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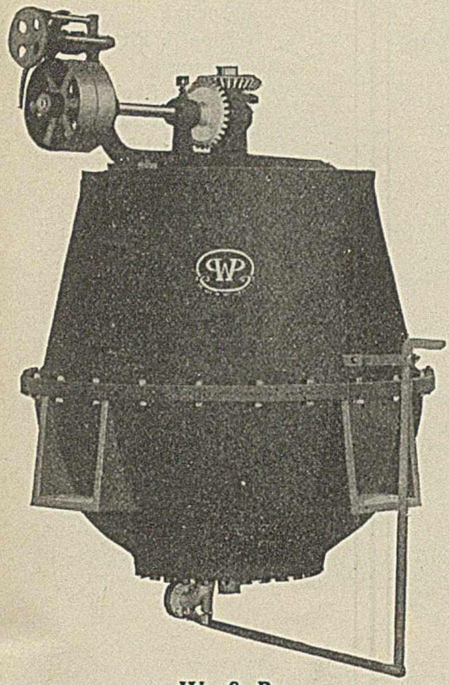

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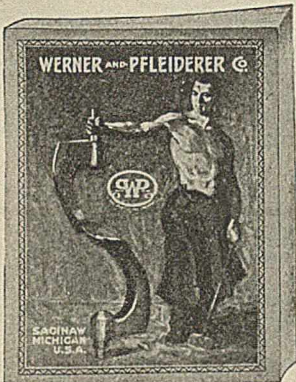

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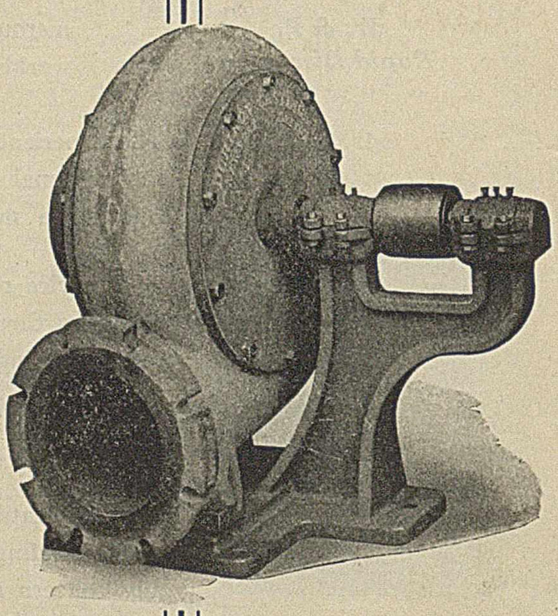

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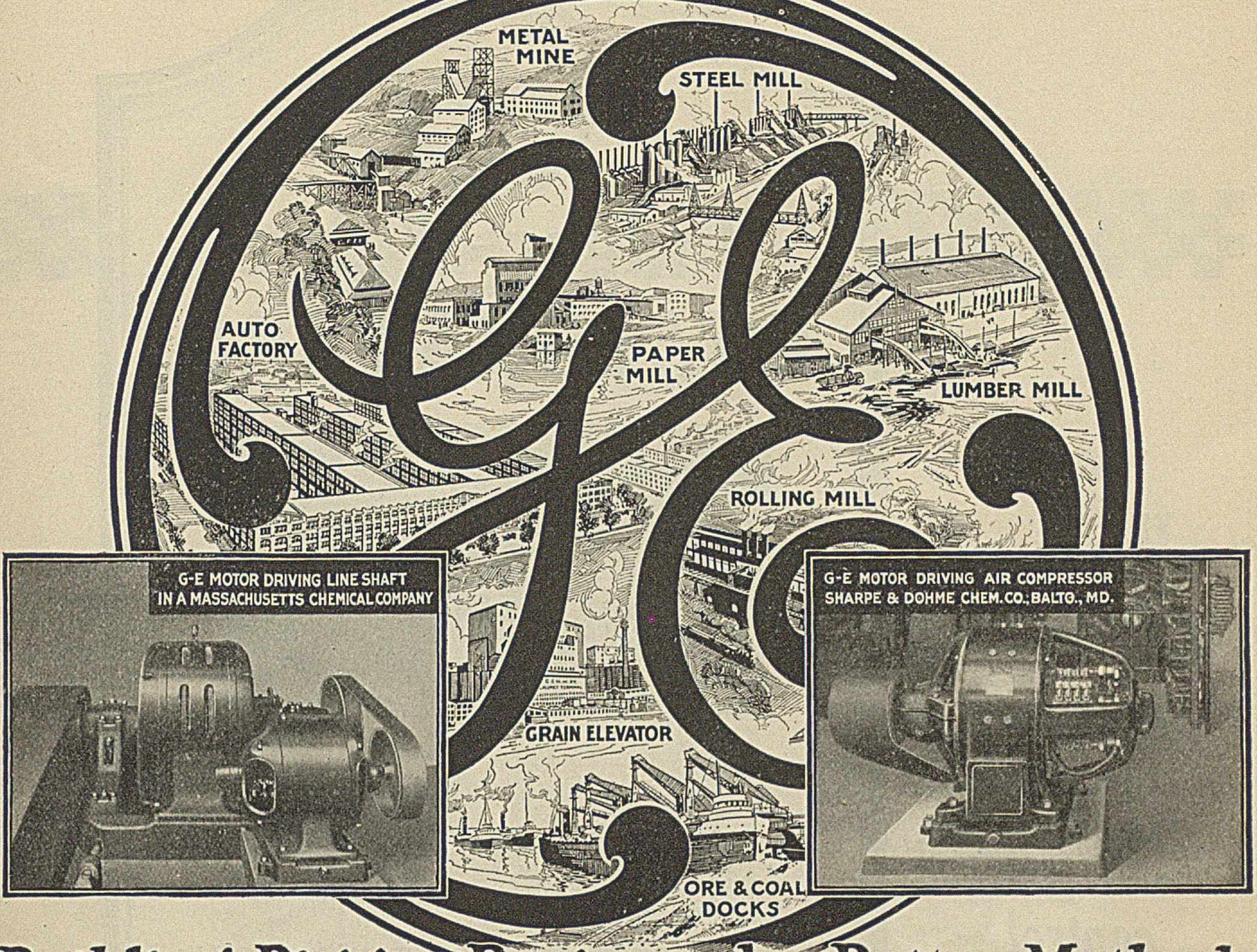

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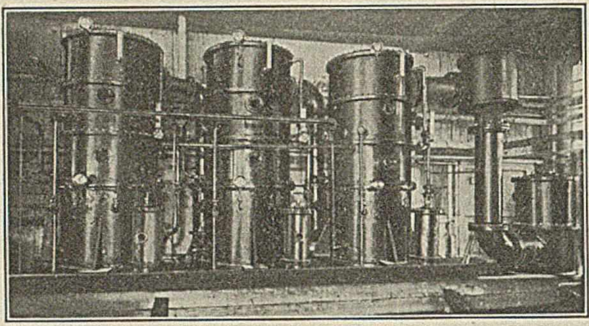

The general features incorporated in all of our Evaporator Designs require:—

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6. No moving parts.
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We build evaporators adapted to the particular work in hand. Under certain conditions it is more advantageous to use horizontal tube effects, and we have designed for this work an evaporator that has been tried out and proven to be excellently adapted to the purpose in hand.

For liquids which foam and for solutions containing a large proportion of incrusting material we have designed and constructed a fast circulation evaporator which has proven entirely successful.

Under certain other conditions we recommend the use of a basket type Evaporator.

We would like to inform our friends that in line with the general policy of the **THOROUGH BADGER SERVICE** we have a very competent staff of experienced men who make a specialty of studying all evaporator and heat problems, evolving such equipments as are best suited to the precise conditions of operation, not only as regards the material being treated, but also taking into careful account plant conditions, such as steam supply, water supply, and the limitations imposed by already existing apparatus. It is with pleasure that we solicit an opportunity of demonstrating the high quality of this branch of our service.

E. B. BADGER & SONS CO.
BOSTON, MASS., U. S. A.

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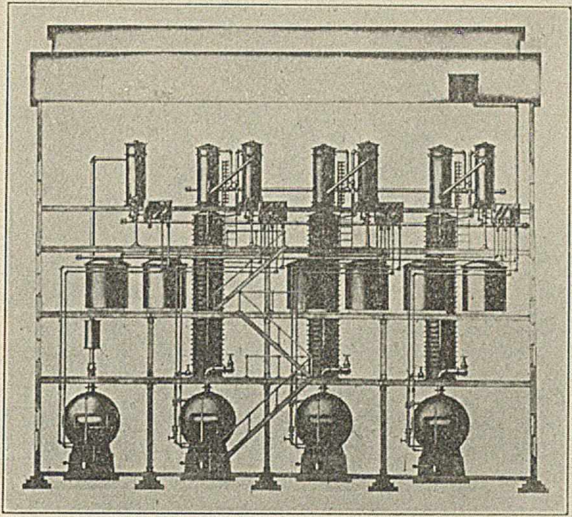
can be had for the Largest Work and Highest Degree of Refinement



Outside of the general design of distilling equipment, which includes the general arrangement and proportion of the parts involved, BADGER STILLS possess many special features, such as perforated boiling cap construction, liquid distillate flow and feed regulators, pressure regulators, etc., such as are not embodied in the design and construction of any other stills.

We construct practically all of our apparatus in our own shops. We furnish not only the famous Barbet Continuous Stills, but also BADGER Stills of continuous and discontinuous design. In carrying out their specifications the work of construction is placed in the hands of expert workmen, who have a complete knowledge of metal working and its application to still building. Our object is to furnish the most efficient still possible, to give an apparatus which will be easily operated, durable, and which will give the highest grade of product desired.

We shall be glad to furnish you with any information which you may require regarding your problems of distillation and condensation.

E. B. BADGER & SONS CO.
BOSTON, MASS., U. S. A.

ALCOHOL

The production of pure ethyl alcohol from fermented mash was for many years handled in a very inefficient manner. The perfected stills which we have developed for this purpose, in conjunction with the Barbet Company, give a better product with enormous economy.

ACETIC ACID

Units for the distillation, refining or concentration of acetic acid require expert knowledge of construction and design. We have recently furnished and have under construction, several complete plants for this work.

SALICYLIC ACID

The special design of still, with special lining required for handling this product, is right in our line. Successful apparatus which we have furnished involves special features which only long experience in design and construction has put us in a position to work out.

ANILINE

The economical production of aniline of proper boiling point requires the use of stills of the same general design as our phenol stills.

ACETALDEHYDE

The distillation of acetaldehyde from solutions is a problem requiring very careful design; our continuous stills for this purpose have solved the problem.

ETHER

We are the constructors of probably 95% of the ether stills required for smokeless powder work in the United States and Canada. Of more than 75 units installed for this work, there are none giving other than absolute satisfaction.

BENZOL

Our cast iron construction, automatic flow, pressure regulators and general design of stills are distinctly advantageous for this work.

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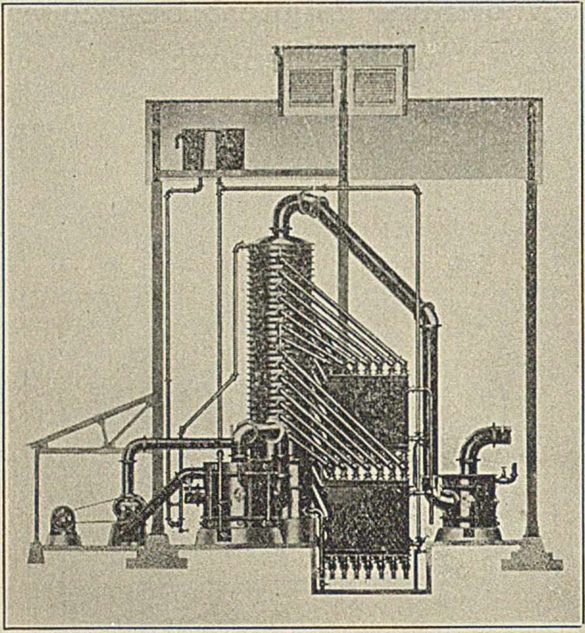
SOLVENT RECOVERY
and
ABSORPTION EQUIPMENT



The great development in the chemical industry and the increasing high price of solvents have made the question of proper solvent recovery a very important one. There are, furthermore, many processes where extraction with volatile solvent can be employed to great advantage. We have had a great deal of experience in handling many problems of this kind.

The laws of vapor pressure, which govern practically all recovery operations of this type, are little understood in their commercial application. By scientific study of the problems involved we have been able to effect enormous economies in operation, and obtain recoveries which were not considered possible.

Solvent extraction and absorption can be employed in place of compression and distillation. It requires expert knowledge and careful study to determine which method is the best. We are familiar with the whole field and can advise clients properly in this regard.

It would be well for concerns having problems of this kind on hand to consult with us and get the benefit of our experience in many industries.

We are enumerating on this page a few of the special problems in solvent extraction and recovery to which our equipment has been applied.

BENZOL

The recovery of benzol from coal gas by absorption and subsequent distillation is very important at the present time. We have developed special forms of vacuum and gas heated stills, as well as absorption towers, which are exceedingly efficient and economical.

ACETONE

The application of our cap plate water or brine-cooled absorption system to the recovery of acetone and alcohol employed in the manufacture of Cordite not only gives exceedingly high recovery, but permits the obtaining of a concentrated solution which can be distilled with great economy.

AMMONIA

The application of our special designs of distilling and scrubbing columns to the production of pure ammonia gas has been productive of very satisfactory results.

ETHER

The recovery of ether in the manufacture of smokeless powder by absorption and suitable solvent requires long experience and efficient apparatus. We have given a great deal of study to this particular problem.

PHENOL

In many cases solvent extraction can take the place of distillation with considerable economy and the production of higher quality product. The production of carbolic acid from coal tar distillate, which is a problem of this kind, has been successfully solved.

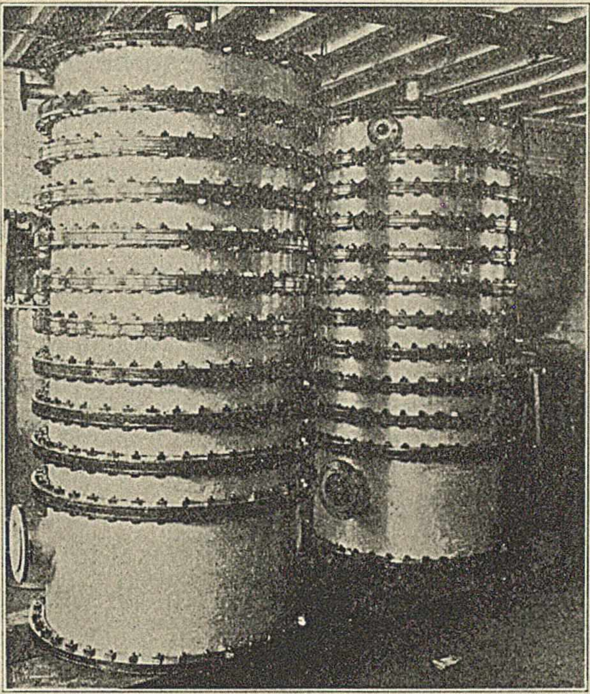
ETHYL NITRITE

The recovery of this material from gas from a particular manufacturing plant illustrates the advantages of expert knowledge and careful design. It is also an example of absorption of material insoluble in water or in other solvent, such as, in this case, alcohol.

ACETALDEHYDE

In process recently developed for the manufacture of acetic acid, the efficient absorption of acetaldehyde from air and gas has been of great importance. This is a problem which we have solved, and the successful working out of the process made unnecessary the use of refrigerating plant otherwise required, which would have cost not less than $100,000.

E. B. BADGER & SONS CO.
BOSTON, MASS., U. S. A.



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IS ONE OF THE MANY ACCOMPLISHMENTS OF THE

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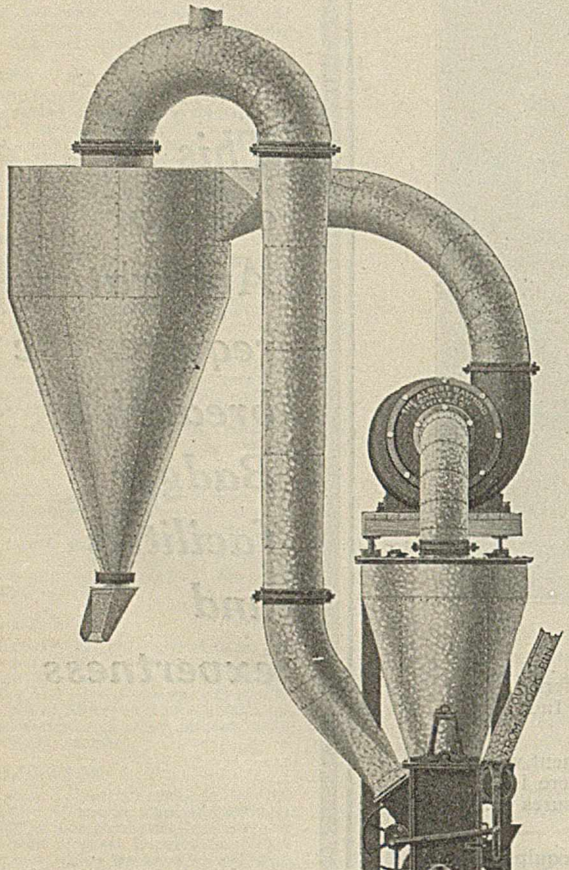
In many of the new industries developed since the beginning of the war fine ground materials have been required. And being able to produce an economical fine grind on these materials has in many cases determined the successful commercial solution of a particular process.



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If you require a powdered material of any kind investigate the ***Raymond System*** which will handle many materials that cannot be screened.

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We design special machinery and methods for Pulverizing, Grinding, Separating and Conveying all powdered products. We manufacture Automatic Pulverizers, Roller Mills, Vacuum Air Separators, Crushers, Special Exhaust Fans and Dust Collectors.

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TABLE OF CONTENTS

EDITORIALS:

ORIGINAL PAPERS:

LABORATORY AND PLANT:

FORMAL OPENING OF THE NEW CHEMICAL LABORATORY, UNIVERSITY OF CINCINNATI:

ADDRESSES:

SCIENTIFIC SOCIETIES:

EDITORIALS

THE PLATINUM SITUATION

The movement for the conservation of platinum, inaugurated at the recent Kansas City Meeting of the American Chemical Society, is making satisfactory progress. The phrase "conservation of platinum" is accurate, for by conservation is meant not the hoarding but the wise use of resources. In this case it signifies the diversion of large supplies of the precious metal, from decorative but unnecessary use in jewelry, into those chemical industries, such as the sulfuric acid industry, where it is essential. Fortunately the jewelers, in a spirit of patriotism, have endeavored to meet the situation by passing resolutions, printed elsewhere in this issue, pledging themselves to aid in this phase of conservation.

If confirmation of the wisdom and necessity of such conservation were needed it was furnished during the present month by Judge Sheldon Amos, the Munitions expert of the British Commission. At the luncheon given in his honor by the executive council of the National Civic Federation, Judge Amos said: "One of the first things we did, and which must be done in every country under such conditions, was to put a 'stopper' on certain kinds of trade; that is, stop speculation in munitions and army supplies and, in fact, all materials in connection with which there would be danger of the Government being embarrassed by competition. *The first on the list of such materials are the rare metals, notably platinum*" (our italics).

In the light of the appeals by scientific organizations, the testimony of the British expert, and the resolutions of the jewelers, we confess that we are surprised and puzzled by a statement attributed to the Secretary of Commerce. While commending the resolutions of the jewelers, as "wise, patriotic and unselfish action," he is quoted in the *New York Evening Sun*, of May 7, 1917, as saying: "The country will in no way be aided by the cutting down of purchases by the public, and the women of New York would be acting under a false impression if they accept as true the erroneous newspaper statements that they should stop buying platinum jewelry in order to show their patriotism."

Meanwhile, so far as laboratory utensils are concerned, chemists are busily engaged in trying to find suitable substitutes for platinum. The advance in recent years of 500 per cent in the price of platinum makes this search imperative. In this issue we print an article by Dr. Fahrenwald giving the results of tests on "rhotanium." Laboratory ware made of "palau," a palladium-gold alloy, is now being marketed. Mr. Haynes' tungsten-chromium-cobalt alloy, "stellite," presents interesting possibilities, and is being thoroughly tested. To coördinate work in this field, Prof. Bogert, Chairman of the Chemistry Committee of the National Research Council, has appointed Prof. James Lewis Howe, of Washington and Lee University, Lexington, Va., Special Committeeman in charge of substitutes for platinum. The Bureau of Standards is likewise actively at work on this subject. Necessity is still the mother of invention.

THE WILLARD GIBBS MEDAL AWARD

At the meeting of the Chicago Section on May 18, 1917, the Willard Gibbs medal was awarded to Dr. Edward Williams Morley. This event carries pleasure to the hearts of all American chemists. By his painstaking, accurate work on the densities of oxygen and hydrogen, Dr. Morley has made permanently valuable contributions to the very fundamentals of chemistry and has set an example of patience, skill and scientific integrity.

It is, however, not alone as one of our most distinguished chemists that we love to think of him, but also as a man who has endeared himself to all with whom he has ever been associated. Throughout a long life he has preserved always an innate simplicity and lovableness; toward his fellow-man his heart has been always deeply sympathetic, especially toward the younger men. We remember well an incident at an early meeting of the American Chemical Society. Dr. Morley made for us, in answer to a hesitating request, a sketch of a hand blowpipe. Long after the blowpipe had finished its day of usefulness the drawing was preserved as a valued souvenir of the generous response of a great man to the request of a very young chemist.

The Chicago Section is to be congratulated upon this happy occasion and upon the wise decision of its jury of award.

IN THE LIGHT OF EXPERIENCE

President Wilson, in his statement given to the press after signing the selective draft bill, said: "It is not an army we must shape and train for war; it is a nation." In this work of preparation we have just had the aid of the British and French High Commissions. Their visits were made for the avowed purpose of advising us of the mistakes made by their governments at the outbreak of the war. Through the testimony of these officials, both in the private conferences held in Washington and in their public addresses, we have undoubtedly profited greatly. Unfortunately the membership of the Commissions included no chemists who could speak to our people and to the authorities in Washington of early mistakes made as regards the position of their chemists in the plans for modern war. Fortunately abundant evidence exists concerning such errors and their tardy correction. A year passed before England realized that her chemists had rushed to "the front" among the early volunteers. Then began the recall from the spectacular and thrilling scenes of battle to those laboratories and plants, where through their special training they could render greatest service in that critical moment of national peril.

As a nation we would be foolish indeed did we not profit by that experience. If, however, we are so to profit, it is high time for the chemists of the country to make their united voice heard, regardless of any possible misunderstanding of motive in so expressing themselves. We traveled a little last week and learned some things which gave rise to serious concern that already we may be well advanced in that same path of error, the disastrous effects of which have been so clearly pointed out.

Our military forces are to be raised by two methods: voluntary enlistment is now in daily progress; the selective draft system will soon be inaugurated. Under the volunteer system we found in our very limited journey one research in coke oven practice badly crippled by the absence of its leader, a member of the National Guard, now in service. At another place, a research, bearing probably on the solution of the submarine menace, had been discontinued—the student assistant had enlisted. Such men should not be allowed to enlist. Under the prospective draft system we found a commercial laboratory whose staff of well-trained assistants may be completely taken by the draft and yet that laboratory has been most successful in the construction of high explosives plants. Finally, we have watched closely all authorized statements as to probable exemptions under the draft system and we have as yet found no mention of the chemist.

A census of the chemists has been taken and we are informed that more than eleven thousand have replied, giving the information requested. Is this information to be used simply to secure the extra men who may be needed for the special problems which now confront our very much overworked government chemists? Should it not rather constitute in addition an official reserve for the adequate and uninterrupted output of the greater chemical industry which must immediately arise if this nation is to throw all of its resources into this war?

President Wilson has just said: "The business now in hand is undramatic, practical and of scientific definiteness and precision." This thought recalls a visit we had not long ago from two students of Lehigh University, representing their fellow students in chemistry in that institution. Both were former Plattsburgers and filled with eager enthusiasm for active service in the army, but they were also students in chemical engineering. Their question was—how can we best serve our country? Their final decision was to await the call to the colors by our country, meanwhile to organize their fellow students into groups according to raining, so that in case of need they can promptly offer their services to those industrial chemical plants for which they are best qualified, no matter how menial the work may be. Such a decision was essentially undramatic, thoroughly practical, marked by scientific definiteness and shot through with highest patriotism. It will be interesting to watch the subsequent career of these two young men as indicative of how we, as a nation, have met the problem of best utilizing chemists in this war which has so frequently been characterized as a war of chemists and engineers.

In this connection there is another problem which deserves the most serious consideration of our university executives, and that is, the provision of increased facilities for instruction in chemical engineering, facilities both as to men and to means. The uncertainty as to student attendance next year, the absence of many professors and the probability of decreased funds have already led many institutions to formulate plans for curtailment of normal university activities. However strong the convictions which led to such contemplated restrictions, it is sincerely to be hoped that the outworking of the policy will not make itself felt in the chemistry courses, especially in the instruction in chemical engineering. On the contrary, it is sincerely to be hoped that ways may be found to increase rather than decrease this particular feature of the university curriculum.

Here again we may find guidance from British thought and conviction. In the *Journal of the Society of Chemical Industry*, Vol. 30, page 247, it was recently pointed out that "the chemists of this country were and are in the position of an army without reserves." That remark applies also to America. In such a war as we are now entering there will be increased work to be carried out at home by chemists in the peaceful arts and in the munition and directly related factories, while large numbers of chemists will be required in active field service. This country should at once proceed to train an adequate "reserve army" of chemists to meet the shortage that will be heavily felt beginning next spring and lasting until the end of the war, indeed until at least five years after peace is declared.

Whether or no that reserve army is to be supplied depends most largely upon the character of university budgets now in the making. The changes in the curricula of many educational institutions which are now being planned to admit of more intensive military training are fundamentally based upon the same necessity which demands increased chemical training. After nearly three years of bloodiest war, with more than five million men in service, and with the accompanying enormous tax on British resources, Sir Robert Hadfield, the President of the Faraday Society, said on March 6, 1917, during a discussion of the training and work of the chemical engineer: "It is most desirable that increased facilities should be given to training chemists to meet those conditions which will arise after the war."

THE CHEMIST AS A RAILWAY INDUSTRIAL AGENT

Congratulations to Mr. V. V. Kelsey, the recently-appointed industrial agent of the Carolina, Clinchfield and Ohio Railway! And congratulations to that Railway which, by this promotion from the laboratory to the broader executive field, has officially recognized that the chemist can do more in its service than analyze boiler waters and determine whether or no specifications are met in purchases of coal, lubricating oil and paints, however important such functions may be. We hail this event as another distinct step upward in the constantly increasing regard of America for its chemists.

In itself this promotion is the natural outcome of the broad vision of a chemist as to his possibilities of usefulness to the railroad which employed him and to the country traversed by that railroad. In turn he has always received the generous support of officials in the adequate supply of laboratory equipment, services of assistants and freedom in the working out of his plans for industrial development. While the promotion is therefore perfectly logical, nevertheless it carries with it much food for profitable reflection, both on the part of railway officials and of the chemists in their employ.

This business of pioneering is not new to Mr. Kelsey, for he was the first chemist to be put in charge of an exhibit of natural resources, by a railway company, at the National Exposition of Chemical Industries. We beg to extend best wishes for his success in the new position and confidently predict that through his thorough surveys, interpreted by sound chemical common sense, many new industries will arise. From such industries the country will receive needed finished products, manufactured from raw materials which so long have lain dormant.

A REGRETTABLE FAILURE TO COÖPERATE

During the past two years we have continuously pleaded for coöperation as the most effective means for chemical progress. It has been our pleasure to record many forward-looking movements based primarily upon this rational principle. It is, therefore, with keen regret that we now record a failure to inaugurate an important work in which coöperation was the essential factor. We refer to the matter of coöperation on the part of the chemical industries with the U. S. Bureau of Foreign and Domestic Commerce in the compilation of a fully itemized census of chemicals other than dyestuffs during a typical pre-war fiscal year such as 1913–14. The importance of such a census was discussed in the editorial section of the March 1917 issue of THIS JOURNAL.

A committee was appointed by the American Chemical Society to raise $2000 from the chemical industries. This amount was to be added to a similar sum which the Bureau of Foreign and Domestic Commerce agreed to set aside out of its present appropriations. The joint amount, $4000, had been estimated by the Bureau as the cost of the preparation of the census from the Treasury records. The census was to be published by the Bureau and to be available to all at the usual price of such government publications—fifteen cents.

In order to raise the required $2000 from the chemical interests, the committee asked by letter more than one hundred firms to contribute fifty dollars each, believing that sufficient favorable replies would be received to cover fully the amount needed. Any excess was to be returned to the contributors.

The committee's confidence, however, was not justified by the results, as only a little more than half the required amount was raised.

Having failed through this method, it was hoped that the balance might be subscribed by the National Association of Manufacturing Chemists. Its officers replied that as an Association it had no funds which could be used for such a purpose and that they considered this to be a matter for subscription by individual firms.

Loath to give up the effort we again conferred with Dr. Pratt, the Chief of the Bureau, laid the facts before him and urged that in view of the need of such government statistics the work be undertaken solely from government funds. In his courteous reply he pointed out that he would gladly authorize the undertaking if congressional appropriation could be obtained, but that present appropriations did not justify the favoring of the chemical industries with such expenditure of funds, while other industries were freely coöperating financially with the Bureau. The argument was sound, the position reasonable. So ended the effort.

We take pleasure in recording here the list of those who have subscribed to this fund:

The Barrett Company
The Calco Chemical Company
Corning Glass Works
The Dow Chemical Company
General Chemical Company
Globe Soap Company
Heller & Merz Company
Alcan Hirsch
Hooker Electrochemical Company
The Institute of Industrial Research
Eli Lilly & Company
Madero Brothers
Mallinckrodt Chemical Works
Merck & Company
National Aniline & Chemical Company
National Gum & Mica Company
The New Process Metals Company
Pfaudler Company
Chas. Pfizer & Company
Smith, Kline & French Co.
Solvay Process Company
Arthur H. Thomas Company

However deeply we are convinced of the fundamental need of such a census of imports and however important this step might prove as a preliminary to more adequate current statistics for the chemical industries, we do not feel that the raising of this $2000 is a matter of a house-to-house canvass.

If, therefore, no further interest is developed in the matter, the committee will on June 15th return the subscriptions already made, which subscriptions were originally asked on the condition that the full amount be secured. We still have hopes!

PROSPECTIVE ENLARGEMENT OF CHEMISTS' CLUB BUILDING

The growth of the membership of the Chemists' Club, now numbering nearly fifteen hundred, and the large proportion of non-resident members, give to the record of the annual meeting of that organization on May 2, 1917, national rather than mere local interest.

The reports of its officers showed strength in all the departments of this unique organization. Under the wise and effective leadership of President M. C. Whitaker, the finances have been placed upon a sound basis, and his unanimous re-election for the ensuing year assures the continued growth of the Club and the safe administration of its affairs.

The most striking item in the report of the House Committee was the over-demand for bedrooms in the Club building. This feature of the Club, regarding the wisdom of which there was at first so much skepticism, has proved to be a success far beyond the highest expectations. For the visiting chemist, the isolation of New York Hotel existence has been changed to the more congenial atmosphere of club life. Indeed, so successful has this feature proved that it is seldom there is a vacant room, and many applications for rooms have to be denied.

To meet this condition, the trustees of the Club have, upon the recommendation of the members present at the annual meeting, promptly taken steps to secure by lease or purchase the four-story building adjoining on the west side. The two buildings could easily be thrown into one, and, by slight rearrangement of the annex, twenty-seven extra bedrooms could be provided. It is believed that the negotiations now in progress will be successfully terminated at an early date.

AN EMBARRASSMENT OF RICHES

The opening sentence of the last annual report of Secretary Parsons was as follows: "The growth of the American Chemical Society during the year 1916 has broken all precedents." In support of this statement it was shown that the membership had increased during the year from 7417 to 8355. All who have followed closely the growth of the Society felt that this increase probably would mark the high point in the curve of numbers of members, that 1916 would always be characterized as the "Banner Year" in the Society's growth.

On May 11th we were informed by the Secretary that the Society's membership had increased to 9325, exceeding even at that early date the increase for the entire previous year. What the final showing for 1917 will be is a purely speculative, but highly entertaining and enthusing matter. As indicative, however, of the outcome it is extremely interesting to learn that the increase during the past month has been twice as great as during the corresponding period in 1916.

One result of this expansion is the complete upsetting of all estimates of the number of copies of each of the Society's publications which would be necessary to meet current demands. The necessity of such estimates will be readily recognized, especially at this time, in view of the increased cost of materials, labor, etc. Secretary Parsons now finds that it is impossible to supply new members with the early issues of this year, and writes us as follows:

"The half-year membership will begin for members elected after June 1, 1917; that is, dues will be five dollars and the journals will be furnished from July 1, 1917. Of course, all members elected on this basis will be expected to continue as regular members and pay ten dollars covering 1918 dues upon receipt of statement."

In connection with the question of new members we wish that some Local Section would attempt the following simple experiment: get each member to agree to carry a membership application blank in his pocket for one month, provided he has not prior to the expiration of that month forwarded it to the Secretary filled out with the requisite data for action by the Membership Committee. We are confident that the psychological effect of the constant presence of that paper would result in stimulating initiative in getting new members where in many cases it had not previously existed. Such initiative is all that is needed to increase the membership far beyond the loyal hopes and expectations of even the most optimistic.

AN ADVANCE IN ADVERTISING RATES

At the Urbana Meeting, April, 1916, the Directors passed a resolution urging the Advertising Committee to advance the advertising rates as soon as practicable. In view of impending changes in the management of THIS JOURNAL the Committee felt that it would not be wise to make such advances until the new organization was completed and familiar with the details of the work.

At a meeting held on May 7, 1917, the Committee decided that an advance in rates should at once become effective and fixed the amount of the advance at a forty per cent increase over existing rates, providing, of course, that existing contracts should be filled as per contract.

The justification of this advance lay in the marked increase in the circulation of the journals incident to the rapid growth of the membership of the Society; its necessity was made evident by the cost sheets exhibited by the Advertising Manager.

Members of the Society can render valuable aid at this time by using their persuasive powers in holding all former advertisers to their appreciated connection with the Society's publications and by urging the establishment of such connections by those firms which at present do not utilize this valuable medium of publicity.

ANNOUNCEMENT

Beginning with the July issue we shall publish each month a Washington letter. Mr. Paul Wooton, Metropolitan Bank Building, Washington, D. C., has been engaged as our special correspondent. Mr. Wooton is an experienced journalist in thorough touch with matters legislative and scientific. Even under normal conditions, Washington news is of great interest to American chemists; and especially is this true at the present time when so much activity is centered there and when so many matters of deep concern to chemists are in process of development. Too often these topics are completely overlooked by the daily press correspondents. It is hoped through this monthly letter to furnish our readers with an accurate account of what is taking place in the National Capital of interest to chemists.

THE CANADIAN CHEMICAL JOURNAL

As indicative of the highly stimulated condition of the Canadian chemical industries incident to the war period, we have received the initial number of *The Canadian Chemical Journal*, published at Toronto, Canada, under the same management which founded *The Canadian Engineer*, *The Canadian Textile Journal*, *The Pulp and Paper Magazine*, *The Canadian Woodworker* and other technical publications.

The new journal, to be issued monthly, is "devoted to the chemical and metallurgical interests of Canada." The present issue contains interesting articles on Canadian water powers, the nickel industry of the Sudbury region, potash from Canadian feldspar, chemistry in Canadian woods, etc., and many up-to-date industrial notes. Confidence in the future of the Canadian industry is shown in the following excerpt from one of the leading articles: "Of course the present abnormally high prices of almost all kinds of chemicals make this an attractive field of investment, but it may fairly be assumed that the natural resources of Canada are such that the Canadian chemical industries will stand the readjustment of peace conditions and that in many of the more important branches they will become permanent. This assumption is based mainly on three factors: First, the enormous water powers of Canada, as yet undeveloped, which are becoming more and more the deciding element in the cheap production of many chemicals—such, for example, as atmospheric nitrogen; second, the mineral deposits of Canada whose extent and variety are as yet scarcely comprehended by our own people; and third, the intelligence and energy of the younger generation of Canadian students, who will naturally turn their talents to the possibilities opened up under the new conditions."

We desire to extend best wishes to the new journa and to predict for it a career of marked usefulness in the industrial chemical life of our neighbors across the almost imperceptible northern border-line.

CHEMICAL INDUSTRY IN CANADA

Address delivered by H. E. Howe at the 54th Meeting of the American Chemical Society, Kansas City, April 12, 1917

The force with which the importance of chemical industry has been brought home to the world since August 1914 has led to a stock-taking in various countries, and a review of the situation at present, followed by prophecies for the future. The extent to which our complex civilization depends upon chemical products and chemical processes is greater now than at any time in the world's history, and the accomplishments of chemical industry are in many cases so like magic that the popular press seizes upon the spectacular items and frequently overlooks extensive developments in localities which to the ordinary mind seem to lie outside the chemically active countries. We have had German chemists, German research and German efficiency thrust upon us until one can scarcely mention the subject of chemistry without seeming to hear "Die Wacht am Rhein" or "Deutschland über Alles." But Switzerland before the war was producing more dyestuffs per capita than was Germany, notwithstanding the fact that she had to import her raw materials, including coal itself. The world can learn much about advertising from Germany.

Since the war we have heard more of chemical industries in other countries, and steps have been taken to organize thoroughly and bring into a state of coöperation the many successful chemical laboratories, institutions, and individual chemists so as to place the British Empire and the United States upon a firm chemical foundation.

The work confronting chemical industry and chemical engineers in the production of enormous quantities of munitions is almost beyond our imagination. It may be measured in a way by the munitions production record of England. At the beginning of 1917 the production of high explosives was sixty-two times what it was in 1915. British munition factories are now making more heavy gun ammunition every 24 hours than they manufactured during the entire first year of the war. The monthly output of heavy guns is more than six times what it was during the year 1915. The output of bombs increased thirty-threefold during the single year May 1915 to May 1916. Fourteen times the quantity of machine guns made during the period of August 1914 to August 1915 were made during the year 1915 to August 1916. The daily output of the twelve national projectile factories, which have as yet not developed their total capacity, would fill a train one mile long requiring eight engines to pull it. Where production is speeded up to this extent there are sufficient chemical and chemical engineering problems to tax the resources of a nation, not to mention the important work which England is doing on dyes, optical glass, refractory materials, porcelain, metallurgy, etc.

In France the organization is such that the chemical plants make only what is required for war and of a quantity and quality according to the specifications of the military departments.

You are so well acquainted with the active steps taken in America since 1914 that it is unnecessary for me to emphasize the importance of the work accomplished or under way, but in considering the position of the North American Continent, what attention has been paid to the achievements and potentialities of Canada, our neighbor to the north? Let me tell you something of what is being done there and suggest the possibilities in a brief account of the natural resources.

CHEMICAL PRODUCTION

In one locality, destined to become an electrochemical center of some magnitude, there will ultimately be available approximately 500,000 24-hour horse power. Here acetone is being made by a new process and the production of metallic magnesium in quantity is going forward, the metal being supplied in bars and also in the variety of powders so necessary on the battlefields to-day for star shells. Aluminum, and carbon electrodes are also made, and other chemical products are now under serious consideration as additions to the chemical chain which has been started.

Explosives are, of course, being produced on a large scale and they are of high grade. Trinitrotoluol is produced at one plant by a process involving crystallization from a medium which results in crystals of great uniformity and unusual purity.

Heavy chemicals are produced in sufficient quantities, the plants being located principally in the Province of Ontario. Sufficient potassium chlorate to meet the requirements, together with a considerable supply of phosphorus are produced, utilizing hydroelectric power. There is the usual by-product recovery from coking plants, including ammonia in its various forms. The electric furnace and electrolytic industries are established in Canada, producing bleach, caustic, cyanamide, abrasives and carbides. Ferro-molybdenum is manufactured in the electric furnace, and in many localities the electric furnace has been brought into play in the production of steels.

Steps have been taken, and a large plant is now under construction, for the electrolytic refining of nickel matte produced in Ontario, where we find nickel deposits valued at $500,000,000, and supplying over two-thirds the world's requirements for nickel.

PHARMACEUTICAL PRODUCTS AND DYES

In pharmaceutical chemistry a large number of the finer preparations are manufactured in Canada. Phenacetin has been produced since the war started, as has also acetyl salicylic acid, otherwise known as aspirin. There is one plant with a capacity of over a ton a day making acetyl salicylic acid, which is possibly due to the fact that the patent situation is not in the same condition as in the United States. Para-amidophenol, so essential in fur dyeing, is being produced. Bismuth salts, iodine preparations, resublimed iodine, iodides and iodoform, are being made in quantities sufficient for domestic use as well as for export. Hydrogen peroxide by the barium process is manufactured in quantity, and many sodium and potassium salts are purified and recrystallized. Alkaloids are prepared, and salvarsan of a grade equal to the best German product is now being prepared commercially.

Aniline, phenol and salicylic acid may be added to the list.

A few dyes are now being made in Canada, but the economic conditions are such as to make it improbable that any extension of the work can be carried out.

WOOD PRODUCTS

Hardwood distillation was carried on in Canada previous to the war, but since its beginning new articles have been prepared and refinement of crude materials carried to a greater extent.

The very important pulp industry must be mentioned in passing. At the present rate of expansion and development Canada bids fair to lead in the world's production of pulp in the very near future, if she does not do so already.

ORGANIZATIONS FOR RESEARCH

So much for the material accomplishments which show that considerable is being done. The future would seem to offer much. An Advisory Council on Scientific and Industrial Research has been appointed by the Government, and this Council is about to make a complete inventory of the research men and their facilities throughout the Dominion, of the problems which have been solved, of those upon which work is now being done and of those which merit immediate attention. Immediate steps are being taken to assist researchers in the completion of their education and to provide studentships, scholarships and fellowships to encourage the prosecution of scientific research at the educational institutions. The Council, modeled after the Commission in Great Britain, will be generally concerned with research work in Canada and will consider many problems of national importance.

The Forest Products Laboratory is carrying on work similar to that of the Forest Products Laboratory of the United States and has under way work on other problems which are peculiar to Canadian conditions.

The chemists of industrial laboratories are carefully studying the problems pertaining to their particular industry and are endeavoring to plan their work to meet conditions after the war as well as at the present time.

Under the auspices of the Canadian Pacific Railway, Arthur D. Little, Limited, are not only conducting research upon problems, the solution of which would mean much for the Dominion at large, but are also conducting a survey of the natural resources in order that data regarding them may be made quickly available for practical application and for those interested in industrial development work. So far as I am aware the plan of the Canadian Pacific Railway, as put into operation by Lord Shaughnessy, the President and Chairman, is without precedent and indicates, on the part of a public service corporation, a fundamental interest in the country at large, which is not ordinarily ascribed to such an organization.

PLANT UTILIZATION AFTER THE WAR

The question of utilizing plants, which have been erected primarily for war purposes, when peace comes, is receiving the attention of managers and engineers. Some of them strongly advocate the production of nitrates from the air, especially in those locations where plants own their own hydroelectric development works.

CANADIAN RESOURCES

Let us now consider for a moment some of the resources of Canada, which may conceivably form the basis for industries in which chemists and chemical engineers will find their tasks awaiting them.

AGRICULTURE—Agriculture is the greatest natural resource of Canada, and while many chemists do not think of agriculture as a natural resource in which they can be interested, it is well known that the products of agriculture frequently claim the chemist's attention. When it is recalled that most of Canada's arable land lies at a great distance from the present centers of population, it is evident that many problems will present themselves relative to preparing the produce from mixed farming, in order that it may reach the markets in a condition to command a good price. Methods for drying to avoid high freight costs enter in, as well as the establishment of suitable plants in the vicinity of growth to work upon products of agriculture as raw materials. The large corporation farms of the prairies may quite conceivably come to utilize the services which a chemist can render in many departments, from the proper treatment of seed grain and the maintenance of soil fertility and proper rations for stock, to the utilization of the products themselves. The products of agriculture raised in 1913, which is taken as a year not influenced by war prices, were worth nearly $700,000,000. The production of the Prairie Provinces has increased nearly 600 per cent in the last ten years.

FORESTS—The forests of Canada, while not as extensive as is generally supposed, already form the basis of a great chemical industry in the manufacture of pulp and paper. For the year 1916, nearly $22,000,000 worth of paper was exported, of which 88 per cent came to the United States. The industry is growing at an enormous rate, the daily production of sulfite pulp approaching 1500 tons daily and that of paper exceeding 2000 tons daily. There is considerable activity in hardwood distillation, and the Forest Products Laboratory is now carrying on softwood distillation experiments hoping to produce oils suitable for use in the oil flotation processes. The west contains some wonderful primitive forests, the Island of Vancouver being a notable example. Here Douglas fir 8 feet in diameter and 300 feet high are to be found, 70 trees to the acre, setting with shallow roots on beds of clay in which glacial boulders are scattered. Timber has been so plentiful there that naturally its values were not appreciated a few years ago, for, in 1876, a lumbering concern refused to pay $12,500 for 30,000 acres of timber which is still standing and which is now estimated to cut approximately $300,000,000 worth of timber. There is one timber limit in British Columbia of 72,000 acres of spruce averaging 18 inches in diameter from which about 4,000,000,000 board feet of lumber may be cut, not to mention the pulpwood available.

Reforestation is already receiving careful attention, although the foresters are somewhat divided as to the proper methods. Some believe in planting trees, while others contend that if nature be properly assisted and the young seedlings protected better results are obtained. As in the United States, active measures to prevent and subdue forest fires are receiving consideration. In the Province of Quebec a fire must not be started, excepting for cooking or for warmth, without a government permit, it having been determined that most fires start from bush fires set by settlers when clearing the land. However, 1200 permits were issued last year, and none of the fires got beyond control.

MINERALS—The minerals of Canada already yield an annual income approaching $200,000,000, and yet vast areas have not been prospected. Many of the deposits are inaccessible at present. There is said to be one deposit of gold-bearing gravel in British Columbia containing 3,000,000,000 cubic yards running 10 cents per yard. Dr. O'Neill, the geologist of the Stefanson expedition, reports an ore body of 6,000,000,000 tons bearing copper distributed through the mass, in a quantity, however, which is not commercially attractive, but the indications are that more careful prospecting will disclose concentrations that may make it feasible in future to commence mining operations on the shores of the Arctic Ocean.

The wonderful mineral resources of Northern Ontario are just being uncovered, while in the vicinity of Cobalt another gold mining camp is already establishing itself.

The nickel deposits in the vicinity of Sudbury, Ontario, are also a part of the great Pre-Cambrian Shield which conforms to Hudson Bay and which continues into practically unexplored portions of the extensive Province of Quebec.

Eighty per cent of the world's supply of asbestos is mined in the Province of Quebec.

Canadian apatite cannot be mined by steam shovel as are the phosphate deposits in Florida, and consequently at present it is not a commercial factor but may be looked upon as a reserve supply of this important substance. Some of the material runs from 83 to 87 per cent phosphate, but it is enclosed in a hard Laurentian rock: that now used for phosphorus compounds of a chemical grade occurs with the mica and may be considered a by-product of this industry.

There are two varieties of soda rock which are interesting as being nearly identical with the German phonolith extensively used in the manufacture of Apollinaris water bottles. It is high in soda, contains very little iron, and approaches a natural fused glass in its composition.

Canadian magnesite has already come to the aid of steel manufacturers in providing a satisfactory refractory and is the source of much of the magnesium now made in the United States as well as all of that made in Canada. I need not emphasize the importance of molybdenum to you, but may say that ferro-molybdenum is being produced from Canadian molybdenum.

A great variety of minerals are to be found in the Dominion, and in the recovery of some of them the chemist and metallurgist, as well as the electrometallurgist, will find much to interest and perplex them.

MISCELLANEOUS—Other resources include fisheries which reach a very large annual business and which may soon become so organized that the chemist will find the problem of waste disposition confronting him. It is interesting to note in passing that thousands of soldiers at the front receive rations of Canadian fish twice a week at the present time, and that in order to encourage the fish industry the Government stands prepared to defray a portion of the expense for transporting fish to the interior of Canada from both the Atlantic and Pacific coasts.

The fur-bearing animals yield an export trade of nearly $6,000,000, besides furnishing furs to a large domestic market.

WATER POWER

As of especial interest to you, I would emphasize the water powers of Canada, which with other resources may be expected to form the basis of electrochemical and electrometallurgical industries when the population has become sufficiently concentrated and the industries utilizing these products of greater size than at present. The water powers of Canada are more abundant than in any other country. Already nearly a thousand waterfalls are known to be suitable for the generation of electric power, and every expedition into the wilds brings back reports of additional powers which may be developed when the demand comes: 1,712,193 24-hour horse power has been developed, and of the 18,000,000 24-hour horse power which is in sight 8,000,000 is within reach of present markets. In the central portion of Canada 2,000,000 sq. mi. of territory lies at an average elevation of 1500 ft. This area drains into rivers emptying into the St. Lawrence or the sea and affords water power of dimensions difficult to compute. When the potential horse power is considered with respect to area or population, Canada is as favorably situated as other nations, and when horse power per square mile is considered it is well up in the list with other large countries. When it is remembered that 4 lbs. of coal are required on an average to develop one horse power hour under favorable conditions, the amount of coal which would have to be burned to give power equivalent to the Canadian water powers is quite beyond our comprehension. The price at which hydroelectric power is sold varies with the amount of power taken and is ordinarily based on power at the power-house, leaving the cost of transmission to the consumer. Contracts now in force run from some old ones, about to expire, at $4.50 per horse power year to $20.00 or more. There are water powers which can be developed at a price per horse power which may make it possible to produce nitrates from the atmosphere in competition with the low prices in Norway where processes have found a home due to cheap power. You may remember that in 1912 Dr. Eyde stated that when the cost of power in Norway reached $6.00 per horse power year they would be unable successfully to compete with other sources of nitrates.

Transmission would seem to be one of the important problems in connection with hydroelectric power in order to make this energy available in places where economic conditions make it possible to establish electrochemical industries. At present Niagara power is being transmitted 242 miles to Windsor, Ontario. This is the world's record and such a radius enables power to be used over an area of 180,000 sq. mi. We may confidently rely upon the future to provide us with still greater improvements in power transmission.

FUEL

Fuel is always of paramount importance where any sort of industry is under consideration. At present the domestic fuels in use are principally the coals of eastern and far western Canada, including the anthracite in the vicinity of Bank Head, Alta. The tremendous tonnage of lignite which exists in Alberta, Saskatchewan, and Manitoba has been limited in its use, due to its slacking during drying, but work is now under way which gives promise of making this lignite a satisfactory fuel for power purposes and the prairie provinces thereby more attractive for fields of industry. Research work looking toward the utilization of cereal straws for fuels in certain areas is also under way with considerable promise of ultimate success.

There is also a great quantity of oil shale in New Brunswick and smaller deposits in the western provinces which may be considered reserve supplies of oil.

NEED FOR COÖPERATION OF UNITED STATES WITH CANADA

In closing, I wish to point out the need, the desirability and the advantage of coöperation between Canada and certain forces in the United States. Until Canada can train men to take their places with those now engaged in chemical industries, it is desirable that specialists from the United States seriously consider Canada as a place for their endeavor, in connection with American capital, which will find a favorable field for employment. There is every indication that there will be a preferential duty in favor of Canada of at least 25 per cent so far as the rest of the British Empire is concerned. Therefore, if Americans wish to benefit from this trade arrangement they must manufacture in Canada. The ten years ending with 1913 saw carried out in Canada an industrial program, probably unapproached in other countries of the world, involving an investment of more than $1,300,000,000 of European capital, for we must remember that, so far as construction is concerned, Canada may be compared with the United States at the close of the Civil War. It is obvious that British, Belgian, Dutch and French capital which formerly found investment in America will not come again for a period of years, while it will be the privilege of the United States to finance new industries the world over. And if America is called upon to aid Canada with specialists and with money she will be doing no more than the "old lion" has done for her, since during the last century nearly $5,000,000,000 has been loaned to industrial America, principally by Great Britain. Canada will welcome American capital, and her natural resources awaiting economic development offer an attractive and legitimate field for enterprise.

To those of us who are endeavoring to study Canada it would seem that opportunity not only knocks at the door of industrial and financial America but stands with her finger pressed against the doorbell. Will we hear the ring before the bell batteries run down, or will we, like a mining engineer who visited Cobalt to report on a property which proved of no value, travel over a silver sidewalk and not realize it?

A. D. Little, Ltd.
137 McGill Street
Montreal, Canada

ORIGINAL PAPERS

FACTORS CAUSING VARIATION IN THE YIELD OF CAMPHOR IN THE FLORIDA CAMPHOR TREE

By Samuel C. Hood

Received April 10, 1917

The recent increase in the price of camphor gum has renewed the interest in the commercial cultivation of the camphor tree in Florida, and has stimulated the laying down of new plantations as well as the increase of those already under way. Since the methods of production in Florida are widely different from those in Japan and Formosa, and are commercially possible only by the application of special equipment on large areas, it is important that full information be available on the several factors which cause variations in the yield of the gum in the leaves and twigs of the tree.

The present paper is based on observations made on camphor trees growing under various conditions in Florida, during the years 1907 to 1912, at which time the writer was a member of the scientific staff of the Bureau of Plant Industry, United States Department of Agriculture. Samples were taken from nearly 2000 trees over a range of latitude of about 250 miles. This paper presents only the more important results having a direct bearing in the methods of commercial production.

METHODS

The production of camphor in Florida depends upon the planting of the trees in hedges and utilizing either the trimmings, consisting of leaves and small twigs, or by severely pruning the trees each year and distilling the resulting branches and leaves. Such trimming or pruning is done during one of the dormant periods, *i. e.*, during May to June or November to February.

SAMPLES—All tests made during this investigation, unless otherwise stated, were on leaves and twigs of the previous growth taken during the dormant season. Percentages of crude camphor were calculated on the green weight of the material taken at the time of collection.

DISTILLATIONS were made in Remington stills of 5 gal. capacity using about 2 kilos of material in each sample. It was found by repeated tests that with leaves and small twigs of the last growth, no cutting or grinding was needed. In the case of larger wood, the material was cut into small chips crosswise of the grain with a hatchet or power chipping machine constructed for this purpose. The stills were equipped with false bottoms to prevent burning of the charge; about 2 qts. of water were added with each charge, and distillation was carried on over a gas flame. Comparative tests with steam and direct heat distillation showed that better results were secured with the method described, which permitted the more complete recovery of the camphor dissolved in the water.

CONDENSER—The camphor was condensed in a 1-meter Liebig condenser, and was removed to the receiver with the aid of a slender wooden rod and a piece of closely woven cloth moistened with water. It was usually necessary to clean out the condenser once during the distillation, otherwise so much residue was collected as to make its final removal difficult. The distillate was received in a 200-cc. beaker fitted with a double siphon tube, and the water was returned to the still from time to time.

CRUDE CAMPHOR—After the distillation was complete, and all camphor removed to the receiver, the pasty mass of camphor and oil was moved about with a spatula to gather up separated oil globules and made up into a mass. This was transferred to a folded square of closely woven cotton, moistened with water and placed over the container of a small meat-extract press. The cloth was folded over the mass and the water, with part of the oil, pressed out into the small beaker used for weighing. By means of a capillary pointed pipette the water was drawn out from beneath the oil and the cake of camphor added to it. This mixture of oil and camphor was weighed as crude camphor.

PURE CAMPHOR in the crude was determined as follows: the crude camphor was stored in well-stoppered bottles for at least two weeks until it had recrystallized, forming large crystals about the size of sugar grains. The mass was well mixed with a spatula and about 5 g. weighed into the tubes used for oil extraction.

Spun brass cups about $^3/_4$ in. in diameter and $2^1/_2$ in. long were fitted with pressed bags of 100-mesh brass gauze projecting into the cup about one inch. The top of the bag was then trimmed even with the top of the cup and soldered to a $^1/_2$-in. band of the same material as the cup. The solder was drawn into a ring forming a shoulder resting on the top of the cup.

The cup and bag were each weighed, and the sample of crude camphor weighed into the bag. The cup was then placed in the pocket of a Babcock milk centrifuge and run at full speed for 5 min. By this process the oil was thrown out into the cup, and from the weight of the cup and oil, the weight of the oil removed could be determined.

CORRECTION—It was found that the amount of camphor dissolved in the oil was nearly uniform for each kind of material distilled. This correction was determined by fractional distillation of considerable samples of crude oil, and the camphor removed from the fraction coming over at 195 to 215° C. by repeated freezing.

DISTRIBUTION OF CAMPHOR IN THE TREE

In order to gain some information on the distribution of camphor in the various parts of the tree, and to determine which portion would provide the most valuable commercial material with the minimum handling costs, distillations were made of the various parts of growing trees 8 to 12 years old.

Leaves and twigs of the two previous growths were collected in May from tree W5. This material consisted of leaves and twigs of the spring growth just matured, and leaves and twigs of the previous summer growth. Of this entire year's growth, 70 per cent was leaves of the spring growth. The percentages of crude camphor and per cent of pure gum in the crude are given in Table 1.

TABLE 1—RELATIVE PERCENTAGE OF CAMPHOR IN LEAVES AND TWIGS

Material	Per cent Crude Camphor	Per cent Purity	Per cent Pure Camphor
LEAVES: Spring	1.72	76	1.31
Summer	1.46	78	1.14
TWIGS: Spring	2.14	70	1.50
Summer	1.22	73	0.89

These results are typical of a large number of similar tests made on many trees over a period of several years. In all cases the camphor content was found to diminish if allowed to remain on the trees through another growth season. The percentage of purity was, however, higher in the older leaves. This was probably due to the evaporation of the more volatile terpenes of the camphor oil.

In March 1908, two 13-year-old trees were cut to the ground and the material sorted into leaves, twigs and wood of various ages. The results of the determination of camphor in these materials are presented in Table 2.

TABLE 2—DISTRIBUTION OF CAMPHOR IN 13-YR.-OLD TREES
Results in Percentages

	TREE B_1		TREE B_2		PURE CAMPHOR	
	Crude Camphor	Purity of Crude	Crude Camphor	Purity of Crude	Tree B_1	Tree B_2
LEAVES	1.12	78	1.17	75	0.87	0.88
TWIGS:						
Last growth	0.55	88	0.59	81	0.48	0.48
1-yr. old	0.36	76	0.53	74	0.28	0.39
BRANCHES:						
3/4 to 1 1/2 in.	0.53	73	0.52	74	0.39	0.38
4 in.	0.83	70	0.53	70	0.58	0.37
7 in.	..	..	0.92	65	..	0.60
WOOD: 8-yr. branch						
Outer 4 rings	1.26	59	1.87	71	0.74	1.33
Inner 4 rings	1.03	51	1.21	68	0.53	0.82
BARK:						
Branches	0.56	90	..	..	0.50	..
Trunk	0.11	67	..	..	0.07	..

It will be noted that there is a gradually diminishing percentage of camphor from the youngest mature leaves to the wood about 5 years old. After this age there begins a redeposit of camphor in the older wood and this increases as the wood grows older. The results from the 8-year-old wood show a considerable increase, more being present in the outer rings than in the inner.

It has been found by the writer that this increase continues until in 15-year-old wood as much as 3 per cent has been found. This increase in percentage of camphor in the wood is associated with a greatly diminished percentage of purity, often in old wood falling to 50 per cent, and the resulting product of distillation being liquid, from which camphor crystals separate only on long standing.

The camphor oil from the wood is an entirely different product from that derived from the leaves, and contains considerable quantities of safrol, while leaf oil is nearly or quite free from this compound, but contains more of the terpene constituents.

EFFECT OF SHADE

It has repeatedly been shown in the case of many essential oil plants, that partial shade tends to retard the formation of oil in the plant. In order to determine if this would hold for camphor, tests were made on a number of groups of trees of uniform age and history, where one or more of the number were shaded by buildings or other large trees. The results are given in Table 3 as averages of the total determinations made on each group of trees.

TABLE 3—EFFECT OF SHADE ON YIELD OF CAMPHOR
(Results given are averages)

		IN OPEN		PARTIAL SHADE		PURE CAMPHOR	
TREES Group	No.	Crude Camphor	Purity of Crude	Crude Camphor	Purity of Crude	Open	Shade
L.	3	1.95	74	1.39	75	1.44	1.04
C. H.	7	2.18	73	1.84	71	1.59	1.31
T. H.	2	1.83	73	1.24	77	1.34	0.95
D.	2	1.28	76	1.12	78	0.97	0.87
C. (Hedge)	3	2.04	70	1.95	70	1.43	1.37
Nursery(*a*)		1.26	..	0.74(*b*)	..	..	..

(*a*) Leaves of 1-year nursery trees. (*b*) Grown under half shade.

Since they showed to a marked degree the effect of only slight shade, it was thought of interest to determine if there existed a difference between the North and South sides of the trees, since this might have an effect on the direction in which commercial hedges were laid out. The results of a number of such tests are given in Table 4.

TABLE 4—CAMPHOR PERCENTAGES IN NORTH AND SOUTH SIDES OF TREES

	SOUTH		NORTH		PURE CAMPHOR	
TREE No.	Crude Camphor	Purity of Crude	Crude Camphor	Purity of Crude	South	North
W9	1.41	77	1.41	77	1.06	1.09
W30	1.68	71	1.70	72	1.19	1.22
W45	1.00	80	1.00	75	0.80	0.75
W159	1.61	82	1.62	78	1.32	1.26
W160	1.83	72	1.77	73	1.32	1.29
AVERAGE	1.51	72	1.50	75	1.13	1.12

These data indicated no appreciable difference in camphor content between the North and South sides of the trees, and lead to the conclusion that commercial hedges may be laid out in either direction without influence on the yield of camphor.

YEARLY VARIATION IN CAMPHOR YIELD

It was to be expected that a certain variation in camphor yield would be found from year to year in the same trees, and in order to determine the limits of this variation, it was intended to run a series of tests over a number of years. Press of other duties, however, made it impossible to continue these after the second year, but the results of these two seasons (Table 5) show that a considerable variation may be expected. The tests each year were made during the hot, dry period which nearly always occurs during May, so that the weather conditions were the same each year.

TABLE 5—YEARLY VARIATION IN CRUDE CAMPHOR YIELD (PERCENTAGES)

TREE No.	May 8, 1908	May 25, 1909
H852	1.38	1.47
H842	1.57	1.34
H849	1.63	1.38
H845	1.47	1.42
H847	1.68	1.42
H846	1.41	1.47
H848	1.57	1.48
H844	1.45	1.40
H851	1.88	1.78
H843	1.48	1.30
H850	1.85	1.83
AVERAGE	1.57	1.48

The writer has found that the summer growth is subject to much greater variation than the spring growth, since as a rule the trees do not start the summer growth until the beginning of the summer rains, and these are often much delayed. While the spring growth is uniform over the entire tree, the summer growth is inclined to start first at the top and may develop in a very irregular manner over the tree so that new shoots are continually coming out during the entire summer. It has therefore been found advisable to use only the spring growth for tree-testing purposes.

EFFECT OF PRUNING AND TRIMMING

It has often been recommended that the harvest of commercial plantations be done by pruning the trees severely each year, and at the present time such a method is being used in one instance. Experiments conducted by the writer in 1907 show that this method necessitates the handling of large amounts of wood material of very low camphor yield, and also that even the percentage of camphor in the leaves is reduced in growth of this sort. Tree L1, which was about 10 years old, was cut to the ground in March 1907 and the following May the mature new growth from the stump, consisting of leafy shoots 3 to 5 ft. long, were collected and sorted into several types of material. Tree W225 was very severely headed back in February 1907 and the following May the new growth of a watersprout type was collected and sorted in the same manner as in the case of tree L1. The percentages of camphor in the material from these trees are given in Table 6.

TABLE 6—CAMPHOR PERCENTAGES IN WATERSPROUT GROWTH

	TREE L1		TREE W225		PURE CAMPHOR	
	Crude Camphor	Purity of Crude	Crude Camphor	Purity of Crude	L1	W225
LEAVES	1.15	75	1.89	74	0.86	1.40
TWIGS: 1/4 in.	0.49	85	1.15	80	0.47	0.93
1/4 to 1 in.	0.06	86	0.31	86	0.05	0.27

In the total growth harvested, the leaves made up about 65 per cent of the weight, and gave a much higher camphor yield than did the twigs and woody material.

Further data on this point were secured from the distillation of material grown in Texas, and consisting of 2-year-old nursery trees. These trees had been cut to the ground at one year old so that the material received consisted of the growth of one year of two growing seasons. The material consisted of 10 entire trees and was sorted into the leaves and twigs of each growing season: 65 per cent of the total was leaves and 53 per cent was leaves of the spring growing season. The tests were made after the spring growth had become dormant. The camphor yields found are presented in Table 7.

TABLE 7—CAMPHOR YIELD (PERCENTAGES) OF TWO-YEAR-OLD TEXAS TREES

Material		Crude Camphor	Purity of Crude	Pure Camphor
LEAVES:	Spring	1.13	76	0.86
	Summer	1.10	86	0.95
TWIGS:	Spring	0.69	81	0.56
	Summer	0.16	81	0.13

In order to gain still further data on the effect of severe pruning on the camphor content of the succeeding growth, one side of each of two trees was severely pruned in January and the following May, after the spring growth was fully dormant, leaves and twigs were collected from the watersprout growth on the pruned sides and also from the normal growth on the unpruned sides of the tree. The results are given in Table 8.

TABLE 8—CAMPHOR YIELD (PERCENTAGES) OF WATERSPROUT AND NORMAL SPRING GROWTH

		NORMAL GROWTH		WATERSPROUT		PURE CAMPHOR	
Tree	Material	Crude Camphor	Purity of Crude	Crude Camphor	Purity of Crude	Normal	Watersprout
W8	Leaves	2.13	76	2.03	71	1.62	1.44
	Twigs	1.60	79	1.69	80	1.26	1.35
W152	Leaves	1.60	73	1.40	71	1.17	0.99
	Twigs	1.40	80	1.18	72	1.12	0.85

These results show that the percentage of camphor is less in the growth of a watersprout nature induced by severe pruning, than in the normal growth of the tree. This is important in connection with commercial operations, since any harvesting method used should supply crude material of a maximum percentage of camphor, and should reduce to a minimum the amount of low-yielding wood material to be handled.

It was found in the earlier hedge trimming experiments that the first cuttings tended to stimulate a heavier growth during the succeeding growing season. In order to determine if this also varied from the normal, the following experiments were carried on with 10-year-old trees which had never been trimmed or pruned. One side of the trees was trimmed with hedge shears to remove the leaves and twigs of the last growth, while the other side was left untrimmed. This trimming was done in January and the following May after the spring growth had become dormant, the leaves and twigs of the spring growth were removed at the last growth scar on both the trimmed and untrimmed sides of the trees. The camphor yields of the different collections are presented in Table 9.

TABLE 9—EFFECT OF TRIMMING ON CAMPHOR YIELD

TREE No.	UNTRIMMED SIDE		TRIMMED SIDE		PURE CAMPHOR	
	Crude Camphor	Purity of Crude	Crude Camphor	Purity of Crude	Normal	Trimmed
H852	1.38	72	1.90	71	0.99	1.35
S1	1.49	80	1.83	75	1.19	1.37
S2	1.67	81	1.81	80	1.35	1.45
S3	1.60	76	1.63	74	1.22	1.21
S4	1.66	81	1.94	76	1.34	1.47
AVERAGE	1.56	78	1.82	75	1.22	1.37

These results show that the trimming had caused an increase in the camphor content of the succeeding growth of 14 per cent, but with a slight decrease in the percentage of purity. It is probable that this is due to the more rapid growth caused by the trimming, since it is shown elsewhere in this paper that a very definite relation exists between the rate of growth and the percentage of camphor in the leaves and twigs.

VARIATION DUE TO RATE OF GROWTH

The camphor tree is often planted as a windbreak along the edge of orange groves, and under such conditions, the trees sometimes receive some commercial fertilizer by sending feeding roots into the area fertilized for the orange trees. It also often occurs that a few missing orange trees in a grove are replaced by camphor.

Under these conditions, where the trees receive more or less commercial fertilizer, it has been noted repeatedly that such trees yield a percentage of camphor above the average, and that the growth of the tree was more rapid. In order to find if a relation existed between the rate of growth of the tree and the camphor content, localities were found where a considerable number of trees of uniform age had been planted in a block, and tests were made on trees of different heights to determine the camphor content. Results obtained from Groves S and H were similar (Table 10).

TABLE 10—VARIATION IN CAMPHOR CONTENT DUE TO RATE OF GROWTH

Tree No.	Height ft.	Crude Camphor	Purity of Crude	Average Percentages: Crude Camphor	Average Percentages: Purity of Crude	Average Percentages: Pure Camphor
S3	20	1.90	74	1.93	75	1.45
S76	20	1.88	78			
S5	18	2.10	73			
S77	8	1.76	69	1.75	70	1.23
S75	8	1.76	71			
H848	14	1.57	71	1.63	72	1.17
H849	14	1.63	71			
H845	13	1.47	72			
H850	12	1.85	72			
H847	11	1.68	72	1.60	71	1.14
H851	10	1.88	70			
H844	8	1.45	71			
H846	8	1.41	70			
H842	6	1.57	73	1.52	72	1.09
H843	6	1.48	72			

These results seem to indicate that the more rapid the growth of the tree, the greater will be the percentage of camphor in the leaves and twigs, as well as the much greater yield of this material. This is important in the handling of commercial plantations, and methods of culture should be used which will force the growth of the tree as much as possible.

VARIATION DUE TO SOIL CONDITIONS

Throughout the entire investigations on Florida camphor, the writer has noted that the character of the soil has much to do with the percentage of camphor in the trees. In order to gain some definite information on this point, a number of trees were tested in selected groves growing in widely different soil conditions. The trees selected were from 8 to 12 years of age, and the total number of trees in the selected groves varied from 50 to 4000 trees. The soil conditions in each grove were as indicated in Table 11.

The highest yield of camphor was secured on the heavy clay soil and the percentages decrease rapidly as the soil becomes more sandy and less retentive of moisture. The lowest yields were secured on the soils Nos. 7 and 8, known locally as "sand scrub." No. 6, although located on a light soil, shows a rather high percentage due doubtless to the fact that these trees have been well fertilized with commercial fertilizer and have made good growth. Nos. 9 and 10 seem to be exceptions since although located on an extreme type of sand scrub, they show a high percentage. This is probably due to the very much dwarfed conditions of the trees, and that an abnormal camphor formation has resulted. This same condition has been found frequently on this class of soil where no fertilizer is applied.

TABLE 11—CAMPHOR VARIATIONS DUE TO SOIL CONDITIONS
RESULTS IN PERCENTAGES

Grove	No. of Trees Tested	Crude Camphor	Purity of Crude	Pure Camphor
1	8	2.14	75	1.61
2	5	1.95	74	1.44
3	6	1.76	75	1.32
4	5	1.62	73	1.18
5	8	1.56	76	1.19
6	16	1.74	76	1.32
7	12	1.58	71	1.12
8	16	1.50	76	1.14
9	6	1.86	73	1.36
10	32	1.85	76	1.41

GROVE 1—A heavy black clay prairie soil of great fertility, and containing a high percentage of vegetable matter.

GROVE 2—A clay loam soil with clay subsoil, and high in vegetable matter. The trees in this grove have also received small amounts of commercial fertilizer.

GROVE 3—A sandy loam with clay subsoil. A much more sandy soil than No. 2.

GROVE 4—Good grade of sandy high pine land with clay about 4 ft. below surface. A soil similar to No. 3 but lower in vegetable matter and with clay deeper down.

GROVE 5—A light sandy soil, low in vegetable matter and with clay about 8 ft. below surface.

GROVE 6—A soil very similar to No. 5 but these trees had received considerable amounts of commercial fertilizer.

GROVE 7—A very light sandy soil with clay 12 ft. below surface.

GROVE 8—Soil formed from old sand dunes and with almost no vegetable matter. Clay 20 ft. below surface. These trees had received very small amounts of commercial fertilizer.

GROVE 9—White sand similar to No. 8 but no commercial fertilizer had been given to these trees. Trees were badly dwarfed and were yellow in color.

GROVE 10—Same soil as No. 9 but rather more extreme. Trees very yellow and dwarfed.

From the commercial standpoint, the data presented in Table 11 is very important, and shows that it is essential that a good grade of soil be secured for camphor culture. Taking the yield of hedge trimmings as 10,000 lbs. per acre per year, the difference in yield of crude camphor between Grove 1 and Grove 8 would be 64 lbs. of crude gum, or 48 lbs. of refined camphor per acre. This sum would most certainly pay a good interest on the additional investment in a good grade of land. In fact, however, the difference would be far greater than this, since no account has been taken of the increased growth of the trees under the better conditions.

CONCLUSIONS

From the data presented, the following conclusions can be drawn concerning the factors causing variation in the yield of camphor in commercial plantations under Florida conditions.

I—The maximum yield of camphor is secured from leaves and twigs of the last growth, taken during the dormant season. If allowed to remain on the tree through another growing season, the yield is reduced. The yield from young wood is very small and not to be considered from a commercial standpoint.

II—Severe pruning to induce a watersprout growth gives a low yield in the leaves and small twigs, while the yield of wood so produced is extremely low.

III—The clipping of leaves and twigs, as in hedge trimming, tends to increase the yield of camphor in the next growth.

IV—Any commercial plan of harvesting should provide a material high in yield with a minimum amount of low-yielding wood, and with the least injury to the trees.

V—Even slight shade tends to decrease the percentage yield of camphor in the leaves, and to reduce the leaf area of the tree.

VI—A considerable yearly variation may be expected under varying climatic conditions and rainfall.

VII—To a considerable extent, the yield of camphor is proportional to the rate of growth, and forcing the growth of the trees will give a larger tonnage of material yielding higher in percentage of gum.

VIII—Higher percentages were secured on the better soils, especially those containing considerable amounts of clay. Under extreme stunted conditions of growth, the percentage of camphor in the leaves may be rather high.

ORLANDO, FLORIDA

THE CHEMISTRY OF WOOD

I—METHODS AND RESULTS OF ANALYSIS OF SOME AMERICAN SPECIES

By A. W. Schorger

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The chemical composition of woods is of considerable importance to many wood-using industries. Of special interest to the pulp and paper industry is the actual yield of cellulose from the different woods as compared with the yield of pulp obtained from them. The various methods of manufacturing chemical pulp destroy from 25 to 35 per cent of the cellulose when based on the cellulose content as found by laboratory analyses. However, knowing the cellulose content of the wood, the manufacturer may strive so to regulate cooking conditions that the yields of pulp will more nearly approach the theoretical.

Spruce wood contains approximately 61.00 per cent cellulose, yet the yield of pulp by the sulfite process rarely exceeds 45 per cent. The remaining 55 per cent of the wood, consisting largely of decomposition products of the lignin, is practically a waste product, and it is believed that a better knowledge of the chemistry of lignocellulose will lead to a much better solution of the problem of its utilization.

The results obtained by the chemical analysis of a wood cannot always be directly correlated with those obtained by processes, such as wood distillation, in which there is a profound decomposition of the wood. The yields of methyl alcohol from softwoods and hardwoods is much smaller than might theoretically be obtained from the percentage of methoxy groups present. This is possibly due to the formation of methyl ethers that remain in the tar. The yield of methyl alcohol from the two classes of woods is not even proportional to the amounts of methoxy groups present, the hardwoods giving decidedly the largest amounts of alcohol. Practically the same condition obtains for the acetic acid.

One of the requirements for the woods used as separators in lead storage batteries is that the amount of organic acids, especially acetic acid formed by the action of the sulfuric acid, be a minimum to prevent destruction of the grids. Analysis has shown that the conifers give the least acid on hydrolysis with sulfuric acid so that the probable value of the various woods may be predicted on this basis. In fact, longleaf pine, giving only 0.76 per cent of acetic acid, has already proved to be very serviceable for separators.

The data available in the literature on the composition of American woods especially are very meagre. Dean and Tower[1] studied the chlorine method of estimating cellulose and determined the cellulose content of several species; and De Chamot[2] has determined the yields of furfural from a large number of woods. The solution of the wood waste problem appears to lie mainly along chemical lines so that in connection with the industries already utilizing wood by chemical processes a systematic survey of several typical American woods seemed essential. Owing to the highly complex, colloidal nature of wood the results obtained by analysis are not capable of an exact interpretation. A large amount of work will be necessary to secure a correct insight into the true composition of the lignocellulose.

GENERAL COMPOSITION OF WOOD

Wood belongs to the widely distributed class of lignocelluloses of which jute probably represents the purest form. In addition to lignocellulose as the main constituent, hemicelluloses are always present in wood, while resins, gums, tannins, carbohydrates, dyes, ethereal oils, etc., occur as secondary constituents.

Lignocellulose is generally considered to consist of cellulose chemically combined with lignin. Cross and and Bevan[1] dissolved jute lignocellulose in zinc chloride-hydrochloric acid solution; on addition of water the greater portion of the lignocellulose was reprecipitated. Since the soluble and insoluble portions showed no difference in their reactions the lignin and cellulose were considered as chemically combined. Several writers have looked upon lignin as an incrusting material,[2] the combination with the cellulose being purely physical. In support of this view, König and Rump[3] have recently published results showing the selective action of certain chemicals. They consider the natural cell membranes to consist of cutin, cellulose and lignin. When thin sections of wood were oxidized with hydrogen peroxide and ammonia only the lignin was removed; further treatment with 72 per cent sulfuric acid removed the cellulose leaving a residue of cutin. Since the residual cellulose plus cutin, and the cutin, still conformed to the cellular structure of the original membrane, it was assumed that the lignin, cellulose and cutin are not chemically but physically combined. Owing to the powerful reagents employed this assumption is scarcely justified.

Lignins are characterized as being carbohydrate derivatives containing methyl, methoxy, formyl and acetyl groups, and consequently have a higher carbon content than cellulose or the hemicelluloses. Lignin is partially soluble in water under pressure,[4] readily soluble to a greater or lesser extent in dilute acids and alkalies, and is more readily attacked by oxidizing agents than cellulose. The early investigators considered lignin to be a definite compound. Schulze[5] gave the elementary composition of lignin as 55.55 per cent carbon, 5.83 per cent hydrogen, and 38.62 per cent oxygen. The experiments of Lange[6] showed that lignin consisted of possibly two substances having a higher carbon content than that previously reported. Isolated lignin has the properties of an acid and the term lignic acid is frequently applied. Lange isolated two lignic acids differing in their solubility toward alcohol. The following analyses were reported:

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[1] *J. Am. Chem. Soc.*, **29** (1907), 1119.

[2] *Am. Chem. J.*, **16** (1894), 224, 611.

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[1] "Cellulose," p. 114.

[2] This subject is discussed at length in Schwalbe's "Chemie der Cellulose," pp. 451–4.

[3] "Chemie und Struktur der Pflanzen-Zellmembran," Berlin (1914).

[4] Tauss, *Dingler's polytech. J.*, **1890**, 276, 411; König and Sutthof, *Landw. Verst.*, **70** (1909), 402.

[5] *Chem. Zentr.*, **28** (1857), 324.

[6] *Z. physiol. Chem.*, **14** (1890), 15–30, 217–226.

		Beech	Oak	Fir
Soluble in Alcohol	C	61.47	61.61	61.28
	H	5.48	5.47	4.95
Insoluble in Alcohol	C	59.04	58.83	60.51
	H	5.37	5.15	5.22

It is possible that the two acids may be identical since it was found that by dissolving the material insoluble in alcohol in caustic soda of specific gravity 1.10, precipitating with H_2SO_4, filtering and washing, the material was then in great part soluble in alcohol.

Streeb[1] isolated from the alkaline liquor of a soda cellulose plant some lignic acids that contained 62.94 to 65.50 per cent C and 5.11 to 5.39 per cent H. The methyl content amounted to 5.78 to 5.98 per cent corresponding to about $C_{36}H_{44}O_{14}$ or $C_{36}H_{40}O_{12}$.

Cross and Bevan,[2] studying the action of chlorine on lignocellulose, obtained a yellow lignone chloride, $C_{19}H_{18}Cl_4O_9$, whose properties led them to believe that lignin has a quinone structure. The lignone chloride is readily soluble in a solution of sodium sulfite, a deep reddish purple solution being obtained.

Lignocellulose gives acetic and formic acids on treatment with steam and weak hydrolytic reagents. Oxidation with chromic acid gives about 6 per cent acetic acid. Cross and Tollens[3] believe that formyl (H.CO—) and acetyl (CH_3.CO—) groups are actually present as such in the wood, while Cross and Bevan believe also in the presence of acetic acid residues (—CH_2.CO—). Characteristic of lignin is the occurrence of methoxy groups, the estimation of which has been suggested as a means of determining the degree of lignification.[4]

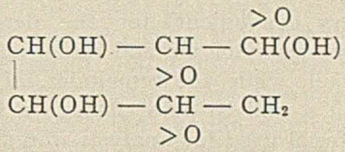
Klason[5] has assigned to lignin the formula $C_{40}H_{42}O_{11}$. The reactions of lignin and coniferyl alcohol being analogous in many respects, it is assumed that the lignin is derived from coniferyl alcohol by condensation and oxidation of four molecules of the alcohol.

HEMICELLULOSES

Hemicelluloses are defined by Schulze as carbohydrates insoluble in water but soluble in dilute acids and alkalies. They are further characterized by being more easily hydrolyzed by dilute acids than the true celluloses. The principal hemicelluloses in wood are pentosans $(C_5H_8O_4)_n$ although small amounts of hexosans $(C_6H_{10}O_5)_n$, such as mannan and galactan, are frequently present. The most widely distributed pentosan is xylan or "wood gum," which, on hydrolysis, gives xylose, usually with small amounts of arabinose. Since on distilling wood with 12 per cent hydrochloric acid, methylfurfural is obtained, in addition to furfural, it is generally assumed that methyl pentosans are also present. The determination of pentosans in wood by means of the furfural reaction is of doubtful accuracy as is pointed out below.

CELLULOSE

Among the various formulas proposed for cellulose that of Green[6] is widely accepted since it is most in harmony with the general reactions of the cellulose molecule:



$$\begin{array}{l} \qquad\qquad\qquad\qquad\quad >O \\ CH(OH) — CH — CH(OH) \\ | \qquad\qquad\quad >O \\ CH(OH) — CH — CH_2 \\ \qquad\qquad\quad\;\; >O \end{array}$$

Green's formula may be applied to cotton cellulose but scarcely to wood cellulose. Cotton cellulose gives but a trace of furfural when distilled with 12 per cent hydrochloric acid while from 6 to 17 per cent of furfural may be obtained from wood cellulose, according to the species. This fundamental difference is explained by assigning to wood cellulose an oxycellulose structure.[1] König and Hühn[2] and many other investigators have rejected this explanation as a result of the low yields of furfural obtained from prepared oxycelluloses. Wood cellulose isolated by the chlorine method, in addition to giving furfural, exhibits a strong methoxy reaction.

METHODS FOR THE ANALYSIS OF WOOD

The methods employed in this work were the following:

SAMPLING—A cross-sectional disc about 2 in. thick is taken from the tree about 20 ft.[3] from the ground and from this disc two diagonally opposite sectors are split out, the size of the sectors depending upon the diameter of the tree. The material employed for analysis consists of two forms—sawdust and shavings not more than 0.005 in. in thickness. In some cases, especially with very soft woods, a satisfactory sample is best obtained by scraping the wood with a piece of window glass or a cabinetmaker's scraper. Some woods work best while wet and others when nearly air-dry. The best conditions for a particular species are determined by experiment.

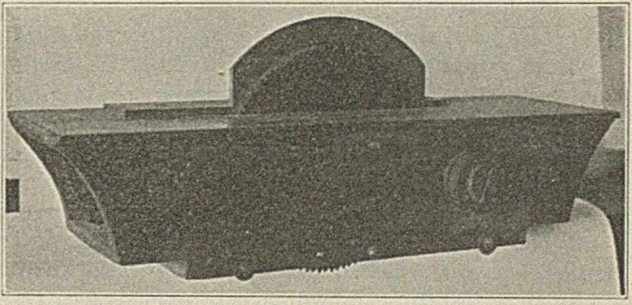


FIG. I—SAW WITH GUIDE ARRANGED TO CUT JUST THE WIDTH OF THE SAW, SO THAT A PIECE OF WOOD IS CUT ALMOST ENTIRELY INTO SAWDUST

Shavings obtained with a plane should be passed through a grinder having a shredding effect, the resulting fragments being 3 to 5 mm. long and 1 to 2 mm. wide. The material is then screened and all that passes through a 40-mesh sieve is rejected. The residual material is thoroughly mixed to insure a uniform sample. The remaining portions of the sectors are cut into sawdust (Fig. I) and the sawdust thor-

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[1] Dissertation, Göttingen (1892), 25.

[2] "Cellulose," p. 135.

[3] *J. Landw.*, **59** (1911), 185–186.

[4] Benedict and Bamberger, *Monatsh.*, **11** (1890), 263.

[5] "Beitrage zur Kenntnis der chemischen Zusammensetzung des Fichtenholzes," Berlin (1911).

[6] *Z. Farben- u. Textilchemie*, **3** (1904), 97, 197, 309.

[1] Cross and Bevan, "Cellulose," p. 82.

[2] "Bestimmung der Zellulose in Holzarten und Gespinstfasern," p. 51.

[3] Strength tests at the Forest Products Laboratory have shown that this portion of the tree is most uniform.

oughly mixed. A portion of the sawdust from coniferous woods is kept in a sealed container (Mason jars are very convenient) for the determination of moisture by the xylol method and the determination of volatile oil, while the remainder, after air-drying, is ground in a mill to pass through a 40-mesh sieve. All the material used for analysis would be in the air-dry form, the moisture content being determined in a separate sample. All results are calculated on the oven-dry basis.

The 40-mesh sawdust is kept in a rubber-stoppered flask so that after the moisture has once been determined the samples taken out for analysis can be easily reduced to the dry weight by calculation.

MOISTURE—Three g. of 40-mesh sawdust are weighed out in a glass-stoppered weighing bottle and dried for 6 hours in an air oven at 105° C. Dry wood is very hygroscopic and should always be weighed in a closed vessel. In the case of coniferous woods the moisture figure also includes most of the volatile oil.

VOLATILE OIL—Twenty-five grams of sawdust from the sealed container are quickly weighed, placed in a 250-cc. Erlenmeyer flask, and 75 cc. of water-saturated xylol added. On distillation the xylol and water distil over together, the distillate being collected in a graduated funnel. The amount of water present can then be read off directly.[1]

Ten grams of sawdust are weighed into a tared wide-mouthed, stoppered Erlenmeyer flask. The flask is then provided with a rubber stopper containing a tube extending nearly to the bottom of the flask for the introduction of steam and an outlet tube for connection with a condenser. The flask is heated in an oil bath maintained at 110° C. and steam passed in gently until oil ceases to distil over. This point can be readily ascertained by catching a few cc. of the distillate in a test tube in which case even traces of oil are distinguishable on the surface. When all the oil has been driven over, the stopper is withdrawn and any adhering sawdust is washed down into the flask. Heating the flask in the oil bath is continued until practically all the water is expelled. This operation is greatly facilitated by inserting a tube into the mouth of the flask and applying suction with a water pump. The exterior of the flask is then carefully cleaned and the drying completed in the air oven. The stoppered flask is then weighed after cooling.

In this way the weight of wood substance is obtained, the water and volatile oil having been removed. Since the moisture content of the original sample has been determined by the xylol method, subtracting the combined weight of residual wood substance and moisture from the original weight of the sample gives the amount of volatile oil.

The determination of volatile oil by heating a sample in the oven and subtracting the water found by the xylol method from the resulting total loss in weight usually does not give the true oil content. The "pine oil" of longleaf pine can be quite readily expelled with steam but only partially by heating for a brief period in the oven.

The volatile oil determination may be neglected in the case of only slightly resinous conifers.

WAXES, FATS, RESINS—Three or four grams of 40-mesh sawdust are extracted with ether 4 to 5 hrs. in a Soxhlet extractor, the amount of material extracted being determined by weighing the residue remaining after evaporation of the solvent.

ASH—Five grams of sawdust are incinerated in a shallow platinum dish in the electric muffle at a dull red heat. The contents of the dish are stirred occasionally, if necessary, to insure complete combustion of the carbon. If the combustion is incomplete the carbon will appear as a black suspended material on treatment with dilute hydrochloric acid.

ALKALI-SOLUBLE—Two grams of 40-mesh sawdust are placed in a 250-cc. beaker, 100 cc. of 1 per cent $NaOH$ added, covered with a watch glass, and the beaker placed in a pan of boiling distilled water for exactly one hour, the height of the water in the pan being maintained level with the solution in the beaker by the addition of boiling distilled water. The contents of the beaker are occasionally stirred. The material is then collected in a tared alundum crucible, washed thoroughly with hot distilled water, dilute acetic acid, and water successively, and dried. The difference is the portion soluble in alkali and consists of pentosans, lignin, resin acids, etc.

HOT WATER-SOLUBLE—Two grams of 40-mesh sawdust are digested with 100 cc. of H_2O in a 300-cc. Erlenmeyer flask provided with a reflux condenser. After the water has been boiled gently for 3 hours, the contents are transferred to a tared alundum crucible, washed with hot water, dried and weighed. Duplicate determinations should check within 0.2 per cent.

COLD WATER-SOLUBLE—Two grams of 40-mesh sawdust are placed in a 400-cc. beaker, 300 cc. of water added, and allowed to digest at room temperature with frequent stirring for 48 hours. The sawdust is then transferred to a tared alundum crucible, washed with cold distilled water, dried, and weighed in a weighing bottle.

PENTOSAN AND METHYL PENTOSAN—Two grams of 40-mesh sawdust from coniferous woods (1 g. from hardwoods) are placed in a 250-cc. flask[1] provided with a separatory funnel and attached to a condenser (Fig. II). Add 100 cc. of 12 per cent hydrochloric acid (sp. gr. 1.06) and distil at the rate of 30 cc. in 10 min. The distillate is passed through a small filter before entering the receiver. As soon as 30 cc. of distillate are collected, 30 cc. of HCl are added to the distillation flask and the distillation is continued in this manner until 360 cc. of distillate are collected. To the total distillate, add 40 cc. of filtered phloroglucine solution that has been prepared at least a week previously by heating 11 grams of phloroglucine in a beaker with 300 cc. of 12 per cent HCl, and after solution has taken place making up to 1500 cc. with 12 per cent HCl. After addition of the phloroglucine, the solution soon turns greenish black. After standing

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[1] For details of this method see *Forest Service Circular* **134**.

[1] The flasks are easily made by fusing an outlet tube and funnel to the ordinary all glass wash bottles.

16 hours[1] the furfural phloroglucide will have settled to the bottom of the beaker. If a drop of the supernatant liquid gives a pink color with aniline acetate paper[2] the precipitation of the furfural is incomplete. A further amount of phloroglucine solution is then added and the beaker allowed to stand over night as formerly. In no case so far has it been necessary to use more than 40 cc. phloroglucine solution.

The furfural phloroglucide is filtered, using a tared Gooch crucible having a thick asbestos mat and washed with exactly 150 cc. of water. The crucible is then dried for 4 hours in a water oven and weighed in a weighing bottle.

The crucible is placed in a narrow beaker and 20 cc. of 95 per cent alcohol are added to the crucible. The beaker is then placed in a water bath maintained at 60° for 10 minutes. The alcohol is removed with a suction pump and the process repeated (usually four or five times) until the alcohol that runs through is practically colorless. The crucible is then dried for 2 hrs. in the water oven and again weighed. The weight of the residual phloroglucide subtracted from the weight of mixed phloroglucides gives the weight of methyl furfural phloroglucide. From the weights of furfural phloroglucide and methyl furfural phloroglucide obtained, the amounts of pentosan and methylpentosan present in the wood are calculated from the tables of Kröber and Tollens.[1]

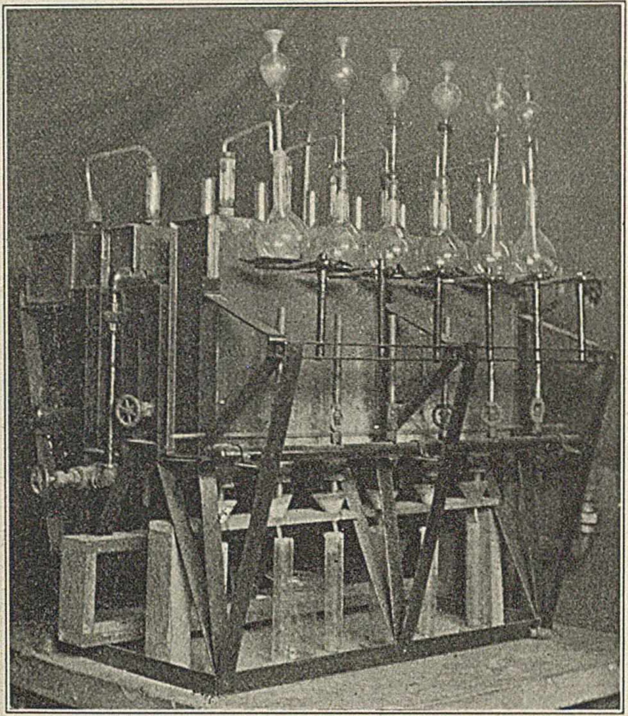

FIG. II—APPARATUS FOR DETERMINING PENTOSANS AND METHYL PENTOSANS

CELLULOSE—Two grams of shavings in an alundum crucible are extracted 3 to 4 hrs. with a mixture of equal parts of benzol and alcohol. After evaporation of the solvent the shavings are thoroughly washed with hot water using the suction pump. The moist shavings are then transferred with a pointed glass rod to a 250-cc. beaker, evenly distributed over the bottom, and subjected to a stream (about 40 bubbles per minute for each sample) of washed chlorine gas for half an hour. The end of the tube delivering the chlorine gas should be about $^1/_2$ in. above the shavings. At intervals of 6 to 7 minutes the contents of the beaker are stirred to insure uniformity of chlorination. After the chlorine treatment the shavings are treated with a solution of SO_2 until the chlorine odor disappears, transferred to the alundum crucible, and washed with hot water. The shavings are again returned with the glass rod to the beaker, and 100 cc. of a 2 per cent sodium sulfite solution are added and the beaker, covered with a watch glass, placed in a boiling water bath for 30 minutes. The fibers are then transferred to the crucible and washed with water. The above procedure is seldom sufficient to remove all the lignin, so that the treatment with chlorine and subsequent treatment as outlined above is repeated until the fibers are practically a uniform white. The second and following treatments with chlorine should not be longer than 15 to 30 min. After all the lignin has been removed the fibers are given a final bleaching by adding 20 cc. of a 0.1 per cent solution of potassium permanganate, allowed to stand 10 minutes and rendered colorless with SO_2 solution. The fibers are then thoroughly washed with hot water, dilute acetic acid, then with alcohol and finally with ether, and dried for two hours at 105° in the air oven. When cool, the crucible is weighed in a weighing bottle.

ACID HYDROLYSIS—Approximately 2 g. of 40-mesh sawdust are placed in a 250-cc. Erlenmeyer flask and 100 cc. of 2.5 per cent H_2SO_4 added. The flask is connected with a reflux condenser and the contents are boiled gently 3 hours and then allowed to cool. The interior of the condenser is washed down with a little distilled water and the contents of the flask are transferred to a 250-cc. graduated flask. Distilled water free from carbon dioxide is added to make up to the mark and the solution allowed to stand several hours with frequent shaking, and then filtered.

A wide-mouthed, round-bottomed, 750 cc. flask is provided with a rubber stopper containing: (1) a dropping funnel; (2) a glass tube drawn out to a capillary, closed with a rubber tube and pinchcock, and extending to the bottom of the flask; and (3) a Soxhlet connecting bulb-tube. An ordinary condenser is used, to the end of which is attached for a receiver a 500-cc. distilling flask cooled with a stream of water and connected with a manometer and suction pump.

A few pieces of pumice are placed in the boiling flask to which is added 200 cc. of the filtrate obtained

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[1] Böddener and Tollens (*J. Landw.*, **58**, 232–7) have found that if the distillate containing the phloroglucine is heated to 80 to 85° and then allowed to stand for 2 hours the precipitate of furfural phloroglucide can be filtered off without waiting for the solution to stand over night. The latter method, however, is preferable.

[2] The aniline acetate paper is conveniently prepared by dipping strips of filter paper into aniline acetate. The latter is prepared by adding acetic acid, drop by drop, to a mixture of equal parts of aniline and water until a clear solution is obtained. It is preferable to use hydrochloric acid in place of acetic acid since the latter sometimes contains impurities that give a red coloration with aniline.

[1] Abderhalden's "Handbuch der biochemischen Arbeitsmethoden," Vol. II, pp. 137 and 154.

TABLE I—ANALYSIS OF WOODS—RESULTS IN PERCENTAGES ON BASIS OF OVEN-DRY SAMPLE

SPECIES	Sample No.	Ash	SOLUBILITY OF SAMPLE IN				Acetic Acid by Hydrolysis	Methoxy Groups (CH_3O-)	Pentosan	Methyl Pentosan	Cellulose	Ash in Cellulose	Ash-free Cellulose	Pentosan in Cellulose	Methyl Pentosan in Cellulose	Volatile Oil
			Cold Water	Hot Water	Ether	1% NaOH										
Longleaf Pine (*Pinus palustris*)	11	0.40	7.75	8.20	6.70	24.62	0.70	5.00	7.33	3.48	55.33	0.31	55.02	8.38	1.26	1.60
	12	0.34	5.60	6.03	6.70	21.07	0.93	4.90	7.52	3.29	57.53	0.30	57.23	7.19	1.39	1.12
	13	0.35	5.40	6.78	2.65	18.89	0.62	5.26	7.57	3.87	61.41	0.32	61.09	7.39	1.03	0.87
	20	0.38	6.05	7.57	9.23	24.87	0.79	5.03	7.43	3.67	59.67	0.36	59.31	7.90	0.96	1.60
	Mean,	0.37	6.20	7.15	6.32	22.36	0.76	5.05	7.46	3.60	58.48	0.32	58.16	7.71	1.16	1.30
Douglas Fir (*Pseudotsuga taxifolia*)	1	0.40	3.79	6.62	0.94	15.82	0.93	4.81	6.03	4.24	61.97	0.21	61.76	5.56	1.26	....
	2	0.37	3.16	6.07	1.00	16.76	1.01	5.17	6.30	4.64	57.00	0.10	56.90	....	....	....
	3	0.35	2.94	6.36	1.11	15.12	1.13	4.88	6.00	4.38	63.08	0.07	63.01	....	....	....
	5	0.38	4.25	6.96	1.02	16.72	1.07	4.92	5.73	4.38	63.82	0.26	63.56	5.12	1.15	....
	Mean,	0.38	3.54	6.50	1.02	16.11	1.04	4.95	6.02	4.41	61.47	0.16	61.31	5.34	1.20	
Western Larch (*Larix occidentalis*)	1	0.21	10.45	12.57	0.72	22.07	0.61	5.08	11.15	2.47	58.25	0.24	58.01	9.12	1.40	....
	2	0.32	11.00	12.40	0.74	21.93	0.91	4.91	11.04	2.83	58.71	0.18	58.53	8.41	1.22	....
	3	0.22	8.16	10.08	0.93	19.44	0.76	5.08	10.22	3.14	60.91	0.29	60.62	8.67	1.24	....
	4	0.16	12.83	15.30	0.83	25.11	0.55	5.05	10.78	2.80	53.31	0.45	52.86	9.55	0.90	....
	Mean,	0.23	10.61	12.59	0.81	22.14	0.71	5.03	10.80	2.81	57.80	0.29	57.51	8.94	1.19	
White Spruce (*Picea canadensis*)	1	0.33	1.28	1.88	1.95	11.33	1.58	5.31	10.78	3.08	62.61	0.27	62.34	10.26	0.83	....
	2	0.29	0.92	2.28	0.90	11.58	1.57	5.26	10.31	3.52	63.29	0.39	62.90	9.29	0.68	....
	3	0.30	1.45	2.52	0.97	12.75	1.49	5.29	10.04	3.95	60.43	0.25	60.18	....	....	....
	4	0.32	0.82	1.88	1.63	10.63	1.73	5.32	10.42	3.64	61.09	0.30	60.79	9.33	0.66	....
	Mean,	0.31	1.12	2.14	1.36	11.57	1.59	5.30	10.39	3.55	61.85	0.30	61.55	9.63	0.72	
Basswood (*Tilia americana*)	1	0.80	2.04	3.84	1.50	23.43	5.78	6.23	19.82	3.72	62.92	0.11	62.81	24.48	1.19	....
	2	0.74	1.63	2.94	1.14	21.61	6.14	6.05	19.54	3.85	62.41	0.14	62.27	23.54	1.46	....
	3	0.96	3.14	5.66	3.59	26.93	5.46	6.11	20.37	3.68	54.66	0.24	54.42	26.61	1.62	....
	4	0.94	1.23	3.22	0.89	21.46	5.41	5.91	19.14	4.16	63.13	0.22	62.91	21.89	1.45	....
	5	0.85	2.55	4.67	2.68	25.38	6.18	5.72	20.79	3.23	63.08	0.22	62.86	24.86	2.00	....
	Mean,	0.86	2.12	4.07	1.96	23.76	5.79	6.00	19.93	3.73	61.24	0.19	61.05	24.28	1.54	
Yellow Birch (*Betula lutea*)	1	0.58	2.88	4.21	0.55	20.02	3.99	6.12	24.26	3.18	60.49	0.11	60.38	28.40	1.11	....
	2	0.57	2.58	3.87	0.67	20.20	4.39	6.03	25.40	3.12	61.08	0.12	60.96	29.96	1.32	....
	3	0.54	3.15	4.66	0.54	19.51	3.81	6.19	23.00	2.25	61.82	0.14	61.68	26.55	1.04	....
	4	0.37	2.06	3.15	0.63	19.65	5.02	5.92	25.86	2.21	61.85	0.16	61.69	....	....	....
	Mean,	0.52	2.67	3.97	0.60	19.85	4.30	6.07	24.63	2.69	61.31	0.13	61.18	28.30	1.16	
Sugar Maple (*Acer saccharum*)	1	0.46	2.60	4.27	0.29	16.98	4.26	7.22	21.10	2.50	60.78	0.13	60.65	21.08	1.04	....
	2	0.51	2.73	4.22	0.22	17.20	4.25	7.23	21.90	2.14	61.67	0.28	61.39	25.82	1.05	....
	3	0.40	2.94	4.87	0.30	18.04	4.60	7.25	22.21	2.05	60.20	0.33	59.87	25.83	1.00	....
	4	0.38	2.33	4.15	0.20	18.35	4.74	7.28	21.62	2.85	60.48	0.39	60.09	25.20	0.77	....
	Mean,	0.44	2.65	4.36	0.25	17.64	4.46	7.25	21.71	2.39	60.78	0.28	60.50	24.48	0.96	

above (in the case of hardwoods use 100 cc.). The flask is heated in an oil bath maintained at 85° C., while the pressure is reduced to 40 to 50 mm. When the contents of the flask are reduced to about 20 cc., distilled water is added through the dropping funnel, drop by drop, at the same rate that distillation takes place. When 100 cc. of wash water have been distilled over, the distillate is titrated with $N/100$ NaOH by using phenolphthalein as indicator. If (*a*) 200 cc., or (*b*) 100 cc., of solution were taken for distillation, the number of cc. of NaOH used is multiplied by (*a*) 5/4, or (*b*) 5/2, respectively, and calculated as acetic acid.

All the distilled water used in this determination should have been recently boiled to expel carbon dioxide.

DETERMINATION OF METHOXY GROUP (CH_3O)—The principle of the methoxy determination depends upon heating the substance to be examined with hydriodic acid, whereby methyl iodide is formed. The methyl iodide is swept from the reaction flask by a current of CO_2 into vessels containing an alcoholic $N/10$ silver nitrate solution, the methyl iodide being decomposed with the formation of silver iodide. When only a few determinations are being made it is preferable to weigh the silver iodate directly.

The silver nitrate solution is prepared by dissolving 17 grams of the salt in 50 cc. of distilled water and making up to a liter with absolute alcohol. The silver nitrate and potassium sulfocyanate solutions are standardized as follows: a known weight (0.5 g.) of pure dry sodium chloride is made up to 250 cc. with distilled water; to 25 cc. portions of the salt solution known volumes (about 15 cc.) of the alcoholic silver nitrate solution are slowly added with constant stirring. The beakers are placed in the steam bath and heated until all the alcohol is expelled. A few drops of nitric acid are added, the precipitated silver chloride is filtered off, washed, and the filtrate titrated with the solution of potassium sulfocyanate by using 5 cc. of a saturated solution of ferric alum acidified with nitric acid as the indicator. From the weight of sodium chloride employed the strength of the silver nitrate and potassium sulfocyanate solutions can be readily calculated.

Twenty-five cc. of the silver nitrate solution are placed in the absorption bulb. To the heating flask containing 0.30 to 0.35 g. of sawdust are added 15 cc. of hydriodic acid (sp. gr. 1.70). The flask is heated in a glycerin bath kept at 130°. As soon as all the connections are made, a current of washed CO_2 gas is passed through the apparatus at the rate of 45 to 50 bubbles per minute. The heating is continued until the silver nitrate solution, at first milky, has become perfectly clear; this requires about 2 hours. The contents of the absorption bulb are washed into a beaker, made up to 300 cc. and heated on the steam bath until the contents have evaporated to 50 cc. A little nitric acid is added, the AgI[1] filtered off, washed with hot water containing a little nitric acid, and the silver nitrate remaining in the filtrate is titrated with the potassium sulfocyanate solution. One cc. of $N/10$ $AgNO_3$ is equal to 0.0031 g. of methoxy (CH_3O).

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[1] According to Rosanoff and Hill [*J. Am. Chem. Soc.*, **29** (1907), 269] in using the Volhard method no appreciable error is involved in titrating directly solutions containing AgI and AgBr but AgCl must be removed.

RESULTS

The results of the analysis of seven American woods are summarized in the accompanying table. A discussion of the results, as well as a discussion of methods of analysis, is given in the following paper.

Forest Service
Forest Products Laboratory
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THE CHEMISTRY OF WOOD

II—DISCUSSION OF METHODS AND RESULTS

By A. W. Schorger

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In order to obtain a better understanding of the reasons for adopting the methods given in the previous paper, it is essential to consider both methods and results in detail.

DRYING—The methods of drying wood and other forms of cellulose have received considerable attention. Ost and Westhoff[1] believe that a temperature of 120 to 125° C. is necessary to drive out all hygroscopic moisture, but it is very doubtful if water retained near this temperature should be considered as merely hygroscopic. Winterstein[2] found that cellulose that had been heated at 105° for 48 hours gave increased amounts of glucose when boiled with dilute sulfuric acid.[3] Suringar and Tollens[4] obtained 2 to 3 per cent less cellulose from jute that had been heated at 97° for $3^{1}/_{2}$ hrs. than from air-dry material. Renker,[5] however, reached different conclusions. Unbleached sulfite cellulose that had been heated at 105 to 107° for 48 hrs. showed no difference in the yield of cellulose by the chlorine method of Cross and Bevan as compared with the air-dry material. Furthermore, unbleached sulfite cellulose that had been heated several days at 105 to 107° gave no greater yields of glucose than the air-dry cellulose, when hydrolyzed with sulfuric acid. The statement of Hofmann[6] that unbleached sulfite cellulose lost 8.175 per cent by weight in a vacuum over phosphorus pentoxide and 9.36 per cent by heating at 100° was not confirmed.

Methods of drying should be considered from the standpoint of convenience as well as accuracy. Renker[7] found that 3 to 4 weeks are required to dehydrate cellulose standing over phosphorus pentoxide, but by employing a vacuum the time could be reduced to about 20 hrs. for air-dry material; if saturated with water a longer time is required. He agrees with Schwalbe[8] that all hygroscopic moisture can be removed by heating at 105 to 107° C., and in fact this method was employed as the standard of absolute dryness. König and Hühn[9] also accept this method of drying. Accordingly heating to constant weight at 105 to 107° may be accepted as reliable except in case of special cellulose derivatives, or when it is desired to subject the cellulose to a critical subsequent examination as in the work of Renker.

Some confirmatory experiments were made in the drying of sawdust that showed that the moisture content could be determined in the oven with sufficient accuracy. Samples of sugar maple sawdust were dried in an electric oven at 105 to 107° and in a vacuum desiccator containing fresh concentrated sulfuric acid for three consecutive periods of 7, 6 and $8^{1}/_{2}$ hrs., respectively. The results are given in Table I.

TABLE I—DRYING SUGAR MAPLE SAWDUST BY VARIOUS METHODS

Sample	APPARATUS	Temp. ° C.	Heating Hours	Sample Grams	LOSS IN WEIGHT Gram	LOSS IN WEIGHT Per cent
A	Electric Oven	105–107	7	4.0963	0.2811	6.86
B_1	Vacuum: Pressure 60 mm.	55	7	3.2229	0.2128	6.60
B_2	Vacuum: Pressure 30 mm.	57	6	3.2229	0.2181	6.77
B_3	Vacuum: Pressure 30 mm.	60	$8^{1}/_{2}$	3.2229	0.2178	6.76

The sample heated *in vacuo* reached constant weight after 13 hrs. heating; and after $21^{1}/_{2}$ hrs., the total loss in weight is still 0.10 per cent less than that produced by heating for only 7 hrs. in the electric oven. That drying in the oven was more efficient is shown by the following experiment: A sample of spruce sawdust that had been digested with dilute alkali and then thoroughly washed with hot water was placed in the oven for 7 hrs. The sample then weighed 1.6635 g. After remaining in a Hempel vacuum desiccator containing concentrated sulfuric acid for 7 hrs. at 55° C. and 60 mm. pressure the sawdust actually increased in weight 0.0070 g. After an additional heating for 6 hrs. at 57° and 30 mm. pressure the sample weighed 1.6670 g., showing that half of the absorbed moisture was still retained. This is in harmony with previous observations that the various forms of cellulose have a greater affinity for moisture than the reagents ordinarily used for drying.[1]

The effect of prolonged heating on the weight of wood in an air oven at 105 to 107° was determined in the case of basswood and longleaf pine sawdust sufficiently fine to pass through a 40-mesh sieve. The weighing bottles used were of about 40 grams weight.

TABLE II—EFFECT OF PROLONGED HEATING ON WEIGHT OF WOOD GROUND TO 40-MESH

SAMPLE HEATED:		BASSWOOD NO. 1		LONGLEAF PINE NO. 20	
HEATING Periods	HOURS Total	Weight Grams	Decrease Per cent	Weight Grams	Decrease Per cent
0	0	3.3487	....	2.9172	....
3	3	3.1517	5.91	2.7731	4.94
4	7	3.1524	5.89	2.7732	4.95
4	11	3.1517	5.91	2.7731	4.94
10	21	3.1504	5.95	2.7699	5.05
8	29	3.1476	6.03	2.7676	5.13
15	44	3.1501	5.96	2.7686	4.98
48	92	3.1486	6.00	2.7646	4.23
69	161	3.1410	6.23	2.7523	5.65
98	259	3.1491	5.99	2.7551	5.56
191	450	3.1361	6.38	2.7409	6.04
261	711	3.1342	6.43	2.7321	6.35

The results given in Table II indicate that the weights of both woods remain practically constant during 18 hrs. heating. The total loss for the longleaf pine during the last 708 hrs. heating is 1.41 per cent, while that for the basswood during the same period was only 0.52 per cent. Since the longleaf pine contained 9.23 per cent of resin the increased loss in this case is to be expected.

VOLATILE OIL—Determination of the volatile oil

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[1] *Chem.-Ztg.*, **33** (1909), 197.

[2] *Z. physiol. Chem.*, **17** (1893), 393.

[3] Schwalbe ("Chemie der Cellulose," p. 26) is of the opinion that the results observed by Winterstein were due to the presence of considerable amounts of oxycellulose in the material examined.

[4] *Z. angew. Chem.*, **9** (1896), 749.

[5] "Bestimmungsmethoden der Cellulose," **1910**, p. 16.

[6] *Papier-Ztg.*, **1907**, p. 2558.

[7] "Bestimmungsmethoden der Cellulose," p. 18.

[8] *Ber.*, **40** (1907), 1347.

[9] "Bestimmung der Zellulose in Holzarten und Gespinstfasern," p. 13.

[1] Jentgen, *Z. angew. Chem.*, **33** (1910), 1544.

by subtracting from the total loss in weight by drying in the oven at 105 to 107° the amount of moisture found by the xylol method, gives lower results than if the volatile oil is expelled with steam. The "pine oil" in longleaf pine is difficult to expel completely by heating in the oven. Table III presents comparison of the amount of volatile oil obtained from longleaf pine by the two methods.

TABLE III

Longleaf Pine Sample No.	PER CENT VOLATILE OIL BY METHOD OF	
	Heating in Oven	Steaming
11	1.20	1.60
12	0.98	1.12
13	0.65	0.87
20	1.47	1.60

WAXES, FATS, RESINS—The portion of the wood soluble in ether, consisting of waxes, fats, resins, etc., was determined in two ways: *viz.*, loss in weight of the wood extracted, and direct weight of the extract after evaporation of the solvent. Only the latter method was found to give reliable results.

ALKALI-SOLUBLE—The action of alkali on wood is not specific. Among the products rendered soluble are lignin, resin acids, pentosans, and other carbohydrates. The action of alkali on hardwoods is much more pronounced than on conifers. This is in part due to the slight solubility of the pentosans of the conifers in alkali as compared with dilute acids.

The action of alkali is determined by Cross and Bevan[1] by boiling the solution containing the material to be examined. This method was not adhered to since the temperature is difficult to regulate and the solution bumps violently. The material should be washed with dilute acetic acid in order to remove the alkali adsorbed.

WATER-SOLUBLE—It is necessary, especially in the case of hot water, to limit the time of extraction as there is no sharp limit to the solubility of wood in boiling water. Klason[2] found that the wood of the Norway spruce, which had been first extracted with ether to remove resins, etc., lost about 12 per cent of its weight by alternate extraction with alcohol and water.

The chief materials extracted by water are tannins, bitter principles and carbohydrates. The lignin is also partially attacked, methyl alcohol and acetic acid being formed. The method employed would not be applicable to woods containing tannins with phlobaphenes rendered insoluble by boiling.

The water-soluble portion of the western larch was found to consist largely of a galactan, apparently $(C_6H_{10}O_5)_{20}$, that yielded only galactose[3] on hydrolysis. Investigation showed that galactans were characteristic of the water-soluble portions of the conifers.

PENTOSAN AND METHYL PENTOSAN—The determination of pentosans and methyl pentosans as worked out by Tollens and his pupils is very accurate so far as duplicating results is concerned. A comparison was made of the two methods of extracting the methyl furfural phloroglucide. Extraction with alcohol in a beaker[1] at 60°, while more tedious than extraction in a modified Soxhlet as recommended by Ishida and Tollens,[2] was found to give more accurate results, since by the latter method it is difficult to determine when the extraction is completed.

The weights of wood chosen (2 g. for conifers and 1 g. of hardwoods) give about 0.20 g. mixed phloroglucides. The results of extraction of pure furfural phloroglucide (about 0.20 g.) by Ellett and Tollens[3] show that about 0.0014 g. are dissolved during each extraction. A correction cannot be easily applied for this loss since when the table[4] is examined giving the amounts of arabinose and rhamnose found when known amounts of these sugars were taken, it is seen that the amount of arabinose found is 3 per cent too high and the rhamnose is 1.7 per cent too high. It was also found that 3 to 4 extractions with alcohol as recommended were usually insufficient to give even an approximately colorless extract. In Table IV are given the number of extractions necessary for approximately 0.20 g. of mixed phloroglucides from the various woods and celluloses, using 20 cc. of alcohol:

TABLE IV

Douglas Fir	White Spruce	Yellow Birch	Basswood	Sugar Maple	Longleaf Pine	Western Larch
8 to 9	6 to 7	4 to 5	6 to 7	5 to 7	4 to 6	5 to 6

It is evident that appreciable errors will result from a large number of extractions. These errors might be reduced by correcting for the solubility of the furfural phloroglucide were it not for the fact that the amounts of arabinose and rhamnose found by Ellet and Tollens were both too high in spite of the 3 to 4 extractions. In view of these facts it is highly probable that the figures given for methyl pentosans especially are too high.

It would probably be better to give the results as furfural and methyl furfural since wood not only contains true pentosans but apparently "furfuroids." Furthermore the true pentosans are mixtures of at least arabans and xylans, each giving different amounts of furfural. The source of the methyl furfural is attributed to the presence of methyl pentosans, though no methyl pentose of methyl pentosan has so far been isolated from wood. The various wood celluloses also give considerable amounts of furfural. To account for this behavior Cross and Bevan[5] have assigned to wood cellulose an oxycellulose structure. König and Hühn[6] are firmly of the opinion that wood cellulose obtained by the chlorine method contains residual pentosans, and that their method of determining cellulose (digestion with glycerin and sulfuric acid followed by oxidation with hydrogen peroxide and ammonia) is the only one giving true cellulose, *i. e.*, cellulose free from lignin, pentosans, etc. It seems essential, however, to look upon wood cellulose from a broader view-point. Cotton is doubtless the typical cellulose of the $(C_6H_{10}O_5)_n$ group, but it is no more reasonable to expect cotton to be the only cellulose in nature than

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[1] "Cellulose," p. 117.

[2] "Beiträge zur Kenntnis der chemischen Zusammensetzung des Fichtenholzes."

[3] Schorger and Smith, THIS JOURNAL, 8 (1916), 494–9.

[1] Ellett and Tollens, *J. Landw.*, 53 (1905), 20.

[2] *Ibid.*, 59 (1911), 61.

[3] *Ibid.*, 53 (1905), 20.

[4] *Ibid.*, 53 (1905), 22, Table IV.

[5] "Cellulose," p. 82.

[6] "Bestimmung der Zellulose, etc.," p. 50.

glucose to be the only sugar. It is probable that wood celluloses should be looked upon as definite compounds of hexosans with varying amounts of pentosans. The methoxy reaction may also be due to the presence of small amounts of methyl glucosides or similar derivatives. In this paper *cellulose will be defined as the residue remaining after alternate treatment with chlorine gas and sodium sulfite up to the point where the chlorine-sulfite color reaction, or the Mäule reaction, ceases.*

Analysis has shown that the hardwoods can be sharply divided from the conifers by means of the pentosan content of the cellulose. In general, the pentosan content of the hardwood cellulose is greater than that of the original wood, while with the conifers the reverse is true. The pentosan content of the cellulose from the conifers varied from 5 to 10 per cent and that from the hardwoods from 24 to 28 per cent. The methyl pentosan content of the various celluloses is fairly uniform but in the case of the woods there are considerable variations. Douglas fir contains the largest amount of methyl pentosan, the latter amounting to 42 per cent of the total pentosans and methyl pentosans present. In general the conifers contain more methyl pentosan than the hardwoods.

Table V—Pentosan Content (Percentages) of the Celluloses

Hardwoods			Conifers		
Basswood	24	Mean 26	Western Larch	9	Mean 8
Sugar Maple	25		Longleaf Pine	8	
Yellow Birch	28		Douglas Fir	5	
			White Spruce	10	

CELLULOSE—Owing to the work of previous investigators, especially that of Renker,[1] no attempt was made to determine the relative merits of the various methods of determining cellulose, since the chlorine method of Cross and Bevan is the only one that can be applied satisfactorily to woods.

One of the most important points in determining cellulose consists in obtaining the sample in the proper physical condition. Dean and Tower[2] rasp the wood. This method with dry woods gives mainly dust and even with wet wood the use of the rasp is tedious and unsatisfactory. In the case of such softwoods as basswood very satisfactory material may be obtained from the damp wood with a small sharp plane. However, with most woods the use of the plane is not to be recommended since the resulting material is not of uniform thickness. For the purpose of chlorination the thickness of the material should not exceed 0.005 in. The best method of attaining this result for the harder woods is by the use of a cabinetmaker's scraper, which is a thin rectangular steel plate with square edges. Unfortunately, considerable skill is required for the proper use of this tool. Unless the user has had a large amount of experience, dust rather than a fine excelsior is obtained. The wood to be scraped should contain 25 to 30 per cent moisture. Nearly as good results are obtained with broken window glass as with the scraper. Certain soft woods do not scrape well, so that it is necessary in some cases to choose a method by trial. All the material that passes through a 40-mesh sieve should be rejected.

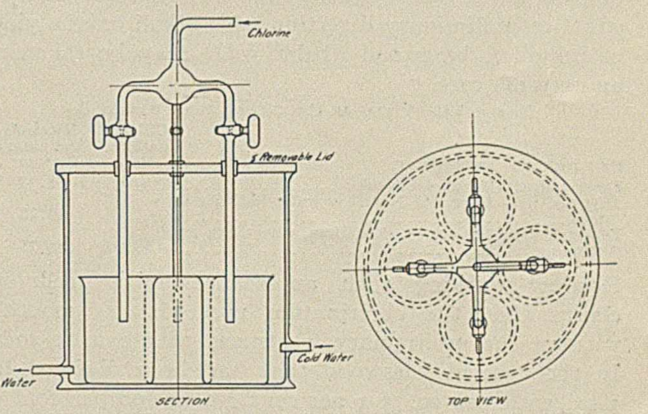
The material placed in beakers was chlorinated in a specially designed apparatus of the type shown in the accompanying figure. This apparatus has several advantages: four samples can be chlorinated at the same time; the material is maintained at a low temperature by means of a constant stream of cold water; the stream of chlorine gas can be regulated by means of the stopcocks; and the excess chlorine is carried off by the water.

At intervals of 6 to 7 minutes the contents of the beakers were stirred with a glass rod and the position of the beakers changed in order to insure uniformity of chlorination. The chlorination was limited to half-hour periods or fractions thereof in the results herewith. A first chlorination of one hour, as usually recommended, is too long in some cases. The number of chlorinations or fractions thereof necessary to obtain the cellulose practically free from lignin were as given in Table VI. The conifers are decidedly more resistant to the action of chlorine than the broad-leaved trees.

Table VI—Chlorination Necessary to Obtain Lignin-Free Cellulose

Douglas Fir	White Spruce	Longleaf Pine	Western Larch	Yellow Birch	Basswood	Sugar Maple
4 to 5	4 to 5	4	5	2 to 3	2	2 to 3

To determine the freedom of the cellulose from lignin the yellow color produced by chlorine and particularly the pink to purple-red color which the lignin chloride gives with sodium sulfite were found very sensitive. The latter reaction is considerably more sensitive for broad-leaved woods than for the conifers. Mäule's[1] reaction that has been considered a very sensitive test for lignin is the following: the fibers are covered with a dilute solution of potassium permanganate and allowed to stand for several minutes; the permanganate is then washed out and the manganese dioxide adhering to the fibers is removed by allowing them to stand in contact with hydrochloric acid of sp. gr. 1.06. After solution of the manganese dioxide, the fibers are thoroughly washed with water and then a few drops of ammonia are added. In case lignin is present a deep red coloration is produced. This reaction was found to be sensitive for the hardwoods but in the case of the conifers no red color was ever obtained. When the original wood is given the above treatment, it is colored dark brown. As the chlorina-



Apparatus for Chlorinating Wood

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[1] "Bestimmungsmethoden der Cellulose," Berlin (1910).

[2] *J. Am. Chem. Soc.*, **29** (1907), 1125.

[1] *Beitr. z. wissensch. Botanik*, **4** (1900), 166.

tion proceeds, and more and more lignin is removed, the Mäule reaction changes from a brown to a dirty brownish gray that is not at all characteristic. Besides being indefinite, this reaction requires considerable time and manipulation. The reaction of the wood fibers with ferric chloride and potassium ferricyanide[1] has a certain qualitative value, but quantitative determinations gave inconsistent results.

The yields of cellulose do not apply to material that is absolutely free from lignin. There are invariably present small fragments of wood that are only partially reduced. However, in obtaining the cellulose absolutely free from lignin the error involved by destruction of the cellulose is out of all proportion to that caused by the presence of the small amount of lignin.

In the original method of Cross and Bevan[2] the ligneous material was boiled for half an hour with 1 per cent NaOH previous to chlorination. Also the chlorinated fibers were boiled for 5 min. with a solution of sodium sulfite containing 0.2 per cent NaOH. Renker[3] states that in both cases the alkali attacks the cellulose and reduces the yield. These statements have been confirmed.

A number of experiments were made to determine the effect of alkali on the yield of cellulose. In the results that follow only the mean results of duplicate determinations are given. As an example of the effect of preliminary digestion with alkali on the yield of cellulose, basswood (Table VII) may be taken as an extreme case.

Table VII—Yield of Cellulose from Basswood

Preliminary Treatment:	Chlorinations Hrs.	Yield of Cellulose Per cent
Extracted with Alcohol-Benzol	1/2, 1/2	62.92
Digested with 50 cc. 1% NaOH in water bath for 30 min.	1/2, 1/2	59.78
Digested with 100 cc. 1% NaOH in water bath for 60 min.	1/2, 1/6	58.25

Renker[4] working with unbleached sulfite cellulose claims that the chlorination proceeds as smoothly without the preliminary treatment with caustic soda. In the case of basswood that had been digested for 1 hr. with 100 cc. of 1 per cent NaOH, 90 per cent of the fibers showed no lignin reaction (Mäule) after the first half hour's chlorination and an additional chlorination of 5 min. was sufficient to remove the lignin entirely. In all cases the preliminary digestion with alkali was found to assist in the removal of the lignin, the time of chlorinating being perceptibly shortened. This is especially true of hardwoods. In the case of the conifers the effect is less evident. As a general rule, however, the treatment results in an appreciable reduction in the yield of cellulose.

Yellow Birch

Preliminary Treatment:	Chlorinations Hrs.	Yield of Cellulose Per cent
Extraction with Alcohol-Benzol for 5 hrs.	1, 1/2, 1/2, 1/2	61.33
Digested with 100 cc. 1% NaOH in Water Bath for 30 min.	1, 1/2, 1/4	60.69

The effect of the alkali on hardwoods is appreciable even in the case of fairly thick shavings, as is evident in the case of the above experiments in which yellow birch was used.

Douglas Fir was digested both with acids and alkali, the result being that the yield of cellulose was lowered by the acid hydrolysis but not appreciably affected by the alkali in this case.

Douglas Fir

Preliminary Treatment:	Cellulose Per cent
Extracted with Alcohol-Benzol	62.90
Digested with 50 cc. 1% NaOH on Steam Bath for 1/2 hour	63.10
Boiled with 100 cc. 2.5% H_2SO_4 for 3 hours	55.12
Boiled with 100 cc. 2.5% H_2SO_4 for 3 hours (material that would pass through a 40-mesh sieve was used in this case)	49.00

The effect of addition of alkali to the sodium sulfite solution in the removal of the lignin from Douglas Fir is also illustrated by the following:

Douglas Fir

Remarks:	Chlorinations Hrs.	Cellulose Per cent
All samples were first extracted with Alcohol-Benzol:		
a Sulfite Solution without Alkali	1, 1/4, 1/4	60.53
b Sulfite Solution with 0.2 per cent NaOH	1, 1/4, 1/4	59.35

Since sodium sulfite gives decidedly alkaline solutions owing to hydrolysis it appeared probable that sufficient alkalinity might be developed to attack the cellulose. Bleached sulfite pulp that had been thoroughly washed with hot water and then dried at 60° was employed. In all cases 2 g. of the pulp were heated with 100 cc. of 2 per cent sodium sulfite solution. The moisture in the pulp was determined by heating a separate sample in the air oven at 105° and the samples treated with sodium sulfite were reduced to the dry weight by calculation. After treatment with the sulfite solutions the pulp was washed with dilute acetic acid, hot water, alcohol, and ether in succession and finally dried at 105° for 2 hrs.

Bleached Sulfite Pulp

Manner of Heating:	Period of Heating Hrs.	Loss in Weight Per cent
a Boiling Water Bath	1/2	1.99
b Steam Bath	1	2.03
c Steam Bath	2	2.15
d Sample *b* Second Treatment on Steam Bath	2	1.24
e Sample *d* Third Treatment on Steam Bath	2	1.26
f One of samples *e* given Fourth Treatment on Steam Bath	2	1.16
g Second sample *e* treated with 2% $NaHSO_3$ on Steam Bath	2	0.57

The loss by the first treatment, approximately 2 per cent, may have been due to the presence of oxycellulose, but the total loss of Sample *b* (5.69 per cent) by the four treatments can hardly be attributed to this one cause.

Some experiments were made in which the sodium sulfite solution containing the chlorinated wood was boiled for 5 min. In one series the boiling solution was maintained saturated with SO_2 by passing in the gas. In some cases this gave an increased yield of cellulose while in others the yield was slightly lowered.

A marked difference in the rate of removal of the lignin from spruce was noted when a 2 per cent $NaHSO_3$ solution was used in place of a 2 per cent Na_2SO_3 solution. After each chlorination the fibers were heated with 100 cc. of the sulfite solutions in a water bath for half an hour.

White Spruce

Treatment:	Half-hour Chlorinations	Cellulose Per cent
a With Sodium Sulfite	3 1/6	63.50
b With Sodium Bisulfite	6	62.80

After three chlorinations *a* did not show the Mäule reaction but chlorine colored the fibers a slight yellow. They were accordingly given an additional chlorination of 5 min. In the case of *b*, after the third chlorination the Mäule reaction was brown; after the fifth chlorination the Mäule reaction was pale saffron,

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[1] Cross and Bevan, "Cellulose," p. 124; Sherman, *J. Am. Chem. Soc.*, **19** (1897), 304.

[2] "Cellulose," p. 95.

[3] "Bestimmungsmethoden der Cellulose," p. 44.

[4] *Loc. cit.*, p. 44.

and the chlorine reaction decidedly yellow; after the sixth chlorination there was no Mäule reaction but chlorine gas gave a pronounced yellow color. The fibers were bleached with potassium permanganate, washed as usual and dried. The cellulose obtained with sodium bisulfite was a dark grayish yellow. The sodium bisulfite solutions were never colored more than a pale yellow. This fact, combined with the large number of chlorinations required, shows that it is essential to have a slightly alkaline reaction in order to render the method efficient.

ACTION OF THE CHLORINE—The rate at which the chlorine is passed into the beaker containing the ligneous material does not appear to have received special attention. Cross and Bevan state that a "slow stream" should be used. It was found that the amount of lignin removed was almost directly proportional to the rate of the introduction of the chlorine as will be seen from the following:

White Spruce		Yellow Birch	
Chlorine Bubbles per Min.	Per cent Loss in Weight	Chlorine Bubbles Per Min.	Per cent Loss in Weight
52 to 53	12.84	44 to 45	15.31
77 to 78	16.31	72 to 74	21.57
120 to 122	22.58	120 to 122	26.11

According to Cross and Bevan the action of the chlorine is mainly additive and substitutive. It is difficult to conceive of the reaction being other than mainly an oxidation. This is also borne out by the considerable excess of chlorine obtained as hydrochloric acid over the chlorine combined with lignin as found by Heuser and Sieber.[1]

The presence of a chlorine carrier such as iodine appears to be without effect. Yellow birch wood was saturated with alcohol containing a few crystals of iodine and after standing a few minutes washed with cold water. After chlorinating at the rate of 64 to 65 bubbles per minute for 30 min. the wood lost 19.83 per cent by weight. When this loss is compared with the losses in the above table it is evident that the iodine did not improve the reaction.

There is an advantage in using a rapid stream of chlorine gas during the first half-hour chlorination, but during succeeding chlorinations, except possibly the second in some cases, the rate should not exceed about 40 bubbles of chlorine per minute. The loss in weight during chlorination was followed by using the regular method for determining cellulose. After heating with sodium sulfite solution the residue was washed with acetic acid, alcohol, and ether. After drying and weighing, the chlorination was repeated. The results obtained appear in Table VIII.

TABLE VIII—ACTION OF CHLORINATION

Western Larch 40 Chlorine Bubbles per Minute			Yellow Birch			
			35 to 40 Bubbles per min. after first Chlorination		70 Bubbles per Minute throughout	
Chlorination Period	Per cent Loss	Mäule Reaction	Chlorination Period	Per cent Loss	Chlorination Period	Per cent Loss
1st $^1/_2$ hr.	29.05	Brown	1st $^1/_2$ hr.	27.71	1st $^1/_2$ hr.	21.57
2nd $^1/_2$ hr.	8.82	Brown	2nd $^1/_2$ hr.	8.08	2nd $^1/_2$ hr.	12.73
3rd $^1/_2$ hr.	2.65	Saffron	3rd $^1/_2$ hr.	2.73(a)	3rd $^1/_2$ hr.	11.79(a)
4th $^1/_2$ hr.	1.23	None	4th $^1/_2$ hr.	1.10	4th $^1/_2$ hr.	4.38
5th $^1/_2$ hr.	0.57	None	Next hr.	1.35	5th $^1/_2$ hr.	1.57
Next hr.	0.94	None	Next hr.	1.45	.....	...

(a) Residual cellulose free from lignin.

The greatest difficulty of the present method of estimating cellulose lies in judging just how long the final chlorination should be in order to remove all the lignin and not attack the cellulose. It is probable that the attack of the cellulose consists in the formation of oxycellulose, and possibly hydrocellulose, which are soluble in the sodium sulfite solution. However, Heuser and Sieber[1] by using methylene blue found that practically no oxycellulose was formed up to the point where all the lignin was removed.

For analytical purposes formation of degradation products of the cellulose would be of little consequence providing that they did not pass into solution. All attempts to accomplish this end were negative. The chlorinated fibers, when extracted with 95 per cent alcohol in a Soxhlet extractor to remove the lignin chloride, still lost 1.5 to 2.0 per cent by weight for each half-hour chlorination following the point where the lignin reaction ceased.

The accuracy attainable in determining cellulose is dependent largely on following the same procedure exactly with duplicate determinations. Two half-hour chlorinations followed by treatment with sodium sulfite are by no means equivalent to a one-hour chlorination. Unfortunately check analyses do not necessarily indicate that the correct amount of cellulose has been secured. The results obtained by different chemists, even if following the same directions, may be considered satisfactory if they check within one per cent. The results attainable by experience may be judged from Table IX, the figures being taken at random.

TABLE IX—PER CENT CELLULOSE—NOT ASH-FREE

Sample No.:	1	2	3	4
Sugar Maple	60.75	61.64	60.10	60.58
	60.82	61.70	60.31	60.37
Western Larch	57.82	58.66	61.20	53.34
	58.67	58.77	60.82	53.28

The conifers invariably are harder to reduce than the hardwoods and less satisfactory checks may be expected. In spite of the fairly good agreement it is doubtful if the results may be considered as having an accuracy greater than one per cent.

The cellulose content of the various species is quite uniform, especially when the cellulose is calculated on the weight of the wood free from material soluble in hot water and ether. On this basis the figures in Table X were obtained:

TABLE X

Softwoods	Per cent Cellulose		Hardwoods	Per cent Cellulose	
Western Larch	66.40	Mean 65.92	Sugar Maple	63.43	Mean 64.26
Longleaf Pine	67.20		Yellow Birch	64.38	
Douglas Fir	66.30		Basswood	64.97	
White Spruce	63.79				

The conifers appear to contain more cellulose than the hardwoods. Occasionally the cellulose content of a tree will be unusually high or low. For example, the cellulose content of basswood No. 3* was only 54.42 per cent; microscopic examination showed no evidence of fungous attack; the analysis was repeated with the same result. On the other hand, the cellulose content of longleaf pine No. 4* is high by several per cent. It was thought that by subtracting the per cent soluble in ether and hot water from a fixed number (70 in the case of longleaf pine) the cellulose content of the various species could be approximated

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[1] *Z. angew. Chem., Aufsatzteil.*, **26** (1913), 805.

[1] *Loc. cit.*

with fair accuracy. That marked exceptions may exist is shown by the following:

LONGLEAF PINE Sample No.:	11	12	13	20
Hot water-soluble	8.20	6.03	6.78	7.57
Ether-soluble	6.70	6.70	2.65	9.23
Cellulose	55.02	57.23	61.09	59.31
Total Percentages	69.92	69.96	70.52	76.11

The analysis of Sample No. 20 was checked by several determinations but the cellulose content remained about 5 per cent higher than the average of the remaining three samples, the calculations being based on the wood free from materials soluble in ether and water.

ACID HYDROLYSIS—The method of determining the acids formed by hydrolysis was adopted after considerable experimentation and, while somewhat tedious, very accurate results may be obtained. Duplicate determinations should agree within 0.1 per cent. It is absolutely necessary to use low temperatures and pressures to prevent decomposition of the carbohydrates, etc., by the sulfuric acid before all the volatile acids are removed. The distillate obtained in a blank determination remained pink for 30 seconds, when phenolphthalein was used as the indicator and one drop of $N/10$ NaOH was added, showing that no sulfuric acid had been carried over. The distillate consists mainly of acetic acid. The volatile acids formed from a sample of yellow birch amounted to 4.0 per cent, calculated as acetic acid. After heating on the steam bath with mercuric oxide to destroy formic acid, 3.6 per cent of acid was recovered. It is probable that the formic acid results from decomposition of carbohydrates and not from formyl groups.

It is not yet certain that all the acetic acid is formed by hydrolysis of acetyl (CH_3CO—) groups. A 2-g. sample of yellow birch by digestion with 100 cc. of 2.5 per cent H_2SO_4 lost 31.41 per cent by weight, showing that a decided decomposition is produced. It is accordingly not impossible that a portion of the acetic acid is formed from acetic acid residues (—$CH_2.CO$—). The effect of the strength of acid on the yield of volatile acids from yellow birch was the following:

Strength of Sulfuric Acid (Per cent)	2.5	5.0	10.0
Yield of Acetic Acid (Per cent)	3.99	4.31	4.53

Small amounts of acetic and formic acids appear to be present in some woods in the free state or at least in very feeble combination. Guijo, a Philippine wood, has a marked corrosive action on metal fastenings. Investigation showed that about 0.2 per cent of acetic and formic acids could be leached from guijo sawdust with cold water. The silver salts in aqueous solution were heated on the steam bath to decompose the silver formate. After filtering off the metallic silver the filtrate deposited beautiful crystals of silver acetate on cooling. The crystals were analyzed as follows:

0.2712 g. silver salt gave 0.1746 g. Ag = 64.38 per cent Ag.

Silver acetate, $CH_3.COOAg$, requires 64.64 per cent Ag.

METHOXY GROUPS—The efficiency of the apparatus and the quality of the acid were tested by using a compound, vanillin, the methoxy content of which is known: Vanillin, $C_7H_5O_2.OCH_3$, requires 20.40 per cent CH_3O—. Found, 20.26 per cent CH_3O—.

Maple gave the highest yield of methoxy groups. The yields of methyl alcohol obtained by destructive distillation are much lower than would be expected from the methoxy numbers.

YIELD	Birch	Maple	Western Larch
Methyl Alcohol (Per cent)	1.53(*a*)	1.94(*a*)	0.65
Methoxy (Per cent)	6.07	7.25	4.95

(*a*) Palmer, *Bull.* **129**, U. S. Dept. Agr., Forest Service.

As previously mentioned, the low yields are probably due to the formation of methyl ethers.

FOREST SERVICE
FOREST PRODUCTS LABORATORY
MADISON, WISCONSIN

ON THE TOXICITY TO A WOOD-DESTROYING FUNGUS OF MAPLEWOOD CREOSOTE AND OF SOME OF ITS CONSTITUENTS AND DERIVATIVES, TOGETHER WITH A COMPARISON WITH BEECHWOOD CREOSOTE[1]

By ERNEST J. PIEPER, S. F. ACREE AND C. J. HUMPHREY

Received March 7, 1917

Considerable interest has been shown the past few years in the industrial use of wood creosotes. At the present time they are on the market to a certain extent as wood preservatives, but their high antiseptic properties render them most useful in general sanitation work and for special technical purposes as well. For example, they dissolve rosin and linseed oil soaps and thus form disinfectants which with water give emulsions having coefficients three to five times, or more, that of phenol. They also have a great advantage over coal-tar creosotes in that they do not inflame the skin and are not toxic or poisonous to humans or animals. Indeed, hardwood creosote, U. S. P., and guaiacol have long been administered as internal medicines, whereas carbolic acid is a well-known poison.

As toxicity to fungi and bacteria is one of the very important attributes in the utilization of the oils or their several constituents, the present study has been undertaken to throw light upon their behavior in this respect. These creosotes are by-products in the destructive distillation of wood and can be produced at a moderate cost. The main products of the distillation are charcoal, methyl alcohol, and acetic acid.

The toxicity of beechwood creosote, both crude and refined,[2] has been determined by R. M. Fleming and C. J. Humphrey,[3] of this laboratory, and found to be much greater, 2 to 4 times, than that of the coal-tar creosote. The toxicity of maplewood creosote and of its constituents and derivatives has been found in the present investigation to be equally as high.

METHOD

Toxicity studies at the Forest Products Laboratory have largely been confined to the petri-dish method;[4]

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[1] The present paper is one of four prepared by the junior author in partial fulfilment of requirements for the degree of Doctor of Philosophy in the University of Wisconsin.

[2] Data on refined product not yet published.

[3] THIS JOURNAL, **7** (1915), 652.

[4] Humphrey and Fleming, U. S. Dept. Agr., *Bull.* **227** (1915), for general method.

that is, the preservative and nutrient culture medium are mixed, poured into a petri-dish and inoculated with a wood-destroying fungus. The rate of growth is compared with a check petri-dish containing only the agar culture medium. This method gives approximate toxicity values in a short period of time and has been followed in the present study with minor variations in the technique.

An electrical stirring device,[1] specially developed for toxicity investigations and other lines of work where uniform and very finely divided emulsions or mixtures are essential, was used in the present experiments (Figs. I and II). The oil-agar emulsion thus prepared was made permanent by quickly cooling it in the petri-dish over ice, by which method the small oil globules were imprisoned in the solidifying agar, thus preventing the oil from separating out as a layer on the surface of the medium.

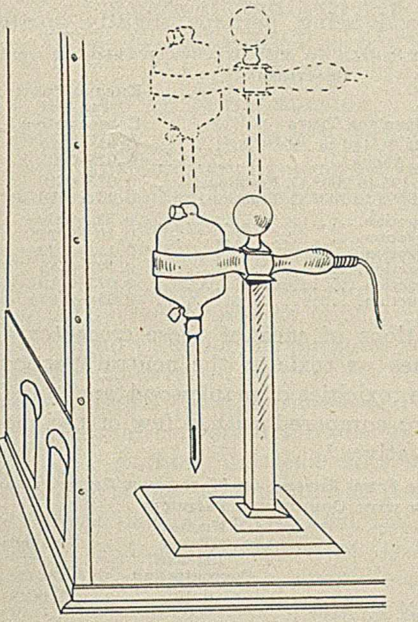

Fig. I—Electrical Stirring Device Operated in a Sterilized Glass Case to Avoid Contaminations

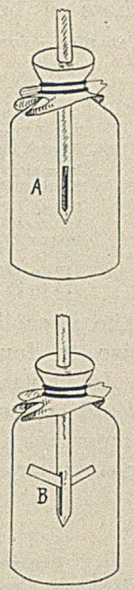

Fig. II—Bottles with Shaft of Stirring Device Inserted
(A) Shaft at Rest with the Arms Collapsed
(B) Horizontal Position of the Arms when in Motion

The culture medium consisted of the following:

Extract of 1 lb. lean beef in distilled water	1000 cc.
Löfflund's malt extract	25 g.
Agar-agar	15 g.

The medium was filtered, but the reaction was not adjusted; it was somewhat acid, around +32 Fuller's scale.

Of the above medium 17.5 cc. were measured from a graduated pipette into 25 cc. glass-stoppered bottles; 0.5 cc. of this was extra to compensate for the amount adhering to the bottles after pouring, leaving 17 cc. to be mixed with the preservative.

The preservative was weighed into 50 cc. glass-stoppered bottles and then diluted to 3 cc. with distilled water. The concentration of the preservative was based on the actual weight in grams in 20 cc. of the agar preservative mixture.

Before sterilizing, the stoppers of the agar and preservative bottles were covered with stopcock grease and capped with muslin fastened around the neck of the bottle by rubber bands. The bottles were then sterilized in the autoclave at 100° C. for 30-min. intervals on three successive days. A bottle each of agar and preservative was then transferred to a sterile culture case and the agar culture solution was poured into the preservative bottle, the mouth of which was then covered with a sterile muslin cap in place of the glass stopper. Two slits at right angles were burned into the center of the cloth through which the stirring-rod of the electric mixer was inserted. This was then set in motion and the contents stirred until the agar was about to solidify. The resulting finely divided mixture was poured into a petri-dish which had been previously cooled over ice. It was then returned to the ice until the contents had solidified.

After all the petri-dishes had been prepared in this way they were returned to the culture case and after their temperature had risen to that of the surrounding air they were inoculated at the center with a transfer of mycelium 6 mm. square cut from petri-dish cultures of *Fomes annosus* Fr. two to three weeks old. For each set of tests a check culture on the nutrient agar was prepared at the same time. The dishes were then placed in an incubator and held approximately at 25° C. for 6 weeks, the extent of growth being noted each week.

SUBSTANCES TESTED

The maplewood creosote used in the tests was prepared[1] by distilling crude tar, obtained from a commercial company in Michigan, from an iron retort. After further distillation from a glass apparatus a fraction distilling between 195 and 265° C. was collected.[2] This was a clear, light lemon-yellow oil whose specific gravity was 1.04 at 21° C.

The toxicity of this oil and the various portions tested is shown in Table I, the duplicate tests being represented by (A) and (B), respectively.

In order to determine if the phenols, or so-called wood tar acids, are the principal toxic constituents, the maplewood creosote was treated two or three times with an equal volume of 15 per cent sodium hydroxide solution in a separatory funnel and the neutral layer separated from the alkali-soluble portion. The neutral oil obtained was washed with water to free it from alkali. It represented 24 per cent of the total creosote. The neutral oil was redistilled and the fraction between 195 and 260° C. collected. This was almost colorless; its specific gravity was 0.97 at 20° C.

The sodium hydroxide solution of the maplewood creosote was then treated with dilute sulfuric acid until no more oil separated on shaking. This oil was separated and washed with water until free of sulfuric acid. It represented 68 per cent of the total creosote, 8 per cent of the creosote being lost in washing. The oil was redistilled and the fraction between 195 and

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[1] This apparatus will be described in a forthcoming article by Ruby J. Tiller and S. F. Acree.

[1] For full description of the chemical work on this material see article by the writers published in This Journal, **9** (1917), 462.

[2] Similar work on a number of creosotes prepared from other commercial maplewood tars shows all of them to have the same general composition, and the results secured in this paper can, therefore, be considered as fairly representative of the American maplewood creosotes.

TABLE I—TOXICITY OF MAPLE CREOSOTE AND ITS FRACTIONS: RESULTS GIVEN IN TERMS OF RADIAL GROWTH OF FUNGUS IN MILLIMETERS

PRESERVATIVE USED	CONCENTRATION (Per cent)	First Week A	First Week B	Second Week A	Second Week B	Third Week A	Third Week B	Fourth Week A	Fourth Week B	Fifth Week A	Fifth Week B	Sixth Week A	Sixth Week B	KILLING POINT
Maplewood Creosote...	0.05	2–5	0	11–15	1–4	23–29	13–28	36–38	22–28	39–44	32–45	*	*	0.10% Solution. Equivalent to 0.0624 Lb. per cu. ft.
	0.10	0	0	0	0	0	0	0	0	0	0	0	0	
	0.20	0	0	0	0	0	0	0	0	0	0	0	0	
	Check	23–30	25–30	*	*	...	...	...	...	...	...	...	...	
Neutral Oil...........	0.05	0	1–3	18–22	19–24	38–42	39–44	*	*	...	...	...	...	0.15–0.20% Solution. Equivalent to 0.0936–0.1248 Lb. per cu. ft.
	0.10	0	0	12–13	11–12	31–32	28–31	*	*	...	...	...	...	
	0.15	0	0	0	0	0	0	0	0	0	0	2–3	1–4	
	0.20	0	0	0	0	0	0	0	0	0	0	0	0	
	Check	24–25	22–26	*	*	...	...	...	...	...	...	...	...	
NaOH-Soluble Portion	0.025	7–10	5–7	12–14	14–16	24–30	30–35	32–42	38–43	*	*	...	...	0.05% Solution. Equivalent to 0.0312 Lb. per cu. ft.
	0.05	0	0	0	0	0	0	0	0	0	0	0	0	
	0.75	0	0	0	0	0	0	0	0	0	0	0	0	
	Check	25–28	21–28	*	*	...	...	...	...	...	...	...	...	
195–230° C. Fraction...	0.05	2–4	0	20–25	12–13	38–39	28–30	*	39–40	...	*	...	...	0.10% Solution. Equivalent to 0.0624 Lb. per cu. ft.
	0.10	0	0	0	0	0	0	0	0	0	0	0	0	
	Check	24–25	25–27	*	*	...	...	...	...	...	...	...	...	
230–265° C. Fraction...	0.05	0	0	0	0	10–11	9–10	17–22	15–19	22–28	20–23	25–33	24–30	0.10% Solution. Equivalent to 0.0624 Lb. per cu. ft.
	0.10	0	0	0	0	0	0	0	0	0	0	0	0	
	Check	...	...	*	*	...	...	...	...	...	...	...	...	

* Surface covered.

255° C. collected. A light yellow oil with a specific gravity of 1.052 at 20° C. was obtained.

The results show that a 0.05 per cent emulsion with the alkali-soluble wood tar acid or phenols prevents the growth of the fungus and is three to four times as efficient as the neutral oils and about seven times as efficient as the coal-tar creosote used for comparison. It is because of this high toxic coefficient of the wood tar phenols and the large proportion, 70 to 85 per cent, present in the hardwood creosote that this oil has such an advantage over coal-tar creosote.

In order to determine whether there was any difference in the toxic value of the higher and lower boiling fractions of maplewood creosote, the refined creosote was redistilled and the fractions 195–230° C. and 230–265° C. separated. The specific gravity of the fraction 195–230° C. was 1.022 at 22° C., while that of the 230–265° C. fraction was 1.043.

A concentration of 0.10 per cent of either fraction entirely prevents the growth of the fungus and a 0.05 per cent emulsion of the 230–265° C. fraction seems to be slightly more toxic than a 0.05 per cent emulsion of the 195–230° C. fraction. As the more toxic phenols occur in the 195–230° C. fraction to the extent of only 65 to 70 per cent and in the 230–265° C. fraction to the extent of 80 to 85 per cent, the higher toxicity of the 230–265° fraction is to be expected.

In all the previous tests those petri-dishes upon which no growth occurred after six weeks were kept in the incubator for two weeks longer. There was no indication of further growth in any case.

Table II gives a summary of the results secured on all the samples.

TABLE II—TOXICITY OF MAPLEWOOD CREOSOTE AND ITS DERIVATIVES: SUMMARY

PORTION USED	KILLING POINT Per cent Concentration	Pounds per cu. ft.
Total Creosote..........................	0.10	0.0624
Neutral Portion.........................	0.15–0.20	0.0936–0.1248
Alkali-soluble (Phenol) Portion...........	0.05	0.0312
195–230° C. Fraction.....................	0.10	0.0624
230–265° C. Fraction.....................	0.10	0.0624

For comparison with the preceding data we give in Table III the results[1] obtained by Ruth M. Fleming and C. J. Humphrey on the toxicity of samples of beechwood creosote.

It is seen from Table II and III that the corresponding fractions of maplewood creosote and beechwood creosote have nearly the same toxicities, those of beechwood being perhaps slightly greater.

TABLE III—TOXICITY OF SAMPLE NO. 3359 BEECHWOOD CREOSOTE AND ITS FRACTIONS

PORTION USED FOR TESTS	KILLING POINT Per cent Concentration
Total Distillate (B. P. 180 to 260° C.).........	0.05 –0.10
Phenol Portion of Above......................	0.025–0.05
Phenol Portion of 195 to 200° C. Fraction......	0.075–0.10
Neutral Portion of 195 to 200° C. Fraction......	0.20 –0.50
180 to 185° C. Fraction.......................	0.25 –0.30
185 to 190° C. Fraction.......................	0.20 –0.25
190 to 200° C. Fraction.......................	0.15 –0.20
200 to 215° C. Fraction.......................	0.10 –0.15
215 to 225° C. Fraction.......................	0.075–0.10
225 to 255° C. Fraction.......................	0.05 –0.075

The phenolic portions of each of these creosotes are two to four times as toxic as the neutral portions.

In Table IV the toxicities of maplewood and beechwood creosotes are compared with a few of the more important preservatives.[1]

TABLE IV—TOXICITY TO *Fomes Annosus* OF MAPLEWOOD CREOSOTE COMPARED WITH COMMERCIAL PRODUCTS

PRESERVATIVE	KILLING POINT Per cent Concentration	Pounds per cu. ft.
Maplewood Creosote.....................	0.10	0.0624
Phenol Portion of Above.................	0.05	0.0312
Beechwood Creosote (Commercial Product Containing Pitch), Sample No. 3183....	0.12 –0.24	0.0749–0.1498
Beechwood Creosote, Refined...........	0.05 –0.10	0.0312–0.0624
Beechwood Creosote, Phenol Portion.....	0.025–0.05	0.0156–0.0312
Coal-tar Creosote(*a*)....................	0.35	0.2184
Sodium Fluoride........................	0.25	0.156
Zinc Chloride..........................	0.50	0.312

(*a*) Grade C of Amer. Ry. Eng. & Maint. Way Specifications.

CONCLUSIONS

From the preceding data it is evident that the representative American maplewood creosote used has a high toxic coefficient. Its general composition and toxicity are practically identical with those of beechwood creosote. These two American hardwood creosotes can, therefore, be used interchangeably in the mixtures often produced commercially from the mixed woods. The results indicate that the toxicity is due to the presence of carbocyclic compounds containing free hydroxyl groups in some cases and in others the corresponding methoxy groups. The alkali-soluble portion of the maplewood creosote is twice as toxic as the total creosote and four times as toxic as the neutral oil. The introduction of an alkyl radical in place of the hydrogen of the hydroxyl group evidently decreases the toxicity.

The killing points of the fractions 195–230° C. and 230–265° C. are practically the same as for the total

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[1] Manuscript covering these data has been prepared for publication; an article describing the methods of preparation of the samples will be published later by R. C. Judd, H. K. Smith and S. F. Acree.

[1] C. J. Humphrey and R. M. Fleming, U. S. Dept. Agr., *Bull.* **227**.

creosote. The toxicity of the higher boiling fraction, however, is slightly greater than that of the lower boiling fraction. This is due to the fact that the higher boiling fraction contains a greater percentage of phenols (80–85 per cent) than does the lower fraction (65–70 per cent). The lower fraction consists principally of the monohydroxy and dihydroxy phenols and their derivatives, the most important being cresol, guaiacol and creosol. The higher fraction consists principally of the trihydroxy phenols and their derivatives, the most important of which are the dimethyl-ether of pyrogallol and its homologues.

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THE DETERMINATION OF PHENOL IN CRUDE CARBOLIC ACID AND TAR OILS

By John Morris Weiss and C. R. Downs

Received March 27, 1917

INTRODUCTION

The purpose of this paper is to describe a method which is capable of giving accurate results for the determination of phenol in mixture with cresols, higher phenols, and neutral oils.

Many methods for the determination of phenol have been proposed. Most of these, however, are designed to serve as an assay of pharmaceutical phenol, and are of no value for the purposes which this paper is designed to cover.

Leube[1] used a colorimetric method with ferric chloride. Nietsch[2] converted the phenols into sulfonates, formed the barium and lead salts and made a determination of the metal content of these salts. Carre[3] converts the phenol to picric acid and makes a colorimetric determination. None of these methods are applicable for even an approximate assay of the real content of phenol in a crude acid or tar oil.

Warnes[4] gives three very empirical methods for testing carbolic acid. The results give only the melting point of a definite volume percentage fraction of the original acid, and though comparative between different samples, does not give any real indication of the actual amount of true phenol present.

Lunge[5] gives a table showing the lowering of the fusing points of mixtures of para-cresol and phenol according to tests made by Lunge and Zschokke. This table is patently useless for estimation purposes, as, in ordinary cases, meta- and ortho-cresols are also present in varying proportions, and affect the melting point to a very considerable degree.

Later, Lunge[6] gives a method of examining crude phenols (carbolic acid) for its yield of crystallizable phenol of a melting point from 15.5 to 24° C. This is known as the method of Lowe, and was in use among English tar distillers for some time. The crude acid is fractionally distilled and the melting point taken on a definite per cent by volume of the crude acids. The test is, however, more a specification for quality of crude acids, than a method of analysis. Lunge further states that for the purpose of "acquiring good practice in this method" mixtures are made of various content of pure crystallized carbolic acid and of cresols (preferably tar cresols, *i. e.*, a mixture of the three isomers) and the melting points of these mixtures are previously determined. No account is taken of variation in melting point due to variation in the interproportion of the three cresols. As Lunge himself points out, the relative proportions of the three cresols in coal tar are not constant. No attempt was made to take advantage of the differences in specific gravity between phenol and the cresols. The method is very crude and would give only very approximate results.

Stochmeier and Thurnauer[1] extract tar acids from carbolic oil, separate the hydrocarbons from the carbolate by steam distillation, liberate the tar acids and fractionate them. They then use Koppeschaar's bromination method[2] on the fractions and use special factors for the fractions containing the cresols. The titration of such mixtures is clearly not allowable as shown by Vaubel.[3]

In 1912 one of the authors[4] made, perhaps, the first published attempt to develop a rational method for the estimation of phenol in crude carbolic acid and tar oils, and it is along the general lines of that method that the method given in the present paper was developed. The method was fairly accurate, giving results within about 10 per cent of the phenol present. The range of the series of mixtures, however, was not close enough and the cresols available at that time were not quite so pure as to-day. Moreover, the recent increase in value of phenol has made a more accurate method necessary, whereas in the past, the close determination of phenol was not of prime importance. In the present method the procedure has been considerably changed and amplified, and a much more complete series of mixtures of known composition has been prepared and studied.

Very recently, while this paper was in course of preparation, René Masse and M. H. Leroux[5] published a method which is in some respect very similar to the method submitted by the authors of this paper. We have taken the liberty of reproducing the determination curve (see Fig. I) given by them, in this paper, and plotting on it points as found in the examination of our own series of mixtures. At some points the error can be very great. They use solidification point alone for their determination, and with variation of the proportion of the cresols among themselves, there might be an error of 2.6 per cent at the 75 per cent point, of 1.8 per cent at the 70 per cent point and of 4.4 per cent at the 65 per cent point. This will be seen more clearly by a reference to the curve. Masse and Leroux used a mixture of 50 per cent *o*-cresol and 50 per cent of a mixture of 60 : 40 of meta- and para-

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[1] *Dingler's polytech. J.*, **202**, 308.

[2] *Jahresber. der. chem. tech.*, **1897**, 1036.

[3] *Compt. rend.*, **1891**, 139.

[4] Warnes, "Coal-Tar Distillation," **1913**, 166–7.

[5] Lunge, "Coal-Tar and Ammonia," 5th Ed., **1916**, 277.

[6] *Ibid.*, 782.

[1] *Chem.-Ztg.*, **1893**, 119, 131.

[2] *Z. anal. Chem.*, **1886**, 233.

[3] *J. prakt. Chem.*, **48**, 74.

[4] *J. Franklin Inst.*, **1912**, 683.

[5] "J. Usines à Gas," abstracted in *J. Gas Lighting*, **136**, 587.

cresols, for the basis of the determination. In our work we used a 50 : 50 meta-para-cresol mixture (as naturally occurring in our oils) and varied the relation of the ortho-cresol to this. In the following table we have shown the variation in percentage between their curves and ours on the mixtures which correspond most closely to their proportions:

TABLE I

Per cent Phenol	Per cent o-Cresol	Per cent m50/p50 Cresol	Per cent Variation
95	2.5	2.5	1.0
90	5.0	5.0	0.5
85	7.5	7.5	0.2
80	10.0	10.0	0.0
75	12.5	12.5	0.3
70	15.0	15.0	0.5
65	17.5	17.5	0.2

This shows their curve to be quite correct if the proportion of ortho-cresol to meta- and para-cresol be equal but this is not generally true of tar oils. Therefore, the use of the simple solidification point curve is insufficient.

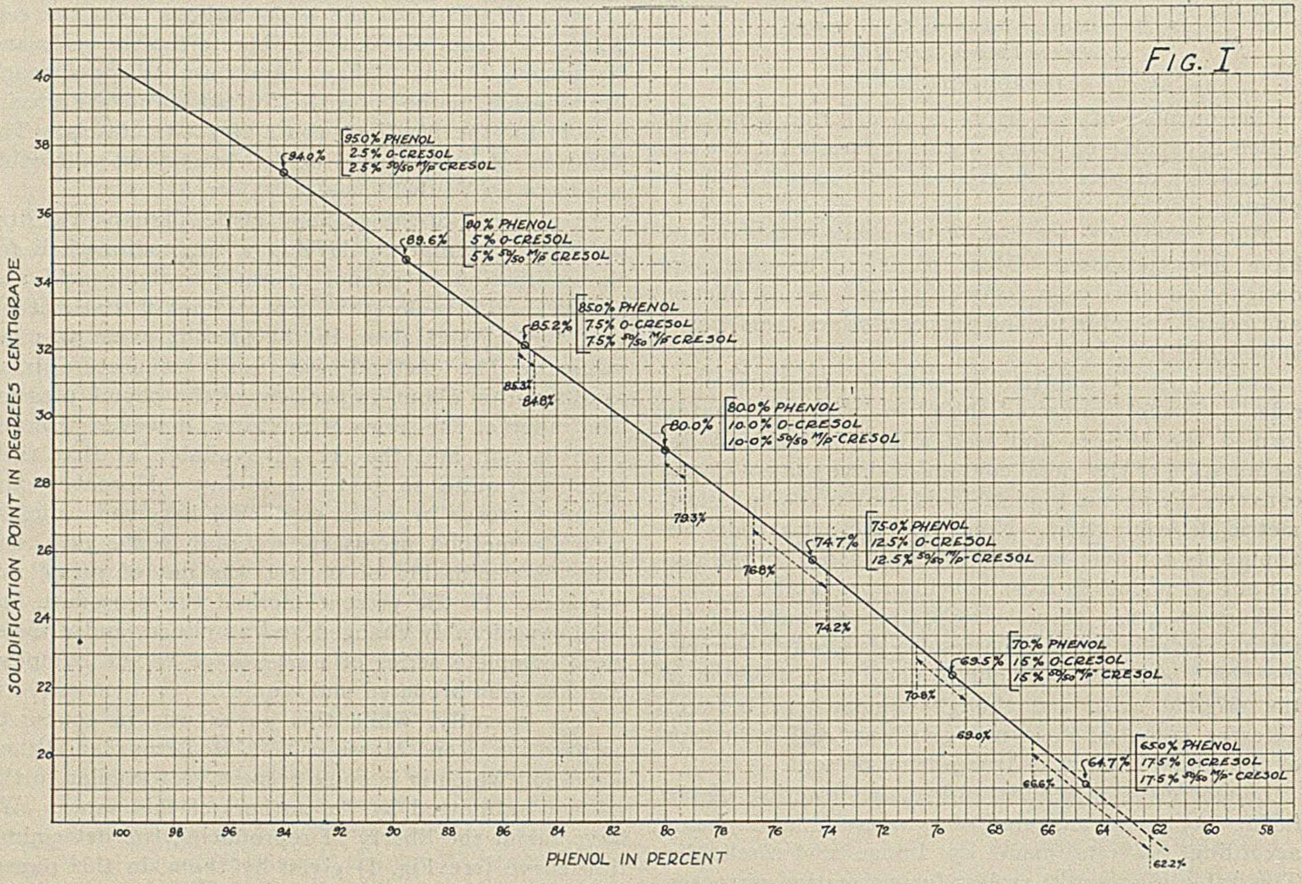
FIG. I

In general, their directions for the distillation of the liberated acids are fairly complete as is also their description of the method for determination of the solidifying point of the fractions. There are, however, a number of points in the handling of tar acids to remove neutral oils, on which their directions are not explicit, and here there is considerable chance for the introduction of serious errors. These errors may be further increased in the procedure for the determination of the phenol content of a tar oil.

The physical properties of the cresols and of phenol have been variously given in the literature (see Table II).

TABLE II

Beilstein(a)	Melting Point	Boiling Point	Specific Gravity
Phenol......	42.5 to 43° C.	178.5 to 184.1° C.	1.0906 at 0°
o-Cresol.....	30	190.8	1.0578 at 0°
m-Cresol....	3	202.8	1.0498 at 0°
p-Cresol.....	36	201.8	1.0522 at 0°
Olsen(b)			
Phenol......	42.5 to 43	182.6	1.0677 at 35°
o-Cresol.....	30	191	1.0511 at 35°
m-Cresol....	3 to 4	202	1.0390 at 35°
p-Cresol.....	36	202	1.0390 at 35°
Landolt-Börnstein(c)			
Phenol......	42.5 to 43	181.5	1.0489 at 50°/4°
o-Cresol.....	30	187.5 to 188	1.0427 at 23°/4°
m-Cresol....	About 4	200.5	1.035 at 13.6°/4°
p-Cresol.....	36	201.1	1.034 at 17.7°/4°

(a) Beilstein, "Hand. d. org. Chem.," 3rd Ed., **1893.**
(b) Olsen, "Chem. Annual," 3rd Ed., **1913.**
(c) Landolt-Börnstein, "Phys. Chem. Tab.," 4th Ed., **1912.**

There are numerous other single references dealing with single properties of these substances but it does not seem worth while to collate them here. We have ourselves made a considerable investigation on the true solidifying point of pure phenol and find it to be 40.5° C. In this we are confirmed by Eger.[1,2] The differences in the literature are probably due to early tests being made on small samples by the capillary tube method which might give higher figures than our solidifying point tests taken on a much larger bulk of material. It is also probable that some of the other melting points given are taken on the same basis although we have not investigated them to the same degree as we have in the case of phenol. A comparison

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[1] *Pharm.-Ztg.* **1903,** 210.

[2] *Chem.-Ztg. Rep.*, **1903,** 86.

of our method for solidifying point (see later) with the ordinary capillary tube melting point will indicate clearly how such differences might arise.

EXPERIMENTAL

The authors have attempted to give the experimental work in chronological order inserting from time to time the conclusions drawn. We have not attempted to give all the details of manipulation in the single experiments, but have collected these together at the end in a description of the method. We wish to express at this point our appreciation of the work of H. E. Lloyd and C. J. Downey who performed most of the laboratory work in connection with the problem.

A—MATERIALS USED FOR STANDARD MIXTURES

(1) *Phenol* was prepared in the plant and represented a close cut middle fraction from the final distillation of synthetic phenol. It did not require further purification by the laboratory.

(2) *Ortho-Cresol*—We selected from a works run an *o*-cresol fraction of high purity, but containing some neutral oil and naphthalene. It was dissolved in caustic soda solution, the carbolate freed from neutral hydrocarbons by steam distillation and the cresol precipitated again by dilute sulfuric acid. The liberated cresol was separated and fractioned through a 3-bulb LeBel-Henninger column, cutting so as to give a cresol of a boiling point of about 2° C.

(3) *Meta-Para-Cresol* was prepared in the same way as the *ortho*-cresol from another fraction obtained similarly. The final material was shown by the Raschig nitration test[1] to contain 49.3 per cent *meta*-cresol and 50.7 per cent *para*-cresol.

The three materials as finally used in the mixtures showed the constants given in Table III.

Table III

	Solidification Point	Specific Gravity at 25° C./25° C.	Boiling Point Range
Phenol	40.4° C.	1.0636(*a*)	180–182° C.
o-Cresol	29.0° C.	1.0444	189–191° C.
m-*p*-Cresol	Below 0° C.	1.0339	199–201° C.

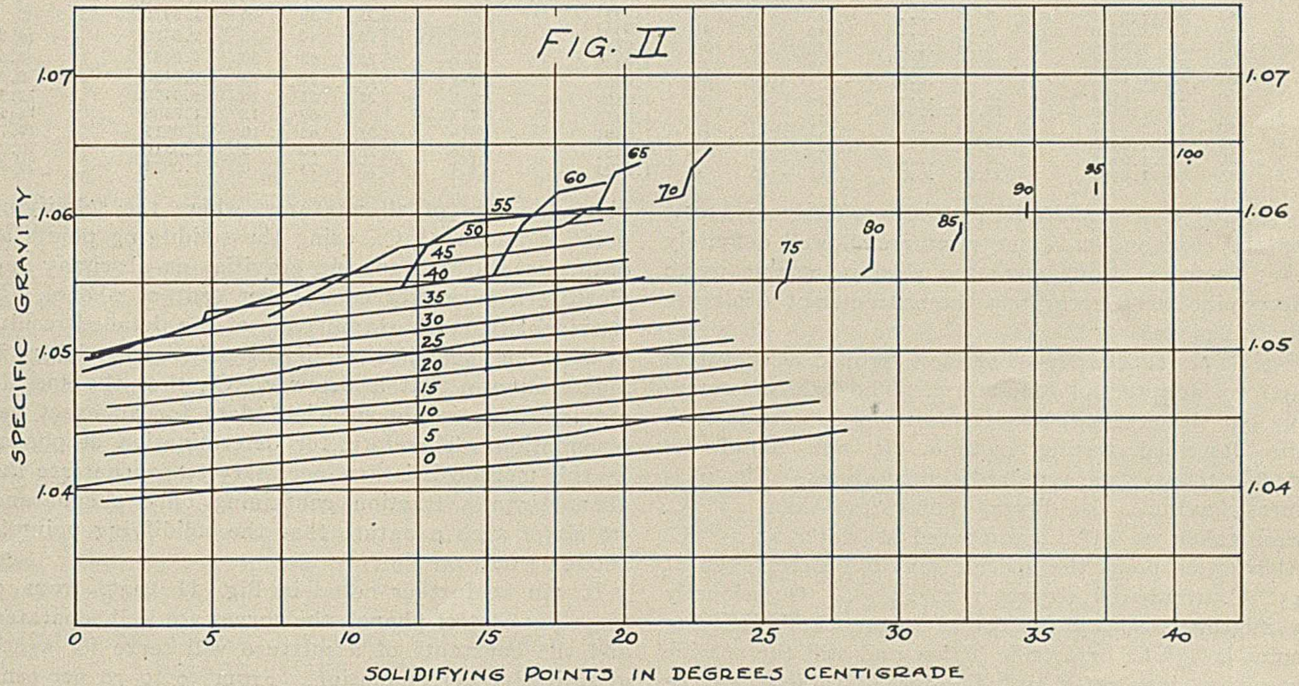
(*a*) Taken at 45°/45° C.

B—MIXTURE OF PURE MATERIALS

Twenty-one so-called series were made up using from 0 to 100 per cent of phenol, each series representing a 5 per cent phenol increment. In the series themselves the relative proportions of the *o*-cresol and the *m*-*p*-cresol were varied for every even 5 per cent. This involved making and testing a total of 231 mixtures.

The mixtures were made up by weight and the accuracy of the weighings was such as to make a maximum possible error in the final percentage of the material in the mixture of not over ±0.2 per cent. Proper precautions were taken to prevent absorption of water both in making up the mixtures and in subsequent handling of them. The mixtures were then subjected to various physical tests as described below.

FIG. II



NOTE: 45 and 40% coincide from 0 to 4.75° C. 35 and 40% coincide from 1.75 to 3.25° C.

(1) *Bulb Distillation*—The first few mixtures were submitted to this test, but it was soon found that sufficiently concordant results could not be obtained. It was necessary to have accurate readings of percentages distilled every even degree in order to differentiate between mixtures made over such a close range as these were. There were so many factors, such as barometric pressure, emergent stem corrections, thermometer lag, rate of distillation, effect of air currents, etc., any of which could produce an error as great or nearly as great as 1° C., that the authors decided it to be utterly impractical to use boiling point ranges as a method of differentiation. After a number of tests had indicated the hopelessness of this method of attack it was definitely abandoned.

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[1] *Z. angew. Chem.*, 1900, 760.

Table IV—Tests of Phenol-Cresol Mixtures: Phenol Varied from 0 to 100 Per cent of Mixture

Phenol	o-Cresol	m-p-Cresol	Sp. Gr. 25/25° C.	Solidification Point, ° C.	Ref. Ind. 50° C.
Series 1					
0	0	100	1.0339	Below —7	1.5281
0	5	95	1.0344	Below —7	1.5281
0	10	90	1.0349	Below —7	1.5281
0	15	85	1.0354	Below —7	1.5290
0	20	80	1.0359	Below —7	1.5290
0	25	75	1.0364	Below —7	1.5290
0	30	70	1.0369	Below —7	1.5291
0	35	65	1.0374	Below —7	1.5294
0	40	60	1.0379	—5.0	1.5299
0	45	55	1.0384	—3.0	1.5299
0	50	50	1.0394	1.0	1.5300
0	55	45	1.0399	4.5	1.5298
0	60	40	1.0404	7.2	1.5303
0	65	35	1.0409	10.1	1.5305
0	70	30	1.0414	12.8	1.5305
0	75	25	1.0419	15.8	1.5310
0	80	20	1.0424	18.5	1.5312
0	85	15	1.0429	21.5	1.5312
0	90	10	1.0434	23.9	1.5319
0	95	5	1.0439	26.0	1.5311
0	100	0	1.0444	29.0	1.5310
Series 2					
5	0	95	1.0347	Below —7	....
5	5	90	1.0352	Below —7	....
5	10	85	1.0357	Below —7	....
5	15	80	1.0363	Below —7	....
5	20	75	1.0370	Below —7	....
5	25	70	1.0377	Below —7	....
5	30	65	1.0384	Below —7	....
5	35	60	1.0391	Below —7	....
5	40	55	1.0397	—4	....
5	45	50	1.0403	0	....
5	50	45	1.0410	2.0	....
5	55	40	1.0416	5.3	....
5	60	35	1.0421	8.3	....
5	65	30	1.0427	11.2	....
5	70	25	1.0433	14.4	....
5	75	20	1.0440	17.4	....
5	80	15	1.0447	19.4	....
5	85	10	1.0453	22.4	....
5	90	5	1.0458	24.7	....
5	95	0	1.0464	27.0	....
Series 3					
10	0	90	1.0374	Below —7	
10	5	85	1.0380	Below —7	
10	10	80	1.0388	Below —7	
10	15	75	1.0394	Below —7	
10	20	70	1.0399	Below —7	
10	25	65	1.0405	Below —7	
10	30	60	1.0410	Below —7	
10	35	55	1.0415	—4	
10	40	50	1.0421	—2	
10	45	45	1.0427	1.2	
10	50	40	1.0433	3.8	
10	55	35	1.0438	7.2	
10	60	30	1.0444	10.0	
10	65	25	1.0450	12.8	
10	70	20	1.0456	15.8	
10	75	15	1.0462	18.2	
10	80	10	1.0467	21.0	
10	85	5	1.0473	23.6	
10	90	0	1.0479	25.8	
Series 4					
15	0	85	1.0393	Below —7	
15	5	80	1.0399	Below —7	
15	10	75	1.0405	Below —7	
15	15	70	1.0411	Below —7	
15	20	65	1.0417	Below —7	
15	25	60	1.0423	Below —7	
15	30	55	1.0429	—6.5	
15	35	50	1.0435	—4	
15	40	45	1.0441	0	
15	45	40	1.0447	2.9	
15	50	35	1.0453	6.2	
15	55	30	1.0459	9.0	
15	60	25	1.0463	11.7	
15	65	20	1.0469	14.8	
15	70	15	1.0475	17.3	
15	75	10	1.0481	19.9	
15	80	5	1.0487	22.6	
15	85	0	1.0493	24.4	
Series 5					
20	0	80	1.0414	Below —7	
20	5	75	1.0420	Below —7	
20	10	70	1.0426	Below —7	
20	15	65	1.0433	Below —7	
20	20	60	1.0438	Below —7	
20	25	55	1.0443	—5	
20	30	50	1.0448	—3	
20	35	45	1.0454	—2	
20	40	40	1.0461	1.2	
20	45	35	1.0467	4.6	
20	50	30	1.0473	7.8	
20	55	25	1.0479	10.4	
20	60	20	1.0485	13.4	
20	65	15	1.0491	16.2	
20	70	10	1.0498	18.8	
20	75	5	1.0504	20.8	
20	80	0	1.0509	23.8	
Series 6					
25	0	75	1.0428	Below —7	
25	5	70	1.0435	Below —7	
25	10	65	1.0442	Below —7	
25	15	60	1.0449	Below —7	
25	20	55	1.0455	—5	
25	25	50	1.0462	—3	
25	30	45	1.0469	—2	
25	35	40	1.0476	0	
25	40	35	1.0480	4.8	
25	45	30	1.0483	6.6	
25	50	25	1.0490	8.4	
25	55	20	1.0498	12.2	
25	60	15	1.0508	14.9	
25	65	10	1.0512	17.7	
25	70	5	1.0517	20.2	
25	75	0	1.0524	22.6	

(2) *Refractive Indices*—A few preliminary tests on the components of the mixtures showed definitely that their constants were too close to enable us to determine at all accurately the character of a mixture by this means.

(3) *Specific Gravity*—This test proved very useful and was adopted for general use. The details of carrying out this test will be given later in connection with the described testing method. It was found desirable to use two standard temperatures. The mixtures having a solidifying point less than 25° C. were taken at 25° C. compared to water at 25° C. Above this point the specific gravities were taken at 45° C. compared to water at 45° C. Occasionally a mixture test will be found where the solidifying point is 25° C. or a trifle higher and still the specific gravities given as of 25° C.—for all these cases the specific gravity was never taken in the solid state or by correction from a higher temperature, but represents an actual determination in the supercooled liquid.

(4) *Solidifying Point*—The old method used ordinarily for phenol, in which the thermometer is used as the stirring rod in the test tube, was given a thorough trial and proved to be too inaccurate, in so far as checks by different operators were concerned, to be used for the present purposes. Accordingly a modified method was devised, the details of which are given later under the testing method. This modification of the method was used throughout the work presented in this paper.

(5) *Detailed Tests*—These are shown in Table IV. The curves in Figs. II, III and IV bring out graphically important features of these tests.

(6) *Consideration of Results*—In Fig. II there are plotted curves of all mixtures, where the solidifying point is above 0° C., using the solidifying points as ordinates and the specific gravities as abscissas and plotting a curve for each 5 per cent of phenol. It is evident from an examination of the detailed results in the tables that if a solidifying point of a mixture is indeterminate, that is below 0° C., that the specific gravity alone is not sufficient data for an exact determination. Therefore, for determination of phenol by this method it is first necessary to concentrate the phenol into a fraction containing only phenol and cresols of such a nature that the solidifying point is above 0° C.

It will be further noted in Fig. II that, from 0 to 30 per cent of phenol, the curves are well separated and the constants of a mixture will serve for exact quantitative determination. From 30 to 70 per cent phenol, the curves cross each other irregularly and points in this area are indeterminate and, therefore, not susceptible of exact determination. From 70 to 100 per cent phenol the curves are well separated and this area can be considered as determinate. The two determinate areas are shown on a larger scale in Figs. III and IV. It is, therefore, evident that to determine phenol, it will be necessary to bring it into a fraction containing only phenol and cresols of such composition that its physical constants will plot to a point within one or the other of the determinate areas shown. The first portion of the remaining problem was, therefore, to develop a method of fractional distillation which would separate all the phenol from a crude acid with only cresols as an impurity. It is evident that with such a fraction, if its constants do not throw it into the determinate area, that a mix-

TABLE IV—(*Concluded*)

Phenol	*o*-Cresol	*m*-*p*-Cresol	Sp. Gr. 25/25° C.	Solidification Point, ° C.
SERIES 7				
30	0	70	1.0448	Below —7
30	5	65	1.0454	Below —7
30	10	60	1.0461	Below —7
30	15	55	1.0467	—4
30	20	50	1.0473	—3
30	25	45	1.0479	—1
30	30	40	1.0486	0.3
30	35	35	1.0493	1.6
30	40	30	1.0500	5.0
30	45	25	1.0506	8.7
30	50	20	1.0513	11.6
30	55	15	1.0520	14.2
30	60	10	1.0527	16.8
30	65	5	1.0534	19.3
30	70	0	1.0541	21.7
SERIES 8				
35	0	65	1.0466	Below—7
35	5	60	1.0473	—4
35	10	55	1.0480	—1
35	15	50	1.0486	0.5
35	20	45	1.0493	0.2
35	25	40	1.0500	1.4
35	30	35	1.0506	3.0
35	35	30	1.0513	4.2
35	40	25	1.0520	7.4
35	45	20	1.0527	10.4
35	50	15	1.0534	13.2
35	55	10	1.0541	15.8
35	60	5	1.0548	18.2
35	65	0	1.0555	20.6
SERIES 9				
40	0	60	1.0487	—1
40	5	55	1.0493	0
40	10	50	1.0500	0.8
40	15	45	1.0507	2.4
40	20	40	1.0514	3.4
40	25	35	1.0521	4.6
40	30	30	1.0528	4.8
40	35	25	1.0535	6.9
40	40	20	1.0542	9.8
40	45	15	1.0549	12.4
40	50	10	1.0555	15.1
40	55	5	1.0561	17.6
40	60	0	1.0567	20.0

Phenol	*o*-Cresol	*m*-*p*-Cresol	Sp. Gr. 25/25° C.	Solidification Point, ° C.
SERIES 10				
45	0	55	1.0507	3.4
45	5	50	1.0514	3.6
45	10	45	1.0521	4.7
45	15	40	1.0528	5.6
45	20	35	1.0534	6.2
45	25	30	1.0541	7.4
45	30	25	1.0548	8.5
45	35	20	1.0555	9.4
45	40	15	1.0562	12.2
45	45	10	1.0569	14.6
45	50	5	1.0576	17.2
45	55	0	1.0583	19.6
SERIES 11				
50	0	50	1.0527	7.1
50	5	45	1.0534	7.9
50	10	40	1.0541	8.6
50	15	35	1.0548	9.1
50	20	30	1.0555	9.8
50	25	25	1.0562	10.3
50	30	20	1.0569	11.2
50	35	15	1.0575	11.7
50	40	10	1.0583	14.4
50	45	5	1.0589	17.0
50	50	0	1.0596	19.2
SERIES 12				
55	0	45	1.0546	11.5
55	5	40	1.0553	11.9
55	10	35	1.0560	12.2
55	15	30	1.0567	12.4
55	20	25	1.0574	12.7
55	25	20	1.0580	12.9
55	30	15	1.0587	13.6
55	35	10	1.0594	14.2
55	40	5	1.0599	16.6
55	45	0	1.0605	19.2
SERIES 13				
60	0	40	1.0565	15.2
60	5	35	1.0574	15.6
60	10	30	1.0581	15.8
60	15	25	1.0585	16.0
60	20	20	1.0592	16.2
60	25	15	1.0598	16.4
60	30	10	1.0606	17.0
60	35	5	1.0615	17.4
60	40	0	1.0621	19.2

Phenol	*o*-Cresol	*m*-*p*-Cresol	SPECIFIC GRAVITY 25/25° C.(*a*)	SPECIFIC GRAVITY 45/45° C.(*b*)	Solidification Point, ° C.
SERIES 14					
65	0	35	1.0590	....	17.6
65	5	30	1.0595	....	18.0
65	10	25	1.0604	....	18.6
65	15	20	1.0608	....	19.1
65	20	15	1.0617	....	19.2
65	25	10	1.0622	....	19.4
65	30	5	1.0630	....	19.6
65	35	0	1.0636	....	20.4
SERIES 15					
70	0	30	1.0608	1.0520	22.0
70	5	25	1.0614	1.0525	22.2
70	10	20	1.0623	1.0530	22.4
70	15	15	1.0632	1.0533	22.5
70	20	10	1.0638	1.0540	22.6
70	25	5	1.0644	1.0547	22.8
70	30	0	1.0650	1.0554	23.2
SERIES 16					
75	0	25	....	1.0536	25.4
75	5	20	....	1.0542	25.4
75	10	15	....	1.0547	25.6
75	15	10	....	1.0553	25.8
75	20	5	....	1.0560	25.9
75	25	0	....	1.0566	26.0
SERIES 17					
80	0	20	....	1.0556	28.6
80	5	15	....	1.0563	29.0
80	10	10	....	1.0570	29.0
80	15	5	....	1.0576	29.0
80	20	0	....	1.0583	29.0
SERIES 18					
85	0	15	....	1.0572	31.8
85	5	10	....	1.0579	32.0
85	10	5	....	1.0587	32.2
85	15	0	....	1.0594	32.2
SERIES 19					
90	0	10	....	1.0596	34.6
90	5	5	....	1.0603	34.6
90	10	0	....	1.0607	34.6
SERIES 20					
95	0	5	....	1.0617	37.2
95	5	0	....	1.0623	37.2
SERIES 21					
100	0	0	....	1.0636	40.4

(*a*) The values for the specific gravity given in this column are plotted on Fig. IV.
(*b*) These values are plotted on Fig. II because if plotted at 45/45° C. the curve for 70 per cent phenol would interfere with the other curves in this figure.

ture of the fraction with a known amount of *pure phenol* can be made, which mixtures can be made to fall in one of the determinate areas. The percentage of phenol can be readily determined in the mixture and from the knowledge of the amount of phenol added, the percentage of phenol in the original fraction can be readily deduced. With this then as a basis, work was commenced on known mixtures of phenol, cresols, higher acids and oils to perfect the manipulative details necessary to bring phenol, no matter what its form, quantitatively into a fraction in which it was accompanied by cresols alone.

C—WORK ON KNOWN MIXTURES TO PERFECT THE DETAILS OF THE METHOD

The preliminary tests involved a comparison of the Hempel and the LeBel-Henninger fractioning tubes. The Hempel tube was of the standard Barrett type (A. H. T. Cat. No. 20496) and the LeBel, the 3-bulb variety: 6-bulb LeBels were tried but those obtainable were not of sufficiently good workmanship to withstand the required temperature without cracking. For these preliminary tests it was necessary to prepare some phenol-free acids of a boiling point above cresols. This was done by taking ordinary crude acids, removing the oil and naphthalene, and fractioning, taking for use the fraction distilling above 200° C. It was shown by careful test to be free of phenol. In these preliminary tests the acids (free of oil and naphthalene) were fractioned and cut at 190 and 202°. The 190–202° fraction was redistilled and cut at 197° C. The total fraction below 197° C. was compared with the curves and the phenol content estimated. If necessary a suitable mixture was made with pure phenol so as to bring the mixture into the determinative range. Needless to say, all details of the distillation were gravimetrically quantitative.

Tests 1 to 7 may be tabulated as follows:

Test No.	MIXTURE—PERCENTAGES Phenol	MIXTURE—PERCENTAGES *o*-Cresol	MIXTURE—PERCENTAGES *m*-*p*-Cresol	Per cent Higher Acids	Per cent Phenol Recovered	Error in Per cent of Phenol Present
1......	33.3	....	....	66.7	32.2	—3.3
2(*a*)...	20.0	20.0	20.0	40.0	16.0	—20.0
3......	20.0	20.0	20.0	40.0	19.2	—4.0
4......	10.0	15.0	15.0	60.0	10.4	+4.0
5......	50.0	10.0	10.0	30.0	48.0	—4.0
6......	25.0	25.0	25.0	25.0	24.2	—3.2
7......	30.0	5.0	5.0	60.0	29.5	—1.7

(*a*) In this test the Hempel column was used, in all others the LeBel was used.

After these tests we decided to abandon the Hempel distillation for this purpose and until otherwise noted all tests were run using the LeBel 3-bulb column. A comparison of the bulb distillations of the "to 197°" fraction of Tests 2 and 3 shows why Test 2 was so much further off than Test 3.

BULB DISTILLATIONS OF TESTS 2 AND 3

	187°	190°	195°	200°	Dry at
Test 2...............	2	22	81	94	205° C.
Test 3...............	1	47	90	98	203° C.

The Hempel had not removed xylenols as well as had the LeBel. The effect of xylenols would be to lower the specific gravity without proportionally lowering the solidifying point, thus giving a lower phenol indication than the truth. This is true to a lesser degree in the LeBel tests and is doubtless the cause of the somewhat low results obtained in a number of the tests. In fact, on Tests 1 to 7, the only

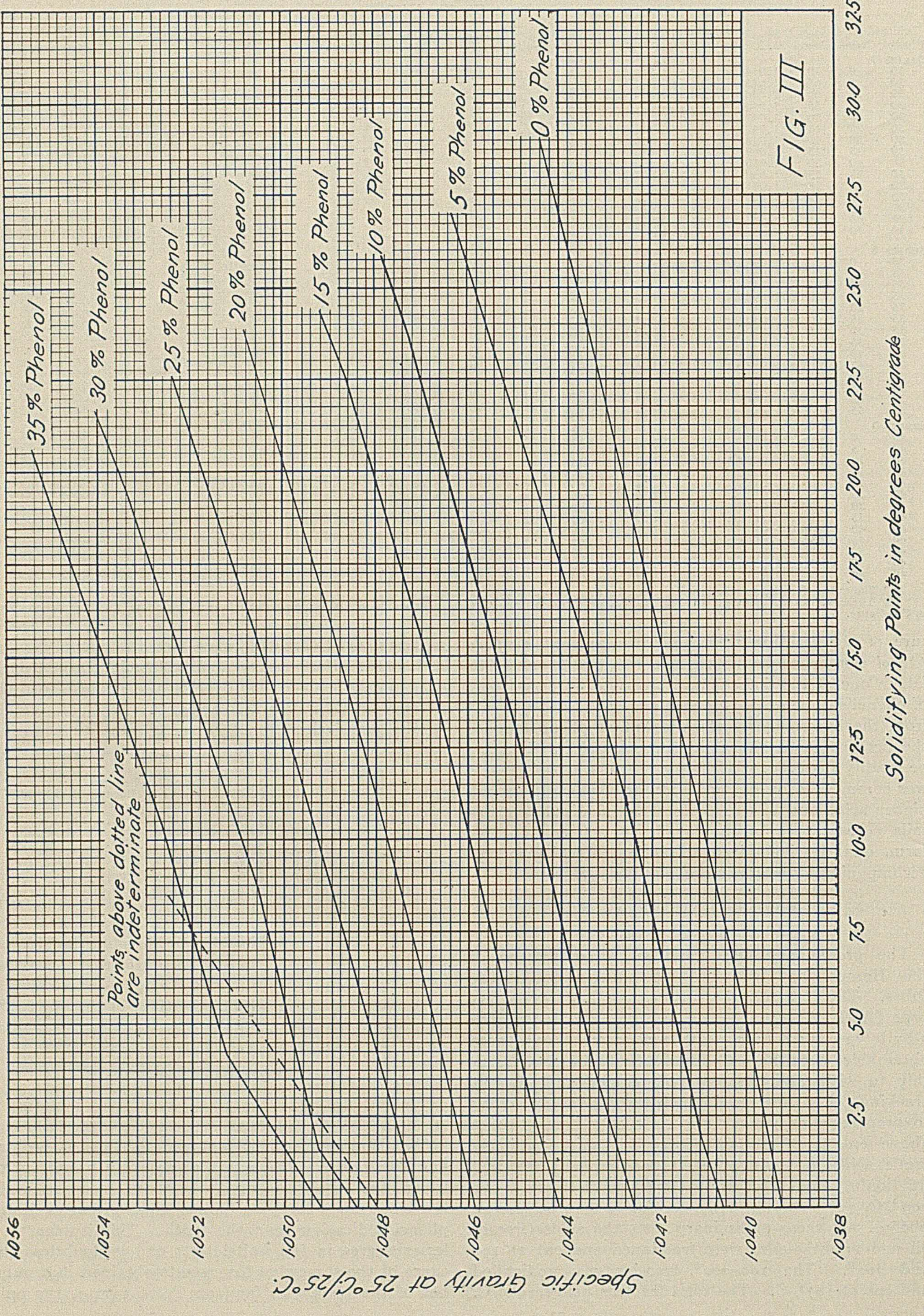
FIG. III
35 % Phenol
30 % Phenol
25 % Phenol
20 % Phenol
15 % Phenol
10 % Phenol
5 % Phenol
0 % Phenol
Points above dotted line are indeterminate
2.5
5.0
7.5
10.0
12.5
15.0
17.5
20.0
22.5
25.0
27.5
30.0
32.5
Solidifying Points in degrees Centigrade
1.056
1.054
1.052
1.050
1.048
1.046
1.044
1.042
1.040
1.038
Specific Gravity at 25°C/25°C.

ones where bulb distillations were made on the "to 197°" fraction, all showed some residue above 201° C. It has not been thought worth while to record these distillations in detail as all the essential information is given in the two already shown.

At this point of the investigation we decided that the fractioning method was fairly accurate, though not entirely satisfactory. It was considered advisable now to proceed to oil mixtures to determine the points in this phase of the investigation which required our attention.

TEST 8—For this test we prepared a quantity of acid-free oil by completely extracting ordinary creosote oil with caustic soda. A mixture was made by using 85 per cent of this oil and 15 per cent of acids containing 20 per cent phenol. The acids were extracted by caustic soda solution from a portion of this mixture. The carbolate was steam-distilled to remove neutral oils and naphthalene, the acids liberated by dilute sulfuric acid and carefully separated from the sodium sulfate layer. The test was then carried on in the usual way, taking care to separate all water from the "to 197°" fraction.

The phenol recovered showed an error amounting to —20 per cent of the phenol present.

TEST 9—Here we used 80 per cent of oil and 20 per cent of acids containing 20 per cent of phenol. The carbolate was formed as in Test 8, and washed with 600 cc. of benzol in three 200-cc. portions. The united benzol washings were agitated with 100 cc. of water in two 50 cc. portions. These were added to the carbolate, which was then boiled gently to expel any benzol present. The sulfate liquor, after acidification, was not washed for recovery of acids.

The general method of fractioning, which was adhered to except where otherwise noted, was to distil to 170°, separate acids from water in the distillate and return the former to the distilling flask. Fractions were then taken to 190° C. The second fraction was redistilled collecting to 197° C. and combining all acid distillates up to 197° for test. The recovery of phenol showed a loss of 22.1 per cent of the phenol present.

TEST 10—In this test no change was made in the relative proportions of the acids and neutral oil. The carbolate was steam-distilled. The NaOH solution was added in three 210-cc. portions. The sulfate liquor was washed with 150 cc. of benzol in three 50-cc. portions and these washings were added to the liberated acids. The mixture used was the same as in Test 9. The error amounted to a loss of 21.7 per cent of the phenol.

The residual acids after fractionation were combined and tested for phenol, but none was found. In this test the benzol was separated from the acids in the fractionation. The benzol recovered had an acid odor, but only a trace of acids could be recovered by solution in NaOH.

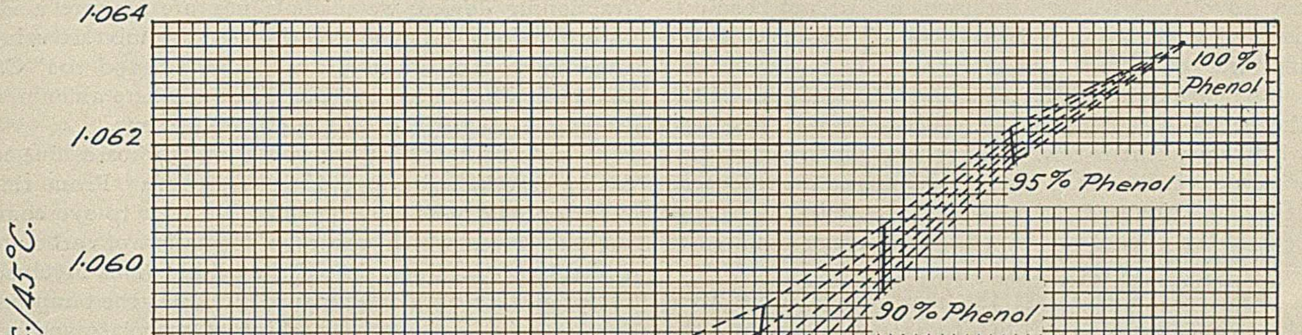

FIG. IV

The results of Tests 9 and 10 showed that the tar oils and naphthalene in the carbolate may be removed with benzol, instead of steam distillation.

TEST 11—In this test the acid-oil mixture was approximately the same as in the previous tests. The method of procedure was the same as in the previous test, except that the liberated acids were washed with 200 cc. of a saturated NaCl solution for removal of sulfate liquor. The solidifying point of the acid mixture made on fractionation of the acids was so low that no further data were taken.

TEST 12—This test in conjunction with Test 14 was made in order to determine the loss due to solution of acids in the sulfate liquor. In the light of further tests, however, it does not indicate much. This is due to other greater losses which had not been discovered at this time. No neutral oil was used in the mixture.

The carbolate was formed using 125 per cent of the theoretical NaOH necessary. The acids were liberated with 50 per cent H_2SO_4. The sulfate liquor was washed with 150 cc. benzol in 50-cc. portions and the recovered acids fractionated. An acid containing 20 per cent of the phenol was taken. The error amounted to —8.5 per cent of the phenol present.

TEST 13—In this test the new modifications were the weakening of the H_2SO_4 to $33^1/_3$ per cent and the washing of the extracted oil with an additional 100 cc. of NaOH solution (making a total of 140 per cent). Benzol was used to wash the carbolate. The sulfate liquor was agitated with 125 cc. of benzol and the washings added to the main quantity of acids. The acids were fractionated in the LeBel-Henninger column. The same mixture was used as in Test 12. The error found was —13.2 per cent of the phenol present.

TEST 14 was made in order to compare the loss of phenol with the loss in Test 12, where a carbolate was made and the acids liberated. The mixture contained no oil and was simply fractionated in the LeBel column. Here the error was —8 per cent of the phenol present.

The results of this test would indicate that the loss in recovery from a carbolate was not much.

TEST 15 was made in order to check the curve with a mixture containing 20 per cent of phenol.

Mixture No. 1	Pure Phenol	20.15 g.
	Pure *o*-Cresol	40.05 g.
	Pure *m*-*p*-Cresol	40.05 g.
Mixture No. 2	Pure Phenol	75.00 g.
	Mixture No. 1	25.00 g.
Solidification Point of Mixture No. 2		28.8° C.
Specific Gravity (45/45° C.) of Mixture No. 2		1.057
Phenol in Mixture No. 2		80 per cent
Phenol in Mixture No. 1		20 per cent

This shows the curves to be correct at this point.

TEST 16—In view of the results of tests to this point it was thought that a considerable part of our losses was due to fractionation and that a change in the temperature at which the fractions were cut might help in the phenol recovery. Accordingly a mixture of acids was made and fractionated using the LeBel-Henninger column as in previous tests. The first fraction collected was to 190° C., the second between 190 and 205° C. This second fraction was redistilled until a temperature of 198° C. was reached. The first and redistilled fractions were collected in the same flask as in previous test. Using the usual 20 per cent phenol acid, an error of —16.5 per cent of the phenol present was obtained.

TEST 17—In this test the temperature of fractionation was again changed. The first fraction collected was that coming over to 190° C.; the second between 190 and 204° C. This fraction was redistilled up to 195° C. With the same mixture as in Test 16 we showed an error of —10.1 per cent of the phenol present.

Changing the temperature of fractionation did not increase the amount of phenol recovered. We then concluded that the column used in fractionation should be changed.

TEST 18—We then decided to try the 12-pear column. To determine the efficiency of this apparatus as a fractioning device, we made a mixture of pure acids as in previous tests, and fractionated the mixture using the original temperature, *i. e.*, to 190° C. (190–202° C.) and redistillation to 197° C. The error here amounted to only +1.4 per cent of the phenol present.

Results of this test showed that the losses due to faulty fractionation had been overcome. From this point on, our tests were made with a view to overcoming the losses which occur on formation of carbolate and subsequent liberation of the acids. This involved the proper strength and amount of H_2SO_4, the temperature of acidification, the washing of carbolate and recovery of the acids dissolved in the sulfate liquors.

TEST 19—In this test we attempted to recover all of the phenol in a pure acid mixture after converting the acids to a carbolate. The acids were agitated with 125 per cent of the theoretical amount of NaOH necessary. The acids were then liberated with 25 per cent solution of H_2SO_4, using a very slight excess of acid (about 10 cc.). The sulfate liquor was washed with 100 cc. of ether in two 50-cc. portions and the washings added to the main quantity of acids. The acids were then fractionated with the 12-pear column. It was impossible to make a clear separation of the ether and acid distillates. The ether and water that came over were agitated with an NaOH solution and the acids recovered. They amounted to about 4 cc. However, they were not added to the acid distillate and so were lost. Better results were obtained here by reducing the strength of the sulfuric acid to 25 per cent. There was a cleaner separation, the acids were lighter colored and there was no gas formation. The error here was —2.1 per cent of the phenol present.

The results of this test indicated that the phenol in a mixture of pure acids could be recovered substantially quantitatively from a carbolate.

TEST 20—In this case 150 per cent of the theoretical quantity of NaOH was used to form the carbolate. The sulfate liquors were washed with benzol. Any acid was recovered from the benzol by washing with caustic soda as described previously. This acid was added to the main bulk of acids. The recovery of phenol showed a very low error, only 0.2 per cent of that present.

TEST 21 was made using a mixture of 20 per cent of pure phenol with crude cresols and higher acids, instead of the purified acids, as in Tests 19 and 20.

125 per cent of the theoretical amount of NaOH was used. The carbolate was washed with 500 cc. benzol in 125-cc. portions. The acids were recovered from the sulfate liquor as in Test 20. The error was —1.3 per cent of the phenol present.

TEST 22 was a check on Test 20, the same procedure being used in each test. The error here was —0.8 per cent of the phenol present.

After running Tests 20, 21 and 22, we concluded that the phenol present in a mixture of acids with small amounts of oil could be recovered with but slight loss. Our next attempt was to recover the phenol from an acid-oil mixture which contained a large amount of neutral oil.

TEST 23—In this test we added enough neutral oil to the acids to make it 80 per cent of the total mixture and attempted to recover the phenol. The acids were extracted from the mixture with 150 per cent of the theoretical amount of a 20 per cent NaOH solution: 800 cc. of the NaOH solution were used in three 200-cc. and two 100-cc. portions. The carbolate was washed with 500 cc. of benzol in 100-cc. portions. The sulfate liquor was washed with ether, and the dissolved acids recovered from ethereal solution with a weak NaOH solution and added, after liberating with H_2SO_4, to the main quantity; 9.2 grams were recovered. This method of recovery and separation from the solvent is a new and important step in the process. The acids were fractionated as before.

Acid-Oil Mixture	Phenol	59.85 grams	20%	20 per cent
	o-Cresol	59.80 grams	20%	
	m-*p*-Cresol	59.80 grams	20%	
	Higher Acids	119.30 grams	40%	
Neutral Oil		1212.50 grams		80 per cent

In the above mixture the acids, with the exception of the phenol, were crude.

The loss of phenol in this test (—16 per cent of the phenol present) was very high (due to imperfect technique, as will be seen later), and it was decided to make tests using smaller quantities of neutral oil.

From this point on, the general method of recovery of the acids was the same, *i. e.*, formation of a carbolate with NaOH (20 per cent strength) washing of the carbolate with pure benzol, washing the benzol solution of carbolate with water, boiling of the carbolate, liberation of the acids with 25 per cent H_2SO_4, washing the sulfate liquors with benzol and recovery of the dissolved acid with weak (10 per cent) solution of NaOH. All fractionations were made with the pear column.

The essential results of Tests 24 to 35 inclusive are shown in the following table:

Test No.	Grams Phenol	Grams *o*-Cresol	Grams *m*-*p*-Cresol	Grams Higher Acids	Grams Oil	Grams Phenol Recovered	Error in % Phenol Present
24	59.75	60.00	60.35	119.90	101.75	55.83	—6.6
25	59.20	59.75	59.85	119.70	10.10	58.08	—1.8
26	60.00	60.10	60.00	119.60	10.30	63.10	+5.1
27	60.00	59.61	59.70	119.10	30.00	60.08	+0.13
28	58.55	60.00	59.60	119.70	120.00	58.10	—0.8
29	29.65	70.00	70.00	129.30	....	31.20	+5.2
30	29.40	70.30	69.70	129.30	....	29.80	+1.6
31	14.65	75.00	74.80	134.70	....	14.05	—4.1
32	15.05	76.00	74.80	135.15	....	15.90	+7.0
33	88.55	44.90	45.10	119.70	....	82.30	—7.0
34	149.90	30.05	29.65	90.15	....	149.20	—0.5
35	15.05	74.85	75.50	134.80	1200.00	14.73	—2.1

In Test 33, a known loss occurred in manipulation, so that this result is natural. The tests seemed to us to be accurate enough for all practical purposes, and checks should be obtained to 0.8 gram of phenol if the work is carried on carefully and conscientiously.

Up to this point we had succeeded in the recovery of the phenol from its mixtures with other pure acids, with unpurified acids in varying portions, and with neutral oil in varying proportions.

To determine the reliability of the test, when applied to crude carbolic acid, we first determined the percentage of phenol in a sample of ordinary crude acids, and next in these acids to which we had added a known amount of pure phenol.

TEST 36—*Determination of the phenol in ordinary crude carbolic acid:* The acids contained 5.32 per cent (by weight) of water and had a specific gravity at 15.5° C. of 1.072.

We first tried to wash a carbolate made with the crude acids as obtained, but there was not a clean separation of the benzol and carbolate layers. We found it necessary to distil the acids first. The crude acids were distilled, after weighing, directly into a 2000 cc. separatory funnel.

Weight of acids distilled	301.5 grams
Weight of distillate (by difference)	271.3 grams—90 per cent
Black pitch residue	30.2 grams

The carbolate was now formed as in previous tests, and washed with benzol. The recovery of the acids and the fractionation were carried out as in the previous tests. We found the phenol content by weight to be 16 per cent on the dry acids or 15.14 per cent on the wet acids.

TEST 37—In this test we added a known amount of pure phenol to the crude acids tested above.

Crude acids	301.15 grams
Pure phenol added	15.85 grams

On determination of the phenol we found that of the phenol added we had recovered 99.2 per cent.

The better results in the later tests were due to constant improvements in the technique with experience. In the following description, we believe, we have given that technique with sufficient wealth of detail to enable any operator following directions carefully, to obtain consistent, accurate results.

D—STANDARD METHOD FOR DETERMINATION OF PHENOL IN CRUDE CARBOLIC ACID AND TAR OILS

I. PREFACE—In this test extreme care and faithful attention to details are necessary to secure accurate results. All instructions given are the epitomized result of considerable experience and are not given arbitrarily, but with a purpose. The test has been a difficult one to develop and requires greater care and skill than do the ordinary tar-testing methods. Even a slight deviation from the directions can cause serious discrepancies.

II. PREPARATION OF THE OIL—If the oil is at all dirty, so that the subsequent separation of the carbolate will not be clean, it must be distilled. This shall be accomplished by weighing a suitable quantity of oil into a clean copper tar-still and taking the total distillate from the start of distillation to coking. The amount of total distillate by weight shall be noted so that results may be figured back to the original oil if desirable. If any foaming-over occurs during the distillation, the run must be rejected and a new one undertaken.

III. EXTRACTION OF THE ACIDS FROM THE OIL—The approximate percentage of tar acids in the oil should be determined by a rough test. A weight of oil which contains between 300 and 350 g. of acids shall be placed in a separatory funnel and treated with 300 cc. (approximate) of 20 per cent caustic soda solution; this caustic soda solution may be made from commercial caustic soda. The oil and caustic soda shall be given a thorough mixing by shaking, care being taken to prevent mechanical loss. The mass shall now be allowed to stand until the carbolate settles completely and a sharp line of division appears between the oil and the carbolate. With a properly distilled oil, from 15 to 30 minutes should suffice. If the oil contains solids, it shall be warmed before extraction to a temperature sufficient to keep solids in solution, and while settling be suspended in a bath of like temperature.

After settling is complete, the carbolate shall be separated and brought into another separatory funnel. The oil shall be washed successively with three more portions of 200 cc. each of the same caustic soda solution, observing the same precautions as to agitation, settling and separation. These separated soda solutions are to be added to the first carbolate. On the last separation it is better to run a few drops of oil along with the carbolate, rather than leave a few drops of carbolate in the oil. After this treatment the oil shall be tested to determine if it is free from tar acids and if so, it may be discarded.

NOTE—If crude acids, not oil, are to be tested, 300 to 350 g. of the crude acid shall be taken in a separatory funnel and dissolved in 800 cc. of 20 per cent caustic soda solution. The treatment from this point on is the same as with carbolate extracted from oil. If the crude acids are tarry it is advisable to distil them quantitatively before dissolving them in caustic soda.

IV. PURIFICATION OF THE CARBOLATE—The carbolate in a separatory funnel shall be extracted five times successively with 100-cc. portions of pure benzol. The manipulation shall be: Add the benzol to the carbolate, mix thoroughly by shaking and allow to settle. Draw the carbolate into another separatory funnel and draw the benzol into a third separatory funnel. Repeat the agitation with fresh benzol four times more, using the two original separatory funnels for this purpose. Finally, after running the washed carbolate into a beaker, rinse each of the funnels which contained carbolate, twice, with 25 cc. each of water, and add the water to the carbolate in the beaker. Take the third separatory funnel, which contains the combined benzol, and wash twice with 50 cc. of water, adding this water also to the carbolate in the beaker. Bring the carbolate in the beaker to a gentle boil until the odor of benzol disappears. No odor of creosote shall be evident in the carbolate at this point. If such odor appears, the washing has not been properly carried out and must be repeated. The carbolate purified of oil is ready for acidification after cooling. The benzol used for extraction may be discarded.

V. ACIDIFICATION—The purified carbolate shall be placed in a separatory funnel and acidified with sulfuric acid of 25 per cent strength (sp. gr. 1.21 at 15.5° C.). The beaker containing the carbolate shall be rinsed with water and the rinsings added to the carbolate in the funnel. During acidification the mass *must* be kept *below 40° C.* by immersion in a bath of cold water. Test for acidity should be made from time to time with litmus paper. *More than a very slight excess* of acid (5 to 10 cc.) must not be added after litmus shows a red color. When acidification is complete, the funnel shall be allowed to stand until complete separation has taken place and the lower layer of sodium sulfate solution is perfectly clear or at most shows a slight opalescence. This settling requires from 3 to 4 hours. After settling is complete the sulfate layer shall be drawn off and kept for further treatment. The carefully separated acid layer shall be drawn down into a weighed 500 cc. round-bottomed short ring neck flask and kept there until further treatment.

VI. TREATMENT OF THE SULFATE SOLUTION—This shall be returned to the separatory funnel which contained the separated tar acids, which still has a thin film of tar acids adhering to the walls. The sulfate liquor shall be extracted three times with successive portions of 100 cc. each of pure benzol and the benzol extracts combined in a separatory funnel of about 500 cc. capacity. The benzol extract shall now be extracted with two successive portions of 25 cc. each of 20 per cent caustic soda solution, or until the soda solution separates nearly colorless. The benzol can then be discarded. The caustic soda solution shall then be warmed until all odor of benzol disappears and acidified with enough 25 per cent sulfuric acid to make slightly acid. This shall be allowed to settle completely, the lower layer of sulfate drawn off and discarded, and the upper layer of acids added to the flask containing the main amount of the separated acid. This flask shall now be weighed and the weight of *wet* crude acids recorded.

VII. FRACTIONAL DISTILLATION—*A. Column*—This shall be the standard 12-pear still head (Eimer & Amend catalogue No. 7156).

B. Flask—This shall be a short neck flask as described under V.

C. Condenser—This shall be the regular Barrett trough type with a 24-in. tube (A. H. T. catalogue No. 20432).

D. Thermometer[1]—This shall conform to the following specifications: (1) It shall be graduated from 170 to 225° C. in $^1/_5$° C. and shall be accurate at total immersion to $^1/_5$° C. (2) It shall be well annealed and shall conform to the following dimensions:

Total length—less than 380 mm.
Bulb length—20–25 mm.
170° mark to bottom of bulb—70–80 mm.
Scale length 170–225°—270–280 mm.
Stem diameter—5–7 mm.
Bulb diameter—4–5.5 mm.

E. Setting up Apparatus—The flask shall be supported over a burner on a 6 in. square of $^1/_4$ in. asbestos

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[1] This thermometer can be obtained from the Taylor Instrument Co.

board with an opening in the center 3.5 in. in diameter. It shall be surrounded by a circular asbestos shield about 3$^1/_2$ in. high and of such diameter as to enclose the flask. A piece of asbestos with a hole cut for the neck of the flask forms the top of the shield. The pear column shall be connected to the flask by a tight-fitting cork, and the condenser to the side tube of the pear column. The thermometer shall be inserted into the top of the pear column by means of a well-fitting cork in such manner that the top of the thermometer bulb is opposite the middle of the off-take of the column. The condenser trough shall be kept full of cold water.

F. Method of Conducting the Distillation—The apparatus shall be set up as described and the distillation started and continued at the rate of one drop per second. The distillate shall be first caught in a 100-cc. separatory funnel until the temperature has reached 170° C. The distillation shall then be interrupted and sufficient salt added to the contents of the separatory funnel to cause any acids coming over with the water to rise to the surface. The aqueous layer is removed and the acids returned to the distillation flask, and the flask with contents weighed. Thereby the weight of *dry* acids is recorded. The distillation shall be again started and the first few drops of acid and water coming over collected in a small, dry separatory funnel. When the last drop of water is over, a fraction to 190° C. is collected in a weighed flask, which shall be closed by a cork as soon as the distillation is completed. The few drops of acid and water which have been collected in the small funnel shall be separated and the acid layer added to the distillate to 190° C. A second fraction from 190 to 202° shall be collected in a 250-cc. round-bottom, short ring-neck flask, which need not be weighed. The distillation shall be discontinued at 202° C. and the flask containing the 190–202° fraction shall be substituted for the first flask, which contained the crude acids. This fraction is now redistilled after drying the condenser tube, using the weighed flask containing the —190° fraction from the first distillate as a receiver. The distillation shall be carried to 197° C. The receiver which now contains the combined fraction up to 197° shall be weighed, the weight of the fraction recorded, and the receiver kept tightly stoppered.

G. Testing the Fraction—The fraction shall be tested for specific gravity and solidifying point as follows:

(1) *Solidifying Point:* A test tube 1 in. inside diameter (this dimension bears no deviation) and 5 in. to 6 in. long is held by a clamp in a vertical position with 3 in. of its lower end immersed in a 500–600-cc. beaker full of water. A standard 0–80° melting point thermometer (A. H. T. Cat. No. 20476), calibrated to an accuracy of *at least* 0.1° C., is fastened rigidly by another clamp in the center of the test tube with the bottom of the bulb 0.5 in. from the bottom of the test tube. An agitator, composed of an iron washer attached to a wire and arranged to work freely up and down the test tube around the thermometer, completes the outfit.

The fraction to be tested for solidifying point shall be poured into the tube to a depth of 2 in. and an approximate solidifying point taken. The contents of the tube shall be again liquefied and the outside bath adjusted to a temperature 4.5 to 5.5° C. below the approximate solidifying point. The temperature relation is very important. The sample is now allowed to cool with constant stirring. The temperature of the sample falls, but when crystals begin to separate a rise is noted which soon reaches a maximum where it remains constant for a varying period of time. This maximum temperature shall be taken as the solidifying point. Two successive tests must give identical results and care must be taken to keep the relation between solidifying point and bath temperature within the 4.5 to 5.5° C. range.

(2) *Specific Gravity*—If the solidifying point is 25° C. or less, this shall be taken at 25/25° C.; if greater than 25° C., the specific gravity shall be taken at 45/45° C. The specific gravities shall be taken with a Westphal balance. Most of the instruments supplied in the United States are standardized for 15.5° C. The balance is set up and the plummet should exactly counterpoise the arm when swinging freely in air. It must be first adjusted to this condition by means of the leveling screw on the base of the column of the balance. The plummet is then immersed in freshly boiled distilled water at 15.5° C. and if the balance and weights are properly made a reading of 1,000 should be obtained. Next, a reading is taken in boiled distilled water at either 25 or 45° C., depending on the temperature desired. After the first adjustment of the balance, the reading in water at 15.5° C. may be dispensed with, but care should be taken to see that the balance in air is correct before readings are made. After the water standard at 25 or 45° C. is established, a reading is taken in the acid fraction at the same temperature. A simple division gives the specific gravity. Precautions to be observed are: keep the balance clean and dry; see that the plummet is properly cleaned and dried when changing from one liquid to another; keep the point to which the wire holding the plummet is immersed constant; control the temperature carefully; and see that the plummet swings freely and does not touch the sides of the vessel containing the liquid.

(3) *Comparison with Curves*—After the solidifying point and specific gravity of the fraction are determined, compare them with the phenol determination curves and see whether the point plotted by the constants falls well within a determination area. If it does not or if it is very close to the limit, it is best to proceed to make a mixture of the fraction with pure phenol.

(4) *Mixture with Phenol*—Phenol for this purpose must possess a solidifying point of at least 40.4° C., tested on the thoroughly dry material. A little judgment is required in making the mixture so as to use the least amount of phenol which will bring the tests of the mixture well within one or the other of the determinate ranges. Needless to say, the mixture must be strictly quantitative, and must be made in perfectly dry containers. The mixtures are made up until one susceptible of determination is obtained.

The specific gravities and solidifying points are taken exactly as described previously.

H. Interpretation of Results—After the tests show the point to be within the determinative areas the method of interpolation is as follows: If the plotted point occurs in the lower set of curves a line is drawn through the point normal to the curves between which it lies. The distance from curve to curve is measured accurately along this line and the fractional distance of the point is interpolated into per cent phenol. If the point occurs in the upper set of curves, the line is drawn through the point parallel to the nearest of the dotted converging lines, the linear distance measured and the percentage interpolated as before.

If mixtures are made, the calculations are somewhat complicated and the following formula may be used:

Let A = per cent phenol found in mixture
B = Grams "to 197°" fraction taken
C = Grams pure phenol taken
X = per cent phenol in "to 197°" fraction

$$X = \frac{A(B + C) - 100\,C}{B}$$

The per cent phenol in the "to 197°" fraction multiplied by the weight of the fraction gives the weight of total phenol in the oil or crude acid taken.

A specimen test on acid with the recorded data may be shown as follows:

TAKEN: Crude Acid, 300 g.

Distilled 170–190°	60.65 g.	
190–202°	178.05 g.	
190–202° redistilled to 197°	78.05 g.	
Total under 197°	138.7 g. =	46.2 per cent
Sp. gr. at 25/25° C.		1.0602
Solidifying point		20.4°
These constants are indeterminate.		
MIXTURE { to 197° fraction	25 g.	
Pure phenol	25 g.	
Sp. gr. at 45/45° C.		1.0576
Solidifying point		31.7° C.
Phenol in fraction mixture (from curve)		84.75 per cent
PHENOL IN ORIGINAL ACIDS (calculated)		32.2 per cent

The method requires about two days to complete a test but several can be run simultaneously. The keynote of success in this work is extremely careful and accurate attention to the details of manipulation. This test has been in use in our laboratories since the early part of 1916, and has given very satisfactory results.

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A NOTE ON SILICON-COATED METAL

By W. E. VAWTER

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The resistance of silicon to the corrosive action of acids and alkalies is taken advantage of in chemical industry by using apparatus constructed of iron which contains a large percentage of silicon. However, the high silicon castings have small tensile or compressive strength and breakage is high. It was therefore thought that, if iron could be satisfactorily coated with silicon, a great saving could be effected. Coating iron by dipping into molten silicon is unsatisfactory, since the melting points of the metals are similar and iron is soluble in fused silicon.

The object of a study made by the author was to investigate a process for coating a metal, preferably iron, with silicon, so that the non-resisting metal would be amply protected from any corrosive chemical, and at the same time stand up under severe handling, without injury to the object or the protective coating. Samples of iron were heated to temperatures of 400 to 600° C. in an atmosphere of silicon hydride,[1] in order to ascertain whether the gas would decompose and form a coating of silicon upon the surface of the iron.

EXPERIMENTAL

1—Iron wire was heated to 550° C. in a current of dry silicon hydride for one hour. Small patches of silicon formed over the surface of the wire, but when the sample was placed in a normal solution of sodium chloride, corrosion immediately started where no silicon was present, totally undermining the silicon coating after two weeks' immersion.

2—A repetition of Experiment 1. In this experiment the silicon did not appear to be undermined; the coating of silicon remained after two weeks' immersion, although corrosion had taken place rapidly at all other places.

3—This sample was heated to 500° C. for two hours. A thin film of silicon was deposited on the wire and the wire became badly pitted where there was no coating of silicon. The silicon coat was intact after two weeks' immersion.

4—This sample was heated for $3^1/_2$ hrs. at 550° C. Very little silicon adhered to the wire, and that which deposited was in scales and could easily be removed.

5—This sample was first pickled in acid and then heated in a current of silicon hydride at 550° C. for 2 hrs. The resulting coating was smooth and uniform in color, and the treated iron remained in salt solution four days before any corrosion was noticed; this was at the end where the wire had been cut and had no silicon protection.

6—The sample was first heated to 700 ° C. and then allowed to cool to 550° C. before exposing to the gas. While an excellent coat was obtained, which stood up well in the salt solution, no advantage in preheating could be observed.

7—This sample, after running two hours at 550° C., was further heated for 5 minutes at 700° C. Ten days' immersion in salt solution completely removed the coat. It was expected that the silicon would attach itself more firmly to the iron by the heat treatment, but the after-heating probably broke the coat at some place and allowed corrosion to set in.

8—An exact duplicate of Experiment 5 with similar results.

9—A piece of wire, which had been pickled, washed, dried and exposed to the air for several hours, until

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[1] The silicon hydride used in these experiments was prepared by treating an alloy of magnesium and silicon with a dilute solution of hydrochloric acid. The evolved gas consisted of about 5 per cent of silicon hydride and 95 per cent of hydrogen. The alloy was prepared by intimately mixing one part of powdered silicon with two parts of powdered magnesium and heating the mixture for two hours at 600° C. in an atmosphere of hydrogen. This alloy has the composition approximately represented by the formula $SiMg_2$.

a film of oxide had formed, was treated as in Experiment 5, but no silicon adhered to the wire.

10—A sample was heated at 550° C. for 10 min. and the temperature was then changed to 700° C. for the same period. This alternation was prolonged 1$^1/_2$ hrs. Little silicon deposited upon the wire and that which did had a tendency to scale off.

11—A sample treated by the method used in Experiment 5 was replaced in the tube and heated to 700° C., but the coat scaled.

12—This sample was heated 5 hours at 750° C. Silicon deposited upon the wire, but a slight bending would break the coat and it could be peeled off with the fingers. The wire did not heat uniformly and that portion which was maintained at a lower temperature did not give this fracture effect. The iron under the scale was unchanged, which would indicate that the silicon merely forms a shell over the iron.

Other metals, copper, nickel and aluminum, were also subjected to treatment, but no coating of any description was obtained.

It is interesting to observe that silicon will coat iron when applied by this method, under proper conditions; but no practical use can be looked for along this line. What was desired was an adherent, homogeneous deposit of silicon on iron; but the above experiments showed that only a thin and fragile shell formed over the iron.

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THE COMPOSITION OF MENHADEN OIL FATTY ACIDS[1]

By E. Twitchell

The object of this analysis was as much to test the use of my melting-point method of determining fatty acid mixtures as to investigate the true composition of the fatty acids found in menhaden oil.

In a previous paper[2] I described a method of determining the composition of mixtures of solid fatty acids, which consisted in adding a certain proportion of the mixture to be analyzed to a solvent consisting of a pure fatty acid of the kind to be determined, then finding the melting point and noting how much the original melting point of the solvent had been depressed. This depression was caused by all the acids in the mixture except the one sought, which, being identical with the solvent, would have no effect on it. Assuming that the other acids, singly or in mixture, produced a lowering of the melting point of the solvent proportional to their total concentration and independent of the kind of acid, it was a simple calculation to find the amount of these other acids and, by difference, that of the one sought.

The assumption that the lowering of the melting point of the sovlent acid is proportional to the percentage of foreign acids is very nearly true in most cases up to 20 per cent, though, if the solvent is an acid of unusually low melting point as compared with the dissolved acid, as for instance, behenic dissolved in myristic acid, the melting-point curve beyond 10 per cent of the dissolved acid is no longer a straight line and the depression caused by 20 per cent of this acid is abnormally great. In such cases only 10 per cent of the acid of higher melting point was used in the mixture.

The assumption that all acids produce the same lowering of melting point is for this purpose near enough to the truth where the more common fats and oils are concerned, the fatty acids of which do not differ greatly in molecular weight. I have generally taken 4° as the lowering of melting point caused by adding 20 parts of one fatty acid to 80 parts of another considered as the solvent. However, where the acids are to some extent known it will be more accurate to take values determined for each fatty acid. A number of these values I shall give below.

This as an analytical method is limited to fatty acids solid at ordinary temperatures, but can be extended by applying it to the solid fatty acids obtained from liquid acids either by the hydrogenation process or by fusion with caustic potash. The hydrogenation process, by the addition of hydrogen, converts unsaturated fatty acids into saturated ones having the same number of carbon atoms; oleic, linolic, linolenic and clupanodonic acids are all converted into stearic acid. Fusion with caustic potash produces a decomposition of members of the oleic series by which the principal product is a saturated fatty acid having two less atoms of carbon; oleic acid is converted into palmitic acid, erucic into arachidic, etc. Linolic acid, with two double linkages, is converted into myristic acid and it may be assumed that all acids of the linolic series on fusion with caustic potash yield a saturated acid with four less carbon atoms. As to the action of caustic potash on the still more unsaturated acids, so far as I know, no experiments have been made, though it has been assumed that acids with three double linkages (linolenic acid) would yield a saturated acid of six less carbon atoms and those having four double linkages (clupanodonic acid) would yield a saturated acid having eight less carbon atoms.

It is easily seen what an aid this power of converting liquid acids into solid acids according to definite laws can be toward arriving at the composition of a fatty acid mixture; and in my previous paper I made use of the hydrogenation process. In that paper I gave results of my analysis of menhaden oil fatty acids as follows:

	Per cent
Palmitic acid	22.7
Other solid, saturated acids	11.8
Unsaturated acids with 16 carbon atoms	None
Unsaturated acids with 18 carbon atoms	26.7
Unsaturated acids with 22 carbon atoms	20.2
Other unsaturated acids	18.6
	100.0

The vacancies in this analysis were due to my not having the pure standard acids to correspond with all of the fatty acids in the mixture. I had at that time only palmitic, stearic and behenic acids. I have since prepared myristic and arachidic acids, making the series of normal saturated fatty acids with even num-

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[1] Read at the 211th regular meeting of the Cincinnati Section, American Chemical Society, March 28, 1917.

[2] This Journal, 6 (1914), 564.

bers of carbon atoms complete between myristic and behenic acids inclusive.

PREPARATION OF MYRISTIC ACID

The fatty acids of nutmeg butter were repeatedly crystallized, first from petroleum ether and then from 76 per cent alcohol. In all there were thirteen crystallizations, the last few of which scarcely made any change in melting point or combining weight. The combining weight by titration with alkali was 228.5 (theory 228). The melting point was 53.76°.

PREPARATION OF ARACHIDIC ACID

No fat could be found that contained any considerable quantity of this acid; but rape oil contains erucic acid, which on fusion with caustic potash, should yield arachidic acid.

The fatty acids of rape oil were fused for 6 hrs. with caustic potash at 300° C. At the end of this time the liberation of hydrogen had ceased. The fatty acids were separated from the other products of the fusion, and, as arachidic acid seemed to be the acid of highest melting point in the mixture, it was a simple matter to separate it from the rest and to purify it by simple crystallization from petroleum ether and 90 per cent alcohol. Eight crystallizations gave an acid having a combining weight of 312 (the theoretical) and a melting point of 74.78°.

The following table gives the melting points of mixtures in pairs of all the series of five acids before mentioned, 80 parts being taken of one, considered the solvent, and mixed with 20 parts of each of the others. The method of determining the melting point was about as described in my previous paper. Correction was always made for the stem of the thermometer which extended above the bath.

	Melting point	M. p. of Mixture containing 20% Behenic acid	Lowering of m. p. of Solvent	M. p. of Mixture containing 20% Arachidic Acid	Lowering of m. p. of Solvent	M. p. of Mixture containing 20% Stearic Acid	Lowering of m. p. of Solvent	M. p. of Mixture containing 20% Palmitic Acid	Lowering of m. p. of Solvent	M. p. of Mixture containing 20% Myristic Acid	Lowering of m. p. of Solvent
Behenic acid	79.99	...	...	76.42	3.57	75.81	4.18	75.45	4.54	75.31	4.68
Arachidic acid	74.78	71.17	3.61	...	...	71.05	3.73	70.79	3.99	70.49	4.29
Stearic acid	69.30	...	4.02*	65.34	3.96	...	...	65.08	4.22	64.91	4.39
Palmitic acid	62.37	...	3.68*	...	3.88*	58.16	4.21	...	..	57.77	4.60
Myristic acid	53.76	...	...	...	3.62*	...	4.42*	49.23	4.52	...	..

*On account of the relatively high melting point of the dissolved acid the mixture was made with 90 parts solvent plus 10 parts dissolved acids. The lowering of melting point multiplied by 2 gives an imaginary value for the 80 + 20 mixture.

The figures given in this table are not quite to be considered as final, as I am not yet satisfied with the accuracy of my method of determining melting points and hope to improve it.

MYRISTIC ACID IN HYDROGENATED MENHADEN OIL

In the course of my previous work on menhaden oil a fractional distillation had been made of the fatty acids separated from the hydrogenated oil. There were twenty-one fractions in all.

In this distillate were found behenic, stearic and palmitic acids. The presence of arachidic and myristic acid seemed probable, but having neither of these acids in the pure state I could not at that time establish their presence in any of the fractions.

If any myristic acid were present it would very likely be found in the first fraction. To further concentrate it, a portion of this fraction was dissolved in alcohol, partially precipitated with lead acetate and filtered. The fatty acids were separated from the filtrate and melted in the proportion of 20 parts with 80 parts of myristic acid:

The m. p. of this mixture was	51.60°	
That of pure myristic acid is	53.76°	
The lowering of the m. p. was therefore	2.16°	
The lowering of the m. p. of myristic acid by 20 per cent of palmitic acid is	4.53°	
The percentage of myristic acid in the fatty acids under examination is therefore: 100 × (4.53 — 2.16)/4.53 =	52.3	Per cent,

which clearly establishes the presence of myristic acid in the hydrogenated oil. Whether it was present in the original oil or was formed from an unsaturated acid by hydrogenation will be determined later.

The value 4.53° (the depression caused by palmitic acid as given in the table) was used in this calculation because it was practically certain that the acids other than myristic in the substance were chiefly palmitic.

For more accurate quantitative results, another mixture should be made as described in my previous paper; that is, using so much of the substance to be examined that the lowering of melting point is about 4°.

ARACHIDIC ACID IN HYDROGENATED MENHADEN OIL

Fractions 15 and 16 of this same distillate had an average combining weight of 308.5 and were the most likely to contain arachidic acid. They were united and crystallized twice from 90 per cent alcohol at 15° C. The mean combining weight of the crystals was 324. In my previous work it had been shown that behenic acid (molecular weight 340) was present in the hydrogenated oil. It would almost certainly be in this fraction. It remained to determine whether the reduction in combining weight was due to arachidic acid,

20 parts of the crystals melted with 80 parts of arachidic acid had a m. p. of	72.95	
Arachidic acid has a m. p. of	74.78	
The lowering of the m. p. was therefore	1.83	
The lowering of the m. p. of arachidic acid by 20 per cent of behenic acid is	3.61	
The percentage of arachidic acid in the crystals is therefore 100 × (3.61 — 1.83)/3.61 =	49.3	Per cent,

which establishes the presence of arachidic acid in the hydrogenated oil.

As a matter of interest a test was made for behenic acid in the same fraction.

20 parts melted with 80 parts of behenic acid caused a lowering of m. p. of the latter of	1.96°	
The percentage of behenic acid in the substance is therefore 100 × (3.57 — 1.96)/3.57 =	45.1	Per cent

The percentage of arachidic acid in the total fatty acids of the hydrogenated oil, not distilled, was determined as follows:

20 parts were melted with 80 parts of arachidic acid. The mixture had a m. p. of	71.67
The m. p. of arachidic acid is	74.78
The lowering of m. p. is therefore	3.11
The lowering of the m. p. of arachidic acid by the other acids in the mixture is taken as	4.00
The percentage of arachidic acid is therefore 100 × (4.00 — 3.01)/4.00 =	22.2

which is either in the original oil or has been produced by hydrogenation.

THE SOLID FATTY ACIDS OF MENHADEN OIL

These were separated from the liquid acids by the method described in my previous paper. About 31 per cent were obtained, having, however, an iodine value of 9.44, equivalent to 10.5 per cent of oleic acid, which, for the purposes of this calculation, I shall assume it to be.

The menhaden oil which I used for this work was unfortunately not the same as that I had used in my former work. It was not so fresh and contained more free fatty acids than I could have wished. This may account for the rather high iodine value of the separated solid acids.

20 parts of these solid fatty acids melted with 80 parts of myristic acid, had a m. p. of	50.57
Myristic acid has a m. p. of	53.76
The lowering of m. p. is therefore	3.19
The lowering of the melting point of myristic acid by 20 per cent of palmitic acid is	4.53
The percentage of myristic acid in the solid fatty acids is therefore 100 × (4.53 — 3.19)/4.53 =	29.6

As a confirmation of this result and to leave no doubt that myristic acid was really present, the above solid fatty acids were dissolved in alcohol and enough lead acetate added to precipitate about one-half. The fatty acids separated from the filtrate, tested as above, gave 45.9 per cent of myristic acid.

20 parts of the same solid fatty acids melted with 80 parts of arachidic acid had a m. p. of	70.57
Arachidic acid has a m. p. of	74.78
The lowering of m. p. is therefore	4.21
The lowering of the m. p. of arachidic acid by 20 per cent of palmitic acid is	3.99
The percentage of arachidic acid is therefore 100 × (3.99 — 4.21)/3.99 = —5.5 per cent =	None

In the same way the percentage of palmitic acid in the solid fatty acids was found to be 52.9 per cent, and of stearic acid 5.7 per cent, and as the oleic acid, from the iodine value, was 10.5 per cent, the composition of the solid acids as separated, is:

	Per cent
Myristic acid	29.6
Palmitic acid	52.9
Stearic acid	5.7
Arachidic acid	None
Oleic acid	10.5
	98.7

As the menhaden oil fatty acids contained 31 per cent of acids of the above composition, the amount of myristic acid in the menhaden oil fatty acids is 29.6 × 0.31 = 9.2 per cent and of stearic acid 5.7 × 0.31 = 1.8 per cent.

In my previous paper I expressed a doubt as to whether there was any stearic acid in the mixture, though the tests gave small positive figures. This doubt might still exist, and to decide this point a fractional precipitation was made of the fatty acids of menhaden oil dissolved in alcohol, using only enough lead acetate to precipitate about 2 per cent of fatty acids. This precipitate, on decomposing, yielded fatty acids testing 26.5 per cent of stearic acid. There can be no doubt of the presence of stearic acid in the original oil.

GENERAL RESULTS

Having found myristic acid in both the original and the hydrogenated fatty acids in equal amounts, it was not produced by hydrogenation. On the other hand arachidic acid was found only in the hydrogenated and not in the original fatty acids. It was therefore produced by the addition of hydrogen to an unsaturated acid with 20 atoms of carbon.

The composition of menhaden oil fatty acids given in my paper of July 1914, can now be filled out as follows:

	Per cent
Palmitic acid	22.7
Myristic acid	9.2
Stearic acid	1.8
Unsaturated acids with 16 carbon atoms	None
Unsaturated acids with 18 carbon atoms = 26.7 less 1.8 per cent stearic acid =	24.9
Unsaturated acids with 20 carbon atoms	22.2
Unsaturated acids with 22 carbon atoms	20.2

As to the series to which the unsaturated fatty acids with the different numbers of carbon atoms belong, the method of fusion with caustic potash above mentioned will give some information. On fusing the menhaden oil fatty acids with caustic potash and separating the higher fatty acids from the product, a mixture was obtained which would not crystallize from 90 per cent alcohol except at very low temperatures, and this already indicated that it contained no arachidic acid. It was crystallized from 76 per cent alcohol.

20 parts of the crystals melted with 80 parts of arachidic acid had a m. p. of	71.04
The m. p. of arachidic acid is	74.78
The lowering of m. p. was therefore	3.74
The lowering of the m. p. of arachidic acid by 20 per cent of stearic and palmitic acids, is	3.86
The percentage of arachidic acid is therefore 100 × (3.86 — 3.74)/3.86 =	3.2

As this was in a small fraction which should contain all of the arachidic acid present, the amount in the total product of the fusion would be very small if, indeed, it can be inferred from the above low figure that its presence has been proved at all. From this it can be concluded that the unsaturated acid with 22 carbon atoms found in menhaden oil and which, like erucic acid, is converted by hydrogenation into behenic acid, is nevertheless not erucic acid, since on fusion with caustic potash it is not converted into arachidic acid. It is probably a more unsaturated acid which is converted into stearic or palmitic acid by the fusion.

The above fraction was also tested for palmitic and stearic acid and found to contain: Palmitic acid, 73.6 per cent; stearic acid, 23.6 per cent.

The factors for lowering of melting point given in the table, although still somewhat uncertain, cannot be far enough from the true ones to affect the results very seriously. Even if the average value, 4°, for the depression caused by mixing 20 parts of one acid with 80 of another had been used in my calculations, the general conclusions as to the composition of menhaden oil would not have been changed.

As to the usefulness of this method, I know of no other way of determining the composition of a mixture of solid fatty acids of more than two constituents, especially where only a small quantity is available. Even with large quantities, though it might be possible by means of numerous fractional precipitations or distillations to separate some of each of the constituents in a pure state, a quantitative result can never be obtained in this way.

Wyoming, Ohio

NOTE ON THE DETERMINATION OF STRONTIUM AND LITHIUM IN WATER

By S. D. Averitt

Received December 13, 1916

There are probably no determinations in water analysis which on the whole require more time and work than those of strontium and lithium.

Several years ago while coöperating with the Referee on Water Analysis for the Association of Official Agricultural Chemists, the methods which are now official for strontium and lithium were tested. The great amount of time and work required for these determinations led the writer somewhat later to investigate the accuracy of indirect methods which were sound in theory and which, with careful work, must prove accurate provided there was a really determinable amount of strontium or lithium present; that is, an amount that would not be too seriously affected by the experimental error unavoidable in the most careful work. This investigation led to the conclusion that the indirect methods employed were as accurate as the official methods and the time and labor saved was a matter of considerable importance.

It is self-evident that pure preciptates and careful work are necessary for indirect methods, the lack of which has undoubtedly had much to do with the unfavorable opinion many chemists entertain relative to them.

It is believed that a brief statement of the methods will be of general interest to water analysts and others who have occasion to determine strontium and lithium, as they differ materially from other indirect methods that have been proposed, at least in their application to the separation of calcium and strontium and the extent to which they are carried in the separation of the alkalies. In the brief statement which follows, no details of procedure will be given as they should be perfectly familiar to any analyst of reasonable experience.

The official method (Stromeyer-Rose) for the determination of strontium begins with the weighed oxides of calcium and strontium (CaO and SrO). These are dissolved in nitric acid and brought to dryness, the separation of strontium depending upon the insolubility of strontium nitrate in alcohol-ether mixture.

The writer's method is as follows: Dissolve the weighed oxides in hydrochloric acid and precipitate again as oxalates as in the first case; filter, wash, dissolve in sulfuric acid and titrate with standard $KMnO_4$, noting the exact volume of $KMnO_4$ required.

If W = weight of CaO and SrO.
O = total oxygen in CaO and SrO (found by the titration with $KMnO_4$).
X = O in CaO.
Y = O in SrO.

Then $X + Y = O$ (1)
and $3.5044X + 6.4769Y = W$ (2)

Solving for Y, determine SrO ($6.4769Y$) then W — SrO = CaO. When the value of Y is found, in order to get SrO a constant factor occurs which is $\frac{6.4769}{6.4769 - 3.5044}$ or 2.179. Consequently it is not necessary to solve these equations in order to get the weight of SrO, which is obtained as follows:

(W — CaO equivalent of $KMnO_4$ titration) 2.179 = SrO. (3)

In order to test the accuracy of the above method, pure $CaCO_3$ equivalent to 0.9954 g. CaO and $SrCO_3$ equivalent to 0.1000 SrO were dissolved in hydrochloric acid and made up to 500 cc.; 50 cc. aliquots equivalent to 0.09954 g. CaO and 0.0100 g. SrO were used, giving the results shown in Table I.

Table I—Determination of Strontium (Results in Grams)

Expt. No.	CaO + SrO Present	CaO + SrO Found	CaO Equivalent of $KMnO_4$	SrO Present	SrO Found
1	0.1095	0.1095	0.1048	0.0100	0.0102
2	0.1095	0.1096	0.1052	0.0100	0.0096
3	0.1095	0.1096	0.1049	0.0100	0.0102
4	0.1095	0.1096	0.1051	0.0100	0.0098
5(*a*)	0.1095	0.1097	0.1052	0.0100	0.0099

(*a*) Precipitated in presence of magnesium chloride.

The oxalates were washed with a 1/2 per cent solution of ammonium oxalate in all cases. Those which were to be titrated were finally washed with 15 cc. of cold water in 5-cc. portions dropped from a pipette around the top of the filter fast enough to cover the precipitate, letting the filter run dry between each washing. This amount of washing with water is sufficient for aliquots containing approximately 0.0300 g. of oxides and it is not advisable to have much larger aliquots for titration nor to use more water in washing, otherwise strontium oxalate will be dissolved.

If the double oxides weigh more than 0.0500 g., aliquots should be taken for the $KMnO_4$ titration. The $KMnO_4$ should not be stronger than $N/10$.

The Official Method for the determination of lithium (Gooch) begins with the weighed chlorides (NaCl, KCl and LiCl) and depends upon the insolubility of sodium and potassium chloride in absolute amyl alcohol.

The writer's method is to make the solution in water of the weighed chlorides of sodium, potassium and lithium up to convenient volume. Take an aliquot for the determination of K from which KCl and the Cl in the same becomes known. Another aliquot is titrated with standard $AgNO_3$ from which the total Cl is obtained.

If W = weight of NaCl + KCl + LiCl.
C = weight of total chlorine (as found by titration with $AgNO_3$).
Cl in NaCl + LiCl = C — Cl in KCl.
X = Cl in NaCl and Y = Cl in LiCl.

Then, $X + Y = C - Cl$ in KCl (1).
and $1.6486X + 1.1957Y = W -$ KCl (2).
Solving for Y find weight of LiCl ($1.1957Y$) in getting the weight of LiCl from Y a constant factor occurs, *viz.*, $\frac{1.1957}{1.6486 - 1.1957}$ or 2.64, and as in the case of strontium it is not necessary to solve the equations.

The weight of LiCl is obtained as follows:

The NaCl equivalent of $C - Cl$ in KCl $-$ ($W -$ KCl) multiplied by 2.64 = LiCl (3).

In order to test the accuracy of the method, 0.5000 g. NaCl, 0.1000 KCl and 0.0100 LiCl were dissolved in water and diluted to 100 cc. Spectroscopic tests showed a small quantity of potassium in the sodium chloride and a trace of sodium in the potassium chloride. This was immaterial since no lithium was shown in either the sodium chloride or potassium chloride nor was there any lithium in the calcium and magnesium chlorides used, but a small quantity of sodium and a trace of potassium were indicated in both. The results appear in Table II.

TABLE II—DETERMINATION OF LITHIUM (RESULTS IN GRAMS)

Expt. No.	KCl + NaCl + LiCl		LITHIUM LiCl	
	Present	Found	Present	Found
1	0.6100	0.6098	0.0100	0.0106
2(*a*)	0.1525	0.1569	0.0025	0.0025

(*a*) Determination in a solution containing calcium and magnesium chlorides.

The determination of lithium by this method is based upon two other definite determinations, potassium and chlorine, than which, in the hands of a skilful analyst and under the conditions of this method, no determinations are more accurate.

The only other factor affecting the accuracy of the method is an impurity in the weighed chlorides and under ordinary conditions this can result only from careless work or inexperience.

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NITRATE DETERMINATIONS IN THE PRESENCE OF CHLORIDES

By W. F. GERICKE

Received February 2, 1917

The presence of chlorides in solutions on which nitrate determinations are to be made by the colorimetric method has long been a source of trouble to the analytical chemist. When nitrates are present in large amounts in solutions containing chlorides, determinations can very easily be made by the use of some of the reduction methods. Since, however, determinations for nitrates are often called for in solutions in which the amounts present are small, the quantity of ammonia produced by the reduction of the nitrates is of such magnitude as to often introduce a considerable error due to manipulation. It is under such conditions that the phenoldisulfonic acid method for nitrate determination is often employed, and in the absence of chlorides has been found to be sufficiently accurate and expeditious.

The effect of chlorides on nitrate determinations has been the subject of some study and has been duly reported in chemical papers. It is not deemed necessary to review here the literature on the subject. Suffice it to mention some of the results of the more important investigations.

(1) Chlorides cause losses of nitrates in determinations made by the phenoldisulfonic acid method.

(2) The loss of nitrates is not occasioned by the evaporation of the aqueous solution to dryness prior to the addition of the phenoldisulfonic acid.

(3) The loss of nitrates occurs when the phenoldisulfonic acid is added to the residue from the evaporated solution.

(4) The use of precipitants to remove the chlorides prior to the evaporation of the aqueous extract is recommended for accurate determinations.

(5) The use of calcium oxide and also calcium carbonate for the clarification of aqueous extracts, especially from soils, is recommended as a precipitant that is both efficient and non-interfering in the nitrate determinations.

The result of investigations, the conclusions of which have been briefly stated above, indicates that the presence of chlorides interferes with the reactions at a certain point in the process of the determinations—namely, when the acid and dry salt containing the nitrates and chlorides come into contact. This results in the production of heat with the liberation of both chlorine and nitric acid, and thus interferes with the proper reaction of the latter with the phenoldisulfonic acid. Working on the principles enunciated by the investigators studying the colorimetric method of nitrate determinations, it occurred to me to try a method by which total evaporation of the nitrate-bearing solution to dryness could be obviated together with the necessity of adding the acid to the dry residue. Since the phenoldisulfonic acid reagent is a mixture of sulfuric acid and phenoldisulfonic acid it seems that the proper condition for the reaction of the phenoldisulfonic acid and the nitrates is in a sulfuric acid solution. By the addition of sulfuric acid to the nitrate-containing solution, a condition is brought about by which the complete evaporation to dryness of the aqueous solution may be obviated. When the phenoldisulfonic acid is then added to the acid-treated nitrate solutions some nitrophenoldisulfonic acid is formed. The complete reaction, however, will take place when the proper concentration of the solution has been attained. To attain this concentration and to employ temperature to accelerate the reaction of the nitrates and the phenoldisulfonic acid is the purpose of the partial evaporation to which the samples are subjected.

In making nitrate determinations one must remember that the theoretical reaction that elucidates the principle of the method goes to completion for quantitative determination only when an excess of phenoldisulfonic acid is used. Therefore, proper care should be taken in the preparation of the sample that the amount of nitrate present in the sample be neither too large nor too small to introduce measurable error due to the excessive or insufficient use of a given measure of the required acid reagents. Too much acid may seriously affect the accuracy of the determinations of samples in which the nitrate content is small, because of the action of the acid on the salts, and the increase in temperature involved.

The phenoldisulfonic acid reagent, as used in the laboratory, was prepared according to the Chamot method, *i. e.*, an addition of fuming sulfuric acid is employed to insure pure phenoldisulfonic acid, free from all traces of phenolmonosulfonic acid.

The quantity of the sample used for analysis must be determined by the amount of nitrates in the aqueous solution. If the nitrate content is from 1 to 5 parts per million, a sample of at least 50 cc. should be used. To the sample, placed in a casserole or beaker, 1.5 cc. of concentrated sulfuric acid are added with constant stirring, then 2 cc. of phenoldisulfonic acid reagent. The casserole is then placed on a water bath, and most of the solution evaporated at the ordinary temperature of the steam bath. The last part of the evaporation, however, should be performed at a temperature preferably not over 70° C.

The evaporation should proceed until the original solution is concentrated to a quantity varying from 6 or 7 to 12 or 14 cc. The point to which evaporation must be continued is determined by the amount of nitrates in the original solution; for a low nitrate content a greater concentration of the original solution will be necessary. The proper concentration is determined by the color of the solution, which resembles that of phenoldisulfonic acid, slightly tinged with yellow. This condition will come, and final evaporation be attained at about the time that acid, due to the presence of chlorine, can be detected in the evaporating vapors. The important thing to observe in this modification is to bring about the final evaporation at a relatively low temperature. In no case should the solution be materially colored and turbid, although it may be somewhat darkened. A colored solution will result in an off tint when the alkali is added and will necessarily interfere with the accuracy of the determination. When the evaporation of the solution to its proper concentration has been accomplished, about 50 cc. or more of water are added; the solution is then neutralized with an alkali, care being taken to avoid the formation of excessive temperature when the acid is neutralized. The solution is then placed in the colorimeter and compared with a standard previously prepared, which may be made up according to the old method of total evaporation to dryness before adding the reagents, or according to the modification here reported, either of which will give the proper tint.

RESULTS OBTAINED

Table I indicates the feasibility of adding the phenoldisulfonic acid reagent before evaporation to dryness. Table II indicates the practicability of the method even if relatively large amounts of sodium chloride are present. If the nitrate content is very low and the salt very high, for instance, 1 part per million of nitrate and 1 per cent NaCl, determinations were subject to serious errors, due to loss of nitrates and discoloration of the solution. In cases of very low nitrate and high salt content, evaporation of the solution to its proper concentration should be performed at a much reduced temperature and preferably under partial vacuum, in order to reduce the action of acids on the salts.

TABLE I

50 cc. Solution in Sample
1.5 cc. Sulfuric Acid Added
2 cc. Phenoldisulfonic Acid Added

NITRIC NITROGEN P. p. m.	Theoretical Amount in cc. of Standard	ACTUAL READING in cc. of Standard	CHANGE IN TINT
1	50	49	None
1	50	50	None
2	100	100	None
2	100	98	None
5	500	500	None
5	500	500	None
20	1000	1000	None
20	1000	1020	None

TABLE II

50 cc. Solution in Sample

NITRIC NITROGEN P. p. m.	NaCl Added mg.	Per cent NaCl Present	Theoretical Amount in Cc. of Standard	Amount Recovered (Cc. of Stan.)	CHANGE IN TINT
1	100	0.1	50	50	None
1	100	0.1	50	50	None
1	250	0.25	50	50	None
1	250	0.25	50	48	None
1	500	0.50	50	49	None
1	500	0.50	50	50	None
1	750	0.75	50	50	Slightly colored
1	750	0.75	50	46	Colored
1	1000	1.00	50	35	Badly colored
1	1000	1.00	50	30	Badly colored
5	500	0.50	250	250	None
5	500	0.50	250	250	None
10	1000	1.00	500	500	None
10	1000	1.00	500	500	None

SUMMARY

I—A satisfactory modification of the phenoldisulfonic acid method of nitrate determination in presence of chloride salts has been found.

II—Total evaporation of the solution to dryness can be obviated by the use of concentrated sulfuric acid, which is added to the aqueous extract.

III—The addition of sulfuric acid and phenoldisulfonic acid to the aqueous solution prevents the loss of nitrates, which in the old method was due to the action of acid added to the dry salt after evaporation to dryness had been accomplished.

IV—The temperature at which the final evaporation and concentration of the aqueous solution is performed should not exceed 70° C.

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A NOTE ON THE PHENOLSULFONIC ACID METHOD FOR NITRATES IN WATERS HIGH IN MAGNESIUM SALTS

By M. STARR NICHOLS

Received February 28, 1917

In the phenolsulfonic acid method for the determination of nitrates in water supplies, as given in the "Standard Methods of Water Analysis of the American Public Health Association," no provision is made for the removal of the hydroxides of magnesium and iron which may form on the addition of the alkali hydroxide used to neutralize the excess phenolsulfonic acid. In this laboratory, until recently, the colored solution containing the nitrated sulfonic acid has been filtered to remove these hydroxides and the filter paper subsequently washed.

In the absence of the hydroxide of iron, the filtration may be dispensed with and the color read immediately if a quantity of an ammonium salt is added to hold the magnesium in solution. There are two advantages to this procedure. In the process of filtering a deeply colored solution there always remains a small quantity of the color in the filter paper; and in the second place if a large number of samples are being handled each day, the time factor is worthy of consideration.

The quantity of ammonium salt required is dependent upon what alkali is used for the neutralization of the excess acid. If ammonium hydroxide is used, 5 cc. of a saturated solution of ammonium chloride will be found sufficient to hold in solution the magnesium which will be present in from 20 to 50 cc. of most waters. If sodium or potassium hydroxide is used there will be required a larger quantity of the ammonium salt, owing to the result of mass action between the alkali hydroxide and the ammonium salt, and the amount required will be in proportion to the amount of alkali added in excess. The addition of the ammonium salt is made after the neutralization and before the final dilution.

The writer has found this slight change in procedure quite serviceable in routine water analysis.

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LEVULOSE THE PREPONDERANT SUGAR OF APPLE JUICES[1]

By JOHN R. EOFF, JR.

Received February 19, 1917

Thompson and Whittier[2] state that levulose is the predominant sugar of apple juice and give the percentages of sugars found in the juice of one variety of apple at different stages of ripeness. Browne[3] states that apples contain levulose in excess of dextrose, but gives no figures. Worcollier[4] says that dextrose and levulose compose 60 per cent to 95 per cent of the sugars present in apple juices and in the proportion of 2.8 to 10 times as much of levulose as of dextrose. As far as the author is able to find out, no systematic investigation of any large number of apple juices from various varieties of apples grown in different localities has been made to prove or disprove that levulose is always in greater quantity than the other sugars present in such juices. The object of this paper is to give the results of some work done on various varieties of apple juices, and is the first of what the author hopes to make a series of several papers treating of the amounts of the different sugars in apple juices.

Twenty varieties of apples were secured through the courtesy of the Virginia Agricultural Experiment Station, at Blacksburg, Va. The fruit had been grown in the orchards of the station, carefully selected as representative and true to name, and picked and immediately shipped when market-ripe. Fifteen of the varieties were native and were the commercial apples best adapted to the section where grown. Five varieties were French cider apples, and had been gathered from trees 14 years old. The juice was thoroughly expressed from the fruit after grinding in a small meat grinder, a very efficient hand press being used for the purpose. The analysis was undertaken immediately after pressing.

The method followed in the examination of the juices was substantially that detailed by Thompson and Whittier: 26 g. of juice were weighed into a 100 cc. flask, clarified with a slight excess of lead acetate, the lead being removed with a slight excess of potassium oxalate after completing the volume. In some instances alumina cream was used in conjunction with lead acetate. The solution was polarized in a 200 mm. tube at 20 and 87° C. before and after inversion with invertase, the instrument used being one of Schmidt and Haensch's make, Ventzke scale. The calculations were made by the following formulas:

$$L = \frac{a - a'}{0.624} \qquad D = \frac{b'}{0.794} - L \qquad S = \frac{a - b}{1.317}$$

ANALYSES OF APPLE JUICES

						PER CENT OF SUGARS AS DETERMINED						PER CENT OF SUGARS OCCURRING AS		
Variety of Apple	Sample Analyzed	Acidity of Juice	Gravity of Juice	Temp.		SUCROSE	LEVULOSE		DEXTROSE		TOTAL			
NATIVE	1916	(a)	Brix	° C.	(b)	(c)	(d)	(e)	(d)	(e)	(d)	Sucrose	Levulose	Dextrose
Mother	10/16	47.0	11.8	23	2.2	2.3	5.8	6.4	1.7	2.2	9.7	22.7	59.8	17.5
Grimes (*Golden*)		63.5	12.3	23	3.6	3.9	5.8	6.4	0.5	1.2	9.9	36.4	58.6	5.0
Arkansas		69.0	11.9	23	1.5	1.5	5.8	6.2	2.6	3.0	9.9	15.2	58.6	26.2
Limbertwig		66.0	11.1	23	0.8	0.7	5.0	5.4	3.5	2.3	9.3	8.6	53.8	37.6
York Imperial		67.5	11.8	23	1.1	1.3	6.3	6.6	2.5	2.7	9.9	11.1	63.6	25.3
Lankford	10/24	59.0	12.6	24	2.4	2.6	...	6.8	...	2.2	..	..	..	..
Gano		63.0	11.2	23	1.3	1.8	5.9	6.0	3.2	2.6	10.4	12.5	56.7	30.8
Peck		67.5	12.0	23	2.4	...	6.3	6.2	3.3	2.3	12.0	20.0	52.5	27.5
Northern Spy		56.0	12.0	23	2.5	3.7	6.2	7.1	1.7	1.7	10.4	24.0	59.6	16.4
White Pippin		50.5	11.9	23	2.4	2.7	5.9	6.8	2.2	1.8	10.5	22.9	56.2	20.9
Plumb Cider	10/27	73.5	10.5	23	0.6	0.6	5.9	6.7	1.4	1.6	7.9	7.6	74.7	17.7
Rome (*Beauty*)		44.5	11.5	22	2.2	1.9	6.1	6.8	1.4	1.9	9.7	22.7	62.9	14.4
Yellow Newton		53.0	13.1	22	3.5	3.1	5.9	6.8	1.8	2.4	11.2	31.2	52.7	16.1
Stayman Winesap		72.0	13.5	22	3.2	3.1	6.3	6.8	2.1	2.4	11.6	27.6	54.3	18.1
Ben Davis		52.0	11.2	22	1.2	1.0	5.9	6.5	2.5	2.7	9.6	12.5	61.5	26.0
FRENCH														
Bonne-de-Frieulles	11/2	29.0	16.3	22	2.6	2.9	8.5	8.9	2.7	3.0	13.8	18.8	61.6	19.6
Amère-de-Berthecourt		16.0	11.1	22	1.0	1.0	6.6	7.3	1.5	1.8	9.1	11.0	72.5	16.5
Godard		30.0	15.4	22	3.6	3.6	7.1	7.4	1.5	2.0	12.2	29.5	58.2	12.3
D'Arroles		148.0	14.2	21	2.2	2.4	6.8	7.2	2.3	2.5	11.3	19.5	60.2	20.3
Amère-du-Surville		41.5	11.7	21	0.2	0.4	7.2	7.6	2.3	2.2	9.7	2.1	74.2	23.7

(a) Cc. N/10 alkali per 100 cc. of juice. (b) Clerget. (c) Acid inversion, Cu reduction. (d) Optically. (e) Browne.

Where

a = Direct reading at 20° C. b = Invert reading at 20° C.
a^1 = Direct reading at 87° C. b' = Invert reading at 87° C.
S = Per cent sucrose. L = Per cent levulose. D = Per cent dextrose.

The different sugars were also determined by Browne's[1] method using Munson and Walker's table,[2] for the reducing sugars and the following formulas for the calculations:

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[1] Published by permission of the Commissioner of Internal Revenue.

[2] Delaware College Agricultural Experiment Station, *Bull.* **102**, 1913.

[3] *J. Am. Chem. Soc.*, **23**, 877.

[4] *Ann. fals.*, **2**, 425–7.

[1] *J. Am. Chem. Soc.*, **28** (1906), 439.

[2] U. S. Bureau of Chemistry, *Bull.* **107**, rev., 243–51.

$$\frac{0.793R + S - P}{2.08} = y \qquad R - 0.915y = x$$

where

x = Per cent dextrose. y = Per cent levulose.
R = Reducing sugars as dextrose.
P = Polarization of a sucrose normal weight on a saccharimeter, 20° C.
S = Sucrose

The data obtained from the investigation will be found in the accompanying table. It will be noticed that in every instance the amount of levulose exceeds the combined amounts of the other sugars present, and though this work is by no means exhaustive, it by so much substantiates the statements previously made that levulose is the sugar predominating in apple juices.

U. S. INTERNAL REVENUE LABORATORY
WASHINGTON, D. C.

THE INFLUENCE OF CALCITE INCLUSIONS ON THE DETERMINATION OF ORGANIC CARBON IN SOILS

By EDMUND C. SHOREY AND WM. H. FRY

Received February 28, 1917

It has been pointed out by McCaughey and Fry[1] and by Robinson[2] that soils derived from limestones, especially those of the Limestone and Uplands Province, are peculiar in containing inclusions of calcite and iron oxide in quartz. In a recent mineralogical examination[3] of a number of samples of Hagerstown silt loam, one of the important soil types of the Limestone and Uplands Province, the almost universal presence of calcite inclusions in quartz was again brought to our attention and some consideration has been given their significance.

Quartz is among the most resistant soil minerals and it is evident that crystals of calcite inclosed in quartz, so long as such quartz particles were intact, could have no agricultural significance. In fact, such calcite probably would be a less likely source of soluble calcium in a soil than the calcium-bearing silicates.

The analytical significance of such calcite inclusions cannot, however, be ignored, for there are several determinations commonly made by agricultural chemists where their presence would introduce an error.

In carbonate or carbon dioxide determination, where the carbon dioxide is set free from carbonates by a stronger acid, it is evident that any carbonate completely protected by inclusion in quartz would not be determined by any of the methods now in use. Since, however, such calcite or carbonate has no immediate agricultural significance, this error may be ignored where the determination is made for the purpose of indicating proper agricultural practice.

Among the methods used to determine approximately the organic matter in a soil is one that depends on determining the total carbon and calculating this to organic matter by the use of an empirical factor. Two methods are in use for determination of total carbon: combustion in a current of oxygen with or without copper oxide; or, oxidation of the organic matter to carbon dioxide by some moist oxidizing reagent and determination of the carbon dioxide evolved by one of the usual methods. In both cases determination of carbon dioxide evolved from any carbonate present is made on a separate sample and the quantity so obtained deducted from that total obtained on combustion, the difference being stated as organic carbon.

The first method, usually designated the cupric oxide method, is not subject to much modification and is considered an absolute method by which others are to be judged. On account of its tedious character, however, other methods, usually designated moist combustion methods, have been devised. These usually depend on the oxidizing effect of boiling concentrated sulfuric acid to which potassium dichromate has been added; and proposed modifications of this method relate chiefly to the method of determining the carbon dioxide and the design of the apparatus used.

In determining the organic carbon in a soil containing inclusions of calcite in quartz by either of these methods, it would seem from theoretical considerations that in the case of the moist combustion method the carbon dioxide of included calcite would not be set free, while in the case of combustion with cupric oxide the high temperature would decompose the calcite, rupture the quartz grains and the carbon dioxide set free would be calculated as organic carbon, since it would not appear in the ordinary carbonate determination.

Examination of 28 samples of Hagerstown silt loam for calcite inclusions disclosed their presence in all samples but one. One of these, a sample from Chattooga County, Georgia, was selected as suitable for determining whether or not these theoretical considerations would be borne out in practice.

A sub-sample ground to pass a sieve 80 meshes to the inch, well mixed and dried at 105° C., was used throughout, all results being stated as per cent of the dried soil. The following determinations were made: (1) Combustion with cupric oxide in a current of oxygen;

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[1] Bureau of Soils, *Bull.* **91**, 1913.

[2] Department of Agriculture, *Bull.* **122**, 1914.

[3] The identification of calcite inclosed in quartz in the soils examined was carried out in the manner usual in work with the petrographic microscope. The soil was mounted in an oil having a known refractive index, preferably very close to the index of quartz as this mineral is thereby rendered practically invisible when the analyzing nicol is removed from the microscope tube.

When the nicols are crossed the calcite grains are readily "spotted" by their high interference colors. The work from that point on is simply confirmatory. Rarely the calcite grains show crystallographic outlines and in these cases the index of the grain can be determined by rotating the stage until the grain disappears and comparing the actual index thus noted with the index calculated for a calcite grain having the same orientation as the grain under consideration. The two indices thus obtained should of course be identical.

The chief difficulty in this procedure is that often the interference figures of the quartz and calcite mutually clash, but with patience grains can be found showing the figures with sufficient clearness to enable the grain to be roughly oriented. When all the conditions are favorable as outlined above, there is no doubt as to the identity of the grains under observation. In other cases the extremely high interference colors practically settle the question, the only probable source of error being the confusion of calcite with dolomite, but so far as any analytical error introduced by the inclusions in quartz are concerned it makes no difference whether they are calcite or dolomite.

So far as is known at present there is no other method of determining roughly the quantity of a given mineral on a slide except actual count of sized material, or measurement of heterogeneous material, and in this latter case, as in rock sections, the material must be of uniform thickness. In the case of inclusions such as those under consideration it is obviously out of the question to apply either of the methods mentioned above, and consequently the quantity of the inclusion present cannot be stated in quantitative terms.

(2) combustion with concentrated sulfuric acid to which potassium dichromate had been added; and (3) combustion of the residue after moist combustion with cupric oxide in a current of oxygen. Determination of the carbonate in the soil gave results less than 0.01 per cent carbon and this has been ignored as a negligible quantity. It was originally proposed to subject the residue from the treatment with sulfuric acid and dichromate after washing and drying to combustion with cupric oxide, but it was found that this material had absorbed a considerable quantity of chromium compounds and gave so much sulfur trioxide on heating that it was not possible to obtain concordant results for carbon when treated in this way, and in place another portion of soil was treated with sulfuric acid and potassium sulfate as in a Kjeldahl digestion, heated until all organic matter was destroyed and the residue perfectly white, washed, dried and weighed.

The following results were obtained, all figures being the mean of three closely agreeing results. The figures obtained on combustion of the residue after treatment with sulfuric acid and potassium sulfate were calculated to original soil.

TOTAL CARBON

	Per cent
Moist combustion	0.71
Cupric oxide combustion of residue from moist combustion	0.12
In soil	0.83
Cupric oxide combustion	0.80

It is seen from these figures that there is a reasonably close agreement between the total carbon obtained by direct cupric oxide combustion of the soil, and that obtained by adding together that obtained on moist combustion and cupric oxide combustion of the residue.

It has been frequently noted that moist combustion methods have given lower figures for total carbon in soils than those obtained with cupric oxide, and with the idea that the latter was an absolute method, it has been held that the moist combustion method frequently gives figures lower than the actual amount. The results obtained indicate that if this discrepancy is found in the case of soils containing calcite included in quartz, the facts are quite the contrary so far as total organic carbon is concerned.

In other words, in the case of soils containing calcite inclusions, the carbon dioxide of such calcite will not appear in the ordinary carbon dioxide determination but will appear in the cupric oxide combustion and be calculated to organic carbon. This is not the case with the moist combustion method and it would seem fair to assume that in case of soils containing calcite inclusions the figures obtained for organic carbon by this method are more nearly the true value than those by the cupric oxide method.

It should be stated in this connection that in order to obtain all the carbon dioxide in combustion of such soils by the cupric oxide method the heating must be prolonged much beyond the time ordinarily taken for the oxidation of the organic matter. It would seem that the decomposition of the included calcite and the escape of the carbon dioxide by the rupture of the quartz grains proceeds slowly, and if the combustion is stopped at the end of the period usually adopted, calcite inclusions can still be found under the microscope and the material will give more carbon dioxide on further heating.

Theoretically if the soil were ground so fine that all calcite inclusions were exposed the carbon dioxide of such calcite would appear in the preliminary carbon dioxide determination and no error would be introduced in the subsequent combustion by the cupric oxide method, but it does seem possible to accomplish this in practice. It is evident that the inclusions are exceedingly minute, for the carbon dioxide calculated to carbon in the preliminary carbon dioxide determination was less than 0.01 per cent, while that obtained from the calcite inclusions by cupric oxide combustion was 0.12 per cent, working with a soil ground to pass a sieve of 80 meshes to the inch. The residue from a moist combustion after washing and drying was ground to pass a sieve 130 meshes to the inch and then leached with hydrochloric acid, and after this treatment calcite inclusions were still abundant when examined under the microscope.

It is, of course, apparent that in any comparison of methods all determinations should be made on samples ground to the same degree of fineness.

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

THE ACTION OF SOLUTIONS OF AMMONIUM SULFATE ON MUSCOVITE

By R. F. GARDINER AND EDMUND C. SHOREY

Received February 16, 1917

Muscovite, one of the micas, is a mineral frequently found in the rocks from which soils are formed and is a commonly occurring mineral in soils. In some soil series it is one of the characteristic or predominating minerals, for instance in a sample of Gloucester stony loam after mechanical separation the sands were found to contain 5 per cent of muscovite and the silt 30 per cent. In the same way a sample of Penn silt loam was found to contain 6 per cent of muscovite in the sand and 9 per cent in the silt.[1]

Muscovite is usually considered one of the most stable of soil minerals. Clark says,[2] "Muscovite, under ordinary circumstances, is one of the least alterable of minerals. The feldspar of a granite may be completely kaolinized while the embedded plates of mica retain their brilliancy unchanged."

McCaughey and Fry say,[3] "The micas are persistent minerals in soils and show but little evidence of alteration, a fraying of the edges at times being quite characteristic. If alteration of the micas has taken place it has not influenced their optical properties. It would appear that micas are the most stable of the potash minerals found in soils except microline."

Johnstone reports that after suspension of mica for a year in water charged with carbon dioxide very little alteration could be discerned.[4]

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[1] U. S. Dept. Agriculture, *Bull.* **122** (1914).

[2] "The Data of Geochemistry," *U. S. Geol. Survey*, **616**, 1916.

[3] Bureau of Soils, U. S. Dept. Agr., *Bull.* **91** (1913).

[4] *Quarterly J. Geol. Soc. London*, **45** (1889).

On the other hand, Steiger found "that by a very superficial treatment with hydrochloric acid approximately one-third of the potassium may be extracted."[1]

In the study of the alteration of soil minerals in this Bureau one of the procedures adopted has been the subjection of the finely ground mineral to the action of solutions of salts, either those known to be present in soils or commonly added to soils in fertilizers. In the course of this work the behavior of finely ground muscovite when treated with a solution of ammonium sulfate was such that while preliminary work only has as yet been done the results seem worthy of record.

Selected laminae of muscovite from a large sample of this mineral were ground in an agate mortar to pass a sieve 130 meshes to the inch. This material in varying quantities was subjected to the action of 10 cc. of a solution of ammonium sulfate varying in concentration from 0.5 to 1.5 per cent. The insoluble material was removed by filtration and the total potassium in the solution was determined by the official method for potash in mixed fertilizers. The total potassium in the original muscovite was determined by the J. Lawrence Smith method.

The results are shown in the accompanying table. The length of time of contact of the solution with the muscovite was in all cases 24 hours; 10 cc. of the ammonium sulfate solution were used in each experiment. The results obtained with orthoclase by the same treatment are also shown.

In additions to these tests, two portions of 0.5 g. of muscovite were heated in an autoclave for 1 hr. at 160° C., in one case with water only, and in the other with 25 cc. of a 4 per cent solution of ammonium sulfate. In the case of water 4.32 per cent of the total potassium as K_2O was rendered soluble, and in the case of the ammonium sulfate treatment 47.3 per cent.

Mineral Muscovite No.	Wt. used, G.	$(NH_4)_2SO_4$ Solution Per cent	Approx. Temp. 0° C.	Per cent K_2O in Mineral	Per Cent K_2O Extracted —On Basis of— Mineral	Total K_2O
1	0.15	0.5	20	8.40	1.68	20.00
2	0.15	1.0	20	8.40	1.67	19.98
3	0.15	1.5	20	8.40	1.87	22.26
4	0.15	1.0	40	6.47	2.20	34.00
5	0.15	1.0	40	6.47	2.20	34.00
6	0.15	1.0	40	6.47	3.73	57.65
7	0.20	1.0	40	6.47	1.55	23.96
8	0.40	1.0	40	6.47	1.75	27.05
9	0.60	1.0	40	6.47	1.65	25.50
10	0.15	1.5	40	6.47	1.87	28.90
11	0.15	1.5	65	6.47	2.07	21.98
12	0.15	1.5	80	6.47	2.33	36.01
13	0.20	1.5	80	6.47	2.95	45.60
14	0.40	1.5	80	6.47	2.80	43.28
15	0.60	1.5	80	6.47	2.33	36.01
Orthoclase:						
1	0.20	1.5	65	7.78	0.25	3.23
2	0.40	1.5	65	7.78	0.13	1.68
3	0.60	1.5	65	7.78	0.08	1.03

In the first three tests, where the concentration of the ammonium sulfate was increased while the quantity of muscovite and temperature remained constant, the differences in K_2O extracted were slight with no straight increase with increase of concentration of ammonium sulfate. In Nos. 4, 5, and 6, where the conditions were approximately the same, there was absolute agreement between Nos. 4 and 5, with No. 6 very much higher. In the case of Nos. 7, 8, 9 and 10, with the quantity of muscovite variable and other conditions constant, the differences in quantity of K_2O extracted are not significant in view of the variation noted where the quantity of muscovite was constant.

The remaining tests, while suggestive of increased extraction at higher temperatures, are suggestive only. Throughout this work the temperature control was approximate only and it would appear from the results that other factors are operative and not sufficiently controlled. The only conclusion that stands out clearly is that at temperatures above 20° C. (room temperature) more K_2O is extracted by this treatment.

So far no results have been obtained throwing any light on the chemical changes involved or the process by which the potassium is rendered soluble.

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[1] U. S. Geol. Survey, Part I, *Bull* **600**, 236.

LABORATORY AND PLANT

NEW ALLOYS TO REPLACE PLATINUM

By F. A. Fahrenwald

Received May 11, 1917

The development of materials to take the place of platinum in many of its applications has become not so much a matter of economic desire, as one of actual necessity.

The present phenomenal activities in chemical research and manufacturing enterprises have resulted in a continually increasing consumption of this material, while statistics show that the world's production has been on the decline. At the present time an actual scarcity of metal is a matter of greater importance than its consequent high cost.

PRODUCTION—The following figures taken from a report by the United States Geological Survey, "Platinum and Allied Metals in 1915," give estimates of production in troy ounces during the period from 1912 to 1915, for the principal mining countries of the world.

Country	1912	1913	1914	1915
Borneo and Sumatra	200	2,000	...	..
Canada	30	50	30	100
Colombia	12,000	15,000	17,500	18,000
New South Wales and Tasmania	778	1,275	1,248	303
Russia	300,000	250,000	241,200	12,400
United States	721	483	570	742

During 1914, the United States imported platinum to the extent of about 75,000 troy ounces, and in 1915 about 65,000 ounces. An additional 65,000 ounces of new metal were recovered by refiners of gold bullion and blister copper, while about 40,000 ounces of metal were derived from refining of various forms of scrap, chiefly from jewelry and dental work.

MARKET—The price of platinum throughout a period of years has shown a steady increase, and there seems small likelihood of a near future return to greatly lowered quotations.

The following figures give average prices per troy ounce for platinum ingots at various times since 1874.

1874	$ 6.12	1898	$17.88	1906	$27.00	1913	$ 44.88
1888	$ 8.19	1900	$19.41	1907	$28.00	1914	$ 45.06
1890	$ 8.67	1901	$19.93	1910	$32.70	1915	$ 49.63 to $ 85.50
1893	$ 9.19	1902	$20.94	1911	$43.12		
1895	$10.22	1905	$21.45	1912	$45.55	1916 1917	$ 85.50 to $110.00

USES—Platinum is employed chiefly in four general fields, *viz.*, Chemical, Electrical, Dental and Jewelry.

A relatively large amount of this metal is actually consumed in chemical applications, as in electrolytic processes, but a large part of it, as in the form of crucibles, dishes, and other apparatus, is not destroyed, and with care may be used indefinitely.

In the electrical field platinum finds a variety of uses, chiefly, however, in the form of contact points on ignition and lighting systems, and in a similar form on numerous types of automatic electric apparatus from automatic telephones and signalling devices to the ordinary every-day temperature-controlled electric iron or heating pad. Most of the metal used in this manner is destroyed. Large quantities of platinum are also used in the construction of electric resistance furnaces for high-temperature work, as in experimental laboratories. The material in this application is not, however, destroyed and may be refined and used again.

Perhaps the greatest actual consumption of platinum occurs in dentistry. It is employed here as pins in artificial teeth, and as foil, plate and sheet in other forms of dental construction. It is estimated that about one-third of the entire consumption of platinum may be accounted for in this manner. As yet no means have been devised to prevent this noble metal from being lost forever. Perhaps actual necessity may yet become sufficiently pressing to overcome false sentiment, which no doubt at this time would greet with horror a suggestion that all teeth or other devices containing platinum be removed from the remains of those for whom it had completed its time of service, and so be made available for further duty.

The jewelry industry is without doubt the greatest offender, and proper steps should be taken at once to prevent the use of platinum in this field. It is not economically sound to permit mere foibles of fancy or dictates of fashion to remove from service a material which is absolutely necessary in the sciences and industries. It is estimated that the jewelry industry consumes nearly one-third of the entire production of platinum, and while some of this is recoverable, most of it, when once in private ownership, is practically lost.

In many instances where formerly it was thought that only platinum could be used, alloys have been developed which, for a given well defined purpose, are quite as satisfactory: in chemistry, laboratory ware of fused quartz, larger apparatus of the various high-silicon iron alloys, nickel chromium alloys and other materials have been adapted to specific phases of the industry, where hitherto only platinum had been found satisfactory.

In the electrical field, platinum for resistance heating elements has been largely replaced by alloys of nickel and chromium. For electrical contact material, as in many forms of ignition and automatic electric devices, platinum has been, to a certain extent, replaced by tungsten. Most of the manufacturers, however, of high-grade magnetos and other apparatus demanding high-duty service, still think it necessary to use platinum as the material of contact terminals, although in the majority of cases the alloys described herein are satisfactory. Platinum is no longer used extensively in the manufacture of incandescent lamps, having been replaced by a copper coated, nickel-iron alloy which has the same coefficient of expansion as platinum.

Many forms of substitution are practiced in dentistry to avoid the use of platinum. Various forms of German silver, gold alloys, and others, are used with varying degrees of satisfaction, but there are many applications in which no other heretofore available material will serve. The writer has developed a method of coating tungsten or molybdenum with precious metals which gives a material of even greater strength and permanence.

For the manufacture of jewelry no other metal or alloy combining the qualifications of workability, appearance, permanence and intrinsic value, has been available.

It is thus evident that while other materials have been developed to successfully replace platinum in a few specific instances, researches have not heretofore produced a material which in all of its characteristics and properties can serve as a general purpose substitute.

In considering a material to replace platinum it is necessary to regard as criteria those properties which make it valuable in applications where no other metal will serve. Its value industrially is chiefly due to a high melting point, and chemical inactivity under those conditions which exist in its application. It is also very malleable and ductile, and may readily be worked into the great variety of intricate forms so often required.

It is apparent, then, that a proper substitute must satisfy the following conditions:

SPECIFICATIONS FOR A SUBSTITUTE

1—Its melting point must be high. For chemical purposes a material that would fuse at the temperature of the ordinary gas flame would find limited application. In electrical uses high fusing point is one of the chief requirements, while in dental manufacture, platinum is subjected to temperatures which are seldom below 1250° C., so that about 1300 to 1400° C. would be a fair minimum temperature that a satisfactory substitute must withstand.

2—It must not be affected by mineral acids, or alkalies, either fused or in solution, and must not oxidize at any temperature up to and including that at its melting point.

3—It must be malleable and ductile, and sufficiently strong to withstand stresses tending to change its form while in use.

4—The chief requirements for jewelry purposes would seem to be rarity and high cost, although the above criteria to a certain extent govern its use in this field also. It is doubtful whether color or appearance are of greater importance than intrinsic value but in order to substitute for platinum in a strict sense, a platinum-white color is desired.

One phase of researches conducted in the writer's laboratories during the past several years has dealt with the development of alloys[1] to replace platinum, and aside from the treatment of special applications, attention has been given to a consideration of the possibilities of producing a material which should practically duplicate platinum in its general characteristics and behavior when judged according to the above set of requirements.

This work has resulted in a material which in most of its general chemical and physical properties is almost indistinguishable from platinum itself, as may be seen from the hereinafter described results.

DISCUSSION OF POSSIBILITIES

It is theoretically indicated, and has been experimentally proved to the satisfaction of the writer, that *no other possible combination of elements can result in alloys of equally enhanced properties, when judged with platinum as the criterion.*

In order to develop this thesis, and in so doing eliminate fruitless fields for further research, it will be necessary before describing these resulting alloys, to consider briefly a few factors involved in their development.

The production of alloys of special properties is no longer a matter of "discovery." In the days of the alchemist men put forth every effort in vain attempts to produce gold artificially, and sought by the use of a philosopher's stone to transmute base metals into a more noble form. At the present time, however, when molecules, atoms, and electrons are a matter of fruitful study, the hope of actually changing one element into another is not so fanciful. But in the meantime, advances can be made only by properly combining the simple elements to form alloys having properties superior to those of the component metals.

The study of metals, as an exact science, is of comparatively recent origin, although some of the devices of metallurgy have been unconsciously employed by men for ages.

The first advances in the art of treating metals to improve certain of their properties were no doubt a matter of accidental discovery, as when some warrior discovered that the metal of his spear and arrowheads could be hardened by hammering between two stones. Even though accidental, an invention of this nature advanced his tribe, more, perhaps, than did the discovery of the metal itself. The forger of the Damascus blade did not know the inner nature of his secret process, but this did not detract from the prowess of its wielder.

So, to the present day, man's advance has been suggestively paralleled by his increasing knowledge of the properties of the metals. With knowledge of the elements came the discovery that certain combinations of metals possessed properties far more valuable than those of any single one, and the continued pursuit of this study has produced materials which have revolutionized industry and warfare. It has produced tools for mining and agriculture; it has made possible steam vessels and railroads, and has permitted the present great diffusion of thought and knowledge by means of the printing press, photographic appliance, telegraph, cable and telephone.

As the results of these early researches radically changed many phases of industry, so in turn, the present changing industrial and economic conditions demand further applications of science in order that new conditions may be properly met as they develop.

In alloys of iron a great number of special steels have been developed which meet any reasonable specifications in that field. In a like manner the alloys of copper, zinc, tin, nickel and other metals have provided a series of brasses and bronzes of remarkable perfection.

Similar successes have not attended efforts to replace the precious metals, and especially is this true of platinum. In undertaking the solution of this problem, it was found, after the entire list of metals had been considered with the above imposed conditions as criteria, that no element, aside from platinum, would satisfactorily meet the above outlined specifications. In view of the fact that no single metal was available for this purpose, it was evident that any search for the desired material must be among alloys, for experience has shown that the properties of a metal may be radically changed by the addition to it of varying amounts of another element, or of several elements, as in the case of steels, brasses, and bronzes.

A review of the literature reveals nothing of direct bearing on this question. The same underlying principles, however, are involved in all investigations of this nature, so advantage may be taken of the broad generalizations which have resulted from the numerous researches on other alloys. Considerable work in the line of thermal analysis has been reported which covers many of the metals under consideration, and in these resulting diagrams of thermal equilibrium the proportion and type of the different constituents of a series of alloys may be easily detected.

It is known that the relation between the constitution of an alloy and its various properties is rather well defined, so, to a certain extent, it is possible, from a study of a given equilibrium diagram, to interpret in terms of characteristic properties, the terms given as constituents.

In view of the fact, then, that the general properties of an alloy are dependent upon the type of its constituents, and that these in turn are dependent upon the nature of their components (the elements) it is desirable that certain applicable laws be briefly pointed out, and that the inter-relationship of the elements themselves be taken into consideration, for without this information it would not be possible to select, from the entire list of the elements, those which might properly be included in an investigation of this nature.

The relationship of the elements is best revealed by some form of the periodic table, as conceived first

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[1] See *Bull. A. I. M. E.* Jan., 1916. Descriptions of these alloys are also subject-matter of professional reports made in 1913.

by Newlands and later improved and enlarged by Lothar Meyer and Mendeléeff. Werner's rearrangement of this table is, perhaps, the most satisfactory for purposes of alloy comparison.

The accompanying table was constructed in this manner, omitting, however, the rare elements not of interest in this connection, and so allowing a better comparison of the metallic elements than does the complete chemical table.

If one begins at the upper left-hand corner and reads bookwise, it is found[1] that the atomic weights show an almost uniform increase from Hydrogen 1 to Thorium 232.5. The horizontal rows represent groups, each containing seventeen elements arranged in two series of seven, each, with a "transition" series of three elements; that is, 1 to 7 constituting one series, 1*a* to 7*a* the second series, and 8 the intermediate or transition series. In the ordinary chemical table the elements are arranged according to valencies, which brings the elements of Column 1 and 1*a*, 2 and 2*a*, etc., together, with considerable confusion as to their physical and chemical properties. In the present table the common elements are shown arranged in accordance with their atomic weights, the group designations 1, 1*a*, 2, 2*a*, etc., being retained merely as an assistance to those familiar with the usual chemical table.

Within any one group there is no sudden change in properties when passing from one element to the next in order. Considering the two series forming one group, the first begins with elements which are strongly base-forming, and ends with strongly acid-forming elements; *e. g.*, potassium, of Column 1, and manganese, of Column 7. The second series begins with elements which are only moderately base-forming and ends with markedly acid-forming elements; *e. g.*, zinc, in 1*a*, and bromine, in 7*a*, of the same group. The elements (8) connecting these series are intermediate in their behavior between the elements on either side, those of the first vertical column (iron, ruthenium, osmium) forming both bases and acids; the others only acids.

In passing, with consecutive readings, from one horizontal group to the next lower, however, one notes a sudden change in the chemical properties of elements of consecutive atomic weights. Thus, the last element of Group II, fluorine, with an atomic weight of 19, is in complete contrast, in its chemical nature, to the next element sodium, with an atomic weight of 23.5, and which belongs in Group III. Iodine, the last member of Group III, forms powerful acids, while cesium, the first member of Group IV, is one of the most powerful base-forming elements known.

If the elements in a vertical column are considered it is found that they are, on the whole, such as would naturally fall together in a classification of the elements according to their general chemical and physical properties. Thus, in the first column are lithium, sodium, potassium, and cesium—the metals of the alkalies; in the second column are calcium, barium, and strontium—metals of the alkali earths, and so on.

Space is not available for a detailed discussion of many other relationships which have been found to exist, but it is apparent that the elements of a vertical column are very similar, and that there is no radical difference in the general properties of horizontally adjacent elements. It is evident, therefore, that a similarity of properties exists among the elements occurring in any one part of the table, and that the general properties of an element can be foretold from its position on the periodic table. The melting points, for instance, which have been included for comparison in this table, range from low to high, or from high to

	1	2	3	4	5	6	7	8			1a	2a	3a	4a	5a	6a	7a	
I	H 1.008 -258?	(ELEMENT) (ATOMIC WEIGHT) (MELTING POINT)																I
II	Li 7.03 186.0	Be 9.1 900 ?	B 11.0 2200?	C 12.0 ?											N 14.04 -210	O 16.0 -235 ?	Fl 19.0 -223	II
III	Na 23.5 97.0	Mg 24.36 651.0	Al 21.1 658.0	Si 28.4 1400											P 31.0 44	S 32.06 (120 114)	Cl 35.45 -102	III
IV	K 39.15 62.0	Ca 40.7 805	Sc 44.1 1400?	Ti 48.1 1850?	X 51.2 1750?	Cr 52.1 1505	Mn 55.0 1225	Fe 55.9 1520	Co 59 1490	Ni 58.7 1450	Cu 63.6 1083	Zn 65.4 419	Ga 70 30	Ge 72.5 900	As 75.0 800?	Se 79.1 217.0	Br 79.96 -7.0	IV
V	Rb 85.4 38.0	Sr 87.6 830	Y 89.0	Zr 90.7 1600	Nb 94.0 2100?	Mo 96.0 2500?	?	Ru 109.17 2000?	Rh 103. 1900?	Pd 106. 1550	Ag 107.93 961	Cd 112.4 321.0	In 114 155	Sn 118.5 232	Sb 120. 630	Te 127.6 451	I 126.5 +114	V
VI	Cs 133.0 28.0	Ba 137.4 850	La 138	Ce 140 645?	Ta 183 2900?	W 184 3200	?	Os 191.0 2700?	Ir 193.0 2300?	Pt 194 1755	Au 197.2 1063	Hg 200.3 -39	Tl 204.1 302	Pb 207.1 327.0	Bi 208 270			VI
VII		Ra 225 ?		Th 232.5 1700?														VII

PERIODIC ARRANGEMENT OF COMMON ELEMENTS

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[1] These simple relationships are, of course, well known. It is thought well, however, briefly to review them in order to draw more clearly a parallelism in the case of alloying characteristics, which is not so commonly recognized.

low, in any given column, and in reading through any one group the successive figures do not represent very great contrasts. This same relationship governs all other properties as, for instance, malleability and ductility. Considering Column 1*a*, as an example, copper is very easily rolled, drawn, or otherwise manipulated; silver, the next lower, is more so, while gold, at the bottom, is the most malleable of known metals.

If one metal, therefore, possesses special properties which make it valuable for given industrial purposes, it is reasonable to suppose that those metals which occupy adjacent positions on the periodic table may possess these same properties to some extent at least, and would, therefore, bear investigation—especially if they come within the same vertical column.

There is no difference between reactions which take place at very high temperatures, resulting in the formation of alloys, and those which take place under ordinary conditions. All reactions and conditions of equilibrium are governed by the same rules, and corresponding constituents result in all cases. It is evident, then, that *the general laws and relationships governing the alloying behavior of the metals are the same as those which have been found to exist in the case of ordinary chemical reactions.*

When two or more metals are brought together in the liquid state, the conditions existing are similar to those found if two or more ordinary liquids are mixed. When the temperature is sufficiently lowered the solidified mass may contain any one of the four following constituents: pure components, solid solutions, compounds, or eutectics, or some combination of these.

A comparison of the properties of different alloys containing these constituents has shown that they impart their characteristic properties to the alloy of which they form a part; in fact, the relation between *the constitution of an alloy and its properties is so clearly defined that the possibilities of industrial application may be predicted for a given alloy, if its constituents are definitely known.* Conversely, *if a certain application is desired, as in the problem under consideration, a definite limit may be placed upon the number and amount of constituents permissible.*

Fortunately, the number of constituents is limited to four as given above. Pure metals impart their own characteristics; solid solutions are, in general, the ductile constituents (if formed from ductile metals, or of a preponderance of one ductile metal); compounds are hard and brittle; while eutectics are usually brittle and hard, and even when present in very small amounts, tend to solidify between the grains of the alloy and destroy its ductility.

Thus another requirement may be added; a substitute for platinum must be a *homogeneous* solid-solution alloy.

This brief discussion has, in a very general manner, outlined the inter-relationship of the elements when arranged in the periodic order of their properties, and has pointed out the similarity of properties of elements grouped in any part of the table. The general properties of an alloy have been shown to depend upon its constituents, and these constituents in turn have been briefly discussed with reference to their characteristic physical properties and with reference to the probability of the occurrence of similar constituents in alloys of closely related elements.

It is not safe, however, to generalize in too broad a manner upon any assumed relationship between certain elements, but it would be impossible to make a logical search for any special alloy, in a field containing all of the elements, without first having definitely outlined the mutual relationship and behavior of the component elements.

With the elements arranged in this manner it becomes at once evident that characteristic chemical or physical properties are confined to definite limited areas of the periodic table. The properties characteristic of silver, for instance, are more or less common also to those eight elements contained in squares surrounding it; this is especially true in the case of those of the same vertical column. In this manner any element on this chart may be analyzed and all other elements contained in non-adjacent spaces will be found markedly dissimilar to it in chemical behavior and in general physical properties.

Considering platinum in this manner, it is at once evident that its general properties will be found in no metal other than those adjacent to it. These surrounding elements are iridium, rhodium, palladium, silver and gold. All metals outside of this block are affected by common reagents and gases, and, without exception, are not stable at elevated temperatures in normal atmospheres.

Considering each of these in turn, however, it is found that not one is suited as a general substitute for platinum. Iridium and rhodium are very refractory; they cannot be readily worked, and are so rare and expensive as not to be economically permissible. Palladium, while meeting most physical requirements, is readily oxidized or carbonized, and is quite soluble in several of the common acids, especially nitric. Silver is readily attacked by acids, and is of too low melting point to serve, except in special cases. Gold resembles platinum in more respects than does any other metal. It is, however, not sufficiently refractory and is too soft to find wide application industrially. A metal falling below platinum in the same vertical column would be possessed of properties still more enhanced, for the "nobility" of the elements of this column becomes greater with increasing atomic weight.

The above consideration is not merely theoretical, for experimental work involving most of the promising metals outside of the above list has supported this view.

Having limited the possibilities to the rather confined area indicated, it was necessary to combine these included metals in such manner as to eliminate or neutralize undesirable features, and to develop those properties which were necessary.

The material of chemical ware must not contain silver because of its affinity for many of the reagents commonly employed. The use of rhodium and iridium in large proportions is obviously not practical, so that

TABLE I—TESTS ON SAMPLES OF RHOTANIUM (Submitted by F. A. Fahrenwald)

Each test piece was about 0.1 mm. thick and presented 10 square centimeters of surface. The palladium was electrolytic metal from Baker and Company. The gold was U. S. mint gold, 0.999 fine. The platinum was the pure foil of commerce.

ACTION OF HYDROCHLORIC ACID

The pieces were boiled in the concentrated acid for 3 hours and showed losses in weight as follows:

5	0.05 mg.	
A	0.05	
A5	0.05	
B	0.0	
C	0.0	
D	0.0	
Pd	2.3	Surface etched
Au	0.05	
Pt	0.0	

CONCLUSION: Hydrochloric acid is practically without action.

ACTION OF HYDROFLUORIC ACID

The pieces were boiled in the concentrated acid for 3 hours and showed the following losses in weight:

5	0.0
A	0.0
A5	0.0
B	0.0
C	0.05
D	0.0
Pd	0.11
Au	0.05
Pt	0.0

CONCLUSION: Hydrofluoric acid is entirely without action.

ACTION OF CONCENTRATED NITRIC ACID

The pieces were boiled with the concentrated acid for 3 hours and showed the following losses in weight:

5	2.85 mg.
A	2.15
A5	2.25
B	2.15
C	2.15
D	2.30
Au	3.25
Pd	Much attacked
Pt	0.05

CONCLUSION: Hot, concentrated nitric acid attacks all the alloys very appreciably, especially those very high in gold.

ACTION OF DILUTE NITRIC ACID

The pieces were boiled with the dilute acid, one part of nitric to two of water, for 5$^1/_2$ hours; the solution concentrating to a ratio of one acid to one water. The following losses in weight occurred:

5	0.1 mg.
A	0.05
A5	0.05
B	0.05
C	0.05
D	0.05
Au	0.05
Pt	0.0

CONCLUSION: Hot, dilute nitric acid is practically without action in all cases.

ACTION OF CONCENTRATED SULFURIC ACID

The pieces were boiled with the concentrated acid for 5$^1/_2$ hours and showed the following losses in weight:

5	0.05 mg.	
A	0.05	
A5	0.15....	0.40 mg.*
B	0.20	
C	0.50	
D	1.60?	
Au	0.05....	0.35 mg.*
Pd	Rapidly attacked	
Pt	2.00	

* After 14 hrs. at 300° C.

CONCLUSION: Hot, concentrated sulfuric acid attacks the alloys high in gold very slightly, the rate of solution increasing with the proportion of palladium. All the alloys are superior to platinum in this test.

ACTION OF FUSED POTASSIUM BISULFATE

The pieces were all immersed together in potassium bisulfate kept at a very low red heat in a porcelain crucible for two hours and showed the following losses:

5	0.65 mg.	gain	Had a silvery appearance showing probable deposition of Pd on its surface.
A	0.25 mg.	loss	
A5	0.40	loss	
B	5.00	loss	
C	16.85	loss	
D	49.4	loss	
Au	0.60 mg.	gain	Had silvery appearance, showing that Pd had probably been deposited upon it.
Pd	Very rapidly attacked		
Pt	0.25		

CONCLUSIONS: The action is similar to that of concentrated sulfuric acid but more intense. It is probable that alloy 5 and Au were slightly attacked, but the Pd taken up more than compensated for the loss. Alloys high in gold are equal to, or better than, platinum.

ACTION OF SODIUM HYDROXIDE IN SOLUTION

The pieces were boiled with 40 per cent sodium hydroxide solution for 3 hours, and showed the following losses:

5	0.0
A	0.0
A5	0.0
B	0.0
C	0.0
D	0.05
Au	0.10
Pt	0.0

CONCLUSION: Hot, concentrated sodium hydroxide solution is entirely without action.

ACTION OF FUSED SODIUM HYDROXIDE

The pieces were immersed in the fused hydroxide in a nickel crucible and kept for 2 hours at a low red heat. They showed the following losses:

5	4.45 mg.
A	4.95
B	4.00
D	9.25
Pd	18.45
Au	7.95

CONCLUSION: Alloys high in gold are more resistant, but all are somewhat attacked. It is well known that platinum is badly attacked.

ACTION OF FUSED SODIUM CARBONATE

The pieces were immersed in the fused carbonate in a nickel crucible and kept for 2 hours at a bright red heat. They all showed slight gains, when there was any change. They were bright and showed no evidence of corrosion. It is difficult to explain the gain in weight, except in the case of Pd, which was undoubtedly somewhat oxidized.

5	Too soft, tended to stick to crucible	
A	0.0 mg.	
A5	0.55 mg.	gain
B	0.25	gain
C	0.25?	gain
D	0.25	gain
Au	Stuck to the crucible	
Pd	3.05	gain
Pt	0.80	

CONCLUSION: The alloys are not appreciably attacked by fused Na_2CO_3 and are equal to platinum in this respect.

ACTION OF SODIUM SULFIDE SOLUTION

The pieces were boiled for 3 hours in a solution of sodium sulfide containing a little polysulfide, and saturated at room temperature.

5	0.0 mg.		Pd	0.75 mg.	loss
A	0.05 mg.	gain	Au	0.45 mg.	loss
A5	0.0		Pt	0.0	
B	0.1	gain			
C	0.1	gain			
D	0.05	gain			

CONCLUSION: The Pd was somewhat tarnished; the others were perfectly bright. The alloys are not all attacked by concentrated hot sodium sulfide solution.

ACTION OF AMMONIA

The pieces were boiled 5 hours in a concentrated ammonia solution.

5	0.0 mg.	
A	0.0	
A5	0.05	gain
B	0.05	loss
C	0.0	
D	0.10	loss
Au	0.0	loss
Pt	0.0	

CONCLUSION: The alloys are not in the least attacked by hot, concentrated ammonia solution.

ACTION AS CATHODE IN ELECTROLYTIC DEPOSITION

The pieces were plated with copper in a solution containing nitric and sulfuric acid, and the deposit was dissolved off by a brief treatment with warm dilute nitric acid, one to one. The losses were:

5	0.0 mg.
A	0.1
A5	0.05
B	0.1
C	0.1
D	0.0
Au	0.05
Pt	0.0

CONCLUSION: The alloys are satisfactory as cathodes in the electrolytic determination of metals.

ACTION AS ANODE IN SULFURIC ACID SOLUTION

The pieces were made the anode in a hot solution of dilute sulfuric acid, one acid to eight water, for 10 minutes using 10 amperes of current. The losses were:

5	26.5 mg.
A	37.6
A5	39.1
B	37.6
C	29.0
D	33.8
Au	48.0
Pt	0.1

CONCLUSION: The alloys are all worthless for use as anodes in electrolysis in acid solutions.

(Signed) H. H. WILLARD
University of Michigan

finally only gold and palladium remain as the permissible *chief* components of an alloy which should more nearly duplicate the general properties of platinum than could any other metal or combination of metals.

A detailed investigation of alloys of these metals has resulted in materials[1] which for many practical purposes cannot be distinguished from platinum.

EXPERIMENTAL

The following results of final tests made to determine the behavior of these alloys under practical conditions of operation were carried out on specimens 0.10 mm. thick and of such size as to present exactly 10 sq. cm. of surface. Earlier work revealed the fact that in order to produce the desired results, it was necessary to make use of small percentages of rhodium; this is not necessary in the case of material for chemical ware; for certain electrical and other uses, however, it is quite essential. It is also permissible to use limited percentages of silver in some of these non-chemical applications.

It is necessary to observe every precaution in the preparation of these alloys, because of the affinity of palladium for many gases and solids ordinarily encountered in the process of manufacture. The most careful heat-treatment also is necessary to insure freedom from segregation. The slightest inhomogeneity is fatal to uniformity of results in practically every application—especially for chemical purposes.

TABLE II—LOSS IN MILLIGRAMS PER HOUR PER 100 SQUARE CENTIMETERS (SUMMARY OF TABLE I)

Alloy No.	Boiling Conc. HCl	Boiling Conc. HF	Boiling Conc. HNO_3	Boiling Dil. 1 to 2 HNO_3	Boiling Conc. H_2SO_4	Fused (Red heat) $KHSO_4$	Boiling 40% Sol. NaOH	Fused (Red heat) NaOH	Fused 1000° C. Na_2CO_3	Boiling Sat. Sol. Na_2S + S	Boiling Conc. NH_4OH
5	0.166	0.0	9.5	0.2	0.1	0.0	0.0	22.2	0.0	0.0	0.0
A	0.166	0.0	7.5	0.0	0.1	1.2	0.0	24.7	0.0	0.0	0.0
A5	0.166	0.0	7.0	0.0	0.3	2.0	0.0	.....	+2.7	0.0	0.0
B	0.0	0.0	7.0	0.0	0.4	25.0	0.0	20.0	+1.2	0.0	0.0
C	0.0	0.0	7.0	0.0	1.0	84.0	0.0	.....	+6.2?	0.0	0.0
D	0.0	0.0	7.0	0.0	3.0	297.0	0.0	196?	+1.2	0.0	0.0
Pure Au	0.166	0.166	10.8	0.0	0.1	0.0	0.3	39.7	....	+1.5	0.0
Pure Pd	8.0	0.33	Rapidly Attacked	Rapidly Attacked	Rapidly Attacked	Very Rapidly Attacked	0.0	92.0	+15.2	+2.5	0.0
Pure Pt	0.0	0.0	0.166	0.0	4.0	1.45	0.0	Rapidly Attacked	+4.0	0.0	0.0

Losses in acid solution, for instance, for different test-specimens made from the same alloy ingot, have been found to vary by over 1000 per cent. Properly prepared material, however, gives absolute uniformity of results.

CHEMICAL PROPERTIES

On page 595 is shown a test-certificate from the University of Michigan Chemical Laboratories which gives results obtained from a series of Rhotanium alloys, together with figures for gold, palladium and platinum for comparison.

When tabulated in terms of loss in milligrams per hour per 100 sq. cm., the results appear as in Table II. It is seen from these figures that for ordinary use with any of the above named reagents, excepting concentrated nitric acid, an alloy may be chosen of a composition that will give service equal to or better than that of platinum. This is especially true in the case of concentrated boiling sulfuric acid.

VOLATILITY AT HIGH TEMPERATURES

In determining losses by volatilization at high temperatures, test specimens taken from the same ingot and duplicating those described above were employed. These were heated in a gas muffle furnace using natural gas and compressed air. These experiments were run in triplicate in two series: first in the muffle with free access to air, but with the products of combustion excluded; and second, heated directly in the gases of combustion with free air excluded. No difference in results was observed under these two sets of conditions. The comparative test pieces of palladium were of vacuum-fused electrolytic metal, while the test-specimens of platinum were cut from a new Baker crucible.

In these experiments the muffle was brought to the desired temperature and the test samples allowed to remain therein for one hour before cooling and weighing preliminary to the test run. An assay balance, reading directly to the fifth place, was used in determining the weight of each specimen before and after treating.

Each experiment consisted in a continuous 10-hour run, with the temperature maintained within ±25° C. of the figures given below. Losses at these various temperatures, in terms of milligrams per hour, per 100 sq. cm. of surface, are given in Table III.

TABLE III—LOSSES IN MUFFLE FURNACE

Sample		1050° C.	1200° C.	1300° C.	1400° C.
Rhotanium No.	5	2.0	Melted	Melted	...
	A	0.8	3.1	Melted	...
	A5	0.6	2.0	Melted	...
	B	0.4	1.4	6.0	...
	C	0.4	1.3	4.0	7.1
	D	0.2	0.6	2.82	5.8
	Au	...	...	..	...
	Pd	3.4 (gain)	14.0 (gain)	37.0 (gain)	120.0 (gain)
	Pt	1.3	2.40(*a*)	3.1	4.5

(*a*) Burgess gives 0.71 to 2.79 mg. (Bureau of Standards, Scientific Paper No. 254).

After heating for 10 hours at these temperatures the surface of each specimen (melted and otherwise) was perfectly bright. At 1400° C. the crystalline structure was clearly shown, due to selective volatilization or grain growth, but no brittleness was produced. The gain in case of pure palladium is due, no doubt, to oxidation, although palladium also carbonizes very easily.

PHYSICAL PROPERTIES

The alloys A5, B, C and D are practically white. C and D especially can only with difficulty be distinguished in appearance from platinum, and then only when in the form of sheet. Wires or articles of

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[1] The name "Rhotanium" has been applied to this series of alloys. Alloys of different composition for distinct purposes are distinguished by sub-letters as shown in the various tables of properties. This name has been registered, and all alloys, both as to composition and use, are covered by patent applications.

intricate design present a true platinum appearance. All are very malleable and ductile, and can be rolled, spun, or otherwise worked into any desired form.

Figures for some physical properties are given in Table IV.

TABLE IV—PHYSICAL PROPERTIES OF RHOTANIUM ALLOYS

ALLOY	Melting point ° C.	Melting point ° F. (Calc.)	Scleroscope Hardness	Tensile Strength Kg. per sq. mm.	Electrical Conductivity × 10^{-4}	Temperature Coefficient
5	1150	2100	7	26	...	...
A	1220	2228	9	35	13.5	0.00097
A5	1280	2335	10	40	9.8	0.00065
B	1350	2462	13	45	7.85	0.00060
C	1410	2570	16	50	5.5	0.00050
D	1450	2642	17	51	3.8	0.00032
Au	1063	1945	5	21	45.5	0.00326
Pd	1550	2822	11	30	9.45	0.00328
Pt	1775	3191	9	24	9.94	0.00348

As may be judged from the above described properties these alloys are well suited to replace platinum in many of its applications in all fields, and have been so used for a long period with entire satisfaction. By taking advantage of the ease with which chemical and physical properties may be modified through adjustment in composition, various alloys of this series have operated fully as satisfactorily as platinum in chemistry, dentistry, jewelry, and in many electrical appliances.

Various grades of this material have undergone extended field trials, the results of which may be summarized as follows for the various fields of application.

CHEMICAL

Rhotanium cannot substitute platinum when exposed to the action of hot concentrated nitric acid, or when used as anodes in electrolytic work, but for all other purposes it is entirely satisfactory *if the proper composition* is chosen, and if *properly* manufactured. It is equal to platinum in the case of hot concentrated hydrochloric or hydrofluoric; hot dilute nitric; fused potassium bisulfate; hot concentrated sodium hydroxide solution; fused sodium carbonate; hot concentrated sodium sulfide solution; and in its resistance to oxidation at high temperatures. It is superior to platinum in its resistance to the action of hot concentrated sulfuric acid or fused sodium hydroxide. It is satisfactory as material for cathodes in the electrolytic determination of metals. Losses by volatilization at temperatures below 1300° C. are less than for commercial platinum.

Rhotanium may be rolled into sheets of any size, and may be formed either cold or white hot into any desired shape. It welds as readily as wrought iron at a white heat without the use of flux or other reagent. Due to greater strength and lower specific gravity, articles of rhotanium weigh only half, or less than half as much as similar articles of platinum. The specific gravity of alloys in this series varies from 18.5 to about 16.0, depending upon the composition: that of platinum is 21.5.

ELECTRICAL

Rhotanium is satisfactory within its temperature limitations as the material of resistor elements in electric heating units. It is not oxidized and is less volatile below 1300° C. than platinum. Its high resistance and low temperature coefficient are valuable in this connection.

It is satisfactory as material for contact terminals in many forms of automatic-electric devices and may be used in this capacity on certain types of telephones, switchboards, signal devices, lighting and ignition systems, and in most other cases except where it has been found necessary to use a high percentage of iridium alloyed with the platinum. Its behavior when tested on certain magnetos was satisfactory, but other experiments performed on a high-duty aeroplane engine magneto gave negative results.

DENTAL

Certain of these alloys have been in the hands of operating dentists for some time and have proved to be equally as good as platinum for many purposes. They have been used for pins and baked into porcelain teeth and as thin foil and heavy sheet for other types of construction, all with the most satisfactory results.

JEWELRY

Rhotanium is superior to pure platinum for use in jewelry. It is harder and stronger, and takes a better finish. It is absolutely not tarnishable or corrodible, and its color is practically platinum-white. It can be as readily worked as platinum, and scrap may be remelted for further use. It may be forged either cold or white-hot and may be "sweated" or otherwise treated as platinum without oxidizing or darkening in color. Finished articles of rhotanium jewelry of the more intricate designs can by no ordinary means be distinguished from platinum. This material passes the common jeweler's and platinum buyers' tests and there will no doubt be some confusion resulting from the passing of this material for platinum.

Exhaustive tests have shown that most of the uses for which platinum has heretofore been considered indispensable can be filled by one of these alloys, thus freeing platinum for those remaining applications where no other material can be employed.

The extent to which platinum may be replaced in this manner is limited by the amount of palladium available, and when it is considered that these alloys contain from 90 to 60 per cent of gold it is evident that the effective supply of platinum may thus be increased by many thousand ounces.

1706 GLENMONT ROAD
CLEVELAND, OHIO

A PRACTICAL METHOD FOR DETERMINING THE VISCOSITY OF STARCH FOR MILL PURPOSES

By G. M. MACNIDER

Received March 16, 1917

Several years ago the author described in THIS JOURNAL[1] a method for determining the viscosity of starch solutions for determining the value of different starches for cotton mill purposes. Since the publication of this article the author has had the opportunity of applying this method to practical mill work and has worked out a modification of the method which is described in this paper.

The original method is briefly as follows: 12 grams

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[1] THIS JOURNAL 4 (1912), 417.

of the starch are weighed into a 600 cc. beaker, 300 cc. distilled water added (thus making a 4 per cent solution), heated over a Bunsen burner with constant stirring to the boiling point, and boiled 10 min.; 200 cc. of this solution are then poured into the cup of a Scott viscosimeter, the temperature being allowed to become constant, and 50 cc. run out into a graduate, the time being accurately measured with a stop-watch. The number of seconds required to deliver 50 cc. of the solution divided by the number of seconds required to deliver 50 cc. of boiling water gives the viscosity.

APPARATUS

The method makes a very satisfactory laboratory method for determining the comparative value of different starches, but in mill work it is frequently of importance to know the viscosity of the starch solution after it has been boiled with steam for an hour or more as is done in the use of the starch in preparing it for warp sizing, etc., in the mill. To accomplish this, a miniature size kettle was constructed, similar in every respect to the large kettles used in cooking starch in the mill. This kettle[1] (see Fig. I) has a capacity of about $1^3/_4$ gallons and is equipped with double agitators of the propeller type, revolving in opposite directions. The steam for cooking enters through a perforated coil on the bottom of the kettle. In order to overcome variations in the amount of steam condensed the steam is passed through a trap just before entering the kettle. The time of boiling is one hour, the time being taken at the time the starch begins to boil. The amount of starch taken for a determination varies with the grade of starch. For thick boiling corn starch, potato starch, and similar thick boiling starches 0.5 lb. of starch per gal. of water is used; for medium thin boiling corn starch, 1 lb. per gal. of water; for very thin corn starch and other chemically treated starches the amount is increased to 2 or 2.5 lbs. per gal. of water.

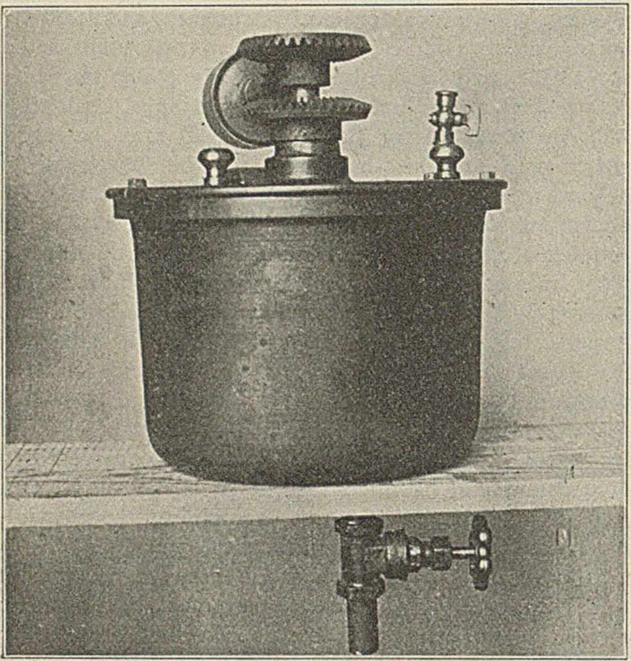

FIG. I

PROCEDURE

One gallon of water is measured into the kettle, the agitators started and the starch, accurately weighed, put into the kettle. The agitators are now run 10 to 15 minutes before turning on the steam in order to produce a perfectly smooth cream to avoid the formation of lumps. The steam is then turned on and the solution boiled for one hour after it comes to a boil. When the boiling is completed, some of the solution is drawn out into a beaker, quickly poured into the cup of a Scott viscosimeter and the viscosity determined as described in the original method.

The figures in Table I illustrate the application of this method in comparing several grades of thick boiling corn starches, using 0.5 lb. starch per gal. of water and boiling for one hour.

TABLE I—VISCOSITY MEASUREMENTS OF VARIOUS GRADES OF STARCH

No.	STARCH	VISCOSITY
1	Regular Pearl Corn Starch	3.10
2	Powdered Starch	4.30
3	Purified Starch	4.30
4	Chemically Treated Starch	2.89
5	Highly Purified Starch	4.58

Some of the gluten and impurities of No. 2 were removed by the powdering process so that this starch shows a higher viscosity than No. 1. No. 3 was another form of starch which had been subjected to the same amount of purification as No. 2. While the viscosity of starch No. 4 was lower than that of starch No. 1, due to the chemical treatment, the actual value of starch No. 4 was greater. The actual mill practice substantiated these figures; *i. e.*, if under certain conditions a size mixing of starch No. 1 made on the basis of 0.5 lb. starch per gal. of water gave the desired results, if No. 2 or No. 3 were substituted, under the same conditions, it would be necessary to use less starch in order to obtain the same thickness of solution and consequently the same results; whereas, if No. 4 were substituted it would be necessary to use more starch.

TABLE II—VISCOSITY MEASUREMENTS OF SEVERAL GRADES OF MODIFIED OR THIN-BOILING STARCHES

No.	LBS. STARCH PER GAL. WATER	VISCOSITY
1	1 Lb.	2.45
2 (same grade as 1)	1 Lb.	2.16
3	1 Lb.	1.94
4	2 Lbs.	1.27
5 (same grade as 4)	4 Lbs.	3.54
6 (same grade as 5)	4 Lbs. (2 oz. KOH added to kettle)	2.63

Nos. 1 and 2 were the same grade of boiling starch; in using No. 2 in the mill it was found necessary to use slightly more than was used of No. 1; the figures show the difference. No. 2 was a higher fluidity or thinner starch than No. 1. Nos. 4 and 5 were the same grade of a very thin starch, the figures showing the difference in viscosity when the amount of starch used per gallon is doubled. No. 6 was the same as No. 5, but with the addition of 0.2 oz. KOH to the kettle. The viscosity figure shows the very marked thinning effect of this amount of caustic potash on

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[1] This kettle was constructed by Mr. J. S. Drake of the Exposition Cotton Mills, Atlanta, Ga., and the author is indebted to Mr. Drake for valuable assistance and suggestions in working out the method.

the starch solution. It frequently becomes necessary to add to a starch mixing some reagent such as caustic alkali, chlorides of calcium, magnesium, zinc, etc., and it is very important to know just what effect the reagent will have on the thickness of the starch solution.

The advantages of this method over the laboratory method for mill work are as follows: the viscosity is determined when the starch has been boiled for the same length of time as it is boiled in preparing it for use in the mill and under the same conditions. The amount of starch used can be in the same proportion to the amount of water as in a regular size mixing; this makes the figures obtained by practical value as they show the viscosity of a size mixing. Also, in comparing two or more starches on such a basis, this fact that the viscosity figures represent the actual thickness of a size mixing makes it possible in case of a variation in viscosity to determine in the small kettle the amount necessary to use, thus saving the time and expense of experimenting on a large scale.

GREENVILLE, SOUTH CAROLINA

FULLER'S EARTH AND ITS VALUATION FOR THE OIL INDUSTRY

By THEODORE G. RICHERT

Received December 11, 1916

An important part of a modern oil refinery is the filtering department, where by means of fuller's earth the refined oils are bleached to the desired lightness in color. As many varieties of such bleaching agents are offered, it might be of interest to describe a cheap and quick method of determining, in the laboratory, the efficiency and economy of fuller's earth.

The earths for bleaching edible oils are clays which are used in the natural state as they occur or which sometimes are especially prepared in order to increase their bleaching power. They include hydrous silicates of aluminum, magnesium and calcium, containing small amounts of other substances, such as iron, sodium, potassium, etc.

Fuller's earth, owing to its colloidal character, when brought in contact with refined oil, forms, with the colloids soap and coloring matter, colloidal aggregates and settles out as such; after separating the oil and the fuller's earth, the oil has an earthy taste and a part of it remains with the fuller's earth.

Essential points in the use of fuller's earth in the oil industry are:

1—Precipitating of minutely suspended particles of soap left in the oil from the refining.

2—Removing of coloring matter, which, owing to the refining, is contained in the oil in an unstable state.

3—The earthy taste imparted to the oil should be removable by means of deodorization.

4—The loss of oil due to absorption should be as low as possible.

For trials like ours, Points 1 and 3 may be neglected; all fuller's earths have the ability of settling out the soap in sufficient degree and the earthy taste can always be removed by means of a well conducted deodorization process. As to the other points, a valuation of fuller's earth concerning its bleaching power and its absorption of oil can be accomplished in the laboratory.

Owing to the varying behaviors of the refined oils, as well as to working conditions in the plants, it is impossible to determine and fix *absolute* values for these properties. The only alternative is to compare the results obtained from working a new unknown earth with the results of one already tested. The principal condition is that for all tests and all earths the same oil and methods be used.

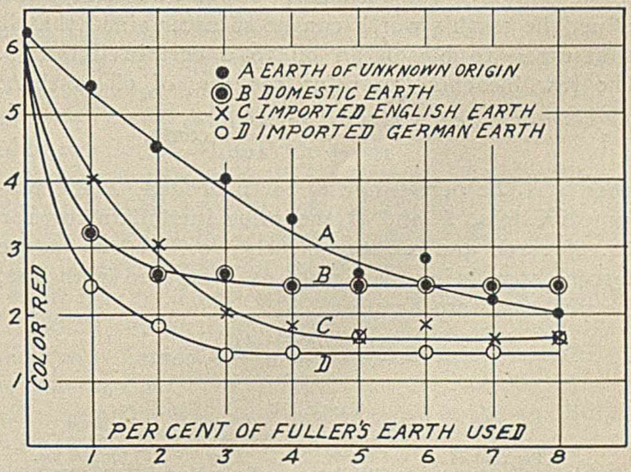
To determine the *bleaching powers* of different earths a number of bleaching tests[1] were made with a well refined cottonseed oil, using from 1 to 8 per cent of the earths (a greater per cent than 8 was not tested, since a higher amount is commercially prohibitory). In Table I, the earth *B* is a domestic, *C* an imported English, *D* an imported German earth; the origin of *A* could not be ascertained.

TABLE I—COLORS (LOVIBOND TINTOMETER) OF OILS BLEACHED WITH DIFFERENT PERCENTAGES OF FULLER'S EARTH

Bleached with Fuller's Earth	BRAND *A*		BRAND *B*		BRAND *C*		BRAND *D*	
	Yellow	Red	Yellow	Red	Yellow	Red	Yellow	Red
1.......	35	5.4	35	3.2	35	4.0	20	2.4
2.......	35	4.5	20	2.6	35	3.0	18	1.8
3.......	35	4.0	20	2.6	20	2.0	14	1.4
4.......	35	3.4	20	2.4	18	1.8	14	1.4
5.......	20	2.6	20	2.4	16	1.6	16	1.6
6.......	20	2.8	20	2.4	18	1.8	14	1.4
7.......	20	2.2	20	2.4	16	1.6	14	1.4
8.......	20	2.0	20	2.4	16	1.6	16	1.6

The results indicate the exhaustion and efficiency of the earth. The maximum effect is reached for Brands *A*, *B*, *C* and *D* with 8, 4, 5 and 3 per cent of earth, respectively.

To show even more distinctly the differences of the several tests, the results are plotted below, with the amount of fuller's earth used and the red color obtained as ordinates. The dotted line indicates the color for "White Oil"—20 yellow, 2.5 red.



The faster a curve arrives at the lowest point of its course, and the nearer it approaches the horizontal axis, the greater is the bleaching power of its earth. In our case, Brand *D* is the most effective; as shown by the curve, small amounts of *B* are highly advantageous, therefore, this earth seems to be well fitted for use in combination with other earths. Brand *C*

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[1] See Official Method of the "Society of Cotton Products Analysts."

is a reliable agent; its run in the factory has confirmed its good qualities. *A* is a slow-acting earth; to obtain any tangible results large amounts are needed, a fact which prohibits its extended application.

Another feature of the curves is to show approximately how much of the different earths would be necessary to obtain the same results. For instance to bleach the oil of our experiment to a "White Oil," there must be used of Brands *A*, *B*, *C* or *D*, 5.8, 3.0, 2.4 or 1.0 per cent of earth, respectively.

To determine *the loss of oil due to absorption*, 300 g. of oil were agitated with 10 g. of each of the fuller's earth samples for a given time at a given temperature. Then the oil was filtered by means of a Buchner funnel.[1] When the earth seemed to be dry, vacuum was kept up for 15 min.; the earth was then removed from the funnel and weighed.

Now when there is known the loss due to absorption when working with one of the earths, it is possible to figure out the loss to be expected using another earth, provided the working conditions remain the same. For instance, the earth *C* was known to cause a loss of 8 per cent; the presumptive losses of the other earths based on this figure are shown in the following table:

TABLE II—ABSORPTION OF OIL BY FULLER'S EARTH

Brand	Earth plus Oil Grams	Oil Absorbed Per cent	Presumptive Loss Per cent
A	11.77	17.7	8.3
B	18.73	87.3	40.8
C	11.71	17.1	8.0
D	14.05	40.5	19.0

THE VALUATION

The value of fuller's earth and consequently the economy of its use is dependent on price, bleaching power, absorption value and the utilization of the residue. The latter, being for all earths practically constant, may be omitted.

On account of the impossibility of determining absolute figures for bleaching power and absorption value, the earth's worth can be so ascertained that it is assumed to bleach an oil to a certain lightness. The raw bleaching cost for 100 lbs. of oil is then:

$$V = \frac{x(100P + AO)}{100 + xA/100} \text{ cents,}$$

when x is the percentage of earth needed, A the presumptive loss, P and O the price in dollars for 100 lbs. of earth and oil, respectively; or, $xA/100$ in the denominator being negligible as compared with 100, the raw costs are simply:

$$V = \frac{x(100\,P + AO)}{100} \text{ cents.}$$

TABLE III

BRAND	Per cent of Earth	Price(*a*) of Earth for 100 lbs.	Presumptive Loss Per cent	Raw Bleaching Cost for 100 lbs. Cents
A	5.8	\$0.70	8.3	9.36
B	3.0	\$0.68	40.8	15.50
C	2.4	\$0.79	8.0	4.01
D	1.0	\$3.15	19.0	5.24

(*a*) All prices are figured f. o. b. Portsmouth, Va.

For the assumed case that a "White Oil" has to be made from our oil, the figures are collated in Table III, the price of oil being taken as \$11.00 per 100 lbs.

CONCLUSION

In spite of the highly developed bleaching power of Earth *D* its application is not to be recommended on account of its excessive price. An extended use would be desirable only with the dropping of its price to about \$1.90 per 100 lbs. or with an oil price of around \$4.50; in both cases the bleaching cost would then be about 4 cents. Brand *C* is the most economical of all; the higher amount of earth used is compensated by the low absorption. Brand *B* is not well recommended because of its high absorption value. Finally, Brand *A* is not suitable for practical work on account of the large amount needed, which calls for large sized filter presses.

In conclusion, it should be noted that the work in the actual run is usually less costly than the preceding figures show; the intense contact of earth and oil under pressure increases the effect. To state that the actual raw bleaching costs fluctuates between 50 and 75 per cent of the above figures would be a fair estimate.

P. O. Box 27, Portsmouth, Virginia

[1] See "Fuller's Earth," by Charles L. Parsons, Washington, D. C., *Bull.* 71, Bureau of Mines.

AN APPARATUS FOR THE PURIFICATION OF MERCURY

By Harrison E. Patten and Gerald H. Mains

Received February 2, 1917

In this laboratory we have need of mercury in a very pure state not only for standard cells, and calomel half-cells, but also in rather large quantities for the filling of thermoregulators used in controlling constant temperature baths. The presence of even a slight trace of foreign metal, such as lead or zinc, after the mercury stands a short time in contact with air, gives rise to the formation of an oxide film on the surface which dirties the capillary tubes and interferes greatly with the delicacy of the thermoregulator.

We have tried out the various methods which have been proposed for the purification of mercury, and have used a number of the types of apparatus described in the literature. The well-known method of Lothar Meyer,[1] in which mercury is passed in a fine stream through a long column of dilute nitric acid, is slow, tedious, and cumbersome. The speed of operation is greatly increased by the modification of J. H. Hildebrand,[2] where the mercury is broken up into numerous extremely fine streams by passing it through muslin into the acid column. L. J. Desha devised a modification[3] by which the mercury, after running through the nitric acid column, was automatically raised to the top and thus kept in continuous circulation. Loomis and Acree[4] incorporated with the Desha apparatus the means of electrolytic purification, *i. e.*, making the mercury the anode in a nitric acid solution.[5]

Even after the above modifications, the purification demanded considerable watching and personal attention. Also there was no means provided for renewing the nitric acid without cleaning and refilling the entire apparatus. We have endeavored to construct a

[1] *Z. anal. Chem.*, **2** (1863), 241.

[2] *J. Am. Chem. Soc.*, **31** (1909), 933.

[3] *Am. Chem. J.*, **41** (1909), 152.

[4] N. E. Loomis and S. F. Acree, *Am. Chem. J.*, **46** (1911), 594.

[5] Wolff and Waters, Bureau of Standards, *Bull.* **3**, 623; **4** (1907), 1.

purifier which would combine the spraying of the mercury through a column of dilute nitric acid, the automatic return and circulation of the mercury, the electrolytic purification, and the automatic renewal of the nitric acid in order to wash away the products of electrolysis and prevent re-solution of impurities. By means of the injector principle (used with some modifications of the Sprengel pump), the mercury is raised through a small-bore tube to the top of the apparatus in small globules in a current of air. During this process some oxidation of the impurities takes place,[1] which we have greatly increased by surrounding the return tube with a heating coil.

The purifier embodying these points has been in satisfactory operation for over eight months, during which time between 50 and 60 kg. of mercury have been run through the apparatus.

DESCRIPTION OF MERCURY PURIFIER

Plans of the apparatus with all necessary dimensions and enlarged details of the more important parts are presented below. The complete apparatus is shown in Fig. 1. It consists of the following principal parts: Mercury reservoirs, nitric acid fall tube, nitric acid reservoir, spray chamber, electrodes, waste tube, injector for returning mercury, and outlet for purified mercury. Fig. 2 is a detail of the main mercury reservoir and of the spray chamber, and shows the placing of the electrodes. Fig. 3 is a detail of the injector, injector cup, and connections. The glass work can be readily assembled by any glass-blower. A convenient stand for the apparatus may be built from wood, or metal rods and clamps may be used.

OPERATION—Impure mercury is placed in the main mercury reservoir *M*, and from this flows into spray chamber *S*, through a stopcock which regulates the speed of operation of the apparatus. Over the lower end of *S* is stretched a piece of bolting silk which breaks the mercury into a fine spray upon entering the nitric acid. The silk is fastened firmly to the end of *S* by silk thread, and a little flare at the end of the glass tube prevents any slipping. We have found No. 7 bolting silk very satisfactory, giving a fine spray and yet not readily clogging up. A No. 20 B. & S. gauge platinum wire is fused through the side of *S* into a mercury contact cup. This wire extends down into the mercury held on the silk. By connecting the wire to the positive pole of a source of current, the mercury, as it sprays through the silk, becomes the anode for electrolysis.

The spray chamber is seated into a ground glass neck on the fall tube *F*. This ground glass neck has a slot provided at one side (Section D, Fig. 2), through which the cathode wire is brought into the fall tube. The cathode is also No. 20 platinum wire, and is made into a loop encircling the end of *S* at the same level as the anode. An electrolyzing current of 1 ampere has proved satisfactory with the dimensions of the spray chamber used.

The overflow or waste tube *W* leads from the fall tube just above the electrode level, so that the products of electrolysis are carried away by the stream of dilute nitric acid. The nitric acid used is a 2 per cent solution. This is fed from the reservoir *N*, into the fall tube at a slow rate, yet fast enough to prevent clogging about the cathode by separation of solid products of electrolysis, principally mercurous nitrate. In the particular apparatus used, it was found that an average rate of flow of 1000 cc. of nitric acid for a 7-hour period sufficed to maintain smooth operation. Some of the metallic impurities are deposited on the cathode. If the amount is excessive a silk bag placed around the cathode wire will prevent falling off of the deposit into the mercury below.[1]

The height of the mercury column in the lower portion of fall tube *F* depends upon the mercury level in the injector cup *C* (Fig. 3), the difference in level being proportional to the weight of the nitric acid column in *F*. The mercury rises inside of the injector bulb *I* until it just reaches the top of the inner tube. This inner tube is brought around up to the top of the apparatus and is connected to the laboratory vacuum system. As soon as the mercury rises above the level, *ml*, in the injector bulb, a drop falls into the inner tube and is carried in a finely divided condition by the difference in pressure up to the auxiliary mercury reservoir *A*. The inner tube must be of $^3/_{16}$ in. inside diameter or less in order to elevate the mercury the required height.

A heating coil, *H*, surrounds the tube through which the mercury is elevated. This coil consists of a copper tube 4 ft. long, just large enough to slip over the glass tube, covered with a layer of asbestos, and then wound with No. 30 nichrome wire in two sections of 19 ft., each paralleled off of a 110-volt circuit. Since the resistance of this wire is 6 ohms per ft., the current in each section is then approximately 1 ampere. A layer of alundum cement holds the wire in place and prevents short circuits. Outside of this is placed a second layer of asbestos for heat insulation.

The finely divided mercury passing upward through the central glass tube in the coil becomes covered with a film, consisting of oxides of the foreign metals present and of mercurous oxide.

The mercury from auxiliary reservoir *A* flows down through a tube back to the main reservoir *M*. A stopcock in this tube serves to keep sufficient mercury in the auxiliary reservoir to prevent air being sucked in from the main reservoir. Also mercury may thus be held in the auxiliary reservoir while cleaning the main reservoir and spray chamber, or when inserting new bolting silk. The stoppers in the various tubes and reservoirs are of cork, except those in the auxiliary reservoir, which are of rubber in order to maintain the partial vacuum.

The optimum charge of mercury for the purifier is about 120 cc. or 1.6 kg. The mercury flow from the main reservoir, when regulated so as to spray evenly and freely through the bolting silk, is approximately 30 cc. per min. Thus all of the mercury circulates through the entire apparatus about one hundred times in a 7-hour period. The mercury is allowed to circulate

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[1] See Crafts, *Bull. Soc. Chim. Paris*, **49**, 856.

[1] N. E. Loomis and S. F. Acree, *Am. Chem. J.*, **46** (1911), 595.

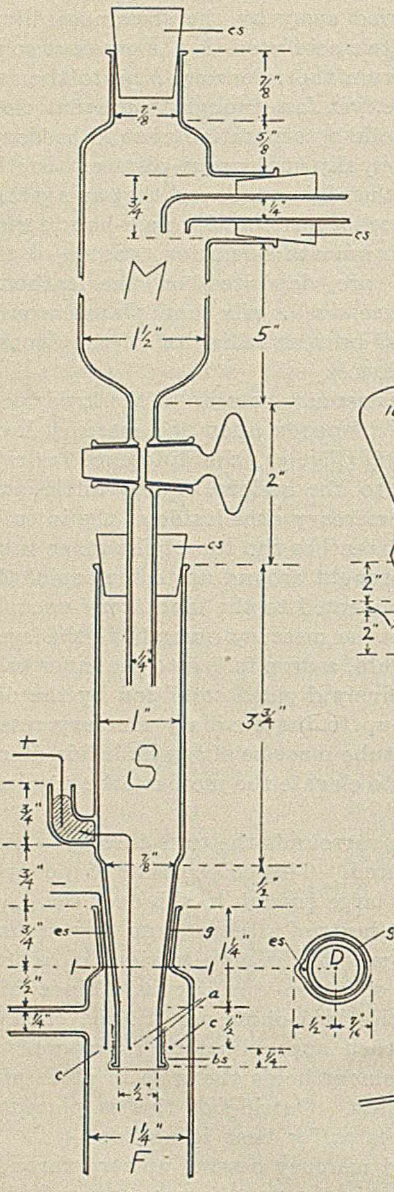

FIG. 2—DETAIL OF RESERVOIR and SPRAY CHAMBER

g, Ground joint
bs, Bolting silk
cs, Cork stoppers
es, Electrode slot
a, Anode
c, Cathode
D, Section through 1-1

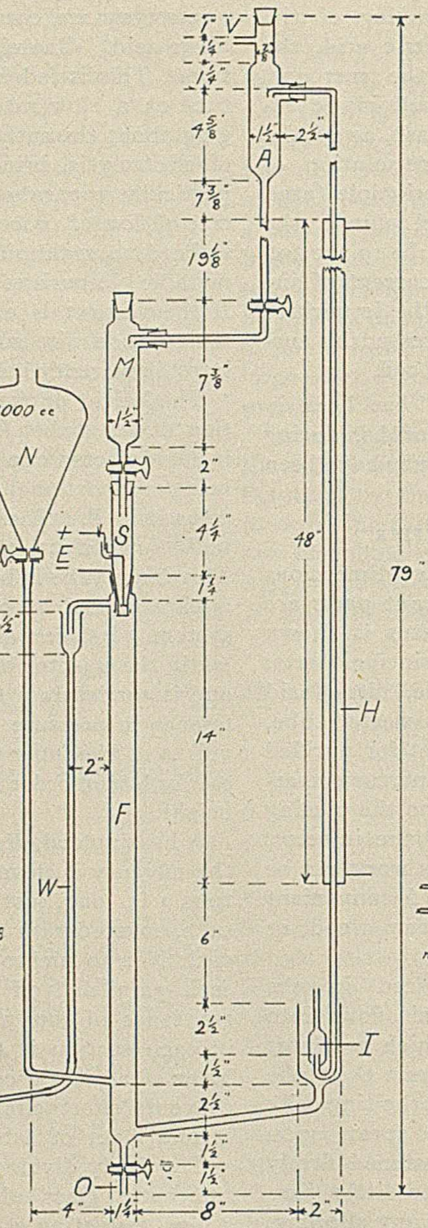

FIG. 1. PLAN OF MERCURY PURIFIER

M, Main Mercury Reservoir
A, Auxiliary Mercury Reservoir
N, Nitric Acid Reservoir
V, Connection to Vacuum
E, Electrode Leads
H, Heating Coil
S, Spray Chamber
O, Outlet for Purified Mercury

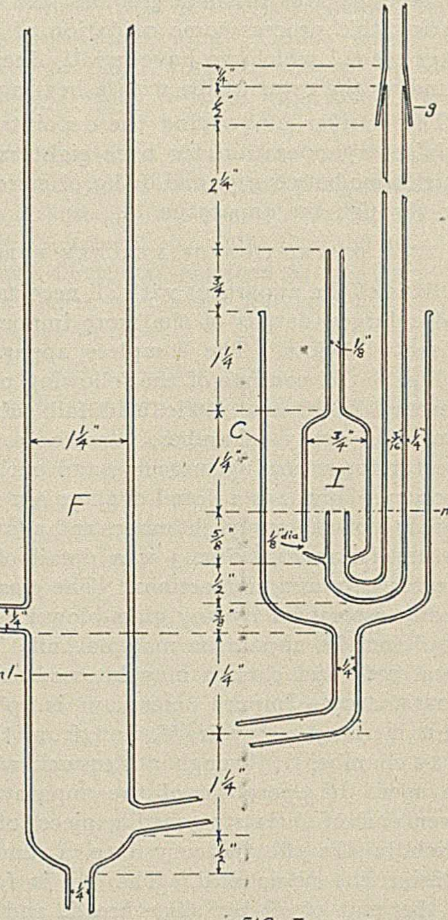

FIG. 3 DETAIL OF INJECTOR

F, Fall Tube
I, Injector
W, Waste Tube
C, Injector Cup
ml, mercury level

three hundred times and is then drawn off through the outlet *O*. At each withdrawal the mercury level in the fall tube *F* is allowed to fall only to a point just above the tube leading to the injector cup. In this way a constant volume of purified mercury is maintained in the lower portion of the purifier, and is not removed except for cleaning the apparatus or shutting it down for a long period.

We have collected the waste nitric acid containing mercurous nitrate and impurities, and have precipitated the mercury with hydrochloric acid in the form of calomel. At the present high price of mercury it is probably worth while to reclaim the waste.

Data from the operation of the purifier show that there is an average loss of 7 per cent of the mercury due to solution in the nitric acid. From two-thirds to three-fourths of this mercury lost by solution may be recovered as calomel.

The apparatus needs very little attention. The nitric acid reservoir is filled once a day, the mercury flow is adjusted when starting up the apparatus in the morning, and looked after once in a while during the day, and the purified mercury is withdrawn when necessary. One piece of bolting silk usually lasts 15 to 20 days.

The purified mercury withdrawn from the apparatus may have a slight amount of nitric acid entrained. We have eliminated this by distilling once with air bubbling through according to Hulett's method,[1] and then redistilling *in vacuo*, using a modification of the Weinhold automatic still.[2]

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[1] G. A. Hulett and H. D. Minchin, *Phys. Rev.*, **21** (1905), 388.

[2] Weinhold, *Carls. Report*, **9**, 68.

We have used the mercury purified in this manner in our thermoregulators and found it to give satisfactory results. Calomel half-cells made up with this mercury have checked closely with each other and with cells made from pure mercury obtained from the Bureau of Standards. An attempt was made by using Hulett's method[1] for determining small traces of impurities, to distinguish between various stages of purification, but no clear-cut differences were obtained.

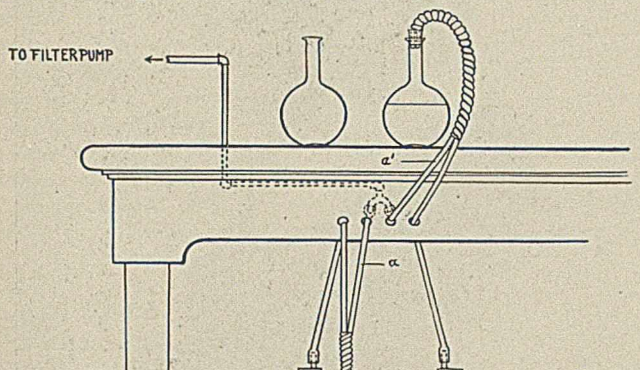
Bureau of Chemistry, Washington, D. C.

HANDLING LABORATORY SOLUTIONS BY SUCTION

By C. L. Beals

Received February 10, 1917

To avoid lifting heavy bottles of solutions constantly employed in the laboratory, an ordinary filter pump provided with a few simple rubber and glass connections may be used to advantage.



The accompanying illustration shows an adaptation of the idea which has been successfully applied at our laboratory for dilute acid and alkaline solutions used in fiber determinations. The cumbersome supply bottles are placed out of the way under the bench. Each is fitted with a 2-hole stopper (one hole acting as an air vent) through which a glass tube extends nearly to the bottom of the bottle. These tubes are connected by means of $^3/_8$-in. light pressure tubing to short glass nipples likewise extending through 2-hole stoppers of a size suitable for the flask in use. Nipples passing through the other holes of the stoppers are similarly connected by rubber tubing and a glass Y to the filter pump. For convenience, the tubes leading from the rubber stoppers are bound together for a short distance with adhesive tape. To handle solutions, one has now only to place the stopper connected with the desired solution tightly into the service flask and start the filter pump. A vacuum tends to form in the system, which is made complete by closing one branch of the Y by pressure of the fingers at α (or α' as the case may be). When sufficient solution has flowed into the service flask it is instantly stopped by releasing the pressure, thus venting the system. The solution remaining in the tubing immediately drains back into the supply bottle. The apparatus works admirably and does away with pouring from heavy bottles or bothersome syphoning.

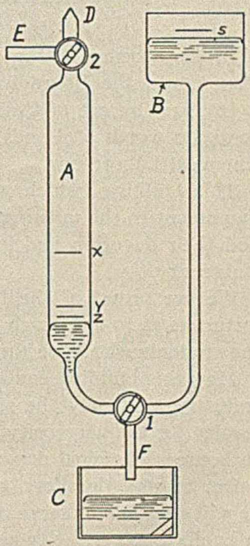
Agricultural Experiment Station
Amherst, Massachusetts

AN IMPROVED BUNSEN DIFFUSION APPARATUS

By Jerome S. Marcus

Received February 27, 1917

In a laboratory, where gas density is determined often and with only moderate accuracy, the Bunsen diffusion method has been found the quickest. There is no weighing, room temperature is used, and there are no liabilities of error from external conditions. The two chief factors of error are due to the difficulty of collecting over mercury and transferring to the apparatus the gas under investigation, and the irregularity of the action of the float.



The apparatus shown in the sketch was designed to eliminate both the float and the transference of mercury and gas, at the same time securing a more uniform pressure on all determinations than by the immersion method. Not only is the manipulation reduced to a minimum, but also the degree of accuracy increased.

The glass tube *A* is fitted at the top with a 3-way cock, opening to the orifice *D* and the tube *E*. At the bottom it is connected by the 3-way cock 1, to the mercury reservoir *B* and the tube *F*. The removable reservoir *C* may be any small vessel. For greater convenience the whole may be mounted on a board.

To standardize against air, cock 2 is opened to communicate with *E*, while the mercury is run out of *A* through *F* by cock 1. With 2 still open, 1 is turned to allow mercury to flow from *B* to *A* till the meniscus reaches the mark *z*. Both cocks are then closed, and *B* is filled from *C* to the mark *s*. At the same time both cocks are turned, so that *A* opens to *D* at the top and *B* at the bottom. By means of a stop-watch, the time for the meniscus to move from the *y* to the *x* is determined.

To determine the rate of diffusion of the gas for the comparison to that of air, 2 is opened to *E*, and *A* filled with mercury from *B* through 1. Cock 2 is then closed. The reservoir *C* is moved up to close the bottom of *F*, which is then connected to *A* by 1. This makes *A* the mercury-filled vessel in which the gas is collected. *E* is connected to the source of dry gas, and then connected to *A* by 2. The gas entering *A* drives out the mercury into *C*, against the difference in pressure between that of the atmosphere and of the mercury column in *A*. The mercury levels are brought to the marks *z* and *s* as before and the rate of diffusion measured.

University of Colorado, Boulder

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[1] G. A. Hulett and H. D. Minchin, *Phys. Rev.*, **21** (1905), 391.

FORMAL OPENING OF THE NEW CHEMICAL LABORATORY UNIVERSITY OF CINCINNATI

The formal opening of the new chemical laboratory of the University of Cincinnati took place on Saturday, April 7, 1917. The exercises, presided over by Judge Rufus B. Smith, were held in McMicken Hall, Burnet Woods. The addresses were as follows:

"Presentation of the Building," Robert Hochstetter.
"Reply on Behalf of the Department of Chemistry," Lauder W. Jones.
"Reply on Behalf of the American Chemical Society," John Uri Lloyd.
Address—"The Swing of the Pendulum in Chemistry," Charles H. Herty.

The chemical laboratory was open for inspection from 9 A.M. to 6 P.M. A brief description of the laboratory is found following the addresses.

A dinner in honor of the occasion was arranged by the Cincinnati Section of the American Chemical Society, and was served at the Hotel Gibson. Dr. Alfred Springer acted as toastmaster. Addresses were made as follows:

"Chemistry in a University," Charles W. Dabney.
"The Relation of Chemical Industry to the University," Robert Hochstetter.
"The Transmutation of the Chemist," Lauder W. Jones.

PRESENTATION OF THE BUILDING

By Robert Hochstetter

In the absence of Mr. Pollak, the Chairman of the Building Committee, the Board has requested me to act in his behalf.

In 1912, by act of Council, the City was authorized to issue $250,000 worth of Bonds, to be used in the construction of a Chemical Laboratory.

This building (which we are dedicating to-day) stands as a monument to the untiring efforts of Dr. Jones and his associates and their devotion and loyalty to Science, Education and this University.

We are proud of the fact that our University has been so fortunate as to have such an able teacher and chemist to guide the students who have the privilege to come under his influence.

May this building continue to serve the cause of Science and Industry, and may the students repay our city in service and loyalty for the generosity of our citizens which made it possible to house a chemical department which is now second to none!

Our thanks, therefore, go out to Dr. Jones with the hope that he will continue to serve our University for many years to come in as efficient a capacity as he has in the past.

It is, therefore, with much pleasure that I, in behalf of the University Trustees, turn over the keys of the new laboratory to you, Dr. Jones, and may the good work you have so nobly begun be carried on indefinitely!

REPLY ON BEHALF OF THE DEPARTMENT OF CHEMISTRY

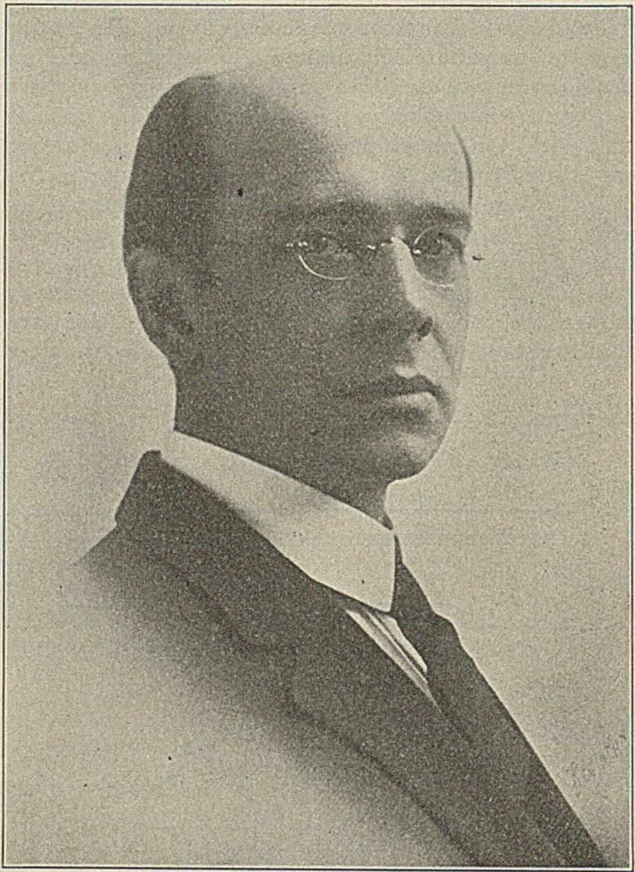
By Lauder W. Jones

In reply to your very generous words, it is my privilege to speak not only for myself, but for the members of my staff, each and every one of whom has assisted in formulating and carrying into effect the plans of this new building. Permit me, therefore, to express to you our individual and composite appreciation of the trust which it is our honor to receive from you as representative of the Board of Directors of this University.

In accepting this laboratory for the services of chemistry and metallurgy, we are aware of the opportunities which you have granted us, and at the same time, of the responsibilities which you have imposed upon us. I assure you that it shall be our endeavor to use our opportunities wisely, and to assume our responsibilities willingly, and that, at all times, we shall strive to make this building, the home of the Department of Chemistry, a place where the things accomplished shall be worthy of the University, and of the community which it is designed to serve.

Although every chemist has built laboratory-castles in the air, and has dreamed out plans for these ideal buildings, not many ever find themselves fortunate enough to see these dream textures fix themselves and become reality. Here, in Cincinnati, it has fallen to my lot to observe and guide a genuine transmutation of this very kind—to see a building develop from the idea of a building, through preliminary sketches, blue-prints, specifications, to the material stage as it stands now in brick, stone and mortar upon our campus. Not my laboratory-castle, perhaps, but better suited for the rough uses of daily life.

So, even though I must count my share in the bringing of these things to pass, a task, nevertheless it was a pleasant task, and one which I was not loath to undertake, since it had inseparably bound up with it something of the joy which an artist must experience in the creation of his work.



DR. LAUDER W. JONES
Professor of Chemistry, University of Cincinnati

On this occasion, it may be interesting to relate briefly the important points in the historical development of the Department of Chemistry in this University. In 1873, the University Board decided to establish an Academic Department. By courtesy of the Board of High Schools, provisional arrangements were made under the management of Mr. G. W. Harper, Principal of Woodward High School, to offer courses of instruction in the High School. In October 1873, under the direction of teachers of Woodward High School, courses in ancient language, German, Mathematics, Chemistry and Physics were offered. Mr.

C. R. Stuntz was the first teacher of chemistry. His class numbered 13.

In 1874 regular courses of study were commenced with five recently appointed professors who offered the following subjects: Mathematics, Ancient Language, English, German, Physics and Chemistry. Dr. Frank Wigglesworth Clarke was chosen Professor of Physics and Chemistry. The Board of Education granted the University permission to use the 4th story of the Intermediate School located on Franklin St.

In October 1875, the first University building, on the McMicken grounds, near the Elm Street Incline, was completed. The Departments of Chemistry and Physics had quarters in the basement of this building. During this year there were in all 62 students enrolled in the academic department of the University.

In 1883, Dr. Clarke left the University to become associated with the U. S. Geological Survey in Washington, and Dr. Thomas H. Norton was appointed in his place.

McMicken Hall in Burnet Woods was completed and opened in September 1895, but the Departments of Chemistry and Physics could not be accommodated in the new building. The old University building on McMicken Ave. was rented by the University to the Board of Education for the use of the 6th District School, with the agreement that the Departments of Physics and Chemistry should occupy a portion of the building. However, in June 1895, a communication from Mr. Henry Hanna announced his intention of presenting to the University $45,000 to be used for the erection of the "North wing of the New University Building." Work upon this building was commenced shortly afterwards, and in December 1896, the Departments of Chemistry and Physics were moved into Hanna Hall which they shared with the Department of Civil Engineering.

I have been told that many citizens wondered how the vast space in this building could ever be filled with students of chemistry. Professor Norton found himself supplied with room more than ample, but with equipment which was practically nil. An interesting item in the University budget for the year 1896 indicates how serious this need must have been. It reads: "For moving to Hanna Hall, $9.30."

Hanna Hall was formally dedicated on May 13, 1897, just 20 years ago, lacking one month.

In 1900, Dr. Norton left the University to become United States Consul to Harput, Turkey. He was succeeded by Dr. Thomas Evans, who had been assistant Professor of Technical Chemistry in the department. During his term of office, the Engineering College, through the coöperative plan proposed by Dean Herman Schneider, started upon its successful career. Dr. Evans at the time of his death, in 1907, was Dean of the Engineering College.

In the fall of 1907, the Department of Chemistry was placed in my charge. At this time there were about 80 students enrolled in chemistry. But the rapid growth of the Engineering College, the affiliation with the Medical School and the organization of the pre-medical course, the introduction of courses in Domestic Arts, brought ever increasing numbers of students into the department, so that Hanna Hall, in spite of the fact that many rooms occupied by the Engineering Department were released and fitted up as laboratories, was no longer adequate to accommodate the students who presented themselves, and the erection of a more ample building for chemistry became a necessity.

In 1897, when Hanna Hall was occupied by the Department of Chemistry, there were 326 students in all academic departments of the University. In 1917, twenty years later, when the new building was occupied, 520 students enrolled for courses in chemistry and metallurgy, that is more than one and one-half times the total number of academic students 20 years ago.

"Chemistry concerns itself with the quantitative study of the changes in composition and constitution which material substances undergo, and with the transformations of energy which accompany them."

The science of chemistry, therefore, is an organized body of knowledge which comprehends within it all of the facts, laws, theories, and hypotheses relating to these changes and transformations, arranged and classified in accordance with the method which custom and convenience have found best suited to meet the needs of those engaged in the study and practice of the science.

Chemistry is called an abstract-concrete science. Since it has for its realm all material substances and the changes in energy which accompany their transformations, there is nothing of a material nature which may not constitute a legitimate problem for investigation by a chemist. If investigations of this kind are conducted without the expectation of making a direct or immediate application of the results to some phase of our daily life, or of industry, the problem is often spoken of as belonging to "pure science." If, however, there happens to be in the mind of the investigator the idea of discovering something of commercial value or of utility to industry, the problem is said to be one in "applied science." Unfortunately, in America in particular, the opinion has been quite prevalent that between these two ways of choosing and pursuing research there exists a chasm which is spanned by no bridges.

A little reflection, however, will convince any reasonable individual that no such antagonism can exist. Michael Faraday, one of the world's greatest men of science, once made the statement that "There is nothing so prolific of utilities as abstractions." If we believe in the uniformity of nature and that the science of chemistry is unified knowledge, it is impossible to conceive of two sets of *unrelated* facts and laws, one in the possession of the "pure chemist" and the other the peculiar property of the "applied chemist." Even the so-called secrets of applied chemistry must be *explainable* by the laws of the "pure chemist." As a matter of fact, in the historical development of the science, applied chemistry came first. The workers in bronze during the bronze age were familiar with metallurgical processes and knew some of the properties of the alloy bronze. In the iron age which followed, the users of iron were perfectly familiar with the property of iron to corrode or rust. Ancient peoples who prepared sugars, gums, starches and dyes, knew intimately the properties and practical uses of these materials. These very substances to-day form the basis of many important industries presided over by applied chemistry.

The relations which exist to-day in nature are the same in essence as they were during the time of the aborigines. All of our laws, theories and hypotheses were latent in the nature of things, waiting for the mind of man to formulate them. We can imagine that very early in the history of the human race there came to be individuals who, in a crude way, observed that certain facts, well known to all their clansmen as of practical value, but looked upon by them as unrelated, in fact, passed analogies, and were co-related or correlated. This stage represents the beginning of the science of chemistry.

Thus, we see that the two phases of the subject, the pure and the applied, were mutually interrelated and dependent upon one another at a very early time, and, in fact, through all ages, they have been inseparably intertwined. In 1845, when Hofmann discovered benzene in the loathsome by-product, coal tar, the fact was of interest, at first, to pure science alone. In 1865, Kekulé proposed the hexagon formula for this substance, benzene; this, too, was apparently the outgrowth of pure speculation and was of importance to those engaged in pure science. Out of these beginnings, contributions of the "pure chemist," the vast industries grouped under the name of coal-tar industries have developed.

After the death of Kekulé, in 1898, 33 years after he had proposed the benzene formula, Japp delivered a memorial lecture in England in which he says:

"Kekulé's work stands preëminently as an example of the power of ideas. A formula, consisting of a few symbols jotted down on paper and joined together by lines, has supplied work and inspiration for scientific organic chemists during an entire generation, and has afforded guidance to the most complex industry that the world has ever seen."

If you will agree with me that my arguments are sound, then it must follow logically that the new laboratory, which has been entrusted to me and to my staff, to-day, can render greater service to the community and at the same time advance the science of chemistry best, if it is so organized as to deny, absolutely, that there exists between theory and practice even so much as a discernible cleavage plane. For the well-being of both they must be wedded—they must take one another for better or for worse.

The consequences of this point of view are almost self-evident. It will be our duty to see to it that elementary students who take courses in chemistry shall receive a most thorough training in the fundamental facts and laws of modern chemistry. As the students advance, their individual preferences will require different outlets for their activities, but, in any event, the department must see to it that each one shall develop along the lines which are best suited to make him an independent thinker and producer in the fields of chemistry.

So, for the advanced students, we should encourage in some the pursuit of research which may be as abstract as he pleases to make it, while in other cases it may be intimately associated with the noise and clangor of industry, believing, at all times, that both pathways lead ultimately to the same goal, the advancement of the noble science of chemistry, as well as the happiness, prosperity and well-being of mankind.

REPLY ON BEHALF OF THE AMERICAN CHEMICAL SOCIETY

By John Uri Lloyd

One of my friends said to me, "Now, Lloyd, Professor Herty is going to tell us about 'The Swing of the Pendulum,' which subject takes in all there is in chemistry, so you won't have anything to say about *chemistry*. Then Professor Jones is going to give us the history of the Chemical Society in Cincinnati, and it will be well for you not to take up much time talking about that." "What am I to talk about?" "Well," said he, "it is to be expected that an old man will talk about the early days, and give incidents that the younger people haven't had an opportunity to hear about, connecting times gone by with the present." So I shall take my text from the following thought, "Remember now thy Creator in the days of thy youth," and aim to put what I have to say within the fifteen minutes given me.

I speak as one who looks back, before there was any University of Cincinnati, before there was even a systematic "Art" of Chemistry in Cincinnati. I remember when the study of chemistry was started here, and believe I know the "Creator" of it all. As I cast my mind back into those days, 1863–4, come to mind those known then as the chemists of Cincinnati. They were Edward S. Wayne, Chemist of the Ohio College of Medicine, Daniel Vaughan of the Eclectic Medical College, and Professor Adolph Fennell, father of Professor C. T. P. Fennell. This was before the Cincinnati College of Pharmacy was established. Dr. J. F. Judge, Dr. W. B. Chapman, and others were co-laborers on the staff that followed. There were also the chemists of the public schools, one of whom you will remember as Professor Stuntz. In those days, the chemist was expected to be a druggist or a teacher in a school. Nothing was there such as we now have in professional chemistry. And yet the principles that dominated pharmacy and chemistry fifty years ago gave birth to the broader vision of the chemist of the present.

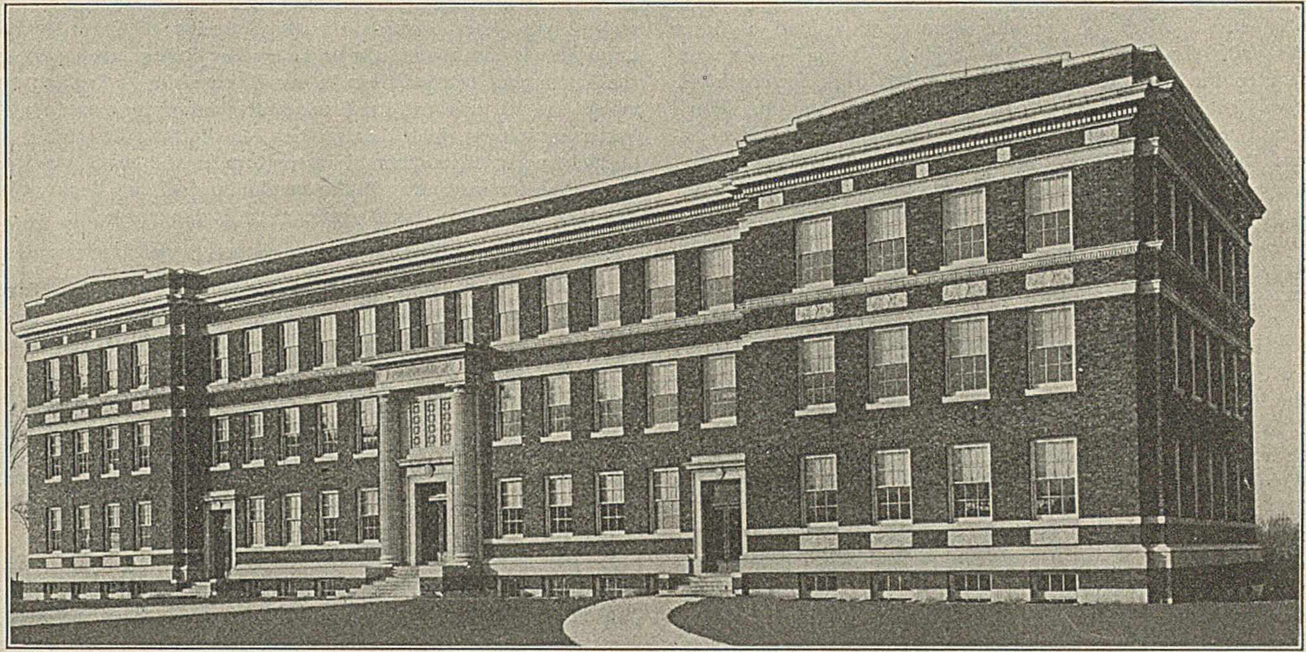
Dr. W. B. Chapman, who established himself as apothecary at 6th and Vine, where the Weatherhead drug store now stands, was one of the *educated* pharmacists in Cincinnati. There were but a few of these, the pharmacists of those days having, as a rule, undergone a very heroic apprenticeship. I can in my mind's eye see Dr. Chapman now. Well did he serve the people of Cincinnati. And with such an ideal before me, I think next of Professor Adolph Fennell, father of our present Professor Fennell, of the College of Pharmacy to-day. Many here cannot but remember him, kindly as a child, a talented, educated, German pharmacist-chemist, a good citizen, a man who did much in the beginning as a creator of that which followed. Comes now to view Daniel Vaughan, that man of extensive learning, who came down the Pike from Lexington, Kentucky, walking to Cincinnati, as also did Rafinesque, the botanist-scientist. Vaughan did what he could in behalf of chemistry, and, at the last he quietly starved and died in a little upstairs corner room at Sixth and John Streets, thus paralleling, in death as in life, the life of Rafinesque, for Professor C. S. R. Rafinesque died a pauper in the rich city of Philadelphia, his body being dissected as that of a homeless vagrant. The room where Vaughan died stands yet at Sixth and John Streets. Vaughan, recognized the world over as a great scientist and mathematician, it is true, starved to death in Cincinnati, but this was his own fault, because he was too proud to beg, too rich in intellectuality to consider physical pain. These men, and such as they, gave to us the chance that comes to us of Cincinnati to-day. Had it not been for them, there could have been no University at the date it was founded, such as stands on this beautiful hillside to-day. Their inspiration gave us Hanna Hall, and the inspiration of which they were a part, gave us that which followed Hanna Hall. Where we stand to-day was then country, only. Burnet Woods had not even been made a part of the city. Indeed, as I recall events, it was long a question whether Cincinnati would receive it as a gift. Fortunately, it was accepted.

Whatever is, had a beginning, but few realize how slight is that beginning. Like the seed from which grew the great redwood tree of the West, was the beginning of this great building here. Its beginning is the very commencement of cosmopolitan art, of alchemistic chemistry, and of connected sciences, which are one and all lost in the shadows of antiquity.

After the days of Chapman and Fennell and Wayne and Judge and Vaughan, those pioneers of the olden time, came the founding of the society that I am honored in representing to-day. The old building in which this start was formulated stands to-day down the hillside, pathetically pleading its own cause. I remember the problems with which McMicken University on the hillside then had to contend. She had no professor of chemistry. The public schools of Cincinnati gave to her the first teacher of chemistry. Give credit to Professor Stuntz, of Hughes High School, whose lectures were given, not in a grand edifice like this, but in the very basement of that old building to a class numbering a possible dozen.

The first authorized Professor of Chemistry in the Cincinnati University, was F. W. Clarke. But partly understood by the men among whom he moved, a pure scientist, his ideals were in investigating lines, not then conceived to be connected with commercial activity. For this reason he did not appeal to most people, but some, like myself, felt like taking off our hats when we met him. Professor F. W. Clarke was working on what he knew to be the very foundation of chemistry, but yet this was seemingly outside the lines of bread and butter, as chemistry was then looked upon.

Came then Professor Thomas H. Norton, and came soon an improvement in location in the building. He was given a much better place than down in the basement. Chemistry was no longer considered a spectacular study, for exhibition purposes as a lecture room show, a something with which to entertain children. Chemistry had now come to be viewed as a science destined to become a feature of the future, and to dominate every-day life. Professor Norton was a man well qualified to utilize chemistry for practical purposes. With him, by good fortune, came Mr. Hanna's gift, Hanna Hall. At that time I was very close to Norton. I knew what he was struggling to accomplish, and time and again spent afternoons and Sundays in his home when the new chemical department was being planned. I comprehend what he and Mrs. Norton did to found that department. Together they made charts for atomic and molecular weights. Together they worked on models to exhibit problems in stereo-chemistry. In other ways Mrs. Norton helped, preparing demonstration maps for chemical lectures explanatory of experiments. Professor Norton and his assistants had little in the way of apparatus, but with that little and what they added, they did well their work. Then came the call to go to Syria, and Dr. Norton gave up his place to his first assistant, Dr. Thomas Evans. He, too, did well his work, but, unfortunately, was taken away from us, in the very strength of his life. But such work as his could no more die than does the redwood tree when it falls to earth, leaving its life seed to become a new tree.



Chemical Laboratory, University of Cincinnati

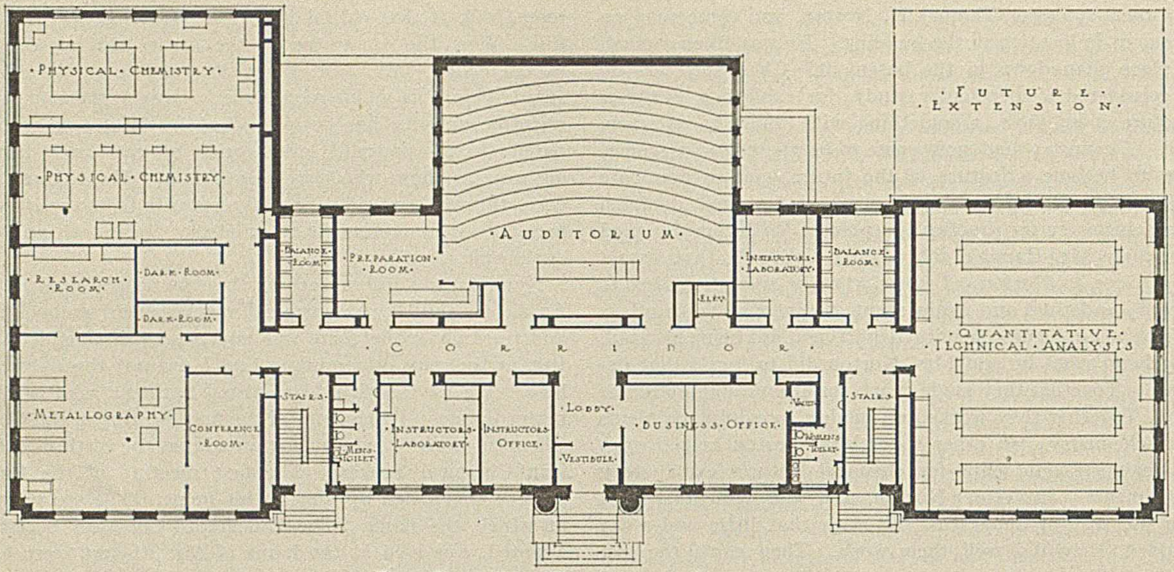
Came then our good friend, Professor Jones, and I must tell you something about the commencement of the chemical department of the building into which he went. When it was first built, there were so few students in that department that one day, as we stood in its uncompleted rooms, Norton said, "Lloyd, I wonder if it will ever be filled with a class. Haven't we bitten off more than we can chew? In other words, shall I not be criticized for spending so much money on a laboratory?" He did not foresee the stupendous growth that would so soon be accomplished.

Came finally Dr. Dabney, a President so competent to study men, and to put the right man in the right place. The Chemical Department that Norton felt might never be filled, grew until the students could not get into it. When you go from this room, look at that old laboratory in Hanna Hall, then consider this. View the apparatus in the great building constructed for chemistry, only. Dr. Jones sits beside us. His mind work stands before us as his monument. Every sphere of American scientific activity has been touched by him, and he has prepared these wonderful laboratories to give back from their outcome to those who have served him. The engineers in science, the whole world over, have contributed to what you find here. It is a monument to scientific Cincinnati, an honor to our people.

Now just a word concerning the American Chemical Society. An old man in the art, some call me, because I go back in study and research before there was any American Chemical Society. But in the way of reminiscences, I am not the only old man here. We have with us Dr. Alfred Springer, who can tell you better than can I, the story of the first meeting of the Cincinnati Chemical Society, which long antedated the birth of the American Chemical Society. The first meeting of the Cincinnati Society was called by him, at his home. The second meeting, at which the name "The Cincinnati Chemical Society" was adopted, was held at the home of Mr. Rheum, over his drug store, Fifth and Broadway. Subsequent meetings were held in the Ohio Mechanics Institute. When the time came at last for the organization of an American Chemical Society, we naturally became the first section of that society in this part of the West. To attend our meetings, came chemists from Columbus, Indianapolis, Lexington, and other cities round about.

I have presented but a superficial idea of that which comes to us from this great building. "Remember now thy Creator in the days of thy youth." We are to-day in our youth, as concerns chemistry. We are but pioneers, struggling, as I believe, over a by-path that leads to unseen avenues. Our path till now has been stony. Who can foresee what will develop to-morrow?

I hold in my hands a slip of paper on which is written, in substance: Three years ago, Cincinnati entertained the American Chemical Society. In order that proper attention might be given to visitors at this meeting, a collection was taken, and a committee appointed, who were given authority to spend all they needed. We took care of our visitors, to their apparent



First Floor Plan, University of Cincinnati

satisfaction. After all the bills were paid, a fund of $1500 was left. To whom did this belong? It had been contributed for a special purpose, which had been accomplished. It was decided, after much consultation, that this fund should form a nucleus to help needy young people who wished to attend the Chemical Department, and who would "make good" in life if given an opportunity. It was decided that this money be loaned them, the principal and interest to be returned to the University when in later years they were enabled to do so, through the equipment they had received. Can a more important fund be established than this? Is the equipping of these young people to be good citizens and so situated as to care for the interests of their country, not an opportunity? I will take the liberty of reading the names of four of those who by their contributions have increased this fund to the sum of $2,000. These are Dr. Ernst Twitchell, Dr. F. W. Weissmann, Mr. Robert Hochstetter, and the firm of Ault and Wiborg. May I not be permitted to predict that those who can help this fund in the city of Cincinnati will be surprised, fifty years from now, to know what this nest egg has accomplished?

As a closing thought, may I not be personal? Much do I appreciate the opportunity of speaking here to-day. To appear on the same platform with the learned men of this great University, including its President, to know that I am listened to by Dr. Herty, past president of the American Chemical Society, to be introduced by Judge Rufus B. Smith, of the Board of Trustees, and to speak to an audience made up of intellectual Cincinnati, is indeed an honor that anyone might well hold precious.

THE SWING OF THE PENDULUM IN CHEMISTRY

By Charles H. Herty

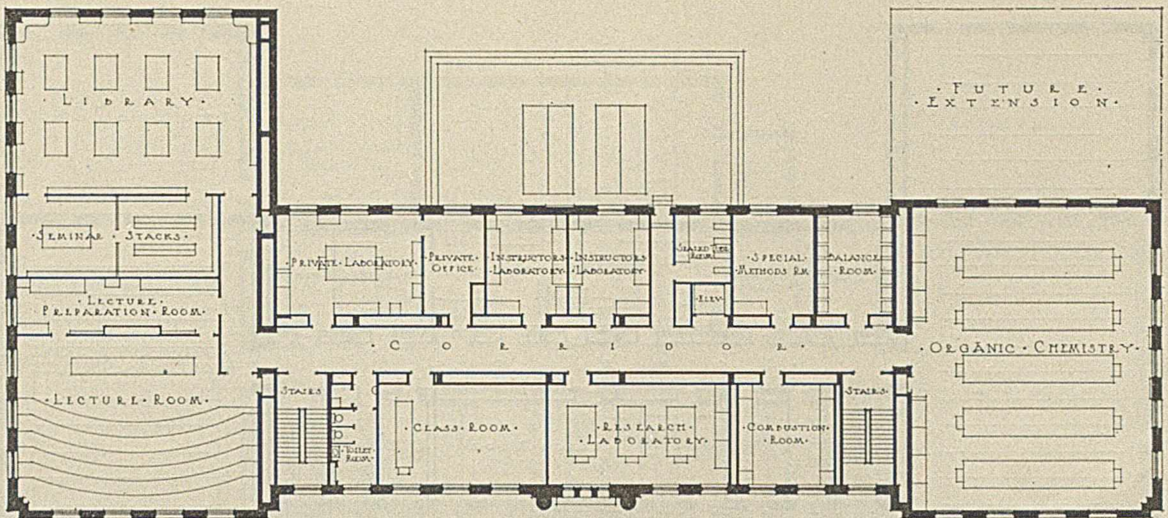
It is a peculiar pleasure to be present on this occasion which marks the formal opening of the new Chemical Laboratory of the University of Cincinnati. Too often of late have we read of loss by fire of historic laboratories; too often have we heard the lament of laboratories overcrowded with students. Here brick and mortar have been promptly joined and a fine housing given to the splendid corps of instructors whose work and whose policies have given to this institution as high rank in chemistry as developed in America. Here, too, has been happily worked out a combination of devotion to the advancement of chemistry as a science and to the application of chemistry in the service of mankind.

The erection of this building gives heart to the hope that the pendulum which has swung so strongly during the past two and a half years towards the development of the chemical industries, is now about to begin an equally strong swing towards the betterment of conditions in our Universities—a betterment which should manifest itself in greater laboratory space and better equipment, in higher salaries for the teaching staff and in such increased number of instructors as will make possible more time for research. If the pendulum does not begin soon to swing strongly in this direction there is danger ahead.

CONDITIONS UP TO 1914

Up to the outbreak of the present great war the chemists of America had been working quietly but with zeal and effectiveness in university laboratories until we had reached the point where no longer was it considered necessary to go abroad to receive thorough and inspiring training in chemical research. This fact had not been widely advertised but it was evidenced by the constant increase in the proportion of young men receiving their higher training in the universities of this country, and by the constantly increasing prestige of the chemical journals of America.

So, too, in the industries of America, chemists had been busily and successfully engaged in solving the many problems of those industries which, because of our resources and environment, were of primary importance. Corn, the great staple of the Middle West, had been converted into a host of useful substances, through the transforming agency of our chemists. Cottonseed, so long neglected in the southern states, had become the raw material of a great industry whose output now approaches in value that of the lint column which for many years had alone given value to the cotton crop. The advances in metallurgy furnished brilliant examples of skill, ingenuity and sound chemical development. Petroleum refining had made possible the tremendous development of the automobile and allied industries. The tanning of leather had been placed upon surer foundations. The great industries of the heavy chemicals, acids, alkalies, etc., received a development of such consummate skill and magnitude of output as to assure success in other lines. At Niagara Falls an electrochemical industry was founded which stands to-day the greatest of its kind in the world, the products of this group now constituting essential equipment of many industries throughout the nation, and assuring us a degree of national preparedness, whose importance no one can overestimate in the great war into which we are entering. These are only a few illustrations of the many-sided and successful efforts of the industrial chemists previous to the outbreak of the present war.



Second Floor Plan, University of Cincinnati

The work, had been done quietly, earnestly and without advertisement and the nation accepted the results with but scant credit to those who primarily were responsible for the splendid achievements. It was, therefore, perhaps natural that with the *shortage* in coal-tar chemicals made manifest through the blockade of German ports, the nation should suddenly have awakened to the importance of its chemists and in that condition which is so often characteristic of rousing from sound slumber petulantly exclaimed: "Why have not our chemists provided us with a dyestuff industry commensurate with our needs?" Some rough jolts were necessary to make clear the situation that chemists alone cannot always develop industries, that combination of capital with chemical skill is essential and that capital is not inclined to invest in industries whose fostering, through adequate tariff legislation, has not only been neglected but strongly opposed by our own consumers. The full wakening, however, was not long delayed and with fine spirit consumers, capitalists, and chemists joined hands, effecting the development of a domestic dyestuff industry which has amazed the world, and which on the morrow, in the annual Easter parades, will give abundant evidence of its life and vigor.

EXPANSION OF CHEMICAL INDUSTRIES SINCE 1914

Along with developments in connection with industries using coal-tar products as raw materials, there has also been a great expansion in all other lines of chemical industries, which during the years 1915, 1916 and the first three months of 1917 has called for a direct investment in these industries of $184,139,000. Not even the greatest optimist could have foreseen such an enormous expansion of the strictly chemical industries in so brief a time. The successful use of this vast sum has aroused the admiration of our people and to-day the chemist occupies a new plane in American public esteem, a position rendered all the more secure by the universal recognition of the important rôle the chemist plays in modern warfare.

To meet the universal demand for thoroughly trained men the industries have drawn heavily on university ranks. Men long habituated to the lecture room and university laboratory have taken new positions in the ranks of the industries and with characteristic American adaptability have fully met the new responsibilities. If this drain on University staffs goes much further there is distinct danger ahead to both the universities and the industries. In the universities should always be found men of the highest possible type, whose researches will carry forward the *science* of chemistry which usually precedes discoveries fundamental to the fullest development of the chemical industries. Such men attract and hold within universities for graduate work the best class of the undergraduate body upon which the future of chemistry in America will largely depend.

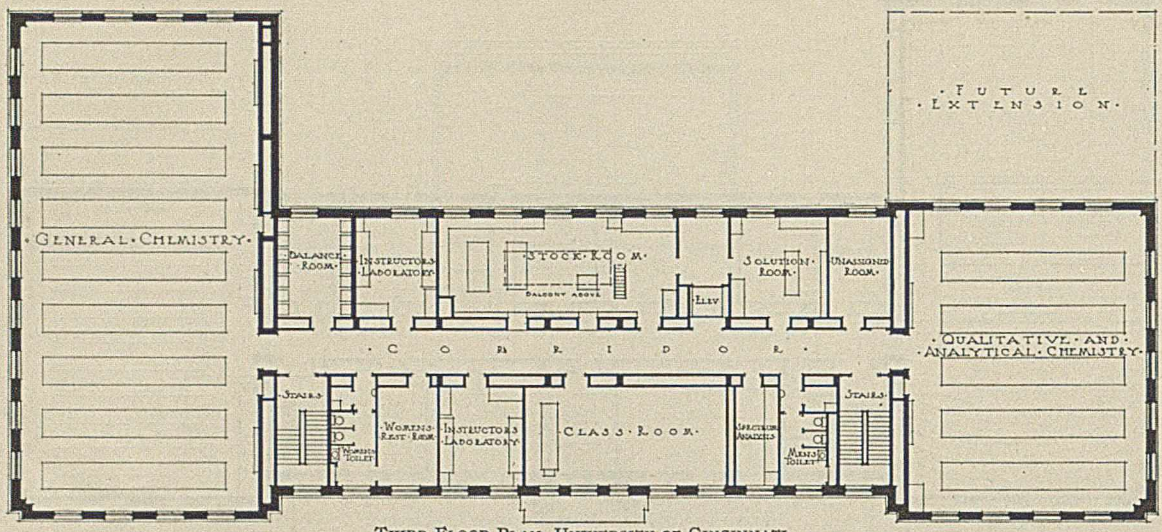
INCREASE IN CHEMISTRY STUDENTS

Furthermore, the wide advertisement which chemical prowess and prestige have recently received has resulted in largely increased numbers in chemistry classes working in laboratories taxed to utmost capacity. In order to secure facts in this connection a questionnaire, that most dreaded of all documents which pass through our mails, was sent to the registrars of all American colleges and universities listed in the report of the United States Commissioner of Education for the year ending June 30, 1916. In the hope of receiving reasonable returns the amount of statistics requested was very limited. Information was asked for the institutional years 1914–15, 1915–16, and 1916–17 as to the total number of students in chemistry, in chemical engineering, and in graduate courses in chemistry, and as to the amounts appropriated to chemistry departments (not including salaries) from funds other than fees paid by students.

Returns from this questionnaire have been slow in coming in and the information is far from complete. Nevertheless statistics have been received from one-third of our colleges and universities and fortunately these figures cover typical institutions, both the larger universities and smaller colleges being represented in approximately the same ratio as in the complete list of the Commissioner of Education.

The figures as to total students in chemistry are not suitable for use as misunderstandings arose as to whether the total number of individuals or the total enrollment of the classes had been requested. Nor are the figures as to appropriations to chemical departments suitable for specific use, as very few institutions answered this question. From those which did answer, however, it would seem that for the most part the chemistry departments of our colleges and universities subsist almost entirely, outside of the professors' salaries, on fees paid by the students. Appropriations from general university funds are in most cases negligible. It is to be regretted that a definite showing cannot be made in regard to this matter. To all who appreciate the unusual cost of material and equipment for research it must be plain that unless special appropriations are made from funds other than those received from students, the untrammeled prosecution of research must necessarily be hampered.

From this questionnaire, however, two reliable sets of figures have been received and these are exceedingly gratifying in view of the increased demand for trained chemists, both in the control work and in research in the chemical industries whose continued development and expansion are so important at this particular time. For the session 1915–16 the registration of students in chemical engineering was 37 per cent greater than during the session of 1914–15, while the increase of 1916–17



THIRD FLOOR PLAN, UNIVERSITY OF CINCINNATI

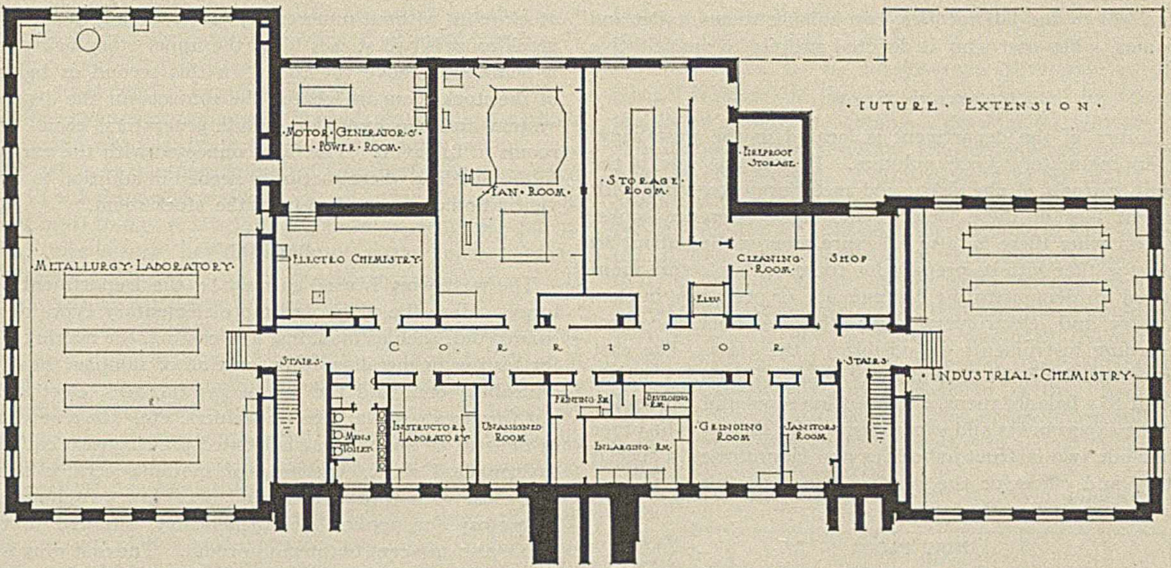
over 1915–16 was 21 per cent. The increase of the current session over 1914–15, which marked the outbreak of the war, is 65.7 per cent. When we consider the great need of chemical engineers this marked response on the part of students gives us a feeling of confidence for the future. Too often during the past two years has there been evidenced by those in charge of the erection of industrial plants an unfortunate lack of knowledge of chemistry from the engineering standpoint. Large sums of money have been lost through errors in plant design, resulting in some cases in the necessity of rebuilding. In many others the result has been inefficient operations which would have brought failure save for favorable contracts and the extraordinary prices resulting from almost famine in certain lines. Such plants cannot hope to exist when days of normal competition are again restored. It is greatly to be hoped that in those institutions training chemical engineers, more and more thought may be given to the important problem of acquainting students with the details of efficient plant design and construction, and especially that continued thought be given to the matter of coöperation between the universities and industries which will result in giving at least some plant experience to students during the days of their collegiate and university training. Interesting experiments are being made along several lines at a number of American institutions of learning. More such are needed, for in this way only, just as in the laboratory and plant, can the wisest solution of the problems be reached. It is unnecessary for me to urge this spirit *here in this institution* where already a definite policy has been marked out and where for years coöperative students have acquired valuable training which is to-day itself felt in many lines of chemical industry.

Another showing made by the questionnaire is one which has given a strong feeling of relief. During the past 2$^1/_2$ years there has been a tremendous demand for young men trained to the extent of an undergraduate course. For the services of such young men good salaries have been offered, as well as opportunities for advancement, and the privilege of taking part in the development of new organizations, which in itself is always a strong attraction for any young man of courage and spirit. It was feared, therefore, that this strong and attractive demand would be at the expense of the number of students in graduate work. If the results from the questionnaire are at all reliable we may comfort ourselves in the knowledge that from the figures received there was in 1915–16 an increase of graduate students of 37 per cent over 1914–15, while during the current session the number has at least not decreased and will probably show a small increase over the preceding session. The importance of this situation cannot be overestimated. If we are to measure up to our responsibilities in the development of chemistry the number of our graduate students must not diminish; nay, it should constantly increase no matter what personal sacrifices are involved in prolonged university residence, for through the work of this class of students the leaders of research will be enabled to carry on most successfully the advance in pure chemistry, while from the training thus received there will be developed minds capable of grappling with the problems of industries and of blazing out new paths of advance so characteristic of this nation. More than this, many *lines* of industry in this country are just beginning to awake to the value of chemical research in their organizations, so that we must supply the need not only for new industries but also for a great number of our older and more established industries in which the trained chemist, through systematic research, will work wonders hitherto undreamed of.

INCREASES IN SALARIES, NUMBER OF TEACHERS AND LABORATORY EQUIPMENT ESSENTIAL

In view of this increase in graduate students in chemistry, chemical engineering and the general courses in chemistry, and in further view of the draft made upon our university faculties by the chemical industries, it is high time influences should make themselves felt which will swing the pendulum, now so far swung to our chemical industries, back to the university side. This is important not only from the standpoint of the university but from the standpoint of the industries themselves, for it is from the universities that the industries draw their trained men, and only with the highest university development will we have more than a spasmodic expansion of the chemical industries. To what influence shall we look for this counterswing of the pendulum? The immediate answer is, to our university and college executives. Their influence has the greatest weight in the making up of the university budgets.

Along what lines can we justly ask their special consideration of the claims of chemistry? In the first place I would urge higher salaries for professors and their associates. While universities cannot hope to equal in their salary lists the offers made by industrial concerns, and while such an increase is unnecessary to persuade the man who loves to teach to remain in the university ranks, nevertheless it is but trite to remind of the constantly increasing cost of living which has characterized this war period. This means that where salaries have not been increased, they have been at least relatively decreased. Small wonder, therefore, is it, that men with responsibilities to others must take into account this decreased earning capacity? In the second place the increased number of students in chemistry signifies clearly that unless teaching staffs are increased in numbers the character of the instruction given must suffer. There is a distinct limit to the number of men that can be success-



Basement Plan, University of Cincinnati

fully taught by a capable instructor. The actual increase in students, therefore, is in itself sufficient argument for more instructors.

In the third place adequate laboratory space and equipment should be provided to meet fully all needs. This *too* has been well met here in this commodious building, but in many *others* such is not the case. Finally, all these cramping and restricting influences *must* make themselves felt in handicapping the efforts of the teaching force to prosecute research to the fullest extent. For such work freedom from financial cares is necessary; time for concentrated thought and continuous experimentation cannot be dispensed with and space and necessary equipment cannot be ignored if that work is to be carried out under the most favorable conditions.

When I think of the exodus of men from the universities to the industries the cure seems to be the same as is now beginning to prove so effective in the problem of urban and country life. It took many years before we fully grasped the thought that the solution of the problem of country life lay in the making of country life more attractive. So in our educational circles, if the universities are to hold their men, conditions which have now become in many cases so hard must be improved.

I recognize fully the tendency for each of us to think that our own work is of greatest importance and should be therefore particularly cared for, and under normal conditions I should hesitate to make this *special* plea for increased funds for chemistry in our universities. But with the signing by President Wilson of that memorable resolution of Congress which enters this nation as an active participant in the great war, this country enters upon a new era, in which every energy of the nation must be devoted to complete consummation of the purposes for which we entered this war and at the earliest possible moment. In this task it is recognized by all that the chemist plays an extremely important rôle. Upon his inventiveness the issue may be largely determined.

REAL TEST OF EFFICIENCY OF AMERICAN CHEMIST NOW BEGINNING

A brilliant representative of the U. S. Navy, Admiral Fiske, I think, pointed out how often success in battle depends upon the unexpected use of some new discovery. Such a possibility will necessarily, through patriotic motives, lead our minds into many new channels, and the real test of the efficiency of the American chemist is now beginning. This test involves not only those men who are directly concerned with the manufacture of munitions, with the development of new explosives, with the perfecting of alloys to meet new demands, with the conservation of food, with the testing of army and navy supplies and with the guarding of the health of the field forces, but it also involves, fundamentally, the work of instruction in our colleges and universities which should be maintained at its very highest possible standard in order that this country, which we all desire to serve even at the cost of our own lives, may be served most efficiently.

With such thoughts in mind I share with you that feeling of deep gratification which must fill your hearts as a result of the recognition of the needs of chemistry as expressed in the appropriation from the funds of the University of Cincinnati which has enabled the erection and equipment of this building.

DESCRIPTION OF THE NEW CHEMICAL LABORATORY UNIVERSITY OF CINCINNATI

Taken from Circular issued by the University of Cincinnati

The new chemical laboratory of the University of Cincinnati has a total frontage of 207 ft. and is, practically, four stories high, since the basement is mostly above ground, and is utilized for laboratory purposes. The west wing is 92 ft. long by 48 ft. 9 in. wide. The east wing, which is to be completed later, is 57 ft. long by 48 ft. 9 in. wide. The cross bar connecting these two wings is 110 ft. long by 52 ft. wide. The total cost of the building was approximately $250,000, including $25,000 for special scientific equipment. It houses the Departments of Chemistry and Metallurgy. At the time of its occupation, in September 1916, 520 students were enrolled in the various courses in chemistry and metallurgy.

FIRST FLOOR

The main auditorium for lecture purposes, which has a seating capacity of about 200, is located on this floor opposite the entrance. Adjacent to it is the lecture preparation room for setting up lecture apparatus. In the west wing are two laboratories for work in physical chemistry, and a laboratory for advanced metallurgy, together with a research laboratory for the Department of Metallurgy. In connection with these laboratories there is a dark room for metallographic work and a constant-temperature room for work in physical chemistry. The other rooms on this floor are devoted to various purposes as follows: the business office in which the records of the department are kept on file and through which supplies are ordered; three instructors' offices and laboratories; two balance rooms, toilet and wash rooms. The east wing is devoted entirely to quantitative analysis.

SECOND FLOOR

The library of the department is located in the west wing. It has approximately 3,000 volumes. It contains sets of the important journals of chemistry and metallurgy, together with the current files of these journals and many reference books. In the west wing there is, also a lecture room seating about 80 students, together with its preparation room. This lecture room is equipped for demonstrating experiments, or processes, in electrochemistry and electrometallurgy; the switchboard control and recording instruments are visible to the class. The east wing is occupied by the laboratory of organic chemistry which is adjacent to a balance room, a combustion room and a room for sealed tube experiments and experiments with gases. The other rooms include two instructors' offices and laboratories, a special laboratory and office for the head of the department, a classroom, and a special methods room.

THIRD FLOOR

The elementary course in general chemistry is given in the laboratory located in the west wing. The table tops are provided at each working place with a hood in which a suction is produced by fan systems. The east wing contains a laboratory now used for qualitative analysis, and is equipped with the same kind of equipment as that of the laboratory of general chemistry. The stock-room from which supplies and apparatus are issued is on this floor; from this stock-room the materials used in all of the laboratories are distributed. A passenger elevator which serves to carry students from other floors, enabling them to reach the stock-room without unnecessary delay, is adjacent to it. The stock-room is two stories high, the upper story being formed by a penthouse above the roof. In this second or balcony floor of the stock-room are located the controls for the distilled water system and the hydrogen sulfide generators, each in separate rooms in the attic. The fans connected with the various rooms in the building where suction is needed in addition to the general ventilation are controlled from the stock-room.

BASEMENT

The west wing is used entirely by the Department of Metallurgy. Here the rough and more elementary types of work are carried out, such as moulding and casting, the making of samples for testing in metallography, cutting of samples, heat treating, annealing, etc., and the use of gas furnaces, electric furnaces, pot furnaces, cupola, electric welders, etc. In a separate room in the rear of the building is located the electrical equipment for experimental work, consisting of motor generator sets, rotary convertor with transformers and necessary switchboards. The laboratory for applied electrochemistry, situated in the cross bar, is also adjacent to the motor room. The east wing is planned for experimental work in industrial chemistry. It will contain typical units of machinery used in industrial work, such as stills, autoclaves, filter presses, nitrating kettles, pumps, fusion pots, vacuum dryers, centrifuges, grinders, etc. A shop, to be put in charge of a mechanician, is located near this room. In addition to these laboratories the basement contains a storage room for supplies, a cleaning room, a fire-proof storage room for combustible materials, a stock-room, a grinding room, instructors' office and laboratory, a storage battery room and the general ventilating system for the entire building.

ADDRESSES

THE CHEAP PRODUCTION OF ALCOHOL

By A. M. Breckler

Received March 19, 1917

The possible uses for denatured alcohol are two: as raw material in manufacture and as fuel.

After eight years of existence, the net result of the denatured alcohol act was the production of 17,000,000 proof gallons of denatured alcohol in 1914, most of which was used in the industries. Here it displaced the potable article, but with so little benefit to the ultimate consumer, according to the Commissioner of Internal Revenue, that in his report he advised a small tax to cover the cost of inspection by government officers during the manufacture.

In 1915, the manufacture of munitions had stimulated the production somewhat, but the benefits to the American people as a whole are doubtful and had the alcohol used in this industry (about 5,000,000 gallons) paid tax, a good portion of our present deficit might not have existed.

It is, of course, as a fuel for explosion engines that we look for the greatest demand for alcohol and it is the production of alcohol for this purpose which will eventually determine the price.

The total alcohol produced in 1914 in the United States from all raw materials was 182,000,000 proof gallons, equivalent to about 5 per cent of our present gasoline consumption. By far the largest source of this alcohol was grain, from which 142,000,000 gallons were produced. To produce enough alcohol to cover our gasoline demands as a motor fuel would take 16 per cent of our total cereal production or about 26 per cent of our total corn production.

The average cost of alcohol from grain (corn) has been 17.5 cents per proof gallon for the past five years. This probably is a minimum figure as it is based on the cost in large distilleries and allows for the sale of the feed recovered at the average market price. The cost this season will not be far from 25 cents per proof gallon or about 45 cents per gallon of 90 per cent alcohol. If the alcohol is furnished in barrels, 2 cents must be added to this per proof gallon. This cost could not be cheapened materially. The overhead here figured is about 3 cents. So far as increasing the yield of alcohol per bushel, the possibility permits of an increase of only 5 per cent at most. It must be remembered that these figures carry no allowance for profit, selling expense and freight.

SOURCES OF CHEAP ALCOHOL

The possibility of making alcohol cheaper on the farm has been given prominence. The idea is to make alcohol and feed the slop to cattle. There are several reasons which would make this impractical in all except rare cases. The cattle fed on slop do not furnish meat which packs well. It is very tender and juicy and perfectly healthful, but becomes flabby on keeping. Therefore, it must be sold on a market which can absorb a large amount of it at a time. The experience of distillers who have fed large numbers of cattle (as many as 12,000 head at one distillery) is that such feeding is more or less of a speculation depending on the markets and the location of the plant, accessibility being necessary in order to enable them to take advantage of favorable prices. Aside from this there are fuel and water requirements which will be taken up later.

In 1912, the Minnesota Agricultural Experiment Station operated a 200-bushel distillery on grain. They employed an experienced distiller, had most careful chemical supervision of the plant and credited the feed at a very good price. Their yield was about 4 per cent under the average prevailing that season. The price charged for the corn was the average farm price as against the market price. The net result was that they were unable to produce alcohol for what they could buy it. If under these favorable conditions, the alcohol cannot be produced at a profit, what can the farmer expect?

The other large source of alcohol is black strap molasses which five years ago was a drug on the market and sold as low as 2 cents per gallon at the sugar house. Coincident with the sudden increase in the demand for alcohol, the demand for black strap increased until to-day it is selling for 12 to 14 cents per gallon. The high price of sugar has stimulated the sugar houses to carry their extraction of sugar as far as possible and hence the yield of alcohol possible has decreased very much.

From black strap of good quality at 5 cents delivered, the cost of production would be around 10 cents per proof gallon, it is true, but if we assume that all the sugar produced in the United States and Cuba represents 80 per cent of that in the juice and all the remaining 20 per cent would be available for fermentation, we would have raw material available for about 162,000,000 gallons of alcohol or enough to supply about 5 per cent of our present demand for motor fuel. It is probable that not more than 25 per cent of this amount is actually available, owing to the increasing yields possible from better sugar extraction processes.

A third source of alcohol is wood waste. Enough money has been spent on sawdust plants to evolve several processes for cracking oils. The fact is that after seventy years of experimenting there are two plants running in this country and were it not for the high price of alcohol at the present time, it is doubtful whether they would run. The bulk of the material handled, the use of strong acids, the complex machinery for leaching and the rapid stripping of the timber tracts under cutting all operate against the process even were other conditions satisfactory. As both the plants now operating are financed by large corporations, it is evident that lack of money has not held the process back and it is very probable that if the process were really profitable, more plants would be in existence.

The sulfite liquors from pulp mills offer a cheap source of carbohydrates. None of the existing processes are exactly satisfactory. Just how much of this is available is a little uncertain. One thing is certain, that the amount of fermentable carbohydrates in this liquor is very variable. At present there are about three plants operating in America on this liquor.

Any process for making alcohol must give careful consideration to the question of yeast nutriment. As the amount of yeast formed is dependent on the volume of liquid rather than the concentration of the fermentable matter, the most economical process in this respect will evidently be that in which the concentration of the fermentable is the greatest practical. About 8 lbs. of dry yeast are formed from every 1000 lbs. of fermented liquor of which 6 per cent or $^1/_2$ lb. is nitrogen. If a liquid contains 10 per cent of fermentable, the amount of nitrogen required is one-thirtieth lb. per gallon 160 proof. The potash requirements are about one-fifth of the nitrogen. Just as much attention to yeast poisons is desirable. This is one of the drawbacks to the use of sulfite liquor for the production of alcohol. Even such a common substance as caramel acts as a yeast poison.

FUEL AND WATER SUPPLY IMPORTANT FACTORS

The factors which most often determine the feasibility of making alcohol from low-grade material are fuel and water supply. The writer has prepared a curve (Fig. I) showing the theoretical heat consumption at various concentrations for distillation of 1 lb. of 160 proof (80 per cent by volume) alcohol. This concentration was selected as being the lowest which would meet all fuel requirements and the highest which could be prepared with comparatively simple apparatus. The values for 95 per cent alcohol are about 1.5 times as great, owing to the heat necessary for the rectification and lower yield in gallons. The factor 3.5 gives the minimum heat consumption while five times the theoretical would give about the average heat consumption.

For example, let us consider the distillation of 160 proof alcohol from sulfite liquor. As concentrated for fermentation, this liquor ordinarily contains 4 per cent fermentable. As this is hexose sugar, the resultant solution will contain theoretically 2.61 per cent alcohol. With a yield of 85 per cent (an average usually obtained), the resultant solution will contain about 2.2 per cent alcohol by volume. The theoretical heat of distillation per lb. 160 proof alcohol from such a concentration is 4,000 B. t. u. As the weight of one gallon of 160 proof alcohol is 7.2 lbs., the heat required under best conditions would be 126,000 B. t. u. while probably it would approach 180,000. Using a good coal running 13,000 B. t. u. per lb., the coal required under favorable conditions would be 10 to 14 lbs. Assuming such coal to cost $3.00 per ton laid down at the paper mill, the costs under good conditions for distillation of 160 proof alcohol would run 1.5 to 2.1 cents per gallon.

Similarly it may be figured that 160 proof alcohol from sorghum juice containing 15 per cent as hexose will cost 0.4 to 0.6 cent per gallon for distillation with coal at $3.00 per ton. This probably represents as concentrated a liquor as it would be possible to ferment, since the resultant alcohol from stronger liquor would probably affect the yeast before the attenuation was complete. In Germany, where thicker mashes are used, the practice is to thin out with cold water during fermentation.

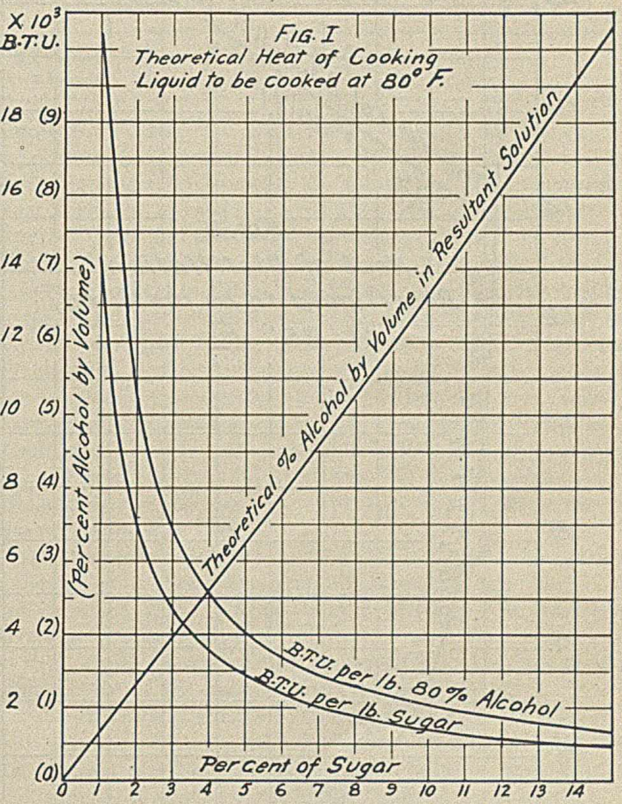


FIG. I

In the case of nearly any material containing starch, and most other materials, a preliminary cooking is necessary and here again the cost is greater the lower the concentration. Fig. II shows the B. t. u. per lb. hexose sugar and likewise per lb. 80 per cent alcohol at various concentrations of sugar. Starch multiplied by 1.11 of course gives the hexose concentration. The factor of 2.5 probably gives the closest figure to actual heat put under the boilers. Fig. II is based on cooking to 212° F. only. In a great many cases, the material is cooled to 310° F. in which case the figures given should be multiplied by 1.69 also.

Considering the case of garbage mash, a sample recently submitted to the writer contained 5 per cent fermentable hexose. This gave a figure of 4,100 B. t. u. per lb. of 80 per cent alcohol theoretically, but as the average yield is 85 per cent of the theoretical, this would become 4,800 B. t. u. Multiplying by 7.2 and 2.5, we would have a theoretical consumption of 86,500 B. t. u. or 6.65 lbs. coal which, at $3.00 per ton, represents 1 cent per gallon for cooking. If the mash should have to be cooked under pressure, the cost would be 1.8 to 1.7 cents per gallon 80 per cent alcohol, making a cost of 2.2 to 2.27 cents for cooking under atmospheric pressure and distilling, and of 2.9 to 3.4 cents at high pressure and distilling. In addition to this, power must be furnished for comminution, pumping, etc., all of which requires fuel. In a grain distillery, the fuel required for this purpose is about half of that for all other purposes. It is, of course, difficult to state this cost for all materials, but this relation would probably not give excessive figures for any other material.

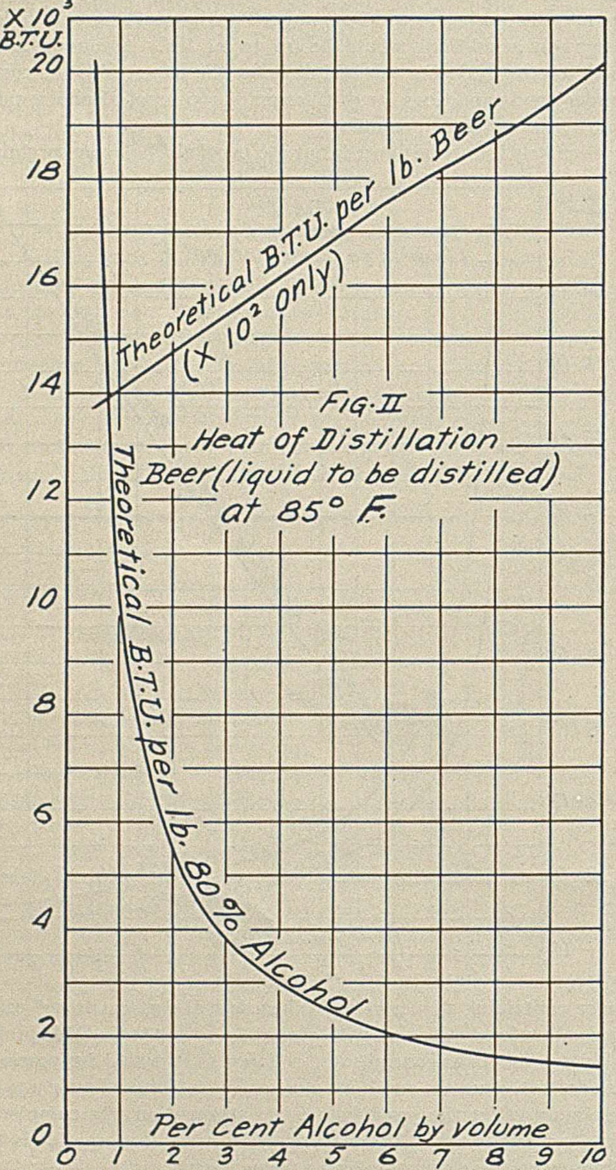


Fig. II
Heat of Distillation
Beer (liquid to be distilled)
at 85° F.

Coal, we can nearly always get, but unfortunately, the problem of cooling is just as pressing. Artificial refrigeration has never been used in distilleries in the United States because of its cost, most reliance being placed on water cooling. For plants which can obtain artesian water or lake water, or plants on the ocean, such a problem is not difficult, but for any proposed inland distillery too much consideration cannot be given to the matter. The water required for condensing a gallon of 80 or 95 per cent alcohol and cooling to 80° F. is theoretically, as in Table I, assuming perfect efficiency. In practice, the amount found thus should be raised to the 3/2 power; this relation is based on experiments made by the writer in distilleries.

TABLE I—GALLONS WATER REQUIRED TO CONDENSE ONE GALLON ALCOHOL AND COOL TO 85° F.

Temperature of Cooling Water:	55	60	65	70	75	80° F.
GALLONS WATER FOR ALCOHOL:						
80 Per cent by Volume..........	4.3	4.5	4.7	4.9	5.1	5.4
95 Per cent by Volume..........	3.4	3.6	3.7	3.9	4.2	4.3

It can be seen that a plant producing 100 gallons 80 per cent alcohol per day will require from 900 gallons water at 55° to 1,250 gallons at 80°, though probably the last figure is too low.

If the mash is heated considerably, more water will be required as is evident by Table II. It is not possible to correlate these figures with practice, unfortunately, as owing to the extreme viscosity of highly concentrated mashes, the factor is much greater here than that calculated as above, and owing to the more efficient apparatus used, the factor for thin mashes much less. At any rate, the figures given are minimal, though a safe margin would be five times the figures given.

MAXIMUM RAW MATERIAL COSTS

Assuming a concentration of 10 per cent fermentable in the liquid as finally prepared for fermentation, Table III represents about the maximum allowable cost per lb. fermentable for producing alcohol of any given cost per proof gallon, assuming an 85 per cent yield. Any feeding value of the residue should be added allowable to raw material cost. As this table is based on an overhead of 2 cents per proof gallon, which is the minimum possible under the most favorable conditions, the figures for raw material cost give the maximum allowable value.

TABLE II—GALLONS WATER PER GALLON 160 PROOF ALCOHOL FOR COOLING MASH TO 70° F.

Per cent Sugar in mash	Water at 55° F.	60° F.	65° F.	70° F.
1	112.5	116.2	120.2	124.4
2	56.3	58.1	60.1	62.2
3	37.5	38.7	40.1	41.5
4	28.1	29.1	30.1	31.1
5	22.5	23.2	24.0	24.9
6	18.8	19.4	20.1	20.8
7	16.1	16.6	17.2	17.8
8	14.1	14.6	15.0	15.6
9	12.5	12.9	13.4	13.8
10	11.3	11.6	12.0	12.4
11	10.2	10.5	10.9	13.4
12	9.4	9.7	10.0	10.4
15	7.5	7.7	8.0	8.3

TABLE III—MAXIMUM ALLOWABLE COSTS FERMENTABLE

COST FERMENTABLE per lb. Hexose Delivered	MINIMUM COST ALCOHOL per Proof Gallon
1.0 cent	9.6 cents
1.2	11.2
1.4	12.6
1.6	14.2
1.8	15.7
2.0	17.2
2.2	18.7
2.4	20.2
2.6	21.8
2.8	23.3
3.0	24.4

Let us suppose that we wished to compete with gasoline at 30 cents. Assuming that absolute alcohol and gasoline deliver the same amount of power per gallon, we must take the cost of alcohol at 15 cents per proof gallon. The raw material must, therefore, cost not over 1.7 cents per lb. fermentable. Alcohol potatoes contain 20 per cent starch. This is the equivalent of 22.2 per cent dextrose. We have the relation 0.222 × 1.7 × 0.38 cent as the maximum allowable cost per lb. of potatoes delivered, provided the feed is not utilized. The feeding value of the residue of 1 lb. of potatoes is generally taken at about 0.1 cent. This would make the maximum allowable cost 0.48 cent or 28.8 cents per bushel delivered.

At present gasoline is selling about 20 cents per gallon. If the Utopian ideas of certain automobile manufacturers were realized, potatoes would have to be laid down at the distillery for not to exceed 20 cents per bushel in order that alcohol might replace this gasoline. The average farm value for potatoes from 1907 to 1915 inclusive was 61.4 cents per bu. It is hard to see how the farmer could be stimulated to produce more potatoes if a lower price were offered as must be if potatoes are to figure as alcohol producers. The popular mind has become so obsessed with the idea of cheap alcohol from potatoes that it is forgotten that the German idea of cheap alcohol is alcohol at about 40 cents per gallon 180 proof.

CONCLUSIONS

Water, fuel and cost of raw material represent about all the factors susceptible of approximate calculation. In addition labor, insurance and depreciation must be taken into account. If starchy substances are used, malt or acids must be used for conversion which is an additional cost. It cannot be too strongly impressed on the prospective manufacturer that sporadic sources of material such as fruit, cornstalks, canning wastes, must always be used under the disadvantage of long periods of idleness resulting in increased depreciation, and disorganization of the working force.

It must not be understood that the writer intends to depreciate the possibility of producing alcohol at a cost to enable it to be used as a motor fuel. Quite the contrary. But it has seemed to him that unless more care is used in experiments on the production of alcohol that capital will abandon the field as unpromising. The newspapers are full of stories about the possibilities of production of cheap alcohol and even our own JOURNAL has not been entirely free from them. As a matter of fact, the production of cheap alcohol presents as many difficulties as the manufacture of cheap gasoline. The alcohol industry is one that requires experience as much as any other chemical industry and in spite of opinions to the contrary, uses at present about as scientific methods as any.

AUTHOR'S NOTE (Received May 16, 1917): Owing to recent increases in grain costs, it is probable that grain alcohol costs will be over fifty cents per proof gallon for the year 1917.

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WILLARD GIBBS MEDAL AWARD

The Willard Gibbs Medal for the year 1917 was conferred upon Dr. Edward Williams Morley, former Professor Emeritus in Chemistry, Western Reserve University, Cleveland, Ohio, at the meeting of the Chicago Section of the American Chemical Society, held May 18, 1917, in the Louis XVI Room of the Hotel Sherman. One hundred and forty-six members and friends of the Chicago Section were present.

Mr. A. V. H. Mory, Chairman of the Chicago Section, opened the meeting and introduced Dr. W. A. Noyes, Professor of Chemistry, University of Illinois, and Editor of the *Journal of the American Chemical Society*, who made the presentation address. The medal address, entitled "Early Researches in Hydrogen and Oxygen," will appear in full in the *Journal of the American Chemical Society*, and we are printing below an abstract prepared by Dr. Morley at our request.

Dr. Morley spoke in his usual happy vein. He showed the distinguishing quality of his mind—that keen sense of proportion that made possible his great work, by fully appreciating the sort of treatment of his subject that would be most acceptable to a mixed audience, including many who had no knowledge of chemistry. There was a personal touch to Dr. Morley's talk that was much enjoyed and he skilfully employed an occasional narrative to drive the points home.

Letters of regret were received from a number of invited guests, including the presidents of the Middle West universities, and a number of the prominent chemists of the country, among them Secretary Charles L. Parsons.

At the speakers' table, besides the chairman, Professor Morley, and Professor Noyes, were President Stieglitz; Harry Pratt Judson, President of the University of Chicago; F. I. Moulton, President of the City Club of Chicago; John H. Long, Professor in Chemistry at the Northwestern University Medical College; William H. Burton, Vice-President of the Standard Oil Company of Indiana; and Edward Bartow, Director of the Illinois Water Survey, each of whom spoke briefly and entertainingly. President Stieglitz was able to give some interesting information concerning the work of our society in these stirring times.

The occasion was a memorable one and established even more completely, if it were possible, the high character of the Willard Gibbs Award.

Edward Williams Morley was born in Newark, N. J., in 1838. He received the degrees of A.B. (1860) and A.M. (1863) from Williams College, and an honorary Ph.D. from University of Wooster (1878). He has also received the honorary LL.D. degree from Adelbert College (1891), Williams College (1901), Lafayette College (1907), University of Pittsburgh (1915), and the Sc.D. degree from Yale (1909). Dr. Morley was professor of chemistry from 1869 to 1906 at Western Reserve College, formerly at Hudson, Ohio, and afterwards removed to Cleveland. He also held the chair of professor of chemistry in the Cleveland Medical College from 1873 to 1888. Dr. Morley is an honorary member of the Royal Institute of London; corresponding member of the British Association for the Advancement of Science; associate fellow of the American Academy of Arts and Sciences; fellow of the American Association for the Advancement of Science, of which he was president, 1895–1896; a member of the National Academy of Sciences, American Philosophical Society, Astronomical and Astrophysical Society of America, the Chemical Society of London, and the American Chemical Society, of which he was president, 1899–1900.

Dr. Morley was honorary president of the Eighth International Congress of Applied Chemistry which met in New York City in September, 1912. The photograph of which a copy is shown here was made at about that time.

The *Chicago Chemical Bulletin* furnished us with the following brief account of Dr. Morley's most important work:

"The attention of chemists was first attracted by his work on a very accurate series of analyses of air which demonstrated that the per cent of oxygen in the air varies between narrow limits and is probably smaller in the higher strata of the atmosphere.

"In 1842 Dumas had made a series of determinations of the composition of water, on the basis of which the value 15.96 was accepted as the atomic weight of oxygen for nearly fifty years. Toward the end of this period a number of different chemists worked upon the problem and it gradually became clear that the value found by Dumas was in serious error, but it was not until Dr. Morley's paper appeared in 1895 that the chemists of the world accepted a quite different value as fully demonstrated. In an elaborate series of investigations, to which he gave more than ten of the best years of his life, the densities of hydrogen and of oxygen gases were determined with an accuracy that has not been excelled or even reached by any other observer. Dr. Morley also effected for the first time a complete quantitative synthesis of water, weighing both the oxygen and hydrogen and also the water formed by their union. The results of all of his studies gave the value 15.879 as the atomic weight of oxygen, or 1.00762 as the atomic weight of hydrogen, and it seems quite certain that these values will never be essentially changed.

"In the domain of physics the Michelson-Morley experiments, upon the transmission of light in the direction of the motion of the earth in space and at right angles to that motion, have been the basis of the modern theory of relativity and have gone far to undermine the confidence of physicists in the existence of the ether.

"Dr. Morley is not only one of the greatest of American chemists, but he is a man whose rare personal qualities have won for him a wide circle of friends."

INTRODUCTORY ADDRESS

By A. V. H. Mory

When William A. Converse, on retiring from the chairmanship and active work in the local section of the American Chemical Society, donated a sum to be used in founding a medal in the name of the Chicago Section for the encouragement of local talent, he builded better than he knew. Certain it is, his native modesty would have deterred him from presuming to found a medal of such dignity and importance as the Willard Gibbs Medal has come to be.

The committee appointed to draw up rules governing the award saw so many difficulties in the way of a local award by a local jury that it broadened the scope of the award. When the now honored president of our national society, Dr. Stieglitz, then chairman of the Medal Committee, proposed that the award bear the name of that most illustrious of American chemists, J. Willard Gibbs, the modest proposal of Mr. Converse then and there took on national importance.

A number of the most prominent chemists of the country were later invited to sit on the jury of award and the distinguished Swedish chemist, Arrhenius, was the first of those recognized in the bestowal of the Willard Gibbs Medal.

That there has been no lowering of the high standard thus set up is attested by the list of illustrious medalists that have followed—Theodore William Richards, Leo H. Baekeland, Ira Remsen, Arthur Amos Noyes, Willis Rodney Whitney, and last of all, our honored guest of the evening—Edward Williams Morley.

It goes without saying that the Chicago Section stands committed to a policy that insures the continued high character and dignity of the Willard Gibbs Medal. And it may be added that we, of the Chicago Section, including the founder of the medal, shall ever remain content to render such humble service as may be our privilege in simply providing, as it were, the machinery for bestowing the honors that the eminent jury shall elect to award.

PRESENTATION ADDRESS

By William A. Noyes

There are a considerable number of fundamental physical constants which lie at the very foundation of all accurate scientific work. Among these the density of the common gases and the relative weights of the atoms of the elements have occupied a very prominent place in the attention of chemists. The earlier determinations of these constants were made by crude and inaccurate methods and the values obtained were displaced by other, better values within a few years. Literally thousands of such determinations have been made, but the large majority, although they usually represent the work of many months, have been displaced by others within a few years or, at most, within a generation. Regnault in Paris in 1845 determined the densities of hydrogen and oxygen with such care that his work stood for a generation without serious question. About the same time Dumas, also working in Paris, determined the ratio of the atomic weights of hydrogen and oxygen and his result also stood for a generation, although we now know that it did not deserve the confidence which it received. Dumas himself considered that the value he obtained was in error, possibly as far as one part in 200. The fact that Regnault did not think of the compressing effect of the pressure of a ton or more on the outside of his empty globe, introduced an error in his value for the density of hydrogen which was almost identical with the error of Dumas's determination of the oxygen-hydrogen ratio. This accidental agreement of two results, each of which was affected by a serious but different source of error gave chemists a very undue confidence and they allowed them to stand without serious question for forty years. After that a number of chemists and physicists gave to the world much better values for both constants. But Professor Morley was not content to give to the world values of the ordinary sort which might stand for a few years and then be displaced by others. He was willing to study, one after the other, and with almost an infinite patience, the sources of error in the determinations, and then he carried out his work with such an ingenuity in the execution of details, and with such accuracy, that at present there is no indication that the work will need to be repeated for a century.

I am glad to express in this way the estimate which the world places on the work of Professor Morley, but there is also a keen personal pleasure in being permitted to make this presentation. I have known Professor Morley for twenty-five years, and with the years there has grown an intimate personal friendship which has become one of the most inspiring influences in my life. Professor Morley is a merciless critic—of himself. He has very little to say about the shortcomings of others, though few see more clearly what some of those shortcomings are. And so I take the greatest pleasure in presenting this medal to a personal friend, to a man who has done work of the very highest quality and to one who has given to the world values for two of our most fundamental constants which will always stand as very close indeed to the truth.

EDWARD WILLIAMS MORLEY
WILLARD GIBBS MEDALIST, 1917

EARLY RESEARCHES IN HYDROGEN AND OXYGEN MEDAL ADDRESS[1]

By Edward Williams Morley

The chemical principles involved in the synthesis of weighed quantities of water from weighed quantities of hydrogen and oxygen require no description; it is only the details of the manipulation by which errors were minimized which are interesting.

Pure hydrogen cannot easily be prepared by solution of a metal

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[1] Author's abstract.

in acid. Carbon and carbon compounds derived from metallurgical operations introduce impurities which cannot be removed. If we remove carbon and carbon compounds, as by distillation of zinc in a vacuum, it is difficult to utilize the hydrogen produced in a given generating vessel to sweep out the nitrogen previously included and still have sufficient gas for the destined use. Electrolysis is accordingly a more convenient source. Electrolysis of caustic alkali would be convenient except that the presence of carbonate introduces impurity which cannot be removed. Dilute sulfuric acid is therefore to be preferred; it gives a gas which, of course, contains nitrogen; it contains vapor of water, and sulfur trioxide; it contains oxygen, ozone and vapor of hydrogen dioxide, diffused through the liquid from the other pole; it also contains sulfur dioxide and hydrogen sulfide. Prolonged evolution finally sweeps out nitrogen. Red-hot copper removes oxygen, ozone and hydrogen dioxide and converts all sulfur compounds into copper sulfide or hydrogen sulfide, which is easily removed by absorption in caustic alkali. Lastly, water is removed by phosphorus pentoxide.

Absence of nitrogen was proved by the absorption of a liter of the gas by means of hot copper, with eudiometric analysis of the one per cent of residue. It was proved that at last less than one part of nitrogen remained in two hundred thousand parts of hydrogen. But in the syntheses, the gas was next absorbed in palladium, the palladium tube was freed from accompanying nitrogen by a prolonged current after absorption had ceased, and this doubly purified hydrogen was utilized.

The amount of moisture remaining in a gas after drying with phosphorus pentoxide was measured; it was given by the loss of weight of a special absorption tube, and was about one milligram in 40,000 liters. The amount of pentoxide taken up by the gas was also determined and found also to be some such quantity as a milligram in 40,000 liters. It is probable that the milligram lost by the absorption tube and at first regarded as moisture was actually pentoxide, and that the amount of moisture remaining after drying with pentoxide is far less than a milligram in 40,000 liters.

The hydrogen used in syntheses was free from carbon compounds and compounds of sulfur. This was proved by burning a slow current of the hydrogen in an atmosphere of the oxygen produced in the same electrolysis. The combustion took place in a closed vessel, so that solution and absorption of possible products of combustion must have been complete. When some 600 cc. of water had accumulated, the liquid was examined; it contained no carbon dioxide and no oxide of sulfur; it contained 0.02 milligram of phosphorus pentoxide.

It was interesting to notice that in this experiment, when the platinum jet had been heated to incandescence in an atmosphere of pure oxygen for some ten days, the inside of the flask was covered with a rust-colored oxide of platinum. When the combustion was stopped, the current of hydrogen was maintained, and the oxide was immediately converted into a deep black coating of metallic platinum.

AUTHOR'S NOTE—Many other details of manipulation are described in the complete address, but they are mostly already on record and need not be here repeated.

CURRENT INDUSTRIAL NEWS

EXTRACTIONS OF LOW-GRADE LEAD ORES

At the Salt Lake City, Utah, Experiment Station of the Bureau of Mines, Department of the Interior, Mr. A. E. Wells, metallurgist in charge, reported as follows to the Washington office of the bureau:

The most successful results of the metallurgical research branch during the month were with low-grade lead ores which have failed to yield to flotation or leaching processes described in previous reports. By mixing these ores with sodium chloride and heating to temperatures of about 800° C. high extractions can be obtained with the lead irrespective of the type of gangue in the ore. The lead volatilizes as a chloride and any silver or gold present is likewise volatilized. These chlorides are precipitated from the fume by the use of the Cottrell precipitator. The precipitated lead chloride after a mixture with lime and a small amount of reducing agent is heated to a red heat when a slag of calcium chloride and metallic lead is formed. The calcium chloride is an advantageous substitute for sodium chloride in the first operation, and from 50 to 75 per cent of the chlorine is recovered in this manner. This process will have the advantage of producing bullion from either oxides or sulfite ores in localities which do not contain sufficient water for milling purposes. Cost calculations indicate a considerable possibility of this process being cheaper than water concentration followed by smelting.

COAL IN CHINA

The *Board of Trade Journal* of December 21, states that a Hong-Kong contemporary had about that time published extracts from a report respecting the coal reserves of China, which had been drawn up recently by the Director of the Chinese Geological Survey. According to the report, coal seems to be very widely distributed in China. There is not a single province in which coal is not found. The northeastern provinces, Shansi, Chihli, Shantung and Honan are, undoubtedly, the most important, the first named being the richest of all. Both Inner Mongolia and Manchuria are fairly well supplied with coal. The northwestern and southeastern regions are, however, much poorer, the geological formation being unfavorable. Both bituminous and anthracite coal occur in China, the latter being more extensive, as the coal in Shansi and Honan is largely anthracite. In fact, the latter variety has been more used in China, as it can be burned in open stoves without chimneys. The bituminous coal industry has acquired increasing importance in recent years as the coal now worked on a large scale is mostly of this variety and, out of twenty mines worked by machinery, only three are producing anthracite. The production of coal of all kinds in China in 1913 was about 15,000,000 metric tons (1 metric ton = 2,204.6 lbs.).—A. McMILLAN.

FRENCH RESINOUS PRODUCTS

According to a report in the *Oil and Color Trade Journal*, 51 (1917), 975, the output in the resin market for the coming year is very satisfactory. The Landes depots and warehouses report stocks of spirit of turpentine amounting to 3,000,000 kgs., to which there must be added 1,041,391 kgs. in Bordeaux warehouses, making a grand total of 4,000 tons. Half of this total has already been sold so that only 2,000 tons are available to keep things going until the new season has opened. Hence prices are very firm at present and it is probable that a rise in price will take place and the present month may close with rates at $45 to $50 per 100 kilos. For dried sorts, there is a pressing demand but very scant stocks. Lots on hand do not, probably, exceed 20,000 barrels of pitch and colophonium. Prices are expected to advance from $15 to $16 delivered Landes stations. The market is greatly favored by the fact that sharp advances will probably occur in America due to higher freights and insurance rates and to shipping dangers. Arrivals are also getting scarcer in England, and London will probably be willing to replace American with French resins.—M.

KAPOK FIBER

According to an article by Messrs. C. F. Cross and F. J. Bevan in the *Journal of the Society of Dyes*, 32, 274, true kapok is the seed-hair of *Eriodendron anfractnosum*, though other vegetable downs pass commercially as kapok. The chief use of the material is for stuffing lifebelts, etc., and the fiber keeps out of the water, not because it contains much water-repelling oils or resins (these are found up to 1.8 per cent), but because the fibers form thin-walled tubes filled with air. The fibers are remarkably uniform as regards diameter (0.021 to 0.028 mm.). The air is not expelled by immersion of the fiber in hydrocarbons. Sinking tests of kapok in water take very long times but, in aqueous alcohol of density 0.928, tests as to grade can be carried out expeditiously. In life-saving appliances 1 g. of kapok occupies about 15 cc., and a jacket containing 700 g. of this stuffing would have a floating power of 10.5 kg. After having been suspended in water for 72 hrs. with a weight of 9 kg. attached, the jacket still required 1.3 kg. more to submerge it and after 192 hrs., 0.9 kg. were required to sink the jacket. The references here mentioned are to tests carried out on kapok in the National Physical Laboratory, London, before the war.—M.

BRITISH MINERAL OUTPUT FOR 1915

The following table, taken from the annual report on mines and quarries for the year 1915, gives the production of the various minerals:

	1914		1915	
MINERAL	Quantity (Tons)	Value £	Quantity (Tons)	Value £
Alum Shale	6,078	607	7,911	791
Antimony Ore	....	....	$2^1/_2$	59
Arsenical Pyrites	....	....	421	233
Arsenic	1,988	19,052	2,496	32,779
Barium Compounds	48,930	43,506	62,477	79,829
Bauxite	8,286	2,159	11,723	3,163
Bog Ore	2,342	585	1,986	496
Chalk	4,291,170	197,154	3,233,897	155,560
Chert, Flint	76,213	13,304	102,698	15,536
Chromite	100	50	....	....
Clay and Slate	13,124,361	1,731,779	8,871,821	1,172,877
Coal	265,664,393	132,596,853	253,206,081	157,830,670
Copper Ore	2,373	16,985	579	3,084
Copper Precipitate	185	5,529	243	9,938
Fluorspar	33,816	11,005	33,123	11,484
Gold Ore	47	318	5,086	3,389
Gravel and Sand	2,498,872	215,351	2,350,267	213,373
Gypsum	265,365	83,268	247,229	78,747
Igneous Rocks	7,135,243	1,369,242	6,085,415	1,200,212
Iron Ore	14,867,582	3,921,683	14,235,012	4,587,651
Iron Pyrites	11,654	4,759	10,535	4,873
Lead Ore	26,013	309,813	20,744	295,071
Lignite	300	150	1,783	536
Limestone	12,158,441	1,295,512	11,115,909	1,306,268
Manganese Ore	3,437	2,931	4,640	4,640
Natural Gas	87,000 c. ft.	Not stated	87,00 c. ft.	Not stated
Ochre, Umber	11,069	10,635	8,989	9,641
Oil Shale	3,268,666	837,249	2,998,652	836,393
Salt	2,069,989	560,893	2,005,605	607,251
Sandstone	3,464,528	1,057,096	2,520,856	758,325
Slate	318,912	806,196	226,037	452,819
Soapstone	180	90	850	575
Sulfate of Strontium	13,157	10,439	640	688
Tin Ore	8,085	661,865	8,144	668,609
Tungsten Ore	205	19,722	331	44,803
Uranium Ore	344	....	82	....
Zinc Ore	15,419	56,652	12,057	70,383
TOTALS		£145,863,332		£170,460,949

—M.

FOOD VALUE OF THE SOYA BEAN

In an article in *Compt. rend.*, 164 (1917), 300, the use of the soya bean as an article of food is strongly recommended. The author says that its food value has long been known in the Far East. It forms an important article of diet in the French Indo-Chinese colonies, in Cochin-China, China and Japan. The oil-content of soya beans is no less than 20 per cent and the protein content 40 per cent, whereas French haricot beans contain only 20 per cent of protein and 2 per cent of fat. The author further suggests that the soya bean should be cultivated in France. The nutritive value of the soya bean meal has led to its being used, in some degree, in military rations in the French army and its use for civilian purposes deserves wider application.—M.

SULFUR SUPPLY IN GERMANY

Before the war, says the *Iron and Coal Trade Review*, the Germans obtained annually about 900,000 tons of iron pyrites from Spain for the manufacture of sulfuric acid and they, themselves, produced about 300,000 tons every year at Megen in Silesia. In 1912 and 1913, however, they increased their purchases from Spain to 1,200,000 tons per annum. Owing to the stoppage of the Spanish supplies by the war, they had to look about for other sources and have, no doubt, found some in Poland and also in Greece and Turkey. Besides this, Germany has received about 400,000 tons of pyrites annually from Norway. The eminent French chemist, M. Cazeneuve, states that the Germans have succeeded in producing anhydride from plaster of Paris with the help of the electric furnace. It is also reported that a process is being carried on in Germany which consists in treating plaster of Paris with sand and producing silicate of lime and cement at the same time as sulfurous acid. The Norwegian supply of pyrites is not now available to Germany.—M.

ASBESTOS PACKING

According to A. Dubose in the *Journal de Caoutchouc et Guttapercha*, attempts have been made with partial success to imitate klingerite, the asbestos preparation of German manufacture, which found favor prior to the war as an excellent packing for steam pipe joints. A sample of the product, 3.75 mm. in thickness, was found to consist of 15 layers of a thin asbestos felting, built up of the best long fibers mixed with 2 per cent of flax threads. Such layer was coated on both sides with some agglutinant which contained sulfur; this sulfur was to be slowly vulcanized, apparently while the material was being used, in order that it might become more resistant and impervious. An analysis of the sample gave: 80.5 per cent of asbestos, 2.2 per cent of cellulose (flax), and 17 per cent agglutinant (consisting of rubber and balata with a small percentage of sulfur, less than 0.5). The incorporation of the flax would render the packing stronger, more pliable, and increase the cohesion of the fine layers.—M.

CEDARWOOD OIL

We have received, says *Nature*, from Messrs. Flatters and Garnett, Ltd., Manchester, England, a specimen of their cedarwood oil for use with oil-immersion microscopic objectives. As the result of tests, the oil was found to be of good consistence and color, does not become cloudy in cold weather and has a high refractive index. The refractive index of cedarwood oil is stated to be 1.510, but that of the specimen examined was well above this, *viz.*, 1.518. Hitherto, Great Britain has been supplied with immersion oil from the Continent, but this is a British-made oil which seems to fulfil every requirement. It is supplied in bottles at from $0.18 to $1.00 each, or in bulk.—M.

EXPORTATION OF JAPANESE CHEMICALS

According to a report in the *Chemical Trade Journal*, 60 (1917), 257, an order has been issued by the Japanese Minister for the Interior granting permission to export the following goods from Japan:

Acetate of lead
Ammonia water
Ammonium carbonate
Aspirin
Bicarbonate of potash
Bicarbonate of soda
Boracic acid
Bromine and salts
Caffein
Camphor monobromatum
Castor oil
Cinchona bark
Citric acid
Citrate of iron and ammonium
Gum arabic
Hydrogen peroxide and preparations therefrom
Lactic acid
Lysol
Milk sugar (lactose)
Oil of turpentine
Potassium sulfate
Saccharine
Sulfur
Tannic acid
Tartaric acid
Urotropin
Vaseline
White wax
Yellow wax

—M.

BRITISH BOARD OF TRADE

During the months February 15 to April 15, the British Board of Trade received inquiries from firms in the United Kingdom and abroad regarding sources of supply for the following articles. Firms which may be able to supply information regarding these things are requested to communicate with the Director of the Commercial Intelligence Branch, Board of Trade, 73 Basinghall St., London, E. C.

- Animal traps for bears and pumas
- Astrachan
- Beech boards for brushes
- Bellows suitable for use in damp places
- Bleached white mercerized sewing yarn
- Canvas
- CHEMICALS: Alphanaphthylamine
  - Aluminum sulfate
  - Arsenic acid (sp. gr. 1.8)
  - Formaldehyde (40 per cent)
  - Dimethyl sulfate
  - Florida earth for bleaching oils
  - Carbonate of copper (commercial)
  - Carbonate of zinc
  - Selenium powder
  - Sodium perborate
  - Tin oxide
  - Ultramarine blue
- Combs, white celluloid
- Crepe paper
- Dyes, blue and mauve for ink making
- Enameled tea kettles
- Eyelet rivets
- Fishing nets for sardine fishing
- Fishing twine
- Glass, hard, heat-resisting
- Gramophone needles
- Hand cleanser, suitable for motorists, mechanics
- Handkerchiefs, colored cotton, for African native trade
- Horseshoe nails
- Iron or steel moulds for casting lead name-plates, numbers, etc.
- Knitted fabric, for gas mantles
- Leather boot laces
- Leather tabs for metal key chains
- MACHINERY AND PLANT FOR:
  - Cane plating
  - Converting mica into micanite and mica cloth
  - Cutting cartons for cardboard box making
  - Decorticating ground nuts
  - Dry-cleaning garments
  - Embossing, printing and cutting out parcel seals
  - Hand-stapling, similar to Hotchkiss machines
  - Making India-rubber stamps
  - Making macaroni
  - Making paper spills
  - Making photographic and other lenses
  - Making rice-starch (for delivery after the war)
  - Making sheet-nets
  - Paper baling machines
  - Printing tickets from the reel
  - Printing, in one or two colors, and punching labels at same time, with or without embossing attachment
  - Stitching the corners of boxes, similar to Brehmer machine
  - Stuffing dolls' bodies
- Micrometers
- Mirrors, celluloid
- Note-books, cheap
- Stearine (240 tons)
- Strawboard, millboard and substitutes therefor
- Vacuum chambers for vacuum flasks
- Wax-tapers
- Wood-moulds for buttons, large oval

—M.

DEARTH OF RAW MATERIAL IN SCANDINAVIA

According to *Engineering*, 103 (1917), 175, the scarcity of raw materials is becoming more and more embarrassing in neutral countries, not only are the prices in many cases rapidly approaching an absolutely prohibitive limit, but many industries are on the verge of stopping or have done so. In this connection iron and coal play an important part. Some Danish cement works have had to stop, municipal power stations and gasworks are short of material and some large Swedish enameled-ware works have had to close down. In Denmark, Sweden and Norway, the telephone centrals are at their wits' end, being entirely unable to satisfy their new subscribers who have to wait for months before they can have their telephones installed. The makers are simply unable to get supplies of raw material. In Denmark the benzene supply is quite exhausted.—M.

JAVA CITRONELLA OIL

The exports of citronella oil from Java, says the *Oil and Color Trade Journal*, 51 (1917), 589, are now stated to have increased materially in 1915 over those in 1914 and a further increase was expected for 1916. The returns, so far available, show that the amount exported during the first six months of 1916 was 229,936 kilos, which, on an average of over 3 florins ($1.50) per kilo, will probably cause the year's export to exceed $500,000. The exports have gone to France, Great Britain, Japan and the United States. Owing to the demand, the area of the grass plantations has risen to 5,500 bouws, and, in view of this increase in cultivation, it is possible there may be a fall in price of the oil. It is probable that Japan may take more, but this extra demand will not compensate for the falling off in other directions. Attempts to increase the Australian trade have not been very successful, as that country appears to be quite satisfied with Ceylon oil.—M.

DYE FROM RICE LEAVES

The discovery of a process of obtaining dye from the leaves of the rice plant is attributed to Mr. Shigeo Kumagiri, of the Japanese Department of Agriculture and Commerce. He claims to be able to produce a dark purple dye by a chemical treatment of the grass of this plant. When treated with acids, the new dye turns red. Experiments have shown that it can be used with success for various purposes in laboratories and also for coloring materials. In the opinion of a Japanese paper, the new discovery is not likely to prove of very great use as the leaves of the rice plant have to be gathered before harvesting. The production of the dye from such a source is, however, interesting.—M.

NORWEGIAN WHALE OIL PRODUCTION

Norway's production of whale oil during 1916, says the *Anglo-Norwegian Trade Journal*, was less than it has been for many years owing to several of the whalers being engaged in ordinary trade and to the difficulties connected with the fishing. The total production during the year amounted to 367,400 bbls. as against 475,000 bbls. in 1915, 575,000 in 1914, and 600,000 in 1913. It is necessary to go back to 1910 to find such a small production of oil. The catch at the South Shetlands aggregated last year 195,000 bbls., and at South Georgia 136,700 bbls., while the fisheries at Australia, Africa, the Faroe Islands and Alaska, only yielded 34,800 bbls. The world's production of whale oil in 1916 amounted to 634,500 bbls., of which 216,000 bbls. falls to the South Shetlands, 319,400 bbls. to South Georgia, 40,000 bbls. to Africa, 13,000 bbls. to Australia and 18,000 to Alaska. During 1915 the world's production was 630,000 bbls., during 1914 735,000 bbls. and during 1913 775,000 bbls. Compared with the world's production, the Norwegian production during 1916 was 58 per cent, during 1915 about 75 per cent, and during 1914 and 1913 about 78 and 77 per cent, respectively.—M.

FRENCH TUNGSTEN

The tungsten requirements of the French industries for 1917, says *Engineer*, are considerable, and the question of wolfram production in France has been taken up by the Government. An official has been specially appointed to keep in touch with producing companies, and formal assurance regarding the supply of labor has been given. An arrangement has been arrived at between the Vaubry et Cieux Co. and the Société du Giffre, an important tungsten producer for the more energetic exploitation of the concession. The deposits in the Valley of Cieux are said to contain tin and gold as well as wolfram. Considerable reserves of ore have been disclosed and, at the present time, the production is about 10 tons wolfram per month. It is hoped that by the installation of new plants the production will be increased to 30 tons per month. Other wolfram deposits have been worked in the department of Ille-et-Vilaine and in the Haute-Vienne. The Puy-les-Vignes Mine in the latter district was the only French producing mine in 1912.—M.

DETECTION OF COCONUT OIL

At the annual meeting of Public Analysts held recently in London, Mr. G. D. Edsdon read a paper on the above subject. The author had studied the method of Shrewsburg and Knapp (*Analyst*, 35 (1910), 385) for the determination of coconut oil in mixtures, and also the modification suggested by Revis and Bolton (*Analyst*, 36 (1911), 334), but it had not been found possible to obtain concordant results by either method. In the present paper, the suggestions of Revis and Bolton are further modified by the use of alcohol of sp. gr. 0.9200 and by thoroughly drying the cake of fatty acids before the solution in alcohol. Experimental evidence is given in support of these changes and the modified process seems to give excellent results.—M.

ITALIAN OLIVE OIL PRODUCTION

According to the figures issued by the International Institute of Agriculture at Rome, the area of olive trees under cultivation in Italy in 1916 was 5,703,169 acres. This is only slightly less than the acreage of 1915 which was 5,704,158. The five years' average, 1909–1913, was 5,744,912 acres. The olive oil yield is estimated at 374,786,000 pounds in 1916 as against 300,401,802 in 1915 and 359,771,837 for the 1909–1913 average. The heavy rain and wind storms have injured the olive trees in some localities where the fruit has prematurely fallen.—M.

FRENCH BAUXITE

According to a report by the Secretary of the British Chamber of Commerce for the French Riviera, there exist rich deposits of bauxite in the territory behind the Riviera. Before the outbreak of war, these deposits were for the most part exploited by Germans and the yearly output is said to have amounted to 250,000 tons. A part of this was shipped direct to Germany, but most of it was transported by rail to Switzerland, where, it is understood, a German-owned plant had been erected for the extraction of aluminum by electrolysis. On the outbreak of war, the enemy mines were sequestrated. In the neighborhood of the mines, sites are available for the erection of an electrolytic plant for refining the ore, and there exists an abundant water supply capable of providing the necessary power for such plant. In ordinary times, labor is plentiful and there is thus every facility for treating the ore on the spot. The ore could be delivered at Monaco, at which port wagons can be brought alongside the steamers at the quays.—M.

JAPANESE EXPORT TRADE

According to a report in the *Chemical Trade Journal*, **60** (1917), 158, the Japanese Government is evidently sincere in its efforts to develop and maintain the export trade of the country. It has been announced that the Department of Agriculture and Commerce is considering a measure for the improvement of the quality of the export goods with a view to retaining, at the close of hostilities, the oversea markets acquired by Japan during the war. Complaints, it is said, are too often heard of the poor quality of the goods Japan, at present, exports; so much so that it is generally feared that the trade, acquired during the war, may be eventually lost. The poor quality of certain Japanese goods is attributed to the backwardness of Japan's manufacturing and trading systems. It has been decided by the government to enforce thorough conditioning of all export goods and the particulars of a measure to be adopted are being considered. According to the official plan, the Association of Manufacturers, or their federations established in accordance with the law, will be empowered to condition all export goods on a compulsory system, and the government will exercise general supervision over the organization of conditioning houses and the actual management of business, at the same time giving bounties to these associations.—M.

ACETYLENE GENERATOR

An automatic acetylene generator, of which particulars are published by Messrs. Perkin and Co., Whitehall Road, Leeds, England, is provided with two generating chambers. Only one of these is in operation at any given time, but, when it is exhausted, the other is automatically brought into action. The first can then be recharged and set to come into operation without further attention when the carbide in the second has been used up, and the supply of gas is thus continuous. All the working parts are on the outside and, as the acetylene is automatically washed, scrubbed and cooled by the generator, purifiers are unnecessary. Carbide of any size, and not merely small pieces can be used for charging.—M.

NEW ELECTRIC FURNACE

Considerable numbers of a new electric furnace made by Messrs. T. H. Watson and Co., of Sheffield, are now at work in Great Britain, says the *Times Engineering Supplement* No. **509**, 70. In it three-phase current is employed, and three single-phase transformers are connected with meshed primaries. The secondary windings are connected to an unequal star, from which two connections go to the top vertical electrodes of carbon, while the third is taken to the furnace hearth, which is made of a conducting mixture of dolomite and magnesia, and forms the third electrode. The proportions of the unequal star are so calculated as to give a balance on the primary supply phases when the upper electrodes are in equal adjustment. Thus, if one arc is broken, the others are not affected, and any current overload passing through any arc must traverse two transformers in series and in different phase thus giving a considerable buffer effect and tending towards the reduction of shock and the maintenance of a steady load. The electro-magnetic effects arising from the arrangement of the electrodes keep the bath of molten metal in continual circulation, the steel being thus uniformly heated and the intense heat of the arcs constantly absorbed. The hearth is always at least 20 in. thick and is not penetrated by water-cooled studs. These furnaces were originally designed to meet the requirements of the Sheffield steel industry, especially in connection with high-speed and high-grade alloy steels; one of them has already made over 950 heats of high-speed steel, and, though it was only designed to melt charges in three hours, it regularly turns out five charges in 12 hours. Its lining did not require renewal until after it had made 480 heats. Several standard sizes are made, rated at 10 cwt., 30 cwt., 3 tons and upwards. The electrical equipment for the 10 cwt. size has a capacity of 260 k. v. a., for the 30 cwt. of 520 k. v. a., and for the 3-ton of 800 k. v. a. For a 6-ton furnace 1,560 k. v. a. is provided, and it is furnished with four top electrodes, instead of two, as in the case of the smaller sizes.—M.

PETROLEUM PRODUCTION IN JAPAN

According to the annual report, the production of crude oil in Japan was 3,015,327 barrels in 1915, an increase of 272,807 barrels over the preceding year. The following figures show the production by districts in 1915 and 1914:

	1915	1914
Echigo	1,975,443 barrels	2,009,603 barrels
Akita	1,008,863 barrels	706,588 barrels
Enshu	2,098 barrels	2,455 barrels
Yamagata	471 barrels	425 barrels
Nagano	195 barrels	148 barrels
Hokkaido	9,287 barrels	5,987 barrels
Taiwan (Formosa)	18,970 barrels	17,314 barrels
Total	3,015,327 barrels	2,747,520 barrels

—M.

RENDERING COAL SOLUBLE BY OZONE

An interesting discovery, says *Engineering*, **103** (1917), 296, has been made by Dr. Franz Fischer, who has found that if ozone is brought in contact with coal in a glass tube at ordinary temperature, the surface of the coal undergoes a change in so far as it presents a fine brownish bloom which is soluble in water yielding a brownish liquid which is slightly acid. This discovery led to a test in which 9.6 g. of coal were exposed to the influence of ozone for 133 hrs., when only 0.7 g. of the original coal was left; hence 92 per cent was dissolved. The discoverer draws the conclusion from this that the principal component part of the coal is a transformed product of a former cellulose substance which can be rendered soluble by contact with ozone. To judge from the attributes of this new substance, it is assumed that the greater bulk is an acid which has been formed by the primary formation of ozonites which were subsequently decomposed by water. This test was made with Westphalian coal. —M.

TRADE BETWEEN JAPAN AND SOUTH AFRICA

An extension of trade between Japan and South Africa appears, according to the *Times Trade Supplement*, likely to take place. Japan's purchasing power has increased during the war and several lines which can be supplied by South Africa with ease are in good demand in Japan. Some of these are asbestos, hides, aloes, wines, wattle bark and extract, and tobacco. The Japanese Consul in South Africa considers that there is a great future for export trade between the Union and Japan. An important new market will naturally supply a strong stimulus to production which it cannot be doubted will be capable of considerable extension after the war. Japan has already had dealings in some of the products of South Africa and the possibilities only need exploiting by properly organized methods. Japanese steamships have been attracted to South African ports in increasing numbers and there is every likelihood of Japanese shipowners interesting themselves in fostering trade between the two countries.—M.

TARPAULINS IN AUSTRALIA

A serious shortage of tarpaulins is reported in the Commonwealth of Australia—particularly Victoria—for covering grain when in transit on the railways. Not merely has it been found impossible to furnish coverings for the goods-trucks which have been added, but no renewals have been made to the stocks of tarpaulins which existed at the commencement of the war. The practice of the Railway Department has been to obtain the necessary canvas and itself complete the manufacture of the tarpaulins. Lately, however, supplies of any material at all suitable have been most difficult to obtain. Of a total quantity of 507,000 yds. of canvas which have been ordered by the Railway Department from different contractors since the outbreak of the war, only about 60,000 yds. had been delivered by the end of 1916. There seems little likelihood of any supplies of flax canvas becoming available yet for the purpose, but an inferior quality would be acceptable or, it is believed, any serviceable substitute which could be used to protect consignments of grain, bran and pollard.—M.

PETROLEUM PRODUCTION IN ARGENTINA

The production of petroleum in Argentina, which is monopolized by the Government, is now increasing very rapidly as will be seen from the returns of the last five years: 1911, 920 tons; 1912, 6,850 tons; 1913, 19,050 tons; 1914, 40,530 tons; 1915, 75,200 tons. The output for 1916 is provisionally estimated at 180,000 tons, which shows a large advance on last year. The director of the monopoly estimates that with an expenditure of three million dollars in increasing the number of wells, a production of 480,000 tons could be secured in 1918 and of 900,000 tons in 1920. The government has directed that the number of wells should be augmented upon the lines of this advice. The transport of petrol is effected by means of two tank steamers (capacity 4,000 tons) belonging to the government. Two other steamers are in course of construction.—M.

HIGH TENSILE *vs.* MILD STEEL FOR REINFORCED CONCRETE

In a paper read recently before the Society of Engineers in London, Mr. A. W. C. Shelf endeavored to show that, whereas it has been customary, at least in England, to use plain round mild steel bars for reinforced concrete, these are not the best for the purpose, but that greater efficiency and economy are obtained by physically developing mild steel bars in order to take out the first yield in the steel which is useless and has a detrimental effect on the concrete. When this first yield or stretch is taken out, a higher yield-point is obtained without any injury to the steel, so that it is safer to employ a stress of 20,000 lbs. per sq. in. (which results in a saving of 20 per cent in the weight of steel required) than it was to employ a stress of 16,000 lbs. per sq. in. before the steel was physically developed and, for this reason, the author of the paper is of the opinion that the existing regulation of the London County Council relating to the stress on steel should be altered to avoid the cramping of industrial progress.—M.

WATER-POWER IN NORWAY

According to a report in a contemporary, a special committee of the Norwegian Storthing has recommended that concessions for the acquisition of waterfalls shall not be granted to foreigners or foreign companies except in very special cases as when, for instance, a going concern is desirous of obtaining possession of or regulating a waterfall. For Norwegian citizens, a concession is to be necessary in cases of waterfalls capable of yielding more than 5,000 natural h. p. The capital of companies or syndicates wishing to secure such concessions must be exclusively Norwegian and they must be domiciled in Norway and have an entirely Norwegian board of directors. The duration of the concessions is to be limited to 60 years. The state is to be entitled to secure the waterfall at latest in the 40th year after granting the concession; its option of purchase applies to waterfalls above 5,000 h. p., while, for smaller ones, the option rests with the corporation or municipality. The amount of power to be ceded to the state and municipalities by the possessor of a concession is to be 5 per cent.—M.

SCIENTIFIC SOCIETIES

CALENDAR OF MEETINGS

American Leather Chemists' Association: Fourteenth Annual Meeting, Atlantic City, N. J., June 7 to 9, 1917.

North Carolina Cottonseed Crushers' Association: Annual Convention, Norfolk, Va., June 19 to 20, 1917.

American Institute of Chemical Engineers: Ninth Semi-Annual Meeting, Buffalo, N. Y., June 20 to 22, 1917.

First Industrial Exposition and Export Conference. Springfield, Mass., June 23 to 30, 1917.

American Society for Testing Materials: Atlantic City, N. J., June 26 to 30, 1917.

American Chemical Society: Annual Meeting, Boston, Mass., September 10 to 16, 1917.

National Exposition of Chemical Industries (Third): Grand Central Palace, New York City, September 24 to 29, 1917.

ANALYSIS OF SPELTER[1]

Supplementary Note from the
Committee on Analysis of Non-Ferrous Alloys
Division of Industrial Chemists and Chemical Engineers

As a result of recent investigation it seems advisable to amend the report on "Spelter Analysis" [THIS JOURNAL, 7 (1915), 547] by inserting the following as a foot-note after 55, second line, under the method for cadmium on p. 548.

"It is recommended that the acid be added slowly, 4 or 5 drops at a time; if added rapidly or all at once, standing over night may be an insufficient time for the acid to dissolve the proper amount of zinc, and small amounts of cadmium may remain in solution."

WM. B. PRICE,
Chairman, Committee on
Analysis of Non-Ferrous Alloys

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[1] Approved by the Supervisory Committee on Standard Methods of Analysis, American Chemical Society.

DIVISION OF INDUSTRIAL CHEMISTS AND CHEMICAL ENGINEERS—MINUTES OF BUSINESS SESSION 54TH MEETING AMERICAN CHEMICAL SOCIETY

The meeting was called to order with Chairman Dr. H. E. Howe presiding.

The minutes of the meeting at New York City were read by the Secretary and approved by the Division.

The Secretary reported on the financial condition of the Division as follows:

Cash on hand, September 25, 1916	$ 19.47
Receipts in response to a request for funds	188.00
	$207.47
Expenditures	30.00
Cash on hand, April 1, 1917	$177.47

The Secretary also reported that the entire roll of members had been carefully gone over, all addresses being brought up to date and the names of members who have ceased to be members of the General Society dropped out. It was moved and seconded that the report be accepted.

The Committee on Soap and Soap Products presented a report of their work. This was turned over to the Supervisory Committee on Standard Methods of Analysis, Dr. Hillebrand, *Chairman*, and went to the Council direct.

After some discussion of the Committee situation it was unanimously decided to abolish the Divisional Committee on Standard Specifications and Methods of Analysis. Upon the suggestion of Dr. Hillebrand, Chairman of the Committee on Platinum, that this Committee be not reappointed because there seems to be no occasion for renewed work on the quality of platinum at the present time, it was moved and seconded that the Committee be not reappointed. Motion carried.

There being no further business to come before the Division the reading of the papers was proceeded with.

SAMUEL H. SALISBURY, JR.,
Secretary

ATLAS PORTLAND CEMENT COMPANY
NORTHAMPTON, PA.

AMERICAN INSTITUTE OF CHEMICAL ENGINEERS NINTH SEMI-ANNUAL MEETING

The Ninth Semi-Annual Meeting of the American Institute of Chemical Engineers will be held at Buffalo, New York, June 20 to 22, 1917.

PROGRAM OF PAPERS

Address of Welcome. DAVID C. HOWARD, 1st Vice-President Buffalo Chamber of Commerce.
Some Machinery Employed in the Manufacture of Glue. A. LOWENSTEIN.
Treatment of Sewage by Aeration in the Presence of Activated Sludge. III. EDWARD BARTOW.
The Manufacture of Linseed Oil. GLENN H. PICKARD.
Trade Wastes Disposal. H. P. EDDY.
Chemical Engineering Aspect of Renovating a Sulfide Mill. H. K. MOORE.
Waste Heat Utilization. H. D. BAYLOR.

Symposium on Potash

The Possibilities of Developing an American Potash Industry. R. K. MEADE.
A New Method of Potash Recovery from Feldspar. J. C. W. FRAZER AND E. MILLER.
Potash from Waste Liquor of Beet Sugar Factories. H. E. ZITKOWSKI.

Joint subscription dinner of the Buffalo Engineering Society and the American Institute of Chemical Engineers. Addresses by F. A. Lidbury, President of the Buffalo Engineering Society, and President G. W. Thompson.

EXCURSIONS

Automobile trip around the city and inspection of the Buffalo Foundry and Machine Company Plant. A day at the Buffalo Canoe Club. Automobile ride to Niagara Falls. Car-ride around the great scenic gorge route.

AMERICAN LEATHER CHEMISTS' ASSOCIATION FOURTEENTH ANNUAL MEETING

The Fourteenth Annual Meeting of the American Leather Chemists' Association will be held at Atlantic City, June 7, 8 and 9, 1917, with headquarters at the Marlborough-Blenheim Hotel.

PROGRAM OF PAPERS

Anthrax and Disinfection of Hides. V. A. WALLIN.
The Future of Hide Supply (by Title). A. SEYMOUR-JONES.
Discussion. DR. HICKMAN of the Bureau of Animal Industry.
Comparative Analysis. R. H. WISDOM.
The Testing of Dyes for Leather. G. T. CREESE.
Solubility of Hide in Salt (NaCl) Solution and Effect of Alkali on Soaking Dry Hides. L. BALDERSTON.
Nigrosines. J. MERRITT MATHEWS.
Analysis of Sulfonated Oils. W. K. ALSOP.
Effect of Hard Water on Tannins. T. A. FAUST.
Sulfuric Acid in Leather. C. R. OBERFELL.
Specifications for Kaolin Used in Tannin Analysis. R. W. FREY.
Analysis of Tannery Effluent. W. A. FOX.
Studies Conducted by the Public Health Service in Regard to the Sanitary Disposal of Tannery Wastes. H. B. HOMMON.
(The Public Health Service built an experimental disposal plant at an important tannery where these studies were conducted.)
Chemistry of the Manufacture of Tanning Extracts from Waste Sulfite-Cellulose Liquors. H. H. HURT.
Drum Tannage. OSKAR REITHOF.
Borax and Boric Acid in the Tannery and Currying Shop. H. L. HARRIS.

Symposium on Anthrax

Anthrax. A. S. ROSS.
Practice and Theory on Treatment and Diagnosis of Anthrax. H. Z. FRISBIE.
The Nature of Anthrax and Anti-Anthrax Serum. J. REICHEL.

Discussions

Tanners and Chemists "Round Table" Discussion.
Discussion of Recently Advanced Ideas on Theory of Leather Formation. JOHN ARTHUR WILSON, LLOYD BALDERSTON, WILLIAM KLABER, H. C. REED, F. H. SMALL, ALLEN ROGERS, JOHN H. YOCUM, ROBERT W. GRIFFITH.

PLATINUM IN JEWELRY

The members of the American Chemical Society will be very glad to learn that the Platinum Committee of the Jewelers Vigilance Committee has passed resolutions, which were presented to the Secretary of Commerce, in which they have shown a very proper attitude in recommending to the jewelry trade that the use of platinum in bulky and heavy pieces of jewelry be discouraged, and that jewelers also discourage the use of platinum in all non-essential parts of jewelry, such as scarfpin stems, pin tongues, joints, catches, swivels, spring rings, ear backs, etc., where gold would satisfactorily serve. Considering the fact that platinum is essential for the production of many of the munitions of war and that it is absolutely necessary for the development of our chemical industry and for the development of chemical knowledge, this action of the jewelers is to be highly commended and will be a great help toward discouraging the unnecessary use of platinum and result in a drop in its price and an increase in the stock supply available for the use of the government and our commercial laboratories. The jewelers should be credited with helpful motives, for they will undoubtedly yield up important profits by declining to encourage the desire for personal adornment and ostentatious display of wealth, which is the chief reason for the use of platinum in such articles as watch-cases, solid platinum rings, bracelets, mesh-bags, etc.

It is expected that these resolutions, added to those passed by the Daughters of the American Revolution, by the National Academy of Sciences, and by the American Chemical Society will help to overcome the abuse of platinum.

The needs of the government itself for platinum will undoubtedly be met during the present war without great sacrifice.

While the government itself uses comparatively little platinum, our sulfuric acid industry, especially for the strong acid used in the production of high explosives, is dependent upon it, and its high price makes it almost prohibitive for scientific research. No American man or woman will wish to feel that by wearing platinum they are interfering with the development of the country's industries and scientific standing, but such is inevitably the case.

CHAS. L. PARSONS, *Secretary*

The resolutions passed by the Platinum Committee of the Jewelers Vigilance Committee are as printed below:

RESOLUTIONS

WHEREAS, the Secretary of Commerce has requested the Platinum Committee of the Jewelers Vigilance Committee to bring to the attention of the jewelry trade of the United States the advisability of conserving platinum in order that our government may have larger supplies to draw upon for war purposes, and

WHEREAS, the jewelry trade has already clearly expressed its desire and determination to assist our government to the extent of its ability in bringing the war to a successful termination,

Be It Resolved that we pledge ourselves to discontinue and strongly recommend to all manufacturing and retail jewelers of the United States that they in a truly patriotic spirit discourage the manufacture, sale and use of platinum in all bulky and heavy pieces of jewelry.

Be It Further Resolved that during the period of the war or until the present supplies of platinum shall be materially augmented, we pledge ourselves to discontinue and recommend that the jewelry trade discourage the use of all non-essential platinum findings or parts of jewelry, such as scarfpin stems, pin tongues, joints, catches, swivels, spring rings, ear backs, etc., where gold would satisfactorily serve.

Be It Further Resolved that the jewelry trade encourage by all means in their power, the use of gold in combination with platinum, wherever proper artistic results may be obtained.

Be It Further Resolved that copies of these resolutions be handed to the Secretary of Commerce, to the trade press, and be sent to all our trade organizations, and to the daily press, in order that they may have the widest possible dissemination.

NOTES AND CORRESPONDENCE

SPONTANEOUS COMBUSTION OF OLEIC ACID CONTAINING IRON

Editor of the Journal of Industrial and Engineering Chemistry:

A recent case engaging the attention of this laboratory was concerned with spontaneous combustion of oleic acid when applied to wool. The circumstances of the occurrence have enough of general interest to warrant publication.

The wool or other hair fibers were first oiled with 10 per cent of their weight of oleic acid, after which the oiled fiber was conveyed to bins through a tinned iron pipe by a current of air. The conveying pipes were very near a bank of heating pipes, the latter being employed to heat the mill.

This, of course, was bad practice, but the investigation was continued by an analysis of the oil employed. The iodine value was normal for commercial oleic acid, and the other constants showed nothing of a suspicious nature.

Samples of the various fibers employed—wool, mohair and the like—were then oiled with the oleic acid, and tested in Mackey's Cloth Oil Tester, as described in Vol. II, Allen's "Commercial Organic Analysis." The rise in temperature against time was normal, but the fact was disclosed that the finer fibers gave the greater rise. This fact we ascribed to more surface for oxidation being exposed for equal weights of fiber with similar weights of oil.

If the investigation had ended here, we should not have discovered the true inwardness of the case, but we were sufficiently interested to follow up the clue of more rapid oxidation the finer the fiber to which the oil was applied. To test this point, we next used cotton, and found a rapid and dangerous rise. This should not have been the case, if the oil were suitable. We checked the apparatus, using technically pure oleic acid of pharmaceutical grade, and got no dangerous rise. Thus, it was obvious that something about the oleic acid was wrong, notwithstanding the correct constants. Briefly, we found it to contain iron equivalent to 0.10 per cent ferric oxide.

We next made an oleate of iron, and dissolved it in the pure oleic acid, so that it contained the same amount of iron as the commercial sample. When this oil was applied to cotton, we found that it gave the same dangerous rise in the Mackey Tester as the commercial sample. Here, then, was an adequate explanation of the matter. The small amount of iron had functioned as a catalyzer to promote oxidation.

Since this work was done, we have tested samples of oleic acid from other sources, and have found in them amounts of iron of the same order of magnitude. Experiments have shown that the dangerous rise applies to these also.

We tried to remove the iron by shaking the oils with diluted hydrochloric acid, but without success. To do this successfully would probably necessitate getting the oil and acid in very fine emulsion.

CHARLES E. SWETT AND WALTER S. HUGHES

LABORATORY OF ARTHUR D. LITTLE, INC.
APRIL 28, 1917, BOSTON, MASS.

THE INCOMPLETE HYDROGENATION OF COTTONSEED OIL—CORRECTION

Referring to the article under the above title (THIS JOURNAL, 9 (1917), 451) the following corrections should be made:

P. 451, Foot-note 1: second reference in third line should be dated 1906 instead of 1916.

P. 456, Table II: amount catalyzer in Run F was 2 per cent instead of 4 per cent.

P. 458, Table IV: iodine number of Sample 2, Run I*h*, should read 83.6 instead of 83.1. Iodine number of Sample 6, Run I*l*, should read 54.6 instead of 5.46.

Catalyzer referred to in Tables II and IV as "No. 1" should be understood to represent the *type* of catalyzer referred to in paragraph "2" of the section on "Catalyzers," rather than as one batch of material: several batches, of differing degrees of activity, are represented by it. The catalyzer referred to in the tables as "No. 2" is described in paragraph "1" of the section on "Catalyzers."

H. K. MOORE, G. A. RICHTER AND W. B. VAN ARSDEL

THE MANUFACTURE OF LINOLEUM AND ITS VALUATION—CORRECTION

In the article under above title printed in THIS JOURNAL, 9, (1917), 6, the following corrections should be made:

Page 13, column 1 (tabular matter, under "Linoxyn") the sentence "By 1 per cent aqueous NaOH in the acid" should read "By 1 per cent aqueous NaOH in the cold."

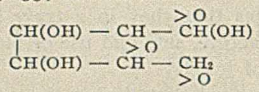
Page 17, column 2, paragraph 4, line 30, "........ poison the solution" should read "........ poison the sol."

ARMAND DE WAELE

THE CHEMISTRY OF WOOD—CORRECTION

In the articles appearing under the above title in this issue, pp. 556 to 566, the following corrections should be made:

The formula on p. 557 should be:



$$\begin{array}{lll} & & >\text{O} \\ \text{CH(OH)} - & \text{CH} - & \text{CH(OH)} \\ | & >\text{O} & \\ \text{CH(OH)} - & \text{CH} - & \text{CH}_2 \\ & & >\text{O} \end{array}$$

On p. 560, 1st column, line 8 of text, *N*/100 should read *N*/10; same column, line 25, iodate should read iodide. Same page, Table I, "Hot Water" column, 4.87 should read 4.78.

On p. 561, Table II, last column, 4.23 should read 5.23.

The following foot-notes are to be inserted:

After 2nd paragraph, 2nd column, p, 562:

The excellent results obtained by Dox and Plaisance [*J. Am. Chem. Soc.*, **38** (1916), 2156] with thiobarbituric acid for determining furfural are unfortunately not easily applicable to the concomitant determination of methylfurfural.

After 24 to 28 per cent, line 18, 1st column, p. 563:

In Technologic Paper No. 88, of the Bureau of Standards, "Studies on Paper Pulps," p. 9, the conclusion is drawn that alkaline processes of cooking pulp do not remove the furfural-yielding complex to the same degree as the acid-sulfite process. Since as a rule only hardwoods are cooked by the soda process the resulting pulps would be expected to yield more furfural from the data given above.

On p. 565, last paragraph, the asterisks should be removed.

A. W. Schorger

FULLER'S EARTH, ETC.—CORRECTION

In my article in this issue, p. 599, the dotted line for "White Oil" (20 Yellow, 2.5 Red) has been omitted in re-making the drawing, so that the sentence referring to same should be omitted.

T. G. Richert

PERSONAL NOTES

The Council of National Defense announces the appointment of a sub-committee on chemicals, to serve under the committee on raw materials of the advisory commission, of which Bernard M. Baruch is chairman. Dr. William H. Nichols, of the General Chemical Co., of New York, is *chairman* of the new sub-division. Besides Dr. Nichols, the sub-committee is composed as follows:

Acids—E. R. Grasselli, of the Grasselli Chemical Co., Cleveland; Henry Howard, Merrimac Chemical Co., Boston.

Fertilizers—Horace Bowker, New York; Chas. E. MacDowell, President Armour Fertilizer Co., Chicago.

Alkalis—J. D. Pennock, Solvay Process Co., Syracuse.

Miscellaneous Chemicals—E. Mallinckrodt, Jr., St. Louis.

Professor Marston T. Bogert, Chairman of the Chemistry Committee of the National Research Council, has moved his office to Room 332, Munsey Building, Washington, D. C.

The Franklin Institute, acting through its Committee on Science and the Arts, has recently made the following medal awards, for especially meritorious papers published in its Journal for the year 1916: The Howard N. Potts Medal to Professor Ulric Dahlgren, professor of biology, Princeton University; Edward Longstreth Medals of Merit to Mr. George A. Rankin, late of the Geophysical Laboratory, Carnegie Institution of Washington; Prof. A. E. Kennelly, Messrs. F. H. Achard and A. S. Dana, Massachusetts Institute of Technology; Mr. John D. Ball, late of the General Electric Company, Schenectady, N. Y.; and Dayton C. Miller, Professor of Physics, Case School of Applied Science.

A number of platinum crucibles were taken from the laboratories of the Agricultural Experiment Station of the Rhode Island State College, Kingston, R. I., just prior to May 7th.

The following resolution was adopted by the Georgia Section of the A. C. S. through mail ballot: "*Resolved:* That the Georgia Section of the American Chemical Society, composed of experienced industrial, agricultural and research chemists, feeling that their services and laboratories can assist in preventing waste and promoting industrial efficiency, offer their services to the Government, individually and collectively."

Prof. J. H. Buchanan, of the food and sanitary chemistry department of the Iowa State College, and his assistant, J. W. Hawkes, were appointed to the Officers' Reserve Corps Camp at Fort Snelling and are at present in attendance.

Mr. W. P. Putnam, of The Detroit Testing Laboratory, Detroit, Michigan, has been elected President of the Detroit Engineering Society for 1917–18. The Detroit Engineering Society includes engineers of all branches of engineering, and numbers over 750 members of the leading men of the community in engineering work.

Prof. A. W. Smith, professor of industrial chemistry and head of the department of chemistry, at Case School of Applied Science, Cleveland, Ohio, was chosen by the Ohio State University as Exchange Lecturer during the week of April 16th to 21st. Dr. Smith lectured each day on chemical engineering topics. The College of Engineering at the Ohio State University made similar arrangements with other institutions, notably Purdue University and the University of Michigan, the lectures from those institutions being electrical and civil engineering. The arrangement is reciprocal.

Dr. H. P. Talbot (*Chairman*), Prof. M. T. Bogert and Dr. W. D. Richardson have been appointed by President Stieglitz as a Committee to consider the proposal of Dr. Bernhard C. Hesse for a Board of Control of National Policies. Dr. Hesse presents his plans in full in the April, 1917, issue of This Journal, page 337.

Professors A. B. Lamb and G. B. Frankforter have been added to the Code of Professional Ethics Committee of the American Chemical Society.

During the past winter, the Wagner Free Institute of Science of Philadelphia has been giving, under the Richard B. Westbrook Foundation, which the Institute administers, special lectures by the members of its faculty. The Chemistry lectures were: "Food from the Air," by Henry Leffmann; "Chemistry of Bread-Making," by Chas. H. La Wall; "Catalysis in the Inorganic Field," by David W. Horn. The lectures will be published.

The Publicity Committee of the Philadelphia Section has elected Dr. Henry Leffmann as Chairman and has begun work. It includes representatives of all the important phases of applied chemistry.

On May 4th, McGill University conferred the degree of LL.D. on Dr. W. H. Ellis of the University of Toronto. Dr. Ellis is a past-chairman of the Division of Industrial Chemistry and Chemical Engineers.

President Stieglitz has appointed Dr. W. F. Hillebrand (*Chairman*), Dr. W. D. Bigelow and Dr. Charles Baskerville as an Advisory Committee to the Bureau of Standards on Analyzed Reagents in pursuance of a resolution passed by the Council at the Kansas City meeting.

Dr. Isaac F. Harris, formerly director of the Lederle Antitoxin Laboratories of New York and later director of the Arlington Research Laboratories of Yonkers, New York, has moved from Bronxville, N. Y., to New Brunswick, N. J., where he is head of the department of biochemistry in the research and biological laboratories of E. R. Squibb & Sons.

Mr. R. T. Will has resigned his position as general superintendent and chemical engineer of the General Coal Products Company of Pittsburgh, Pa., to establish offices and laboratories at Rochester, N. Y., for general testing and consultation in industrial and chemical engineering, under the name of "The Will Corporation." The new laboratories are located at 262 East Avenue.

The John Fritz Medal for 1917 was awarded to Dr. Henry Marion Howe for his investigation in the metallurgy of iron and steel. The presentation ceremonies took place on the evening of May 10th in the Auditorium of the United Engineering Building, 29 West Thirty-ninth Street, New York. Addresses were delivered by Dr. Rossiter W. Raymond, Dr. Ira N. Hollis, Judge Elbert H. Gary and Prof. Albert Sauveur.

The United States Civil Service Commission announces the following open competitive examinations: *Laboratory aid and junior chemist*, for men only, Naval Proving Ground, Indian Head, Md., salaries at $3.28, $3.84, $4.48 and $5.04 per diem. *Assistant chemist*, for men only, Picatinny Arsenal, Dover, N. J., salary $1350 to $1500 a year. Applications for the above positions should be filed at the earliest possible date. They will be rated immediately upon their receipt and appointments made immediately. *Junior chemist* (*qualified in fuels*), for men only, Bureau of Mines, on June 6, 1917, salary $1020 a year. *Assistant analytical chemist and mineralogist*, men only, salary $1800 to $2400 a year, on June 19, 1917; *Associate analytical chemist*, men only, salary $2500 a year, on June 19; *Assistant chemical engineer* (*petroleum*), men only, salary $1800 to $2100 a year on June 12; *Junior chemist*, men and women, salary $1200 to $1440 a year. Until further notice, applications for the last-named position will be received at any time. Papers will be rated immediately upon their receipt by the Commission in order that appointments may be made promptly.

Dr. Charles H. Herty addressed the National Association of Printing Ink Manufacturers during their recent Annual Convention at Delmonico's on the evening of May 7th. The subject of his address was "How the Spirit of Coöperation Can Help the American Color Industry."

On March 9, 1917, Dr. William Louis Jeffries died at Wilmington, Vermont, as a result of poisoning by hydrogen sulfide, while demonstrating the use of niter cake in sulfate pulp manufacture. Dr. Jeffries was educated first at Carson-Newman College, then at the University of North Carolina. From the latter institution he received the degrees A.B. and Ph.D., serving two years as instructor in chemistry. Since November 1915, he had been connected with the by-products division of the du Pont de Nemours Co., Wilmington, Delaware, in which capacity he had been very successful in finding new uses for niter cake. Dr. Jeffries was an enthusiastic member of the Alpha Chi Sigma fraternity and was actively engaged just previous to his death in bringing that organization of young chemists to offer the services of its members to the government in whatever capacity they might be used.

Dr. Wm. H. Taylor, who for 46 years was professor of chemistry in the Medical College of Virginia, and coroner of the City of Richmond, died on April 14th, at his home in Richmond. Dr. J. M. Whitfield, for several years city chemist of Richmond, has been made city coroner to succeed Dr. Taylor.

The University of Illinois Engineering Experiment Station has just completed a preliminary systematic study of the alloys of chromium, copper and nickel, and their properties. The work has been under the direction of Dr. D. F. McFarland, Assistant Professor of Applied Chemistry, and Dr. O. E. Harder, Fellow in Chemistry. Copies of Bulletin No. 93, containing the results of these tests, may be obtained without charge by writing C. R. Richards, Director of the Engineering Experiment Station, Urbana, Illinois.

Dr. L. F. Nickell of the department of chemistry, Washington University, St. Louis, has resigned to accept a position with the Monsanto Chemical Company of St. Louis in their research department.

Dr. E. C. L. Miller, who has been in California for the past year, is returning to the Medical College of Virginia where he resumes his duties as professor of physiological chemistry.

The Municipal Civil Service Commission of the City of New York announces the following opening competitive examination: Assistant Inspector of Combustibles, Grade 2 (Inspector of Combustibles), salary $1200 to $1500 a year. Applications will be received up to June 4th.

Mr. G. A. Burrell, consulting chemical engineer of Pittsburgh, and for eight years in charge of gas investigations of the Bureau of Mines, has been selected by Mr. Van. H. Manning, Director of the Bureau of Mines, to take charge of research work on matters pertaining to gas warfare.

Dr. H. N. McCoy, who has been connected with the department of chemistry of the University of Chicago since 1901 and professor since 1911, has tendered his resignation in order to be free to devote more time to his important technical interests. His process of extracting radium from carnotite is being used by the Carnotite Production Company of Chicago in which he is a director.

At the Chemists' Club on Wednesday evening, April 25th, the Chemical Engineering Society of Columbia University held a dinner, at which former Professors M. C. Whitaker and F. J. Metzger were the guests of honor. The Society presented to each of them a silver desk clock, as a token of appreciation and gratitude for their work in building up the School of Chemical Engineering at Columbia, of which Prof. Whitaker was the head, before he recently resigned.

Mr. M. Landau, president of the Society, introduced Dr. C. F. Chandler, dean of the chemical profession, who acted as toastmaster. Dr. Whitaker, discussing the "Industrial Situation," told about the problems which manufacturers must meet, due to the activity of enemy agents. Dr. Metzger gave a short talk on "Chemical Engineering." Mr. A. F. Smithers, one of the graduating students, talked about "what Columbia has done for us." Mr. A. A. Haldenstein, also a Senior, spoke on "Our Responsibilities to Our Alma Mater."

Prof. D. D. Jackson, acting head of the School of Chemical Engineering, discussed "Preparedness for the Chemical Engineer." Dr. J. Teeple, gave the diners some excellent advice, when he responded to the toast, "Things to Remember." Prof. M. T. Bogert, Chairman National Research Council, spoke on "The Chemist in the Present Crisis." Professors Tucker and Neish both said a few words in appreciation of the work of Doctors Whitaker and Metzger.

Much credit is due the dinner committee, of which Mr. E. C. Brueckmann was chairman, for making the affair such a signal success.

Science reports that six professors of the University of Minnesota have been asked by the war department to act as a scientific research board for the district of the State of Minnesota. The duties asked of them will require a considerable portion of their time from now on. These men are Professor John J. Flather, head of the department of mechanical engineering; Dean George B. Frankforter of the school of chemistry; Dr. L. G. Rowntree, head of the department of medicine; Professor R. W. Thatcher, head of the department of agricultural chemistry; L. W. McKeehan of the department of physics; and F. R. McMillan, of the department of structural engineering. Dean Frankforter and Professor Flather acted upon a special navy board which made an inventory of Minnesota resources last summer and the figures which they gathered at that time will be of inestimable value in the present crisis.

Dr. T. Brailsford Robertson, professor of biochemistry and pharmacology in the University of California, has donated to the Regents of the University of California his patents for the growth-controlling substance "Tethelin," which he has succeeded in isolating from the anterior lobe of the pituitary body and which has been employed to accelerate repair in slowly-healing wounds. The proceeds which may accrue from the sale or lease of these patents are to constitute a fund which will be entitled "The University of California Foundation for International Medical Research" and which will be expended in the furtherance of medical research, preferably research in the physiology, chemistry, and pathology of growth.

Dr. Wm. C. Carnell has resigned the position of Chemical Director of Harrison Brothers & Co., Inc., Philadelphia, to accept a similar position with the firm of Charles Lennig, Inc., Philadelphia, manufacturers of heavy chemicals.

Dr. E. P. Wightman, who has been acting professor of chemistry at Richmond College, session 1916–17, has accepted a position with the research department of Parke, Davis & Co., and will assume the duties of his new position June 1st.

At the request of the National Research Council, the faculty of Wesleyan University have appointed the following local committee, consisting of one representative of each of the scientific departments of the institution with the president, William Arnold Shanklin, *ex-officio:* Professors Walter G. Cady, physics, *Chairman:* William North Rice, geology; Raymond Dodge, psychology; Frederick Slocum, astronomy; Leroy A. Howland, mathematics; Moses L. Crossley, chemistry.

John Faxan Passamore died on March 9th at Wilmington, Delaware, after an illness of six weeks. He obtained his degree in Mechanical Engineering at Cornell University in June 1914. He was Assistant General Superintendent of Joseph Bancroft and Sons Co., Wilmington, Delaware, at the time of his death. Although only 25 years old, he was honored with the Presidency of the Departmental Association of the company, filling the position with marked ability for one of his years.

The New York University is planning the erection of a new chemistry building.

Mr. George D. Rosengarten has returned to Philadelphia very much improved in health.

INDUSTRIAL NOTES

Work has been begun on the erection of a large still-house on the property of the Benzol Products Company at Marcus Hook. It is expected that the building will be completed within six months. The Benzol Products Company, which manufactures coal-tar intermediates, is owned jointly by Semet-Solvay, Barrett and General Chemical interests, and is a constituent in the new National Aniline and Chemical Company, Inc.

The National Aniline and Chemical Company will remove its offices some time in June from 100 William Street, to a new building now being completed at 244 Madison Avenue.

In the West, the waters of various lakes besides Searles Lake are receiving considerable attention, and special mention should be made of Owens Lake, where three companies are now in the field. The last one entering is the California Alkali Company, now building its plant. The lake waters contain soda ash, sodium chloride, some borax, and some potash. The California Alkali Company is closely associated with the Great Western Electrochemical Company, and the salt obtained in the production of the various products will be shipped direct to the Pittsburg, Cal., plant of the Great Western Electrochemical Company and used there for the manufacture of caustic soda and chlorine products.

A fire which caused $20,000 damage is reported to have occurred recently at the plant of the Albany Chemical Company, on Van Rensselaer Island.

The United Chemical and Organic Products Company has been chartered to manufacture glues, gelatine, etc., under the laws of Delaware, with capital of $2,002,700.

The Manufacturers' Association of St. Louis has voted to protest against the proposed tax of 25 cents a gallon on denatured alcohol.

According to a report from Syracuse the Solvay Process Company is about ready to begin the production of potash at its new plant in Solduro, Utah, which is practically ready for operation.

Eimer & Amend are planning alterations and additions, costing $100,000, to their buildings at Third Avenue and East 18th Street, New York City.

The Kalbfleisch Corporation, recently organized, has taken over all the interests of the Franklin H. Kalbfleisch Company, Erie Chemical Works, the Kalbfleisch Corporation, and the Kaloid Company. The new corporation will thus own five plants, one each at Brooklyn, N. Y., Waterbury, Conn., Elizabethport, N. J., Erie, Pa., and Chattanooga, Tenn. The Brooklyn, Waterbury and Elizabethport plants turn out a line of acids and heavy chemicals. The Erie and Chattanooga works (the latter having been recently completed) manufacture the different grades of sulfate of alumina and alum.

The officers of the corporation are: Franklin H. Kalbfleisch, Chairman of the Board of Directors; Robert S. Perry, President; Alfred B. Savage, Vice-President-Treasurer; Richard Sheldrick, Secretary. The directors are the same with the addition of Harry L. Derby. Mr. Perry was formerly president of Harrison Bros. & Co., Inc., Philadelphia.

The Gold Leaf Natural Dye Company, of Manhattan, dyes, chemicals, etc., has been incorporated under the laws of New York with a capital stock of $500,000. Incorporators: G. H. Bruce, R. L. L. Warner, H. Campbell, No. 150 Nassau Street.

The Brunswick Manufacturing Company, dyes and chemicals, has been incorporated under the laws of New Jersey with a capital stock of $30,000. Incorporators: George R. Morrison, J. H. Helm, Horace E. Barwis, New Brunswick.

The Active Chemical Company, of Camden, has been incorporated under the laws of New Jersey, with a capital stock of $150,000. Incorporators: E. C. L. Kressel, R. P. S. Miller, T. Harry Rowland, Camden.

The Bristol-Myers Company, of Brooklyn, has completed plans for additions to its chemical factory at Hillside, N. J., costing $25,000.

The amount of capital stock in new dye companies, according to years during the war, including only companies of more than $50,000 capital stock, has been as follows:

Year 1915	$ 65,565,000
Year 1916	99,274,000
Four months 1917	24,395,000
Total	$189,234,000

The Braemer Air Conditioning Corporation announce the opening of a branch office, with Mr. G. P. Upington in charge as District Manager, at 90 West Street, New York City.

GOVERNMENT PUBLICATIONS

By R. S. McBride, Bureau of Standards, Washington

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.

HYGIENIC LABORATORY

Changes in the Pharmacopoeia and the National Formulary. Martin I. Wilbert. Bulletin 107, 288 pp. Paper, 30 cents. This is a digest of the changes and requirements included in The Pharmacopoeia of the United States (ninth decennial revision) and in The National Formulary (fourth edition) with references to the titles not continued from the preceding editions.

"Because of the fact that The Pharmacopoeia and The National Formulary are in reality separate and distinct publications, it was thought that a comprehensive index of the articles described in recent editions of these two books would be of value to public health officials and to all who are in any way interested in the official standards and requirements for drugs and medicines. To provide a readily available means for determining the present status of any official or recently official article an alphabetical list of the titles included in the U. S. P. VIII, the U. S. P. IX, the N. F. III, and the N. F. IV, has been compiled with an outline of the changes and requirements that have been embodied in the revised editions of the U. S. P. and the N. F.

"In the compilation, the changes in strength and composition are indicated as concisely as practicable, and special attention is directed to changes in nomenclature of official articles as well as additions to and deletions from the list of articles included in the several books.

"The object in mind was to make this publication a practical companion and convenient reference book on the present status of any of the many articles included in either of the four books referred to.

"As noted above, this bulletin is designed to serve as an index to the several recent editions of the official standards and can in no sense be considered to take the place of either of these books. For detailed information regarding the composition, strength, tests, and other requirements in connection with the several official articles, readers are referred to The Pharmacopoeia and The National Formulary."

Pituitary Standardization. The Relative Value of Infundibular Extracts Made from Different Species of Mammals and a Comparison of Their Physiological Activity with That of Certain Commercial Preparations. George B. Roth. Bulletin 109, I, 32 pp.

Pharmacological Studies with Cocaine and Novocaine. A Comparative Investigation of These Substances in Intact Animals and on Isolated Organs. George B. Roth. Bulletin 109, II, 31 pp.

BUREAU OF THE CENSUS

Report on Cottonseed and Cottonseed Products. Post card announcement dated 10:00 A.M., April 24, gives the following figures:

	Shipped out Aug. 1 to Mar. 31	On Hand March 31
Crude oil (pounds)	1,084,946,283	144,122,399(*a*)
Refined oil (pounds)	....	320,930,168(*b*)
Cake and meal (tons)	1,820,172	246,442
Hulls (tons)	769,021	133,774
Linters (500-pound bales)	999,400	136,907(*c*)
Hull fiber (500-pound bales)	325,976	11,357(*c*)
Motes, grabbots, and sweepings (500-pound bales	18,196	11,485(*c*)

(*a*) Includes 4,438,521 and 7,423,786 pounds held by refining and manufacturing establishments on March 31, and 32,666,372 lbs. in transit to refiners and consumers.

(*b*) Includes 10,567,250 pounds held by refiners, brokers, agents, and warehousemen at places other than refineries and manufacturing establishments and 8,477,746 lbs. in transit to manufacturers of lard substitute, oleomargarine, soap, etc., March 31.

(*c*) Includes 11,995 bales of linters, 714 bales of hull fiber, and 110 bales of motes, grabbots, and sweepings stored elsewhere than at the mills.

Imports and Exports of Cottonseed Products from August 1 to March 31

Imports: Oil (pounds)	6,112,022
Exports:	
Oil (pounds)	96,480,617
Cake and meal (tons)	462,463
Linters—Unbleached (running bales)	144,001
Bleached (running bales)	96,041

Manufactured Ice. Report from Census of Manufactures, 1914, issued April 1917. The total cost of ammonia used in the ice and allied industries for the production of ice or refrigeration where ice is a subsidiary product, amounted to $1,887,359.00. For this work 6,780,000 lbs. of anhydrous ammonia were used, about five-sixths of this in the ice industry and about nine-tenths of it in compressor systems. Aqua ammonia used amounted to nearly 2,000,000 lbs., practically all being employed in the ice industry. This material was used in approximately 5,000 machines which had a capacity of 130,000 tons of ice per day of twenty-four hours.

DEPARTMENT OF AGRICULTURE

Sugar-Cane Culture for Sirup Production in the United States. P. A. Yoder. Bulletin 486, contribution from the Bureau of Plant Industry, issued March 19th, 46 pp. Paper, 10 cents. A description of the practices in vogue at the present time in the growing and handling of sugar cane where sirup is the main product.

A Study of American Beers and Ales. L. M. Tolman and J. Garfield Riley. Bulletin 493, contribution from the Bureau of Chemistry, issued March 21st, 23 pp. Paper, 5 cents. A technical study of this subject and of interest to brewers generally.

Studies on the Digestibility of Some Animal Fats. C. F. Langworthy and A. D. Holmes. Bulletin 507, contribution from the States Relations Service, issued March 24th, 20 pp. Paper, 5 cents. This bulletin records studies of the digestibility of chicken fat, goose fat, brisket fat, cream fat in egg yolk, and fat or oil in fish. It is primarily of interest to students and investigators of food problems.

Yields from the Destructive Distillation of Certain Hardwoods. R. C. Palmer. Bulletin 508, contribution from the Forest Service, issued March 6th, 8 pp. Paper, 5 cents. This is the second progress report. Gives the results of experiments in destructive distillation of hardwoods and is of interest to manufacturers of by-products.

The Theory of Drying and Its Application to the New Humidity-Regulated and Recirculating Dry Kiln. Harry D. Tiemann. Bulletin 509, contribution from the Forest Service, issued March 17th, 28 pp. Paper, 5 cents. A technical discussion of a new method of kiln-drying hardwood lumber and of interest to manufacturers of lumber products.

Relation of the Water-Retaining Capacity of a Soil to Its Hygroscopic Coefficient. FREDERICK J. ALWAY AND GUY R. McDOLE. Journal of Agricultural Research, **9**, 27–69 (April 9).

Fixation of Ammonia in Soils. I. G. McBETH. Journal of Agricultural Research, **9**, 141–151 (April 30).

PUBLIC HEALTH SERVICE

The Activity of Wild American Digitalis. GEORGE B. ROTH. Public Health Reports, **32**, 377–80 (March 9). "From the above investigation we may conclude that the wild digitalis which is found in the Northwestern States may be utilized as a source of supply for making the various official preparations of digitalis, and that by using ordinary methods in handling and preparing the leaves we may secure a highly active product, which compares favorably with the activity of cultivated leaves grown under more favorable conditions."

Court Decisions Pertaining to Public Health. A Digest of the Judicial Opinions Published in the Public Health Reports During the Calendar Year 1916. JASON WATERMAN. Public Health Reports, **32**, 453–75 (March 30).

Biological Products: Notice to Those Concerned. Public Health Reports, **32**, 565 (April 20).

In view of the fact that some establishments licensed for the manufacture and sale of biological products located in Germany have not been inspected within the time required by regulation and on account of present conditions will be inaccessible to inspection for an indefinite period of time, the department has revoked the licenses of these establishments, and notification of this fact is hereby given to all concerned.

The establishments in question with their license numbers and products are the following:

No.	NAME	PRODUCTS
12	Chemische Fabrik auf Actien, Berlin, Germany	Antigonococcus vaccine, antistreptococcic serum, diphtheria antitoxin, and tuberculin
24	Farbwerke, vormals Meister, Lucius und Brüning, Hoechst on Main, Germany	Antidysenteric serum, antimeningococcic serum, antipneumonic serum, antistreptococcic serum, antitetanic serum, diphtheria antitoxin, tuberculins
29	The Behringwerk, Marburg, Germany	Antitetanic serum and tuberculin
31	E. Merck, Darmstadt, Germany	Antimeningococcic serum, antipneumonic serum, antitreptococcic serum, diphtheria antitoxin, jequiritol serum, leucofermantin (antitryptic sheep serum), normal horse serum (liquid and dried), tuberculins, and bacterial vaccines prepared from colon bacillus, dysentery bacillus, gonococcus pneumococcus, staphylococci, streptococci, and typhoid bacillus.
32	Kalle & Co., Biebrich, Germany	Tuberculin (Rosenbach)
39	Pharmaceutisches Institut Ludwig Wilhelm Ganz, Oberursel bei Frankfurt a. M., Germany	Antidysenteric serum
55	Chemische Fabrik Güstrow, Güstrow i. M., Germany	Staphylococcus vaccine

Detection of Hydrocyanic Acid Gas: Use of Small Animals for this Purpose. S. B. GRUBBS. Public Health Reports, **32**, 565–70 (April 20).

BUREAU OF MINES

Analyses of Coals Purchased by the Government During the Fiscal Years 1908–1915. GEORGE S. POPE. Bulletin 119. 99 pp. Paper, 15 cents.

Combustion in the Fuel Bed of Hand-Fired Furnaces. HENRY KREISINGER, F. K. OVITZ AND C. E. AUGUSTINE. Technical Paper 137. 61 pp. Paper, 15 cents. "The main object of the investigation described in this report was to determine the conditions governing the process of combustion in the fuel bed of a hand-fired furnace. The results of this investigation furnish data for correct design of coal-burning grates and furnaces and their efficient operation. They also cast light on the important problem of clinker trouble as related to fusibility of ash. They further indicate the possibility of a high rate of gasification of coal in gas producers, as suggested in Bulletin 7."

Asphyxiation from Blast-Furnace Gas. FREDERICK H. WILLCOX. Technical Paper 106, 57 pp. Paper, 15 cents. "This report is issued by the Bureau of Mines in pursuance of its endeavors to increase safety and efficiency in metallurgical industries. It discusses the nature and causes of poisoning from blast-furnace gas, itemizes the places where gas may be expected to be encountered, suggests safeguards and points out the precautions to be taken in working about gaseous places."

Oxygen Mine Rescue Apparatus and Physiological Effects on Users. YANDELL HENDERSON AND JAMES W. PAUL. Technical Paper 82, 83 pp. Paper, 15 cents.

GEOLOGICAL SURVEY

Tungsten Deposits of Northwestern Inyo County, California. ADOLPH KNOPF. Bulletin 640-L, from Contributions to Economic Geology, 1916, Part I, pp. 229–49. Published January 26, 1917. "Tungsten deposits were found in northwestern Inyo County, California, in 1913, but remained practically unknown until the spring of 1916, when they began to be energetically developed. By midsummer two mills, having a total daily capacity of 400 tons, had been completed and were in active operation.

"The ore bodies that are being mined are from 20 to 60 feet wide and from 150 to 200 feet long. They carry from 1.5 to 2 per cent of tungsten trioxide (WO_3). The area in which scheelite-bearing deposits have been found roughly forms a belt 20 miles long, but it is likely that the prospecting now going on will extend the dimensions of the field.

"The mining conditions are favorable; the climate is good, water is easily obtained, transportation facilities are adequate, and an electric transmission line transverses the belt, so that power is readily available. Timber, however, is lacking. The tungsten area adjoins a rich agricultural district."

Antimony in 1915. FRANK L. HESS. From Mineral Resources of the United States, 1915, Part I, pp. 837–843. Published March 13, 1917.

"Antimony prices in 1915 were probably the highest known since the metal became a regular article of commerce. The high prices led to the largest production the United States has made and probably to the largest world's production.

"According to figures collected by the United States Geological Survey the production of antimony ores in the United States in 1915 is estimated at 5,000 tons, containing 2,100 tons of antimony, valued at about $425,000. The largest previous domestic production was in 1892, when 150 tons of metal were produced in San Francisco from Nevada ores and 380 tons of ore carrying 55 per cent of antimony were exported. Practically all operations of 1915 were new; most of them were small; and they were so widely scattered that it has been difficult to obtain accurate figures.

"For years large quantities of antimonial lead were imported into the United States as type metal, for which it is largely used. During 1912, 1913, 1914 and 1915 these imports have been much smaller, but the imports for 1915 were nearly double those of 1914."

Arsenic in 1915. FRANK L. HESS. From Mineral Resources of the United States, 1915, Part I, pp. 845–846. Published March 13, 1917.

"The output of arsenic in the United States in 1915, was much the largest yet made in this country and amounted to 5,498 short tons, valued at $302,116, an increase of nearly 18 per cent over the production of 4,670 tons, valued at $313,147, in 1914, and an increase of about 75 per cent over the largest previous production, that of 1912, when 3,141 tons, valued at $190,757, were produced.

"As usual, the arsenic was all saved as a by-product in the smelting of copper and gold and silver ores.

"White arsenic was not produced in the United States until 1901, when the Puget Sound Reduction Co., at Everett, Wash., made an output of 300 short tons, saved as a by-product in smelting gold and silver ores.

"Prices of white arsenic averaged from 2.45 to 2.83 cents a pound at the works. The prices in New York, according to the *Oil, Paint, and Drug Reporter*, were very steady, starting at 4 to 4.5 cents a pound in January, declining to 3.5 to 4 cents in August, and closing in December at 3.875 to 4.5 cents a pound. Lead arsenate was quoted at 5.5 to 6 cents a pound until November, when it rose to 6.5 to 7 cents a pound. Red arsenic (AsS) was quoted at 8 cents a pound at the beginning of the year, rising to 8.25 to 9 cents in November. No quotations were given in December."

Chemical Relations of the Oil-Field Waters in San Joaquin Valley, California. Preliminary Report. G. SHERBURNE ROGERS. Bulletin 653, 112 pp. Paper, 10 cents. "The writer studied the physical and chemical relations of the water and oil in these fields during the summers of 1914 and 1915. The chief conclusions concerning the chemistry of the waters, deduced by him from a study of several hundred analyses of water from different depths, are as follows:

1—Oil-field water is not necessarily salty, as is generally believed, and may not be even slightly salty to the taste. The degree of concentration of chloride in such water is governed primarily by local conditions and is not affected by the position of the water in relation to oil.

2—Sulfate, which predominates in most of the shallow ground waters on the west side of San Joaquin Valley, diminishes in amount as the oil zone is approached, and finally disappears.

3—The concentration of carbonate increases as the oil zone is approached but depends largely on the concentration of chloride.

4—The horizon, with respect to the oil zone, at which these alterations take place, is different in each field.

On the basis of these conclusions, which appear to be well grounded, at least for the area studied, the following practical suggestions may be made:

In drilling a well in untested territory it may be possible to obtain an indication of the presence or absence of oil and gas below by ascertaining by analysis whether the sulfate is diminishing and the carbonate increasing in the waters successively encountered. In some areas a significant change may be detected as much as 1,000 ft. above the oil; in others the upper limit of alteration may be within a few hundred feet of the oil.

The source of the water in a well that produces a mixture of water and oil may be determined, at least in a general way, by studying its chemical composition. In the Westside Coalinga field, for example, the source of the water may thus be determined rather definitely; in the Midway-Sunset field, where the distinctions are less sharp, the success of this method will depend largely on the number of authentic analyses that are available for comparison.

In this report the writer aims first to present the evidence on which the foregoing conclusions are based and to discuss the interpretation of water analyses and their value from the operator's standpoint, and second, to discuss the chemical relations of water and oil in so far as present information permits. It is hoped that this preliminary presentation will indicate the importance of experimental geochemical work on the interaction of the organic constituents of oils and the inorganic substances found in the oil-field waters. Enough is known already to warrant the belief that systematic experimental work in petroleum hydrology will yield results of practical as well as scientific value.

Cleveland Gas Field, Cuyahoga County, Ohio, with Study of Rock Pressure. G. SHERBURNE ROGERS. **Application of Theory of Probability in Estimating Capacity of New Well.** C. . VAN ORSTRAND. Bulletin 661–A, 68 pp. Issued March 2.

Oil Resources of Black Shales of the Eastern United States G. . ASHLEY. Bulletin 641–L, 33 pp.

Chromite. J. S. DILLER. Bulletin 666–A. The chromic iron imported into the United States in 1916 amounted to 11455 long tons. During the same period the chromic iron produced and sold in the United States amounted to approximately 40,000 tons. The greatly increased trade, especially in steel, and the consequently larger demand for chromite have stimulated the search for it in the United States, as shown by the tenfold increase in production.

The ores west of Riddle are the richest yet mined in the States; in some places they run as high as 55 per cent chromic oxide, and much of the ore contains about 50 per cent. Most of the Oregon ore, however, like that of California, averages about 40 per cent of chromic oxide, and ore of that grade is commonly the basis of sale. The ore generally contains 38 to 45 per cent chromic oxide, 6 to 8 per cent silica, and 17 to 25 per cent alumina. It is claimed that the ore can be concentrated to a content of 55 per cent chromic oxide. If this can be done successfully, it will mean much for the chrome industry of the Pacific coast and will enlarge its possibilities to meet war demands. The difficulty with much of the chrome ore of the Pacific coast has been its low grade and its great distance from the principal markets. The low-grade ore, running 38 to 45 per cent chromic oxide, may be used to advantage chiefly for metallurgic purposes, such as grow out of war demands—for furnace lining and in the manufacture of chrome brick and chrome steel, for example—and by far the greater part of the California output is being so used. For chemical purposes, however, ore of higher grades is desirable, if not essential.

The prices of chromite in California on the basis of 40 per cent chromic oxide ranged in 1916 from $1 a ton f. o. b. early in the season to $20 toward the end of the year. To this must be added for the eastern buyer a freight rate for carload lots ranging from $10 a ton to Chicago, to $1486 a ton to the eastern seaboard, thus making the California 4 per cent ore cost on the eastern seaboard from $28.86 to $34.96 a ton.

With the known supplies of chromite and others whose discovery within the limit of practicable transportation throughout the United States is confidently expected, there is good reason to believe that the domestic output of chromite could be so increased as to go far toward supplying the demand if in the event of war our imports, except those from Canada, were cut off.

The metallurgy of chromite has apparently been so developed in the hydroelectric process as to utilize to advantage relatively low-grade ores such as are most abundant in the United States, and the further development of that process on the Pacific coast, where water power abounds, would greatly diminish the handicap of long transportation.

Sulfur. PHILIP S. SMITH. Bulletin 666B, 4 pp. Even under the conditions that now prevail this country exports much more sulfur than it imports. The excess in 1916 amounted, in round figures, to 107,000 long tons, valued at $2,100,000; in 1915 to 11,000 tons, valued at $250,000; in 191 to 72,000 tons, valued at $1,300,000; and in 1913 to 66,000 tons, valued at $1,100,000. The great falling off of exports in 1915 may have been due in part to the increased use of sulfur in industries in this country. In part, however, probably in large part, the decrease was due to the difficulties and dangers of transportation to foreign ports.

The United States in 1913 produced about 350,000 long tons of pyrite and imported about 850,000 tons. If these figures represent the normal condition of the industry, it is evident that ordinarily the United States uses each year about 50000 tons more pyrite than it produces. The imported pyritic ore has an average sulfur content of approximately 45 per cent, so that 225,000 tons of native sulfur would be required to make up this deficiency if the importation of pyrite were cut off. In 1916 we exported 107,000 tons of sulfur more than we imported. If the exportation of sulfur were prohibited, as it doubtless would be if imports of sulfides were cut off, this excess amount would therefore be available, and at the 1916 rate would make up nearly half of the deficiency that would be created by the shutting out of imported pyrite. The deficiency could be reduced still more by drawing on the reserves of sulfur already mentioned, but doubtless it would be far better not to use the high-grade native sulfur for many of the purposes for which pyrite may be used, but rather to save more of the sulfur from the sulfide ores and to hunt for and develop additional deposits of sulfides.

According to preliminary estimates the United States in 916 produced about 4,500,000 tons of sulfuric acid of a strength of 50° Bé. and nearly 1,000,000 tons of acid of strength higher than 66° Bé. This amount exceeds the amount of similar acids produced in 1913 by more than 50,000 tons of 50° Bé. acid and by more than 900,000 tons of acid of strengths higher than 66° Bé.

Almost no sulfuric acid is imported into the United States, and but a relatively small amount is exported, even under the conditions now prevailing. The reports of the Bureau of Foreign and Domestic Commerce show that in 1916 a little over 600 tons of acid were imported and about 30,000 tons were exported.

Manganese. D. F. HEETT. Bulletin 666–C, 12 pp. The largest domestic production of manganese ore for a single year was but 34,524 tons in 87, and since that year, except for several brief periods, the domestic production has declined rather steadily.

In contrast with the domestic production, the imports of manganese ore, largely from India, Brazil, and Russia, have rather steadily risen in proportion to the production of steel. The extensive deposits of these countries are rich enough to permit the shipment of large quantities of ore without concentration.

MANGANESE RECOVERABLE FROM ORE PRODUCED IN THE UNITED STATES 1915, IN LONG TONS

Source	Ore	Manganese
Manganese ore	9,709	4,660
Manganiferous iron ores (*a*)	48,193	9,040
Manganiferous silver ores	137,045	17,504
Manganiferous zinc residuum	159,318	23,053
	....	54,257
Manganese recoverable as alloys	....	36,792
Percentage of total manganese needed	....	21.0

(*a*) Exclusive of Marquette County, Mich., and Iron County, Mich.

The ferromanganese needed for the American steel industry has been supplied in part by imports of the alloy itself and in part by domestic manufacture from imported ore. It is evident that the domestic manganese ore is the source of little ferromanganese The available data for spiegeleisen, however, show that prior 1914 about half of the domestic product was made from domestic ore. During recent years the imports of spiegeleisen have been negligible. The available supply of these manganese alloys for 1915 and the approximate content in manganese shown in the table. From these results, it has been calculated that for a four-year period an average of 14 lbs. of manganese has been consumed for each ton of steel produced.

AVAILABLE SUPPLY OF MANGANESE ALLOYS IN THE UNITED STATES, 1915, IN LONG TONS

	Quantity	Manganese Content
Imports:		
Ferromanganese	55,263	42,210(*a*)
Spiegeleisen	200	36(*a*)
Domestic production:		
Ferromanganese	144,260	110,134(*a*)
Spiegeleisen	114,556	22,808(*b*)
	....	175,189

(*a*) Percentage of manganese estimated.
(*b*) Percentages of manganese submitted by makers.

The prevailing price of manganese ore has steadily risen since 1914, but the increases have lagged behind those in ferromanganese. For 15 years prior to 1914 the price per unit of 49 per cent ore in the United States ranged from 23 to 30 cents, but during 1916 it rose first to 45 cents and later to 65 cents. The price of 65 cents was maintained through the early months of 1917.

Soon after the war broke out the price of ferromanganese rose suddenly from $40 to $120 a ton for a brief period, then ranged from $70 to $115 for more than a year. During 1916 prices ranged from $115 to $175 a ton, except for a short time in April, when $400 a ton was recorded. This great rise was probably caused by Germany's declaration of a blockade of England. In February 1917, the price reached $250 a ton.

The price offered for standard 18 to 22 per cent spiegeleisen has also increased from about $25 a ton in 1914 to $50 in 1916 and $65 in 1917.

Platinum. JAMES M. HILL. Bulletin 666–D, 4 pp. It will be noted that the normal annual addition of crude platinum to the world's supply has varied between 260,000 and 313,000 ounces. This supply is augmented by the platinum produced by refiners of copper matte and gold bullion. United States refiners of gold and copper produce annually about 1,500 ounces of refined platinum as by-products, chiefly from copper ore, of both foreign and domestic origin.

That the supply of new platinum has not been sufficient to meet the requirements is indicated by the very extensive trade in scrap or used metal. Figures are not available for such trade in foreign countries, but in the United States the yearly sales of secondary platinum normally amount to about 40,000 ounces; in 1916 they increased to 49,400 ounces.

The United States at present is not supplying 10 per cent of its platinum requirements, and while there is some assurance that by systematic geologic investigation, already planned, new placer deposits that will yield platinum may be found and that by a study of the methods of saving platinum a greater yield from all deposits may be had, yet the issue must be squarely faced that in all probability the domestic supply cannot be made adequate to meet the requirements of normal times.

Salt, Bromine and Calcium Chloride. R. W. STONE. Bulletin 666–F, 4 pp. The United States furnishes practically all the salt consumed by its people. In 1916 more than 99 per cent of all the salt used in this country was made here, and the value of the salt exported was more than twice as great as that of the salt imported.

The production in 1916 is estimated at 43,000,000 bbls. of 280 lbs., or nearly half a barrel for each individual in the country, compared with 38,000,000 bbls. in 1915.

Prices of salt are increasing and may continue to increase so long as the present unsettled conditions continue. Some grades of salt have doubled in value since 1915; others have increased 50 per cent. This increase is chargeable not only to increased demands of labor and cost of supplies but also to the larger margin of profit to which producers feel entitled under present circumstances. Heavy charges against the cost of production are wages and fuel. Labor is demanding and receiving higher pay, and coal at some salt works has been hard to get and expensive. One producer reports coal increased in cost from 80 cents to $5.50 a ton.

In 1915 there was a significant increase over the production of the previous year, the total output in the United States being 855,857 lbs., valued at $856,307, or about $1 a pound. The comparatively low price indicated by the total figures is due to the fact that considerable bromine was sold at prices specified in contracts made before the demand increased, and to the further fact that the figures indicate prices at point of production and hence do not include the cost of freight. The price of bromine during the first half of 1916 ranged from $4.75 to $6.50 a pound in New York, as a result, at least in part, of the unprecedented demand from abroad. In March, 1917, it had fallen to $1.30 a pound.

The quantity of calcium chloride produced from natural brines and sold in the United States has recently been about 20,000 short tons a year, valued at $6 to $6.50 a ton. This does not include the output obtained in the manufacture of soda, as calcium chloride so obtained is not an original constituent of brine. Large quantities made in the manufacture of soda have been wasted, and it is hoped that new uses may be found for this by-product.

Since the first half of 1916 there has been a demand for this material which has raised the price. In March, 1917, 70 to 75 per cent fused calcium chloride was quoted in the New York market at $26 to $30 a ton, and granulated calcium chloride has recently been quoted as high as $40 a ton. Fused lump calcium chloride that used to retail for 15 cents a pound was quoted in April, 1917, at 90 cents. It is believed that the supply can easily be kept ahead of the demand.

Sand and Gravel. R. W. STONE. Bulletin 666–G, 3 pp. The sand industry in all its branches grows normally with increase of population. Severe business depression decreases the production of building sand more than that of sand for some other uses, such as engine sand, which is used wherever trains and trolley cars run. Business acceleration increases the output and use of all kinds of sand. The value of all the sand and gravel produced in 1915 of which the United States Geological Survey has a record was over $23,000,000. The statistics for 1916 are not yet available (April, 1917).

"The glass-making industry is in no way dependent on foreign supplies for its sand, salt cake, soda ash, and limestone. Our resources in these materials are ample. Most of the output of glass sand comes from a belt of states extending from New Jersey to Missouri.

"The output in 1916 approximated 2,000,000 tons and was thus larger than it has ever been. On account of the higher wages and the increased cost of machinery and of all supplies, particularly coal, the price of glass sand, which has been decreasing in the last few years, showed an upward tendency in 1916."

The new requirements of armament and munitions have caused foundries and machine shops to increase their output, and this activity is in turn reflected in the molding-sand industry. The total production in 1915 was 3,500,000 tons, more than two-thirds of which came from New Jersey, New York, Pennsylvania, and Ohio. This was 750,000 tons more than the output of 1914. The statistics for 1916 are not yet available (April, 1917), but it is confidently expected that they will show 4,500,000 tons produced in that year.

Sand suitable for grinding and polishing, ranging from small gravel for use in sand-blast work on heavy castings to the fine material used for giving a polish, is abundant, and the country produces about 1,000,000 tons annually. Pennsylvania is the leading producer and in some years furnishes one-half of the output. The present activity in the production of armament will call for an increased production of this material, for it is the practice in large foundries to clean castings by sand-blast.

Asbestos. J. S. DILLER. Bulletin 666–H, 4 pp. The United States is one of the largest manufacturers of objects made from asbestos, but it is not a large producer of crude asbestos. The supply of asbestos of all grades in Quebec, Canada, is so large and so conveniently obtained and the quality of the Canadian asbestos is so excellent as to delay the development of asbestos deposits in the United States. The demand for high-grade asbestos has always been active, but recently, under the stimulus of war conditions abroad, it has become still greater, and the available supply, although larger than before, is frequently not equal to the demand. In 1916 the total output of asbestos in the United States was 1,479 short tons. The imports during the same time, almost wholly from Canada, amounted to 116,162 short tons, making a total supply available for manufacture in the United States of 117,641 tons.

"The prices for asbestos produced in the United States during 1916 are said to have ranged, according to grade, from $15 to $1,000 a ton. In 1915 normal prices ranged from $10 to $400 a ton.

With a view to increasing the available supply of asbestos, especially the spinning grades, which are so much in demand, all areas of peridotite and pyroxenite rocks more or less altered to serpentine should be prospected for cross-fiber veins of asbestos in which the fibers are three-fourths of an inch or more in length. The content of such fiber necessary to yield a profit may be less than 1 per cent of the whole rock quarried, and in obtaining it a much larger percentage of mill fiber will most probably be taken out.

Although the Arizona chrysotile fiber is excellent for certain purposes and finds a ready sale to those who need it for electric insulation, its mode of occurrence necessitates expensive operation and small production, and it should be mined with especial reference to avoiding waste and obtaining all within reach.

Talc and Soapstone. J. S. DILLER. Bulletin 666–I, 2 pp. The total domestic production is nearly twelve times the total imports, over one-third of which came from Canada. The talc of New York is an especially good paper filler and is largely used for that purpose, although much goes into paints. The output of Vermont is used for the same purpose and also for foundry facing, rubber goods, and coating walls. A small amount is used for crayons. Talc for pencils, crayons, burners, and insulators comes almost wholly from North Carolina and Georgia and is cut in Chattanooga. The demand for talc of the highest grade is greater than the supply. Much of the material for toilet powders is imported. With the exception of compact material for pencils, burners, and insulators and the best grade for toilet powders the United States has a large reserve in nearly all the producing States. It is believed that by more searching field investigations the domestic output even of pencil and gas-burner talc as well as the best toilet-powder grades may be greatly increased. The "foot-ease" grade of ground talc, now so extensively used in the Army, may be fully supplied in this country. Although the war has greatly stimulated production it has not greatly increased the price except in emergencies.

In 1916 there was a small production of soapstone in California, but almost the entire output of the United States came from Virginia, where there were four producing quarries. The total yield, including that of California, was 19,652 short tons. The United States has large reserves of soapstone and can, if necessary, greatly increase its output.

Phosphate Rock. R. W. STONE. Bulletin 666–J. Prior to 1914 the United States was producing annually close to 3,000,000 tons of phosphate rock, of which over 99 per cent came from Florida, Tennessee, and South Carolina. Florida produced more than 75 per cent of the total output, including the great bulk of the material exported, which was over 40 per cent of the total. With the beginning of the war the facilities for shipping phosphate rock to Europe were greatly decreased. Many Florida plants were shut down, and they have not resumed operations.

The proportion of exports to total production dropped from about 42 per cent in 1912 and 1913 to 35 per cent in 1914 and less than 14 per cent in 1915. In 1915 the total production was only 60 per cent of that in 1913. In 1916 the industry was in some areas practically demoralized, but there was nevertheless a gain over 1915. The total output in 1916 was 1,980,000 tons, valued at $5,897,000.

Any statement as to probable developments in the phosphate industry when peace is declared is largely conjecture. So long as the war continues phosphate rock cannot be sent to the large consumer, Germany, and high ocean freight rates greatly restrict and practically stop shipments to other European countries. Furthermore, the demand for sulfuric acid for use in making munitions has raised the prices of acid so high that manufacturers of acid phosphate have been obliged to curtail production. This has reduced the quantity of rock phosphate used by manufacturers of fertilizers and increased the quantity of rock ground for direct application to the soil. It seems reasonable to believe that at the end of the war European nations will want increased quantities of phosphate, for their stores of foodstuffs will be low and intensive cultivation of the soil will be necessary. The demand will again fall largely on the Florida and South Carolina deposits, which are close to the seacoast, and the phosphate industry in those States may then look for marked improvement.

Coal. C. E. Lesher. Bulletin 666–M, 7 pp. This separate is devoted to a discussion of commercial conditions, the character of coal application, and the causes of coal shortage. It is essentially an appeal to the consumers of coal to operate in reducing the likelihood of a coal shortage during the coming winter by proper purchase and storage of their fuel supplies at suitable times.

Mineral Production of the United States in 1915. Introduction by H. D. McCaskey. Summary by Martha B. Clark. From Mineral Resources of the United States, 1915, Part I, pp. 11*a*–95*a*. Published April 16, 1917. This is the summary section of the two volumes on Mineral Resources indicating by materials, years, and states, the statistics for production, import, export, and other features of interest. The total mineral production summarized amounted to $2,400,000,000 for 1915.

Cadmium in 1915. Production and Resources. C. E. Siebenthal. From Mineral Resources of the United States, 1915, Part I, pp. 979–981. Published April 30. In 1915 the metallic cadmium produced in the United States amounted to 91,400 lbs. valued at $108,000, an average of $1.19. During the same period 10,600 lbs. of cadmium sulfide valued at $10,000 were produced. The last reports from Germany (for 1913) indicate the metallic cadmium produced in that country, which is the only other producer of this metal in quantity, as 82,000 lbs. which that year was valued on an average of 78 cents per lb.

COMMERCE REPORTS—APRIL, 1917

Large **coal** deposits have been discovered in Iceland. (P. 14)

Coincident with a short lemon crop in Sicily, it has been found that the **lemon essence** had an abnormally high optical rotation. (P. 19)

A plant is to be established at Curaçao, Dutch West Indies, for the extraction of **tannin** from divi-divi; and of **dyes** from dyewoods. (P. 45)

A plant is to be erected in Norway for the production of fuel from **peat** and **wood waste** by the "Rosendable" method. (P. 175)

Two varieties of the lumbang nut are found in the Philippines, the "triloba," a hard nut, and the "trisperma" a soft nut. The oil of the soft nut dries more rapidly than that of the hard nut, and resembles **tung oil.** Some planting has been done, but in general the nuts are gathered from native trees, the industry being very poorly organized. (P. 216)

Efforts are being made to develop in Siberia such industries as **paper, leather, brick, iron,** etc. In several districts coal and iron deposits are in close proximity. (P. 232)

Efforts are being made in Australia to develop the manufacture of **paper** from "lalang grass," which grows over large areas, and resembles esparto. (P. 247)

Barytes deposits of Manchuria are now being developed. (P. 274)

The recent development of the Russian chemical industry is chiefly in the production of **heavy chemicals** and **raw materials,** especially for explosives and dyestuffs. (P. 281)

In a review of the **platinum** deposits of the world, by James M. Hill, the sources in Russia, Colombia, Brazil, Australia and the United States are described. (P. 298)

The Mersey Chemical Works, near Liverpool, built by the Badische Anilin und Soda Fabrik, has been purchased by an English firm to be used now for **explosives** and later for **dyes** and **intermediates.** (P. 321)

Bauxite is now being shipped from British Guiana to the United States. (P. 331)

An extensive deposit of **scheelite** has been discovered in New Zealand. (P. 361)

A marked shortage in the production of **kauri gum** in New Zealand is reported. (P. 365)

Brazil has become the principal source of **manganese** ore, of which it now exports annually 500,000 tons, compared with 122,000 tons in 1913. (P. 379)

In order to increase the available supply of **tin cans** for packing perishable foods, steps have been taken by the Department of Commerce and the manufacturers to increase the use of substitutes for tin, *e. g.*, paper, as containers for non-perishable goods. (P. 389)

Special Reports Issued in April

Spain—15*a*
Canada—23*a*
Honduras—31*a*
Bolivia—39*a*
British India—50*a*
China —52*a*
New Zealand—61*a*
British East Africa—65*a*

Statistics of Exports to the United States (Pp.)

Italy—184
Tartaric acid
Beeswax
Hides
Sienna
Umber
Pumice
Mercury
Soap
Soap stock
Talcum
Citrate of lime
Licorice
Sulfur

Colombia—278
Gold
Hides
Ipecac
Platinum
Rubber
Sugar
Mangrove extract

British India—Sup. 50*a*
Cashew nut
Castor seeds
Coconut oil
Copra
Indigo
Lemon grass oil
Nux vomica
Rubber
Senna
Hides
Turmeric

Spain—Sup. 15*a*
Copper ore
Almond oil
Olive oil
Iron oxide
Garnet
Lavender oil
Thyme oil

Canada—Sup. 23*a*
Coal and coke
Hides
Leather
Aluminum
Asbestos
Copper
Gold
Iron and Steel
Nickel
Silver

Honduras—Sup. 31*a*
Copra
Hides
Fustic
Rubber
Sarsaparilla

Bolivia—Sup. 39*a*
Tin
Tungsten
Copper
Antimony
Lead
Bismuth
Silver
Zinc
Gold
Molybdenum

China—Sup. 52*a*
Antimony
Albumen
Aniline dyes
Camphor
Cantharides
Gall nuts
Indigo paste
Licorice
Musk
Rhubarb
Sodium benzoate
Turmeric
Gold
Hides
Pig iron
Peanuts
Soya bean oil
Castor oil
Cottonseed oil
Peanut oil
Rapeseed oil
China wood oil
Silver
Vegetable tallow
Zinc ore
Aniseed oil
Cassia oil
Paper
Sugar
Tin

New Zealand—Sup. 61*a*
Copra
Hides
Kauri gum
Bullion

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MARKET REPORT—MAY, 1917

WHOLESALE PRICES PREVAILING IN THE NEW YORK MARKET ON MAY 20

INORGANIC CHEMICALS

Acetate of Lime,	100 Lbs.	4.50	@	4.55
Alum, lump ammonia	100 Lbs.	4.00	@	4.25
Aluminum Sulfate, high-grade	Ton	70.00	@	80.00
Ammonium Carbonate, domestic	Lb.	10½	@	11
Ammonium Chloride, white	Lb.	16½	@	17
Aqua Ammonia, 26°, drums	Lb	6	@	6¼
Arsenic, white	Lb.	17	@	17½
Barium Chloride	Ton	85.00	@	90.00
Barium Nitrate	Lb.	10½	@	11
Barytes, prime white, foreign	Ton	30.00	@	40.00
Bleaching Powder, 35 per cent	100 Lbs.	3.75	@	4.00
Blue Vitriol	Lb.	9½	@	10
Borax, crystals, in bags	Lb.	7¾	@	8½
Boric Acid, powdered crystals	Lb.	13	@	13½
Brimstone, crude, domestic	Long Ton	45.00		
Bromine, technical, bulk	Lb.	85	@	1.00
Calcium Chloride, lump, 70 to 75% fused	Ton	30.00	@	32.00
Caustic Soda, 76 per cent	Lb.	6.15	@	6.35
Chalk, light precipitated	Lb.	4½	@	4¾
China Clay, imported	Ton	18.00	@	30.00
Feldspar	Ton	8.00	@	15.00
Fuller's Earth, foreign, powdered	Ton	35.00		
Glauber's Salt, in bbls	100 Lbs.	65	@	70
Green Vitriol, bulk	100 Lbs.	1.05	@	1.10
Hydrochloric Acid, commercial, 18°	Lb.	1⅝	@	1¾
Hydrochloric Acid, C. P., conc., 22°	Lb.	1¾	@	2
Iodine, resublimed	Lb.	3.50	@	3.55
Lead Acetate, white crystals	Lb.	15	@	15¼
Lead Nitrate	Lb.	15½	@	16
Litharge, American	Lb	11	@	11½
Lithium Carbonate	Lb.	1.25		
Magnesium Carbonate, U. S. P.	Lb	24	@	26
Magnesite, "Calcined"	Ton	60.00	@	65.00
Nitric Acid, 36°	Lb.	6½	@	6¾
Nitric Acid 42°	Lb.	7½	@	8½
Phosphoric Acid, sp. gr. 1 710	Lb.	33	@	37
Phosphorus yellow	Lb.	1.15	@	1.25
Plaster of Paris	Bbl.	1.50	@	1.70
Potassium Bichromate, casks	Lb.	36	@	37
Potassium Bromide (granular)	100 Lbs.	1.00	@	1.10
Potassium Carbonate, calcined, 80 @ 85%	Lb.	48	@	70
Potassium Chlorate, crystals, spot	Lb.	57	@	60
Potassium Cyanide, bulk, 98–99 per cent	Mixture	2.15	@	2.20
Potassium Hydroxide, 88 @ 92%	Lb.	85	@	87
Potassium Iodide, bulk	Lb.	2.90		
Potassium Nitrate	Lb.	31	@	34
Potassium Permanganate, bulk	Lb.	3.85	@	4.00
Quicksilver, flask	75 lbs	105.00	@	106.00
Red Lead, American, dry	Lb.	11½	@	11
Salt Cake, glass makers'	Ton	18.00	@	20.00
Silver Nitrate	Oz.	46⅜		
Soapstone in bags	Ton	10.00	@	12.50
Soda Ash, 58%, in bags	100 Lbs.	2.80	@	2.90
Sodium Acetate	Lb.	8½	@	9½
Sodium Bicarbonate, domestic	100 Lbs.	2.10	@	2.25
Sodium Bichromate	Lb.	15½	@	16
Sodium Chlorate	Lb.	23½	@	24½
Sodium Fluoride, commercial	Lb.	12½	@	13½
Sodium Hyposulfite	100 Lbs.	1.85	@	2.00
Sodium Nitrate, 95 per cent, spot	100 Lbs.	4.05	@	4.15
Sodium Silicate, liquid, 40° Bé	100 Lbs.	1.25	@	1.50
Sodium Sulfide, 30%, crystals, in bbls	Lb.	2⅝	@	3
Sodium Bisulfite, powdered	Lb.	.05½	@	.05¾
Strontium Nitrate	Lb.	28	@	30
Sulfur, flowers, sublimed	100 Lbs.	3.05	@	3.45
Sulfur, roll	100 Lbs.	2.85	@	3.25
Sulfuric Acid, chamber, 66° Bé	Ton	30.00	@	32.00
Sulfuric Acid, oleum (fuming)	Ton	40.00	@	42.00
Talc, American white	Ton	10.00	@	12.00
Terra Alba, American, No. 1	100 Lbs.	85	@	90
Tin Bichloride, 50°	100 Lbs.	17.50		
Tin Oxide	Lb.	65	@	66
White Lead, American, dry	Lb.	10	@	10½
Zinc Carbonate	Lb.	25	@	27
Zinc Chloride, commercial	Lb.	16	@	17
Zinc Oxide, American process XX	Lb.	13	@	14

ORGANIC CHEMICALS

Acetanilid, C. P., in bbls	Lb.	54	@	56
Acetic Acid, 56 per cent, in bbls	Lb.	9½	@	10
Acetic Acid, glacial, 99½%, in carboys	Lb.	28	@	30
Acetone, drums	Lb.	27	@	29
Alcohol, denatured, 180 proof	Gal.	72	@	74
Alcohol, grain, 188 proof	Gal.	3.04	@	3.06
Alcohol, wood, 95 per cent, refined	Gal.	1.00	@	1.02
Amyl Acetate	Gal.	3.60	@	3.80
Aniline Oil	Lb.	30	@	30½
Benzoic Acid, ex-toluol	Lb.	6.75	@	7.50
Benzol, 90 per cent	Gal.	59	@	60
Camphor, refined in bulk, bbls	Lb.	89	@	90
Carbolic Acid, U. S. P., crystals, drums	Lb.	42	@	44
Carbon Bisulfide	Lb.	6½	@	7
Carbon Tetrachloride, drums, 100 gals	Lb.	16	@	17
Chloroform	Lb.	60	@	61
Citric Acid, domestic, crystals	Lb.	74	@	75
Creosote, beechwood	Lb.	1.90	@	2.00
Cresol, U. S. P.	Gal.	1.25	@	1.30
Dextrine, corn (carloads, bags)	100 Lbs.	6.05	@	6.10
Dextrine, imported potato	Lb.	15½	@	16
Ether, U. S. P., 1900	Lb.	23	@	30
Formaldehyde, 40 per cent	Lb.	17	@	18
Glycerine, dynamite, drums included	Lb.	56½	@	60
Oxalic Acid, in casks	Lb.	45	@	47
Pyrogallic Acid, resublimed bulk	Lb.	3.25		
Salicylic Acid	Lb.	1.10	@	1.25
Starch, cassava	Lb.	—		
Starch, corn (carloads, bags) pearl	100 Lbs.	4.75	@	4.80
Starch, potato	Lb.	13¾	@	15
Starch, rice	Lb.	10¼	@	11
Flour, sago	Lb.	6	@	6½
Starch, wheat	Lb.	5¾	@	6½
Tannic Acid, commercial	Lb.	45	@	50
Tartaric Acid, crystals	Lb.	79	@	80

OILS, WAXES, ETC.

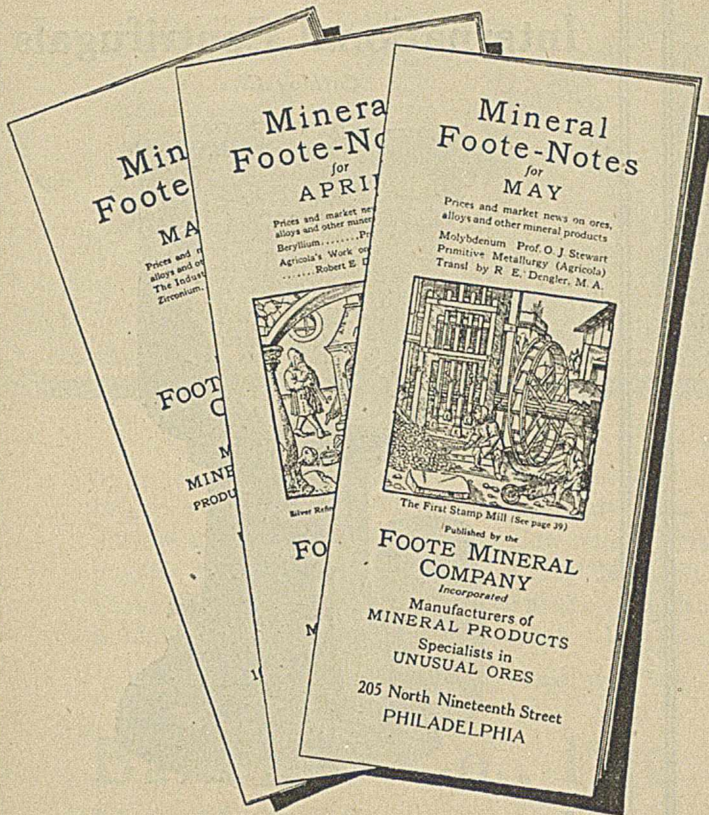
Beeswax, pure, white	Lb.	55	@	60
Black Mineral Oil, 29 gravity	Gal.	13½	@	14
Castor Oil, No. 3	Lb.	24½	@	25
Ceresin, yellow	Lb.	16	@	19
Corn Oil, crude	100 Lbs.	15¾	@	16
Cottonseed Oil, crude, f. o. b. mill	Gal.	1.10	@	1.11
Cottonseed Oil, p. s. y.	Lb.	16¼	@	16½
Menhaden Oil, crude (southern)	Gal.	80		
Neat's-foot Oil, 20°	Gal.	1.60	@	1.65
Paraffine, crude, 118 to 120 m. p.	Lb.	7¼	@	7½
Paraffine Oil, high viscosity	Gal.	29½	@	30
Rosin, "F" Grade, 280 lbs.	Bbl.	6.50	@	—
Rosin Oil, first run	Gal.	37		
Shellac, T. N.	Lb.	57	@	58
Spermaceti, cake	Lb.	25		
Sperm Oil, bleached winter, 38°	Gal.	nominal		
Spindle Oil, No. 200	Gal.	24	@	25
Stearic Acid, double-pressed	Lb.	23	@	23½
Tallow, acidless	Gal.	1.40	@	nominal
Tar Oil, distilled	Gal.	30	@	32
Turpentine, spirits of	Gal.	48	@	48½

METALS

Aluminum, No. 1, ingots	Lb.	59	@	61
Antimony, ordinary	Lb.	26	@	27
Bismuth, N. Y.	Lb.	3.00	@	3.10
Copper, electrolytic	Lb.	32		
Copper, lake	Lb.	32		
Lead, N. Y.	100 Lbs.	10	@	10½
Nickel, electrolytic	Lb.	55	@	56
Platinum, refined, soft	Oz.	105.00		
Silver	Oz.	74⅞		
Tin, Straits	Lb.	66	@	66½
Tungsten (WO_3)	Per Unit	17.00	@	18.00
Zinc, N. Y.	100 Lbs.	9	@	9½

FERTILIZER MATERIALS

Ammonium Sulfate	100 Lbs.	5.75	@	6.00
Blood, dried f. o. b. Chicago	Unit	4.85		
Bone, 4 and 50, ground, raw	Ton	32.00	@	38.00
Calcium Cyanamid	Unit of Ammonia	—		
Calcium Nitrate, Norwegian	100 Lbs.	—		
Castor Meal	Unit	—		
Fish Scrap, domestic, dried, f. o. b. works	Unit	5.00	@	10
Phosphate, acid, 16 per cent	Ton	14	@	15
Phosphate rock; f. o. b. mine:				
Florida land pebble, 68 per cent	Ton	2.00	@	2.25
Tennessee, 78–80 per cent	Ton	5.00	@	5.50
Potassium "muriate," basis 80 per cent	Ton	380.00	@	400.00
Pyrites, furnace size, imported	Unit	nominal		
Tankage, high-grade, f. o. b. Chicago	Unit	4.85	&	10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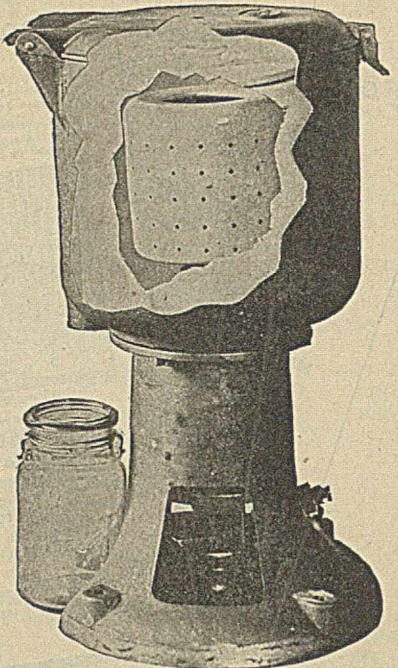

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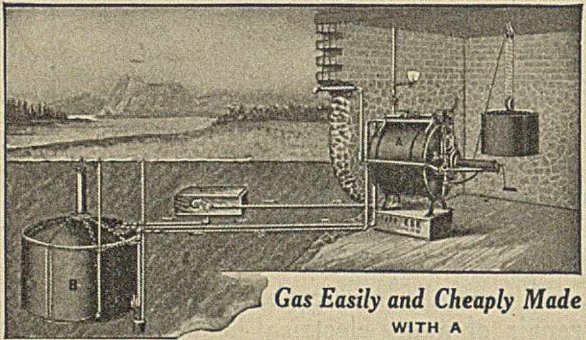

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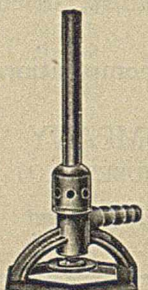

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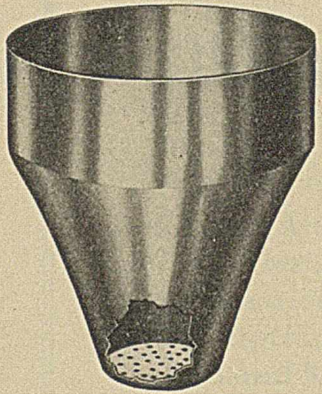

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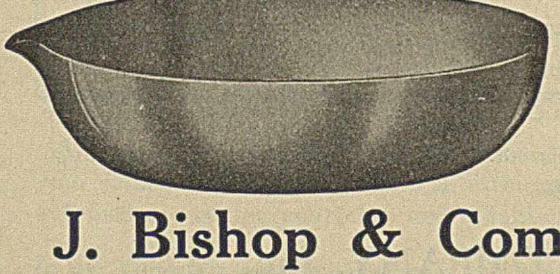

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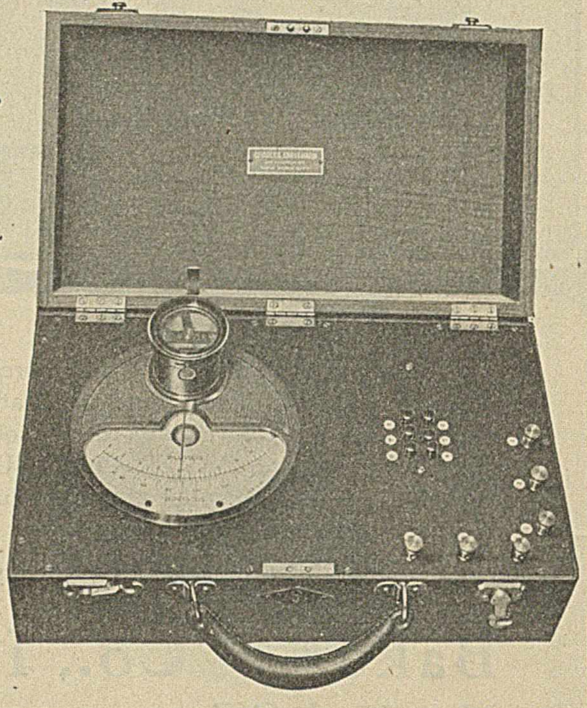

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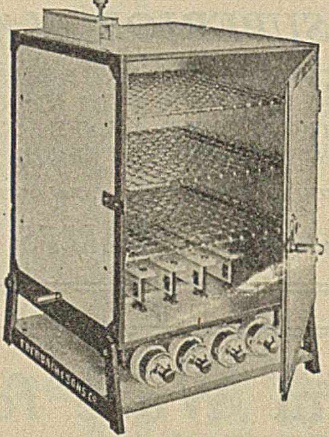

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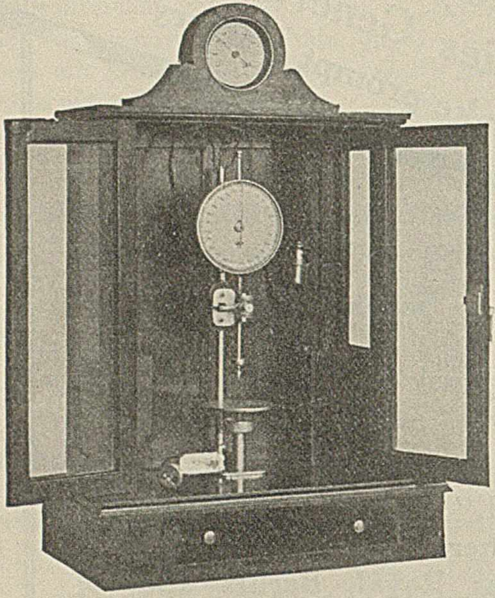

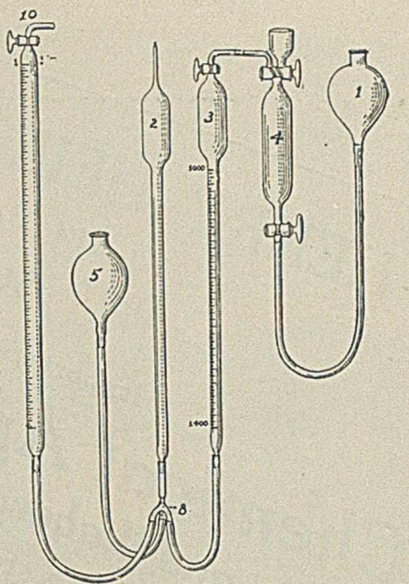
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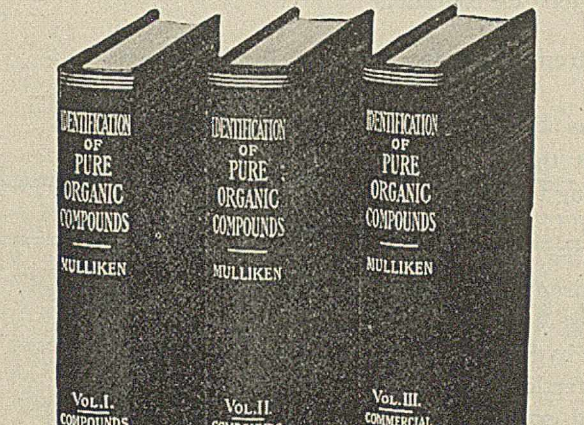
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Platinum Substitute

Crucibles, Evaporating Dishes, Dishes for Sugar, Water and Milk Analysis, also Cones, Electrodes, Spatulas, Tongs, Triangles, etc., made from Palladium Gold alloy, recommended by the Bureau of Standards, and sold under the trade name "Palau."

For laboratory utensils it is superior to platinum ware, and **costs** but **half as much.** It is made in all standard shapes and sizes, and has been used for the past two years by prominent chemists in daily laboratory routine, in varied lines of work including metallurgical and industrial, having been found suited to their requirements.

PALAU WARE has a melting point of 1370° Centigrade and is guaranteed to withstand continuously 1300° Centigrade.

We quote herewith a summary from the report of the Bureau of Standards on Palau Ware:

> "These tests indicate that the 'PALAU' crucible, as far as resistance to loss on heating to 1200° C., is superior to platinum containing about 2.4% iridium, and somewhat inferior to platinum containing about 0.6% iridium. The comparative freedom from iron is an improvement over most platinum ware now in use. It compares favorably with platinum in resistance to hydrochloric, nitric, hydrofluoric, and sulphuric acids, ferric chloride and sodium hydroxide solutions, and fused sodium carbonate. The 'PALAU' crucible is not as suitable for potassium pyrosulphate fusions as platinum. For fusions with sodium carbonate and sodium nitrate the 'PALAU' crucible is less attacked than platinum, but both wares are very seriously attacked under the conditions of our test, the fusion mixture containing no oxidizable material."

We have a considerable stock of this ware on hand, so that we are in position to make prompt shipments.

Write for illustrated catalog, showing the different styles, specifying also sizes and approximate weights.

EIMER AND AMEND

ESTABLISHED 1851

Third Avenue, 18th to 19th Streets, New York City

Branch Offices and Showrooms:

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