1.25
Iodine, resublimed	lb.	4.35	4.35
Lead Acetate, white crystals	lb.	.15	.15
Nitrate	lb.	.15	.15
Red American	100 lbs.	.10½	.10½
White American	100 lbs.	.09½	.09½
Lime Acetate	100 lbs.	3.50	3.50
Lithium Carbonate	lb.	1.50	1.50
Magnesium Carbonate, Tech.	lb.	.12	.12
Magnesite	ton	65.00	65.00
Mercury flask	75 lbs.	80.00	90.00
Phosphorus, yellow	lb.	.35	.35
Plaster of Paris	100 lbs.	1.50	1.50
Potassium Bichromate	lb.	.45	.44
Bromide, Cryst.	lb.	.95	.95
Carbonate, calc., 80–85%	lb.	.18	.18
Chlorate, cryst.	lb.	.15	.15
Cyanide, bulk, 98–99%	lb.	.30	.30
Hydroxide, 88–92%	lb.	.32	.33
Iodide, bulk	lb.	3.35	3.35
Nitrate	lb.	.14	.14
Permanganate, U. S. P.	lb.	.90	.90
Salt Cake	ton	32.00	32.00
Silver Nitrate	oz.	.63	.58
Soapstone, in bags	ton	12.00	12.00
Soda Ash, 58%, bags	100 lbs.	3.65	3.65
Caustic, 76%	100 lbs.	6.50	6.50
Sodium Acetate	lb.	.12	.12
Bicarbonate	100 lbs.	3.00	3.00
Bichromate	lb.	.33	.32
Chlorate	lb.	.10	.10
Cyanide	lb.	.27	.27
Fluoride, technical	lb.	.18	.18
Hyposulfite	100 lbs.	3.60	3.60
Nitrate, 95%	100 lbs.	3.85	3.85
Silicate, 40°	lb.	.02	.02
Sulfide	lb.	.10¼	.10¼
Bisulfite, powdered	lb.	.06½	.06½
Strontium Nitrate	lb.	.27	.27
Sulfur, flowers	100 lbs.	3.75	3.75
Crude	long ton	30.00	30.00
Talc, American, white	ton	20.00	20.00
Tin Bichloride	lb.	.21½	.21½
Oxide	lb.	.60	.60
Zinc Chloride, U. S. P.	lb.	.50	.50
Oxide, bbls.	lb.	.22	.22

ORGANIC CHEMICALS

		June 1	June 15
Acetanilid	lb.	.75	.75
Acid, Acetic, 28 p. c.	100 lbs.	4.00	4.00
Glacial	lb.	.17	.17
Acetylsalicylic	lb.	.95	.95
Benzoic, U. S. P., ex-toluol	lb.	.85	.85
Carbolic, cryst., U. S. P., drs.	lb.	.15	.15
50- to 110-lb. tins	lb.	.21	.23
Acid (*Concluded*)			
Citric, crystals, bbls.	lb.	1.03	.99
Oxalic, cryst., bbls.	lb.	.58	.60
Pyrogallic, resublimed	lb.	2.50	2.50
Salicylic, bulk, U. S. P.	lb.	.55	.55
Tartaric, crystals, U. S. P.	lb.	.84	.82
Trichloracetic, U. S. P.	lb.	4.40	4.40
Acetone, drums	lb.	.22	.22
Alcohol, denatured, 190 proof	gal.	1.13	1.13
Ethyl, 190 proof	gal.	7.00	7.00
Wood, Pure	gal.	3.50	4.40
Amyl Acetate	gal.	4.00	4.00
Camphor, Jap. refined	lb.	1.80	1.65
Carbon Bisulfide	lb.	.08	.08
Tetrachloride	lb.	.14	.14½
Chloroform, U. S. P.	lb.	.40	.40
Creosote, U. S. P.	lb.	.75	.75
Cresol, U. S. P.	lb.	.18	.18
Dextrine, corn	lb.	.06¼	.06¼
Imported Potato	lb.	.14	.14
Ether, U. S. P., conc., 100 lbs.	lb.	.21	.24
Formaldehyde	lb.	.58	.53
Glycerol, dynamite, drums	lb.	.26	.26½
Pyridine	gal.	3.00	3.00
Starch, corn	100 lbs.	5.25	5.25
Potato, Jap.	lb.	.06	.06
Rice	lb.	.25	.25
Sago	lb.	.05	.05

OILS, WAXES, ETC.

		June 1	June 15
Beeswax, pure, white	lb.	.65	.65
Black Mineral Oil, 29 gravity	gal.	.22	.22
Castor Oil, No. 3	lb.	.18	.18
Ceresin, yellow	lb.	.16	.16
Corn Oil, crude	lb.	.16	.16
Cottonseed Oil, crude, f. o. b. mill	lb.	.16	.16
Menhaden Oil, crude (southern)	gal.	.80	.75
Neat's-foot Oil, 20°	gal.	2.25	2.25
Paraffin, 128–130 m. p., ref.	lb.	.11½	.11½
Paraffin Oil, high viscosity	gal.	.45	.45
Rosin, "F" Grade, 280 lbs.	bbl.	20.25	19.05
Rosin Oil, first run	gal.	.90	.83
Shellac, T. N.	lb.	1.00	1.15
Spermaceti, cake	lb.	.30	.30
Sperm Oil, bleached winter, 38°	gal.	1.95	1.95
Stearic Acid, double-pressed	lb.	.27½	.25½
Tallow Oil, acidless	gal.	1.60	1.60
Tar Oil, distilled	gal.	.60	.60
Turpentine, spirits of	gal.	2.45	1.89½

METALS

		June 1	June 15
Aluminum, No. 1, ingots	lb.	.32	.32
Antimony, ordinary	100 lbs.	9.00	8.25
Bismuth	lb.	2.52	2.52
Copper, electrolytic	lb.	.19	.19
Lake	lb.	.18¾	.18½
Lead, N. Y.	lb.	.08½	.08½
Nickel, electrolytic	lb.	.45	.45
Platinum, refined, soft	oz.	95.00	80.00
Quicksilver, flask	75 lbs ea.	80.00	90.00
Silver	oz.	1.01	.90
Tin	lb.	.52½	.50
Tungsten Wolframite	per unit	7.00	7.00
Zinc, N. Y.	100 lbs.	8.50	8.50

FERTILIZER MATERIALS

		June 1	June 15
Ammonium Sulfate	100 lbs.	7.00	7.00
Blood, dried, f. o. b. N. Y.	unit	8.00	8.00
Bone, 3 and 50, ground, raw	ton	48.00	48.00
Calcium Cyanamide, unit of Ammonia		4.00	4.00
Fish Scrap, domestic, dried, f. o. b. works	unit	7.25	7.25
Phosphate Rock, f. o. b. mine:			
Florida Pebble, 68%	ton	6.85	6.85
Tennessee, 78–80%	ton	11.00	11.00
Potassium Muriate, 80%	unit	2.50	2.50
Pyrites, furnace size, imported	unit	.18	.18
Tankage, high-grade, f. o. b. Chicago	unit	7.75	7.75

COAL-TAR CHEMICALS

Crudes

		June 1	June 15
Benzol, C. P.	gal.	.35	.35
Cresol, U. S. P.	lb.	.18	.18
Naphthalene, flake	lb.	.16	.21
Phenol, drums	lb.	.15	.15
Toluol, 90%	gal.	.36	.36
Xylol, water white	gal.	.40	.40

Intermediates

		June 1	June 15
Acids:			
Anthranilic	lb.	2.85	2.85
B	lb.	2.25	2.25
Benzoic	lb.	.85	.85
Broenner's	lb.	1.75	1.75
Cleves	lb.	2.00	2.00
Cresylic, 97–99%	gal.	1.20	1.20
F	lb.	3.50	3.50
Gamma	lb.	2.50	2.50
H	lb.	2.25	2.25
Metanilic	lb.	1.70	1.70
Monosulfonic F	lb.	3.50	3.50
Napthionic, crude	lb.	.85	.85
Neville & Winther's	lb.	1.90	1.90
Phthalic	lb.	.60	.60
Picric	lb.	.25	.25
Sulfanilic	lb.	.35	.35
Tobias'	lb.	2.25	2.25
Amido Azo Benzol	lb.	1.15	1.15
Aniline Oil	lb.	.35	.34
For Red	lb.	.65	.65
Aniline Salt	lb.	.42	.42
Anthracene, 80–85%	lb.	.75	.75
Anthraquinone	lb.	4.50	4.50
Benzaldehyde, tech	lb.	.75	.75
U. S. P.	lb.	1.25	1.00
Benzidine Base	lb.	1.40	1.40
Benzidine Sulfate	lb.	1.15	1.15
Diamidophenol	lb.	6.00	6.00
Dianisidine	lb.	9.00	9.00
p-Dichlorbenzol	lb.	.10	.10
Diethylaniline	lb.	1.40	1.40
Dimethylaniline	lb.	1.25	1.25
Dinitrobenzol	lb.	.36	.36
Dinitrotoluol	lb.	.43	.43
Diphenylamine	lb.	.80	.80
G Salt	lb.	.90	.90
Hydroquinone	lb.	2.00	2.00
Metol	lb.	11.50	11.50
Monochlorbenzol	lb.	.16	.18
Monoethylaniline	lb.	2.15	2.15
a-Naphthylamine	lb.	.45	.45
b-Naphthylamine (Sublimed)	lb.	2.25	2.25
b-Naphthol, crude	lb.	.85	.86
m-Nitraniline	lb.	1.00	1.05
p-Nitraniline	lb.	1.65	1.50
Nitrobenzol, crude	lb.	.16	.16
Rectified (Oil Myrbane)	lb.	.18	.18
p-Nitrophenol	lb.	.80	.80
p-Nitrosodimethylaniline	lb.	2.90	2.90
o-Nitrotoluol	lb.	.25	.25
p-Nitrotoluol	lb.	1.50	1.50
m-Phenylenediamine	lb.	1.15	1.15
p-Phenylenediamine	lb.	2.65	2.65
Phthalic Anhydride	lb.	.45	.45
Primuline Base	lb.	3.00	3.00
R Salt	lb.	.90	.90
Resorcin, tech	lb.	4.00	4.00
U. S. P.	lb.	6.00	6.00
Schaeffer Salt	lb.	.75	.75
Sodium Naphthionate	lb.	1.10	1.10
Thiocarbanilid	lb.	.60	.60
Tolidine, Base	lb.	1.75	1.75
Toluidine, mixed	lb.	.44	.44
o-Toluidine	lb.	.35	.35
m-Toluylenediamine	lb.	1.40	1.40
p-Toluidine	lb.	2.00	2.00
Xylidene, crude	lb.	.45	.45

COAL-TAR COLORS

Acid Colors

		June 1	June 15
Black	lb.	1.15	1.15
Blue	lb.	3.00	3.00

Acid Colors (*Concluded*)

		June 1	June 15
Fuchsin	lb.	2.50	3.00
Orange III	lb.	1.00	1.00
Red	lb.	1.10	1.30
Violet 10B	lb.	6.50	6.50
Alkali Blue, domestic	lb.	4.75	5.50
Imported	lb.	8.00	8.00
Azo Carmine	lb.	4.00	4.00
Azo Yellow	lb.	2.00	2.00
Erythrosine	lb.	12.00	12.00
Indigotine, conc	lb.	3.00	3.00
Paste	lb.	1.50	1.50
Naphthol Green	lb.	1.50	1.50
Ponceau	lb.	1.25	1.35
Scarlet 2R	lb.	1.00	1.00

Direct Colors

		June 1	June 15
Black	lb.	1.00	1.00
Blue 2B	lb.	.70	.70
Brown R	lb.	1.80	1.80
Fast Red	lb.	3.50	3.50
Yellow	lb.	2.00	2.00
Violet, con't	lb.	2.20	2.20
Chrysophenine, domestic	lb.	2.25	2.25
Imported	lb.	3.80	3.80
Congo Red, 4B Type	lb.	.90	.90
Primuline, domestic	lb.	3.00	3.00

Oil Colors

		June 1	June 15
Black	lb.	.70	.70
Blue	lb.	1.65	1.65
Orange	lb.	1.40	1.40
Red III	lb.	1.65	1.65
Scarlet	lb.	1.75	1.75
Yellow	lb.	1.70	1.70
Nigrosine Oil, soluble	lb.	.90	.90

Sulfur Colors

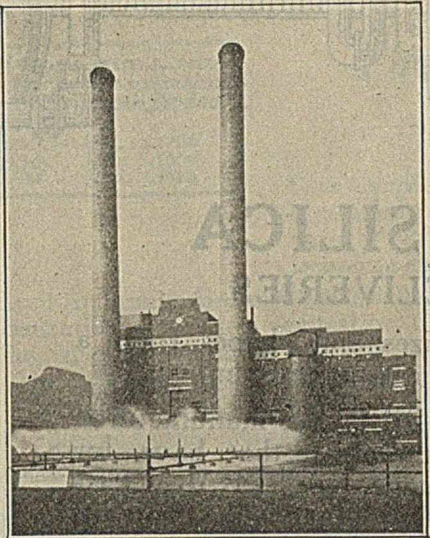
		June 1	June 15
Black	lb.	.20	.20
Blue, domestic	lb.	.70	.70
Brown	lb.	.35	.35
Green	lb.	1.00	1.00
Yellow	lb.	.90	.90

Chrome Colors

		June 1	June 15
Alizarin Blue, bright	lb.	7.75	7.75
Alizarin Red, 20% Paste	lb.	1.10	1.10
Alizarin Yellow G	lb.	1.00	1.00
Chrome Black, domestic	lb.	1.25	1.25
Imported	lb.	2.20	2.20
Chrome Blue	lb.	2.50	2.50
Chrome Green, domestic	lb.	1.50	1.50
Chrome Red	lb.	2.00	2.00
Gallocyanine	lb.	3.25	3.25

Basic Colors

		June 1	June 15
Auramine, O, domestic	lb.	2.50	2.50
Auramine, OO,	lb.	4.15	4.15
Bismarck Brown Y	lb.	.90	.90
Bismarck Brown R	lb.	1.20	1.20
Chrysoidine R	lb.	.75	.75
Chrysoidine Y	lb.	.75	.75
Green Crystals, Brilliant	lb.	6.00	6.00
Indigo, 20 p. c. paste	lb.	.85	.85
Fuchsine Crystals, domestic	lb.	5.00	5.50
Imported	lb.	12.00	12.00
Magenta Acid, domestic	lb.	4.25	4.25
Malachite Green, crystals	lb.	4.50	4.50
Methylene Blue, tech	lb	2.50	2.75
Methyl Violet 3 B	lb.	3.00	3.50
Nigrosine, spts. sol	lb.	.85	.85
Water sol., blue	lb.	.65	.65
Jet	lb.	.90	.90
Phosphine G., domestic	lb.	7.00	7.00
Rhodamine B, extra conc'd	lb.	35.00	40.00
Victoria Blue, base, domestic	lb	6.00	6.00
Victoria Green	lb.	6.00	6.00
Victoria Red	lb.	7.00	7.00
Victoria Yellow	lb.	7.00	7.00



Power Plant, U. S. Government Nitrate Plant No. 2, Muscle Shoals, Ala. The J. G. White Engineering Corporation, Contractors.

This Prominent Government Power Plant

--- as well as the Power Pumping Station and many other structures at Nitrate Plant No. 1, at Sheffield, Alabama, is safe from dampness and other deteriorating influences,

R.I.W. REMEMBER IT'S WATERPROOF REG. U.S. PAT. OFF **PROTECTIVE PRODUCTS**

having been specified and used throughout.
Absolute protection to steel work and concrete against the effects of acid and alkali fumes, as well as dampness, was afforded by the use of Nos. 110 and 232 "R. I. W." Damp Resisting Paint and "R. I. W." Toxement (Pat'd). The first two products were used on the steel work and on wall surfaces, respectively.
No. 110 "R. I. W." in particular, is especially notable for protecting interior steel work from corrosion.
"R. I. W." Toxement was used to render all concrete waterproof, not alone on the surface but *through and through.*
We refer to any user as to results.

For details Address TECH. DEPT.

TOCH BROTHERS

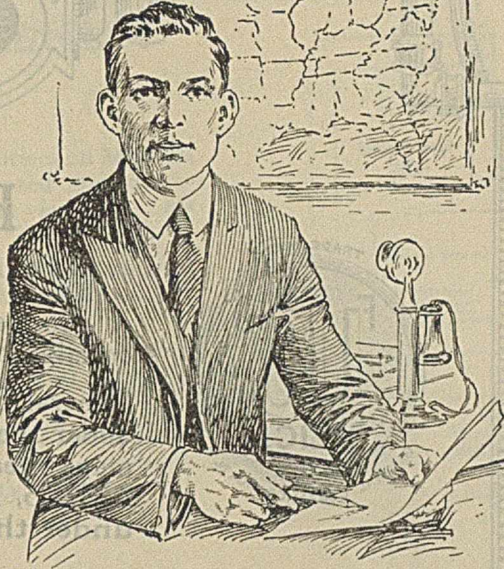
Technical and Scientific Paint Makers since 1848

320 FIFTH AVENUE, NEW YORK

Works: Long Island City, N. Y., and London, England

R.I.W.
REMEMBER IT'S WATERPROOF
REG. U.S. PAT. OFF.

STEEL NEED NOT RUST!
WOOD NEED NOT ROT!
NOR CONCRETE DUST!



$500.00 Saved Monthly in Boiler Labor Alone

After operating a Permutit Water Softening and Filtering System for one and three-quarter years a Light & Power Company in Illinois reported the following:

We have no scale in boilers or pipe lines.

Our saving in coal is quite material—the saving over former conditions is easily 30%.

We formerly had two men drilling boiler tubes all the time, and now have none.

Our saving in boiler labor is easily $500.00 per month.

Zerowater appears just the thing for condensers.

It will pay you to investigate the system that is saving 30% fuel and $500.00 monthly in labor for others. Ask us for more facts—other testimony. Write us today. No obligations.

The Permutit Company
440 Fourth Ave. New York
Offices in all principal cities

FUSED SILICA

QUICK DELIVERIES

TRADE MARK FUSD SILICA GENERAL CERAMICS CO. N.Y.

A complete line of all sizes and shapes from a crucible to a sulfuric acid cascade system.

General Ceramics FUSED SILICA is manufactured under the same rigid requirements that have, for many years, made the name, GENERAL CERAMICS, mean STONEWARE that will **stand up under the most severe conditions.**

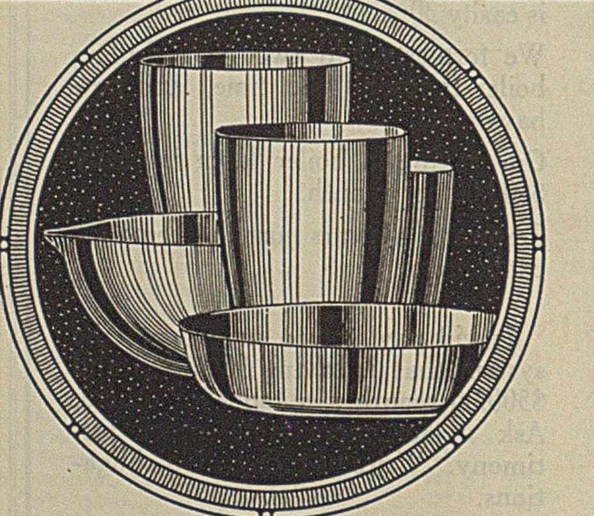
GENERAL CERAMICS COMPANY

"Equipped to Supply a Plant or a Nation"

Manufacturers of Acid-Proof Chemical Stoneware

50 Church Street **New York City**

BAKER PLATINUM APPARATUS



When a manufacturer's name is so closely associated with his product that they are scarcely ever thought of apart—
When, through long years of maintaining a high standard, he has justly earned that mysterious, incalculable asset, good-will—
You may rest assured that high quality, right prices and good service (those essential yet overworked words) are built into and about the product. In no other way could names become actual synonyms, as, for instance—

BAKER PLATINUM

This association ideas is something of which we are proud. We set out to manufacture all forms and sizes of platinum ware for chemical and physical purposes; to produce flawless goods of standard purity at a price consistent with their worth.

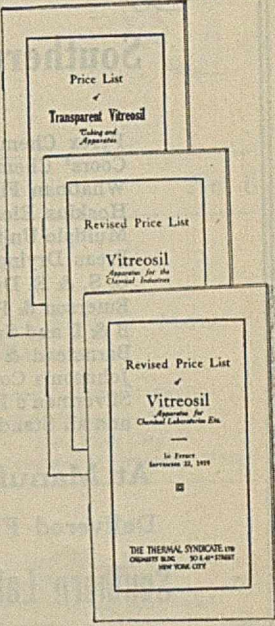
We have succeeded because we *guaranteed* our products and our integrity was never questioned.

In addition to regular stocks of platinum ware, we manufacture special apparatus to order and reshape, repair, purchase or exchange old dishes, crucibles and apparatus of all kinds.

BAKER & CO., INC.

Refiners and Workers of Platinum Gold and Silver

30 Church St. New York
54 Austin St.
NEWARK. N. J.
5 S. Wabash Ave. Chicago.


Price List
Transparent Vitreosil
Tubing and Apparatus
Revised Price List
Vitreosil
Apparatus for the Chemical Industries
Revised Price List
Vitreosil
Apparatus for Chemical Laboratories Etc.
In Force September 22, 1919
THE THERMAL SYNDICATE LTD
CHEMISTS BLDG. 50 E 41st STREET
NEW YORK CITY

"Pioneers of the South"

Southern Distributors

OF

Pyrex Chemical Glassware
Coors' Chemical Porcelain
Whatman Filter Papers
Hoskins Electric Furnaces, Etc.
Multiple Unit Electric Furnaces, Etc.
Freas Drying Ovens & Incubators
C. S. & E. Drying Ovens & Incubators
Emerson & Parr Calorimeters
B & L and Spencer Microscopes
Barnstead & Stokes Water Stills
Johnson's Combustion Tubes & Boats
Silverman's Illuminator
and all Standard Lines

At Manufacturer's Prices

Delivered F.O.B. Birmingham

Southern Laboratory Supply Co.

Warehouse and Offices:
1608-1610—2nd Ave.
Birmingham, Ala.

The Emil Greiner Co.

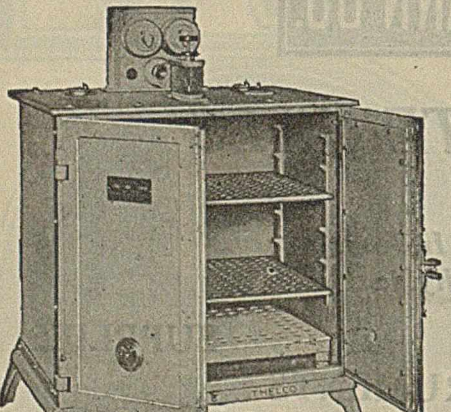
Established 1881
Manufacturers
of
Laboratory Apparatus and Chemical Glassware
55 Fulton and 45 Cliff Sts.
New York, N. Y.

Beckmann Thermometers
Chemical Thermometers
Industrial Thermometers
Hydrometers for Alcohol
Hydrometers for Sugar
Hydrometers for Acids
Volumetric Glassware
Burettes—Pipettes—Flasks
Special apparatus made to order according to sketch.

Our name stands for quality.

Expert glassblowers for over 30 years.

Thelco Electric Oven



Large Thelco Oven 18 x 12 x 14"

Do not fail to ask us for special prices on your Laboratory Requirements.

Glassware as per specifications of the Bureau of Standards, Wash.

Write for special circular

LABORATORY SUPPLIES

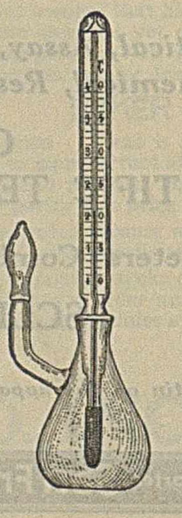
Reynolds, Teschner & Volk Co., Inc.

46 VESEY ST. NEW YORK

THE

NEW YORK THERMOMETER COMPANY

Manufacturers of
THERMOMETERS and HYDROMETERS
36 E. 23rd Street, New York.
Factory: 381 Palmetto Street, Brooklyn, N. Y.



Alcohol Thermometers
Allihn "
Anschutz "
Beckmann "
Calorimeter "
Chemical "
Clinical "
Cold Test "
Cylinder "
Dairy "
Hydrometers
Incubator Thermometers
Lactometers
Long Stem Thermometers
Pentane "
Specific Gravity Bottles
Toluol Thermometers
Urinometers

Write for our complete Catalogue.