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ST. LOUIS MEETING, AMERICAN CHEMICAL SOCIETY

DIVISIONS TO THE FRONT

Each General Meeting of the AMERICAN CHEMICAL SOCIETY has its distinctive note. The 50th Meeting, held at St. Louis, Mo., April 12 to 17, 1920, was no exception in this respect, for above all else there stood out the strength of the divisional and sectional meetings, with their strong programs, fine attendance, and spirited discussions following the presentation of the papers.

We interpret this as the return of the chemist to his normal surroundings and his resumption of problems interrupted by the call of war service. That service was thoroughly performed and its problems have been laid aside, but it is evident that the intense stimulation of the war period is being carried over into the days of peace, hence the rich outpouring of papers for the divisional programs.

The meeting was notably successful. The registration of 976 is an index of the general interest, for while there have been meetings more largely attended, nevertheless the present one was held at a point quite distant from the SOCIETY'S center of membership, and during the week when the uncertainties of travel weighed heavily against the promptings of even the most enthusiastic of attendants of meetings. The classification of the attendance by states shows how widely representative was the meeting.

ATTENDANCE CLASSIFIED BY STATES

Arkansas.....	3	New Jersey.....	21
California.....	6	New York.....	54
Colorado.....	4	North Carolina.....	1
Connecticut.....	2	Ohio.....	80
Delaware.....	18	Oklahoma.....	10
District of Columbia.....	26	Pennsylvania.....	31
Georgia.....	6	Rhode Island.....	1
Illinois.....	152	South Dakota.....	2
Indiana.....	26	Tennessee.....	3
Iowa.....	17	Texas.....	9
Kansas.....	19	Utah.....	4
Kentucky.....	5	Virginia.....	2
Louisiana.....	5	Wisconsin.....	28
Maryland.....	4	Wyoming.....	1
Massachusetts.....	20	China.....	1
Michigan.....	16	Canada.....	4
Minnesota.....	9	Mexico.....	1
Mississippi.....	2	Holland.....	1
Missouri.....	372	Japan.....	1
Nebraska.....	9		
		TOTAL.....	976

The arrangements for the meeting were in the hands of an excellent local committee headed by Dr. Charles E. Caspari. Even the local weather bureau was induced to do its part towards making the meeting a success, and it responded nobly.

Two new sections held their inaugural meetings, the Sugar Section and the Leather Section, while the Dye Section assumed a place as a division of the SOCIETY. The christening of these youngest children of the parent organization was auspicious, and their programs gave proof of the sound judgment of those who had worked for their creation.

An interesting development was the program of the Rubber Division. A marked tendency to "open up" was noticeable, and the process proved delightful. It is believed that the fine example set by one of the chief laboratories in this respect marks the beginning of a new day for that division.

So, too, in the Dye Division the proportion of scientific

papers to general discussion showed a marked increase, and gave promise of future programs which will attract the chemists of the dye laboratories.

Unusual interest attached to the joint symposium on colloids, the attendance at this meeting equaling that at the general opening meeting.

To confirm our judgment as to the marked excellence of the divisional and sectional meetings, we assumed the role of a reporter and secured from the several officers the following statements:

INDUSTRIAL AND ENGINEERING CHEMISTRY DIVISION

The attendance at the meetings of this division and the discussion brought forth by the papers presented may be taken as indicative of the intense industrial interest in the division, notably so in the case of the ideas brought forth in estimating the economic status of the chemist and the high ideas the manufacturers have for their requirements of the chemist's education, and the incentives they offer to research in pure as well as applied chemistry.

H. D. BATCHELOR, *Chairman*

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BIOLOGICAL CHEMISTRY DIVISION

This division completed its program Wednesday by remaining in session until 6:20 P.M., in order that the members might attend the colloid symposium Thursday morning. We had a splendid attendance, standing room only being available for part of the time. The point which stands out most prominently is the fundamental studies which are being made in the field of biological chemistry. The papers presented were not merely reports of general observations, but nearly all of them went down into the deeper fields of the reasons underlying biological phenomena. The most important papers were perhaps those dealing with the vitamine question, and I feel that very important results were accomplished.

R. A. GORTNER, *Chairman*

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SUGAR SECTION

The first meeting of the Sugar Section was attended by about 60 enthusiastic members representing various branches of the sugar industry in all sections of the country. Great interest was manifested in the papers which related to the general topics of saccharimeters, beet sugar chemical control and decolorizing carbons. The sugar chemists spoke of the great benefit of having organized as a section of the AMERICAN CHEMICAL SOCIETY. From the opinions of members present and absent it is believed that the attendance and program of the next meeting will be more than doubled.

C. A. BROWNE, *Chairman*

.....

LEATHER CHEMISTRY SECTION

The first meeting of this section was gratifying in the number who attended and the quality and scope of the discussion of the papers presented. Many chemists working along allied lines were attracted to the meetings, and took active part in the discussions, showing that the field which has just been plowed and sown will undoubtedly bear the fruit of success.

LOUIS E. LEVI, *Chairman*

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AGRICULTURAL AND FOOD CHEMISTRY DIVISION

The meeting of this division was particularly well attended and the papers presented were both interesting and important. Perhaps the most significant feature of the meeting was the earnest and intelligent discussion which each of the papers brought forth, and which was equally as valuable to the author as to the division. Of the fifteen papers on the program, twelve were actually read; the railroad conditions having prevented several members from reaching St. Louis. These conditions also prevented several papers from reaching the secretary in time to be placed on the printed program, so, according to the rules of the SOCIETY, they could not be read at this meeting. The general opinion was that the divisional meeting was in every way successful.

C. E. COATES, *Chairman*

ORGANIC CHEMISTRY DIVISION

A successful session showing the present interest in the making of organic substances, reflecting the great commercial activity in dyes, pharmaceuticals, and other organic products, was held. Organic chemistry is the field in which we have been furthest from national independence, but great strides are now being made in bringing organic research and applied organic chemistry together.

E. EMMET REID, *Chairman*

RUBBER DIVISION

Less and less secrecy is being maintained by the different companies regarding their research work in the development of rubber compounding. The meetings are getting to be of increasing interest as this move gains favor. The division expressed its disapproval of the marketing of compounding ingredients and accelerators under trade names, such compounds being of a poorer grade than standard or mixed with other cheaper material. The composition of these materials will be exposed to the public. The necessity for cooperative research in the rubber industry becomes more and more apparent. The chemists are really starting to get together on this.

ARNOLD H. SMITH, *Secretary*

PHYSICAL AND INORGANIC CHEMISTRY DIVISION

The papers and discussions in this division seemed to divide into three main subjects: chemical reactivity and catalysis, the structure of the atom, and colloids. New studies bearing on the quantum theory seem to indicate that it is possible to calculate the speed of reactions from measurement of energy given off from atoms. It was suggested that all chemical activity may be caused by radiation. Announcement was made of the discovery of meta-chlorine, and the indication therefrom of the existence of a whole new species of atoms. The colloid symposium, arranged jointly by several divisions, aroused so great interest that the general meeting room was filled to overflowing. The papers presented brought out the practical applications of colloidal chemistry to a variety of industries.

W. D. HARKINS, *Chairman*

DYE SECTION

The meeting was very successful and the great interest shown fully justified the Council's action in raising the youthful section to the dignity of a division. Only three of the twenty-one papers were read by title. An account of the dye patents of the Chemical Foundation was very elucidating as to the methods by which licenses can be obtained. The use which will be made of the earnings of the Foundation in fostering chemical research was brought out. A paper on dye research also created considerable interest and discussion. Among other interesting papers was one on the physical constants of aniline, bringing out particularly its hygroscopic quality. The number and quality of the papers from the Color Laboratory of the Bureau of Chemistry of the Department of Agriculture added much to the interest of the session. The members of the section seemed filled with enthusiasm and awake to their responsibilities. Keen interest was shown in the legislative situation affecting the industry represented, and a universal hope for its early enactment. The resolutions passed by the Council were read to the division, and the technical progress made in this country was reviewed.

CHAS. L. REESE, *Chairman*

WATER, SEWAGE AND SANITATION DIVISION

The meeting of this division was very well attended, the program was carried out in full, and the various papers evoked considerable discussion. Dr. Bartow's report of the Sewage Disposal Committee of the National Research Council contained a great deal of interesting material. It is expected that at the Chicago meeting the further report of this work will be one of the most important features of the program. Some of the tests are to be conducted in the vicinity of Chicago, and will probably be ready for inspection in September. The matter of activated sludge and other methods proposed for the treatment of the sewage, especially the effluents from industrial plants, is a matter of extreme public importance, both from the standpoint of health and the general improvement of community conditions. The paper outlining the study of sewage and trade wastes at Bridgeport, Conn., furnished an interesting basis for this latter discussion.

W. W. SKINNER, *Secretary*

PHARMACEUTICAL CHEMISTRY DIVISION

Our meeting was well attended, the most important discussion centering around the subject of the stability, chemistry, and

pharmacology of chloramine antiseptics. New work on hypnotics and anesthetics was reported. The division made important recommendations to be laid before the SOCIETY'S committee on the revision of the forthcoming pharmacopeia at the decennial convention. Steps were taken toward the changing of the name of the division to that of Chemistry of Medicinal Products Division. It is felt that this is a less restricted term, and will place the accent on chemistry rather than pharmacy.

CHAS. E. CASPARI, *Chairman*

MATTERS OF BUSINESS

In spite of the difficulties incident to travel during the week of the railroad strike, 97 members of the Council, some more or less delayed, assembled at the University Club, President Noyes presiding. The proceedings of the lengthy session were broken by the delightful dinner served in the rooms of the Club.

Great interest attached to the many valuable reports of committees which were summarized by the President. Six of these reports are printed on pages 438 to 443 of this issue. By ballot of the Council vacancies on the Committee on National Policies (Advisory Committee) were filled. Dr. H. E. Howe will take the place of Dr. M. C. Whitaker, who was ineligible for reelection, and Dr. George D. Rosengarten will fill the unexpired term of Dr. B. C. Hesse, resigned. This committee was enlarged by the addition of two *ex-officio* members, the editors of the *Journal of the American Chemical Society* and the *Journal of Industrial and Engineering Chemistry*. Dr. A. V. H. Mory was elected councilor-at-large to fill the vacancy caused by the election of Dr. W. A. Noyes to the presidency.

Much interest attached to the selection of the place for holding the next Spring Meeting. The invitation of the Rochester Section was accepted. An invitation from the Pittsburgh Section for the Annual Meeting of 1921 was under the constitution laid on the table. After prolonged discussion and a statement of the preference of the members of the Chicago Section it was voted that it would be inadvisable to hold the 1920 Annual Meeting during convocation week with the A. A. A. S., the Council fixing the second week in September as the time for the Annual Meeting.

Dr. Charles L. Parsons was appointed to represent the SOCIETY at the International Union of Pure and Applied Chemistry in Rome in June 1920.

A measure of international significance was the acceptance by the SOCIETY of the invitation of the American Metric, the London and Manchester Decimal Associations, and the World Trade Club to join in forming a World Metric Standardization Council, and calling a conference in San Francisco in May 1920.

Continued thefts of platinum in various laboratories gave rise to serious consideration of the question of possibly overcoming this by congressional legislation. A committee was appointed to consider carefully this question.

The question of the difficulties attendant upon the manufacture, sale, and use of stills under the Bureau of Internal Revenue regulations was brought to the attention of the Council in a forcible manner by Mr. H. J. Cary-Curr, of Chicago. A committee was appointed to confer with the officers of the Bureau and

to formulate suggestions which might be acceptable to the Bureau and give the needed relief.

The following resolution was unanimously adopted by the Council and ordered telegraphed to members of the Senate Finance Committee.

THE AMERICAN CHEMICAL SOCIETY through its representative Council urges upon our Senators the necessity of prompt passage of the Longworth Bill in the form reported by the Senate Finance Committee. Under the absolute protection afforded by the blockade of German ports and encouraged by the favorable legislation of the preceding Congress the coal-tar chemical industry has grown by leaps and bounds. But to meet the unusual competition threatened from the present unified German industry far greater safeguards must be established than were formerly considered necessary. The delay in enacting such legislation has caused the withholding of large amounts of capital and the creation of an atmosphere demoralizing to research and to the technical development of the industry. We feel that the fate of this industry lies now with the United States Senate and we urge the enactment of the bill as originally reported to the Senate by its Finance Committee, convinced that this legislative support will speed the industry's complete development, thereby assuring American consuming interests freedom from outside domination and providing a powerful factor in national defense.

The question of inadequate pay of professors in the universities, of the injustice of the situation, in view of the high cost of living, and of the danger involved to the whole structure of chemical education was given serious and earnest consideration by the Council. As a result of this discussion a committee was appointed to draft resolutions to be presented to official bodies.

The full proceedings of the Council Meeting are printed in the May issue of the *Journal of the American Chemical Society*.

At a well attended session of the Directors of the SOCIETY two matters were approved which affect to a marked degree the work of the *Journal of Industrial and Engineering Chemistry*. It was voted to appoint the Chemical Catalog Company, Inc., 1 Madison Avenue (Metropolitan Tower, 17th Floor), New York City, to conduct the advertising business of the SOCIETY, under the direction of the Editor of the *Journal of Industrial and Engineering Chemistry*. This arrangement was approved as of April 1, 1920. This company has a strong staff, a wide touch with the chemical industries, and has proved its ability to make a success of its undertakings. It is felt that under this new policy the power of the Journals of the SOCIETY as advertising mediums will be largely increased.

The Directors approved the leasing of space in the Metropolitan Building, 1 Madison Avenue, New York City, for the editorial headquarters of THIS JOURNAL. This change was necessitated by the exorbitant increase in rent at the old location. The move to the new quarters on the thirty-fourth floor of the tower was effected during the past week.

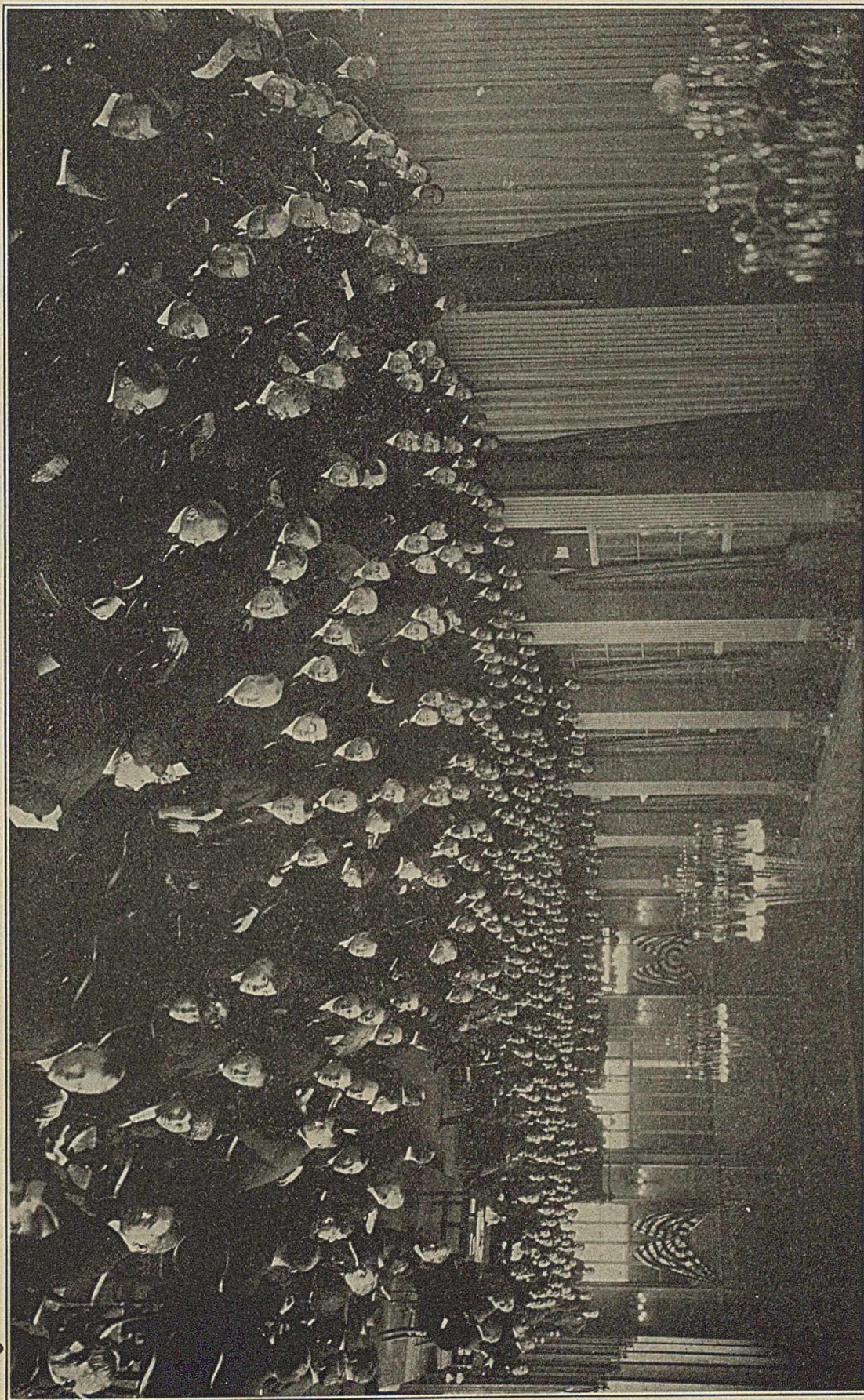
GENERAL MEETING

On Tuesday morning, April 13, the large ballroom of the Hotel Statler was filled with members for the opening general session. Dr. Charles E. Caspari, Chairman of the Local Executive Committee, presided, and introduced Mayor Henry W. Kiel, who extended a cordial welcome to the SOCIETY and expressed his best wishes for a successful meeting. In responding, President Noyes touched upon a vital situation in the life of our science, speaking in part as follows:

It is with very great pleasure that we meet in St. Louis at this time. We remember the long and honorable record of your city in matters of education and the cultivation of the sciences. We remember that here you have the great Washington University

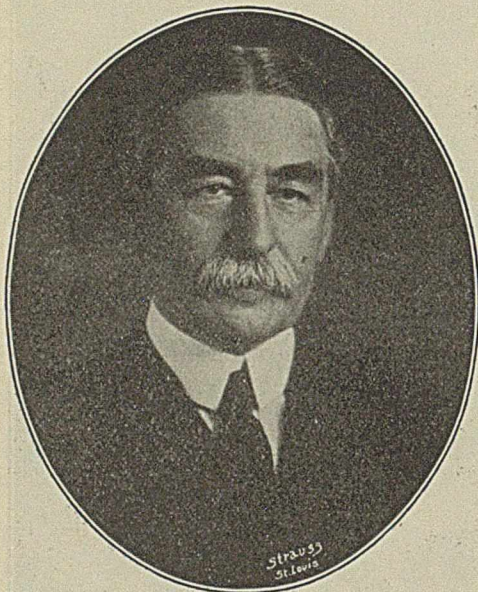
and Medical School. The Missouri Botanical Gardens have made the name of St. Louis famed to the botanists of the world, and we remember many other ways in which St. Louis has fostered education and science. Not least among these, some of us remember with the greatest pleasure the meeting of the chemists in this city seventeen years ago, at the time of the Exposition, which was unique among the expositions of the world, the Congress of Arts and Sciences. In our own science there were gathered such men as Arrhenius, Ramsay, van't Hoff, Moissan, Ostwald, and Sir Ernest Rutherford, six or seven chemists whose names at that time could not be equaled with another six or seven in the whole world, and St. Louis gathered those men here in the Congress of Arts and Sciences, and many of us are very grateful for what she did at that time, so it is with pleasure that we meet in St. Louis now. On our side we are thankful for the growth of this SOCIETY, now numbering, our Secretary tells me, over 15,000 chemists, an organization that is not equaled in its character and numbers by any other scientific organization in the whole world—an organization which is nearly more than twice in number that of any other organization of chemists in the world. When our Secretary assumed office only thirteen years ago we had a budget of perhaps \$30,000, I have forgotten the exact numbers, and a membership of only 3,000; now the annual budget is more than \$200,000. And we are now as chemists standing almost at the parting of the ways in our science in America. Chemistry is going forward into new achievement as never before in the history of our science in this country, almost as never before in the world, and this SOCIETY of ours is the organization upon which devolves the duty of helping in this tremendous development which is upon us. Yesterday afternoon and evening some of us worked for some six or eight hours upon the business of the SOCIETY. Many important things were done: I could not this morning, if I would, adequately summarize the work of that meeting and of literally dozens of men who have been at work upon our committees and have brought to us reports of great value, merely summarized in that meeting last night. Our SOCIETY and its Council took important action in various directions. There is only one, however, that I will mention, because it is most vital to our profession at the present time. This was the action urging upon presidents, boards of trustees and all others who support educational institutions to provide adequate compensation for the teachers of chemistry in our colleges and universities. Many of us in that work know that the present time is critical in the education of chemists, and we know that that education lies at the very foundation of the development which must come in chemistry in America if we would not fail in the work that should be done, and so it is of the greatest importance that the present tendency of our best young men to go into the industries should be checked in some way or other, if we are to have a solid foundation for the future. And more than that, another item which was not emphasized so much in the Council Meeting, one of the pitiful situations in the teaching profession at the present time is the fact that so many of our young men who go into teaching are in their younger years so overloaded with a heavy burden of teaching that research and professional growth are almost impossible. That is almost more serious than the financial difficulty. If our young men have to face both lack of time for research and an inadequate salary, the future of chemical education in America is in a very sad way.

The presiding officer then turned the chair over to President Noyes, and Secretary Parsons presented the nomination by the Council of Ernest Solvay, of Belgium, for honorary membership in the SOCIETY, and a unanimous election resulted. A memorial on the death of Professor Alfred Werner, of Zurich, honorary member of the SOCIETY, was presented and adopted.



OPENING SESSION, 59TH MEETING OF THE AMERICAN CHEMICAL SOCIETY, HOTEL STANTLER, ST. LOUIS, MO., APRIL 13, 1920

It was announced that owing to the legislative situation in Washington, Hon. E. P. Costigan, of the Tariff Commission, had been forced to cancel his engagement to speak. At the last moment the local committee



EDWARD MALLINCKRODT, SR., HONORARY CHAIRMAN, EXECUTIVE COMMITTEE

was fortunate in securing an address by Mr. Paul W. Brown, editor of *America at Work*, who spoke on "The Physical Basis for the Economic Development of the Mississippi Valley." While the subject was an unusual one for an address before the AMERICAN CHEMICAL SOCIETY, the delightful method of presentation held the close attention of the audience. Following Mr. Brown an address was made by the editor of the *Journal of Industrial and Engineering Chemistry*, which is printed on page 429 of this issue.

An interesting and suggestive development of the St. Louis meeting was an informal gathering of all chairmen and secretaries of local sections present, twenty-eight in number. It was felt that the local sections have so many problems in common that a conference of these officers at each General Meeting would bring light upon these problems from many different angles, and that from a joint exchange of experiences in the conduct of local sections valuable points could be gained for furthering this important feature of the SOCIETY'S organization. As a result of the deliberations of this gathering a recommendation was made to the Council that there be formed a section of Local Section Officers, to hold one session at each General Meeting. Pending formal action by the Council on this matter provisional officers were elected: Professor R. H. McKee, of the New York Section, Chairman, and Dr. Edgar B. Carter, of the Indianapolis Section, Secretary, to formulate plans for such a conference at the Chicago meeting.

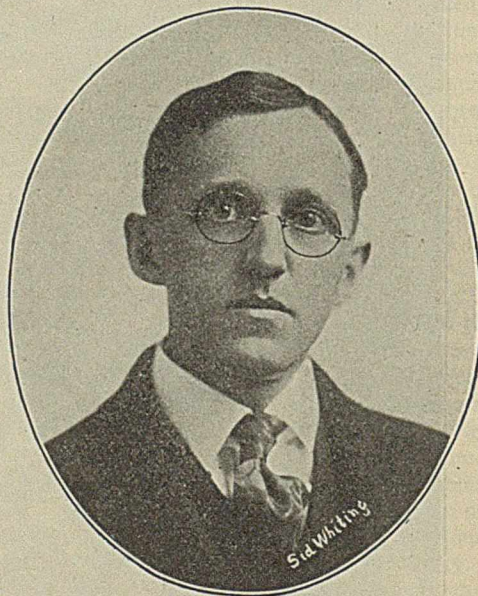
On Wednesday evening in the auditorium of the Central High School an address on "Chemical Warfare" was given before a large and enthusiastic audience by Lt. Col. Amos A. Fries, head of the Chemical War-

fare Service. Colonel Fries brought to the subject many new points of view and his narration of experiences at the Front during the war gripped his audience in deeply sympathetic interest. Colonel Fries paid glowing tribute to individual chemists who had served under him in the trying days of the formation of the Service. At the conclusion of his address graphic moving pictures of gas warfare were exhibited, which showed more clearly than words could tell the actual experiences of the men at the Front.

ENTERTAINMENTS AND EXCURSIONS

On Tuesday evening a delightful smoker was enjoyed by more than 800 chemists. On Thursday evening the banquet was held in the Hotel Statler ballroom, at which Mr. A. C. Boylston, of St. Louis, was toastmaster.

Following the dinner an address was made by Mr. W. Frank Carter, president of the St. Louis Chamber of Commerce, who spoke of St. Louis as a chemical center, pointing out that the chemical industry was second only to the automobile industry in the matter of its expansion during the last few years. Mr. Carter was followed by Chancellor Frederic A. Hall, of Washington University, who spoke of the high ideals which give dignity to the work of the scientist. President Noyes, representing the AMERICAN CHEMICAL SOCIETY, spoke in sincere appreciation of the many hospitalities shown by the sections acting as hosts, and emphasized the necessity of carrying high ideals into one's scientific work if his science is to fulfill its real mission. Brief



T. R. BALL, CHAIRMAN, REGISTRATION AND INFORMATION COMMITTEE

addresses were made by L. V. Redman, Chas. L. Reese, W. D. Bancroft, R. H. McKee, and Ellwood Hendrick, epitomizing the spirit of the various occasions of a highly successful week. In conclusion Dr. Caspari paid a graceful tribute to his co-workers in the various local committees, giving credit to them for the success with which the plans for the meeting had been carried out.

The gathering afforded opportunity for the getting together of various groups of members with common interests, notably reunions of chemical alumni of some of the larger universities. The Alpha Chi Sigma chemical fraternity dinner brought out an attendance of 104 members on Wednesday evening preceding Colonel Fries' address. The members of the American Institute of Chemical Engineers, of whom some forty were present, were hospitably entertained at dinner by Dr. F. W. Frerichs at his home.



L. McMASTER, HEAD OF CHEMISTRY DEPARTMENT, WASHINGTON UNIVERSITY, ST. LOUIS, MO.

A not unusual but always enjoyable feature of the week's program was the excursions of Thursday afternoon and Friday, notably to the LaCleve Gas Works, Monsanto Chemical Works, LaCleve-Christy Clay Products Plant, the Standard Oil Refinery, at Wood River, Ill., and the Illinois Glass Company, at Alton, Ill. A delightful luncheon was served at the Mineral Springs Hotel, Alton, by courtesy of the St. Louis Section.

ADDRESSES

INTRODUCTORY ADDRESS

By W. A. Noyes

Two years ago our Army in France was dependent, among other things, on what the chemists of America could do and upon the men in France who were using the materials the chemists in America could make for them; and some of us chemists have not realized perhaps as we should how far we were dependent upon the men who knew how to use those supplies in the right way in military affairs. I know from friends who were very close to Colonel Fries in the war that the Chemical Warfare Service in France was exceedingly fortunate when he was selected as its head, and we may count ourselves fortunate here in America that he is now the head of the Chemical Warfare Service of the United States. It is our great good fortune to have with us to-night the man who was at the center of things and who did things in France. He will speak to you.

CHEMICAL WARFARE

By Amos A. Fries¹

LT. COL., U. S. ARMY, CHIEF OF CHEMICAL WARFARE SERVICE

The old adage that "There is nothing new under the sun" is often quoted by those who would detract from the glory of a new achievement or condone the evil deeds of those who have broken the faith. The Germans did not invent poisonous gases, nor did they invent the earliest methods used in gas attacks. Neither did the Wrights invent flying, nor the basic methods used in their first flights—but to their everlasting glory they gleaned from a thousand pages the thoughts of many men in many countries, and by applying the knowledge thus acquired made flying a reality. The Germans gleaned from many lands the possibilities of poisonous gases, together with methods of using them—and to their everlasting shame broke faith with the countries then in the war, and on April 22, 1915, launched the first really effective gas attack in the history of the world.

But whatever we may think of that first attack, chemical warfare is to-day an established fact. No modern nation dares to give it up, and ere long no modern nation will want to give it up unless indeed all war can be abolished. But that day, unfortunately, is too far distant for me to take up any of your time to-night talking about it.

I am not going to discuss the chemistry of chemical warfare. I am simply going to tell you what chemical warfare is, tell you what it did in the World War, tell you something of the great part played by the chemists in that war, and, lastly, tell you the future of chemical warfare as I see it.

In the first place, chemical warfare is a complete science in itself. No other invention since that of gunpowder has made so profound a change in warfare as gas is making or will make in the future.

To-day there are only four really distinct arms of the Service, *viz.*, the Infantry, the Artillery, Aviation, and Chemical Warfare. All other forms of warfare are a combination, more or less complete, of these. The gases, smoke, and incendiary materials that make up chemical warfare are used to a greater or lesser extent by other arms, but wherever gas is used it compels precautionary measures that are found in no other branch of the Service.

No officer, however well trained in other branches of the War Department, can handle gas until especially trained in its use. It is the most universal of weapons, and as it drifts with the very air, becomes the most difficult of all methods of warfare to avoid. No form of precaution heretofore used on land or sea is effective against it. The mask and training that go with it are totally unlike any other element of war. Thus, from the laboratory to the enemy, gas warfare is new, requiring new methods, new

¹ Amos Alfred Fries was born March 17, 1873, in Vernon County, Wisconsin, and moved as a boy to Missouri, thence to Oregon. After a short teaching experience he entered West Point by competitive examination, graduating seventh in his class April 26, 1898. He was commissioned Second Lieutenant in the Corps of Engineers, and appointed First Lieutenant in 1901, Captain in 1904, Major in 1912, Lieutenant Colonel in 1917.

He served with Captain John J. Pershing on an expedition into the Moro country, as Chief Engineer Officer, was Director of Military Engineering at the Engineer School, Washington Barracks, Washington, D. C., from 1911 to 1914, and at the outbreak of the war was in charge of the construction of roads and bridges in Yellowstone National Park. Perhaps his chief work with the Corps of Engineers was in connection with the improvement of Los Angeles Harbor.

Leaving Yellowstone Park July 15, 1917, he reached Paris August 14, and was assigned as Director of Roads, but five days later was appointed Chief of the Gas Service, A. E. F., which position he held throughout the war. On August 16, 1918, he was commissioned Brigadier General, U. S. Army, and placed in charge as Chief of Chemical Warfare Service, A. E. F.

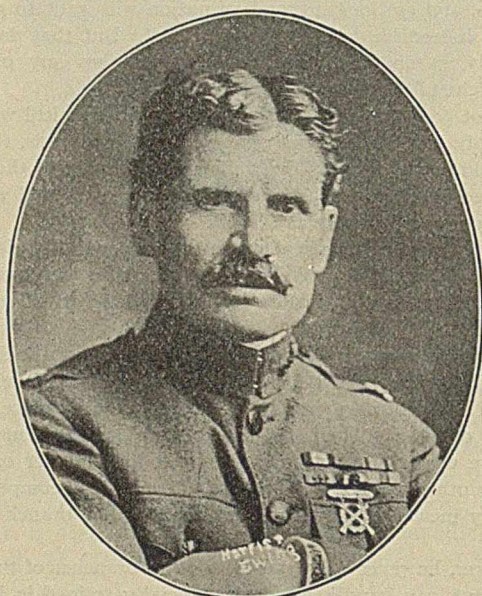
He was decorated by France with the Cross of Commander, Legion of Honor, October 30, 1918, and received the Cross of the Companion of St. Michael and St. George, of England, June 9, 1919. On July 26, 1919, he was awarded the Distinguished Service Medal.

On March 1, 1920, Brigadier General Fries (now returned to his former rank of Lieutenant Colonel) was appointed Chief of the Chemical Warfare Service, vice Gen. W. L. Sibert, transferred.

knowledge and new training. Depending more or less upon the wind, and being affected by the weather, by the character of the ground, and by the verdure on the ground itself, chemical warfare requires the expert to take into consideration conditions which it is not necessary to consider to an appreciable extent in any other fighting branch.

Considering its power it has no equal. Physical vigor is one of the greatest assets in any army. Gas, used properly and in quantities that will be easily obtainable in future wars, will make the wearing of the mask a continuous affair for all troops within two to five miles of the front line, and in certain places, for many miles beyond. If it never killed a man the reduction in physical vigor and, therefore, efficiency of an army forced at all times to wear masks, would amount to at least 25 per cent, equivalent to disabling a quarter of a million men out of an army of a million.

But gas does kill and it does wound, in spite of all precautions, and there is every reason to believe that when handled with proper energy and given its legitimate place it will account for 50 per cent of all casualties in future wars.



AMOS A. FRIES, LT. COL., U. S. ARMY, CHIEF OF CHEMICAL WARFARE SERVICE

One great reason why chemical warfare will continue is that it fills a long-felt want on the part of the soldier, that of shooting successfully around a stump or rock. The gas cloud is inescapable. It sweeps over and into everything in its path. No trench is too deep for it, no dugout, unless hermetically sealed, is safe from it. Night and darkness only heighten its effect. It is the only weapon that is as effective in a fog or the inky blackness of a moonless night as in the most brilliant sunshine. Only the mask and the training that go with it protect. Terror, confusion, lack of discipline and control are fatal.

One naturally wonders why the German decided to start the use of gas. A realization of what has just been said indicates his probable line of reasoning. He hoped to win the war in his first powerful onslaught that actually carried him almost to the outer walls of Paris. He wanted first to separate the English and the French and then by holding the French channel ports make it impossible for England to send troops into France, or else force England to transport her troops long distances by sea to western and southwestern French ports. Besides having far longer sea voyages the ships would have been exposed to a hundred times the danger from submarines. His terrific check at the Marne, coupled with the defeat of his later attempt to break through to the channel ports, showed him that the war could not be won at one stroke, unless he could devise some substance or some method of fighting absolutely unknown, and against which there was no protection. Of all things known or ever

considered gas alone promised such a result. The date when the Germans began active preparations for the first attack is as yet unknown outside of Germany. It must have been quite early in the war, very probably soon after the battle of the Marne—certainly not later than his repulse in the attack of October 1918, when he strove to drive through to the channel ports.

That first attack was extremely well worked out and successful, from the German point of view. The Allied line was completely broken for a distance of three to five miles, and had the German possessed full faith in his weapon he could have pushed whole armies through that gap before reinforcements had closed it on the following morning. Even with his slowness to take advantage of the break caused by the chlorine cloud, he penetrated so far into the Allied lines as to capture nearly all of the dead and most of the seriously wounded. Thus it happens that the total casualties have never been accurately known. The best estimates place it in the neighborhood of 15,000, of which 5000 were deaths, with only 7000, mostly of the less severely wounded, remaining in the hands of the British.

FIRST BRITISH MASKS

Realizing the fatal effects that might follow a second attack, Lord Kitchener made a dramatic appeal to the women of England and France to furnish cotton pads as masks. So quickly were these furnished that within forty-eight hours the greater part of the front line in the area exposed to attack had the crude protection formed by those pads when dipped in sodium bicarbonate and hypo solution. These pads were insanitary, the solution was often misplaced, and the same individual seldom got the same pad twice.

This was followed a little later by the black veil respirator, in which a larger pad was tied over the face. Some glycerol was added, and the solution lasted longer.

It was reported about this time that the Germans had been seen in a gas attack with hoods pulled completely over their heads. This gave rise to what is known as the P. H. helmet. That is simply a sack made of two thicknesses of flannel cloth with two celluloid eyepieces. It was hot, stuffy, and irritating. However, it furnished fair protection and was all that was available until the small box respirator, the model for all American-made masks, came into use between April and October 1916. This development in the mask situation is thus given at considerable length because of the vital bearing it had on the whole later history of chemical warfare.

As indicated above, the death rate in the first gas attack was probably in the neighborhood of 35 per cent of all casualties—and everybody in front of the wave was a casualty. With the development of masks and methods of training in the use of the mask and in taking advantage of the ground, the death rate fell. At the same time the total number of casualties fell, but not at all in the same ratio as the decrease in the death rate. From a probable death rate of 35 per cent in the first attack it fell to 24 per cent, then to 18 per cent, and, as gas attacks by artillery became general, to 6 per cent, and finally with the extended use of mustard gas the rate fell to 2.5 per cent or less. In the meantime phosgene, five times as deadly as chlorine, had been introduced. Tear gases that forced the wearing of the mask, and thus wore out the physical vigor of the man, came into general use. In 1917 mustard was introduced and a little later the famous German sneezing gas which added to the discomfort and difficulty of protection, by penetrating every mask in common use throughout the war.

DIPHENYLCHLORARSINE AND THE CONNELL MASK

And right here is an interesting story. To the average American who served in France, sneezing gas is only a type of the many failures the Germans made in the war. The gas was looked upon with more or less contempt, and yet it had, and still has, enormous possibilities. This diphenylchlorarsine, or sneezing gas, is twice as deadly as phosgene. Being a smoke or dust it

penetrated all masks that depended upon the absorption of gases by charcoal and soda-lime granules as readily as bird shot will trickle through a lot of large-sized broken stone, and in the same manner. It can be kept out only by filters of paper or other materials.

Realizing the possibilities of this gas early in the spring of 1918 those of us in the laboratory and in the field charged with protecting American troops had one of the most anxious periods of our lives. We worked with great haste to develop a filter—and this, first developed by the British and later perfected by the two nations working together, was put into production in April 1918. It took the form of a complete outer covering for the canister that carries the charcoal and chemicals. It added greatly to the breathing resistance, already unduly high. Nevertheless 200,000 were ordered and the majority manufactured, while complete specifications for making them were cabled to the United States and manufacture begun here.

At the same time the Connell mask, the work of an American doctor, was perfected. This mask had a new type canister which inclosed a paper filter that would completely filter out the diphenylchlorarsine. Two hundred thousand of these canisters were also made. This Connell mask is one of the great achievements of the war, and something of which I shall have more to say later. Fortunately the German never succeeded in getting over enough of the diphenylchlorarsine to be dangerous. At the time of our feverish haste in developing this means of protection, we knew how it could be gotten over in quantities that would be terribly dangerous if we could work out the mechanical means of doing so. All of this was communicated to the United States as rapidly as possible, and work pushed here both in getting protection against the diphenylchlorarsine and in developing methods of getting it over. We hoped to beat the German, and we did, and I think that is one of the reasons why he quit when he did. There were other reasons, of course, such as Colonel Wm. H. Walker's hundreds of tons of mustard gas.

MUSTARD GAS

Starting the use of mustard gas in July 1917, the German had the fiendish glee of noting its effect and the number of casualties caused by it for nearly eleven months before he tasted a dose of his own medicine. He told his men that it would win the war, that the Allies had no defense against it, since it burned the body through the clothing as readily as it burned the lungs and the eyes. He told his men that the Allies could not produce it, and in a very large measure he was right. Mustard gas not only produced a tremendous number of casualties, but caused methods of protection to be adopted that were a terrible burden to transportation and a nightmare of discomfort to those using them. The oiled gas-proof suits might be fine for Turkish baths in hot weather, but they were brutal when serving guns in the heat of battle.

Then, too, the Allies had not developed a production of the two ingredients necessary for manufacturing mustard gas by the German method—the only one known prior to the war. While this discovery was a severe blow in the beginning, it turned out eventually to be an extremely fortunate thing for the Allies, as it forced them to develop the now well-known sulfur monochloride method. This in turn enabled them to build up a production that must have been perfectly appalling to the German. That he had some general knowledge of our capacity for manufacturing gas we must assume as certain. We who served in the battle zone through the war got so much information of what was going on in Germany that we knew it was impossible that he was not getting a great deal of the same information from our own country. This information had to be sorted and studied and factors of truth applied, but practice enabled this to be done fairly closely.

FIRST ALLIED USE OF MUSTARD GAS—The French first fired mustard gas against the Germans in the Marne salient about June 18, 1918. From that time the amount of mustard gas

fired by the Allies steadily increased, though neither the British nor the American manufacturing capacity was developed quickly enough to get it into the field in noticeable quantities. But the German must have known it was coming. His men had been told the power of mustard gas and dreaded it. When he found it coming back in greater quantities than he sent it over, he had a problem in the psychology of training that he never fully solved. He had taken so much pains to impress his men with the power of mustard gas that he never succeeded in getting them to look upon it as only one of the incidents of war, and one that, while it caused many casualties, would not necessarily be fatal to his campaign.

Mustard gas made a radical change in methods of fighting—not alone with gas, but with other implements of war as well. It forced a change in the mask which the Americans, with open minds and no binding precedents to bother them, were the first to grasp. The wearing of the mask changed from short periods with everybody quiet to long periods in which men had to fight and do their other tasks. Comfort in the mask became all-important. This to the American spelled the doom of the mouthpiece and noseclip, so that we, in France, began work on this problem as early as November 1917.

You have all read accounts of gas attacks, where the gas was always referred to as a cloud. This arises from the fact that gas was first let loose in large quantities from cylinders placed close together. The rapid expansion of liquid chlorine and other liquid gases into true gases caused condensation of vapor, and thus formed the well-known cloud. At a later date certain smokes, such as tin tetrachloride, were added, because the smoke was irritating and penetrated the early masks in the same manner as diphenylchlorarsine penetrated the later masks.

It was not long until the screening value of the cloud formed by gas began to be appreciated. It was felt that these or similar clouds might be used to screen attacking troops, and enable them to get close to the enemy. This, of course, was not a new idea, but the practical use of the smoke cloud on the field of battle was brought home to those encountering it in gas attacks.

The German after a considerable time hit upon the scheme of generating a non-poisonous smoke cloud and throwing his attacking troops upon the Allies while the latter were wearing the mask. This proved highly successful at first since the English had been taught upon the approach of a gas cloud to adjust their masks and stop all movement. With the coming of mustard gas the formation of a cloud during a gas attack was found not necessary—in fact the absence of a cloud had some distinct advantages. It made the presence of the gas less easy to detect, and that is one of the reasons why the British in the first mustard gas attack in July 1917 thought it was only the ordinary "strafing" of the German. Accordingly they simply took refuge in their trenches and dugouts without putting on their masks, or taking any other precautions against gas. They paid no attention to the pungent smell of the mustard gas, since it caused no discomfort. We can realize the amazement and the extreme anxiety of the officers and medical men and the Chemical Warfare Service when a few hours later men began to go temporarily blind, to get pains in their chests, and to cough, and as the numbers ill rose to hundreds and then to thousands the British realized that a new form of gas had been sprung upon them. Gathering a few duds they rushed them to the field laboratory, 40 or 50 miles away, and soon knew what the substance was, but what the final effects on the men would be was not then known. Severe burns showed on many parts of the body. The eyes in many cases were closed for five to fifteen days. A certain percentage of the men died, whether from the effects on the lungs or from the burns was not known. All this happened just as the British were preparing for their heavy and long-continued attacks against Ypres.

I tell you this in order that you can imagine a little of the feelings of the fellow in the field charged with the responsibility of protecting armies from the enemy's gas, and for developing

means for successfully counter-attacking the German with his own gases. It is no disparagement of the British nor of anyone else to say that they held up the date of their attack for two weeks pending further investigations into the effects of this new gas that had little or no smell, no immediate effects, and that burned the body inside and out, thereby producing casualties that later figures showed to be not less than fourteen times as great as were produced by all methods of gas warfare previously known.

NEED OF AMERICAN LABORATORY IN FRANCE

We Americans then began to call for American chemists and an American laboratory to be located in France, where we could get some immediate emergency work done on such problems as mustard gas presented. We never expected to establish in France any considerable research laboratory. We felt that this could be done better in the United States. But we felt, and in this our judgment was sound, that with a reasonable laboratory equipment and a few of the ablest chemists and research men actually on the field in France, we could learn vastly more quickly and better the problems that had to be encountered, and possibly something of the methods for meeting those problems.

An idea that was probably in the minds of many was voiced by a certain officer who made a trip early in 1918 to France and to the front. He said, "You don't need a laboratory here. All you need to do is to state your problems to us in the United States and we will work them out." He forgot that stating a problem is often half solving it, and perhaps more—that to discover and properly state that problem requires greater ability and maturity of thought than its actual solution. A great deal of your research is done by students while working for their degrees, but the man who lays out the work and supervises it is the ablest you can get.

Two short reports of six and two pages, respectively, written by Prof. G. N. Lewis, of the University of California, in the last days of March during the progress of the great German drive against the English, in which he was caught, brought these facts forcibly home to me. He came to me in France as an able chemist, or as one young doctor put it, "one of the best physical chemists in the world." Our laboratory was then just arriving. What to do with him was a question. I sent him to an English Gas Defense School to learn that side of training. He was put in with the Canadians, where he had the fine experience of making his way back for forty miles during the English retreat from that terrible onslaught of the Germans starting March 21, 1918. His two little reports above mentioned showed a military grasp of the situation that I had not expected from a professor of chemistry. They showed that he realized the powers and possibilities of gas, and had the true winning spirit: that the offensive spirit, the spirit that knows that victories are won not by sitting still but by vigorous offensives.

We were then sorely in need of a man to take charge of training the entire American Army in methods of wearing the mask and otherwise protecting themselves from the enemy's gas. Professor Lewis was promptly made assistant on this work and three weeks later put in entire charge. And he made a tremendous success of it. I need only add that this demonstrates the value of having some of the ablest men at the front. A less able man, without a wide knowledge of men, materials and affairs, would not have grasped the significance of the use of gas in that battle as Professor Lewis grasped it, or have been able to take up and devise methods of training and apply them to meet future gas attacks.

What gas did in the World War is such an absorbing topic, and is of such importance that I could talk to you for hours—but I won't. It almost won the war for the Germans in the huge drive above referred to. It caused 75,000 of the 275,000 casualties that American troops suffered on the field of battle, and yet the German used it in a halting and comparatively feeble manner. It is fortunate indeed for us that he never used it on

the scale that he might have, had he realized its value and could he have gotten the production of gases that we were getting when the war closed.

SMOKE

Chemical warfare includes gas, smoke, and incendiary materials and they can't well be subdivided. As before stated, all the early gas attacks were in the form of clouds. The value of that cloud, not only for carrying gas, but for screening purposes, began to be realized in the fall of 1917. Clouds of smoke may or may not be poisonous and they will or will not be poisonous, at the will of the one producing the smoke. For that reason every cloud of smoke in the future must be looked upon as possibly containing some deadly form of gas. When you consider this for a moment, you can realize the tremendous possibilities for ingenuity that gas and smoke afford the attacker.

The American, trained for 300 years in meeting nature on her great plains and in her vast forests, was early appealed to by this side of chemical warfare. As early as November 3, 1917, the United States was urged, in a cablegram from the Chemical Warfare Service in France, to push the development of a large phosphorus supply for use as smokes. Not only were the early intuitions of the value of gas borne out by later events, but today the future of smoke appears greater still. The battlefield of the future will be covered with smoke—not the all-pervading black smoke of the battles of the Civil War and of earlier wars before smokeless powder came into use, but a field covered with dots and patches of smoke, big and little, here and there, and everywhere.

Every man who has hunted ducks and been caught in a dense fog with ducks quacking all around, and who has tried to get ducks by firing at the quack in the fog can realize the difficulty of hitting a man on the battlefield when you cannot see him and have only a quack, or less, by which to locate him. The smoke will be generated in candles or two- or three-pound cans that can be thrown out in front of trenches; by knapsacks that can be carried and which will give off dense white smoke in large volume for many minutes; by grenades which while they may be thrown by hand will generally be fired from rifles; by artillery shells reaching 10, 15, 20 miles back of the main battle line; and finally from aeroplane bombs whose radius of action is limited only by the size of the earth. And thus smoke becomes one of the great elements of war in the future. It is more or less wholly protective in its nature; but as it costs more and takes longer to train a man in the various problems involved in modern war than ever before in this history of the world, it is worth while taking every precaution to protect him, once you have him trained.

INCENDIARY MATERIALS

Intimately associated with smoke are the incendiary materials. One of the best of all the smokes is that given off by burning white phosphorus, which is also an offensive agent of first-class power. To me, the bursting and burning of quantities of phosphorus in the field is one of the most difficult situations in the world to imagine keeping cool in, and it certainly proved to be so, so far as the Germans were concerned, in the latter part of the war. This brings me to the point where I want to say a few words about the First Gas Regiment, organized originally as the 30th Engineers.

FIRST GAS REGIMENT

"What's in a name?" is an expression known to every man, and often used. The First Gas Regiment, on the starting of the second battle of the Marne on July 18, 1918, realized what there is in a name. They were organized and known as the 30th Engineers. Now the duties of engineers in war have been known, more or less, for scores of years. The two companies of the regiment were ready to put off a big projector gas attack, but General Foch's counter-attack was launched one day too early. The General Staff immediately put the First Gas Regiment to work

on the roads, because they were known as the 30th Engineers. Upon the earnest appeal of Colonel Atkisson, commanding the regiment, they were allowed to go forward and join the infantry in an attempt to take machine gun nests with burning phosphorus and thermite shells fired from 4-inch Stokes mortars. Colonel Atkisson and his officers insisted strongly that they could do this if given a chance; no one outside the Chemical Warfare Service believed they could do it, but as the losses from German machine gunners were becoming exceedingly serious, they were given the chance, and they made good—so good, in fact, that the regiment got little chance to do anything else throughout the remainder of the war. They carried heavy 4-inch Stokes mortars, with their 25-pound shells, for miles, and in many instances kept up with the attacking infantry.

The 4-inch Stokes mortar is a weapon developed by the British for trench warfare and one which they felt could not be used in open warfare. Yet the boys of the First Gas Regiment carried these things and fought with them, and as one infantry officer recently told me, "they were certainly not afraid to go anywhere where they could be of use, whether it was in the front line trench, or in front of the front line trench"—and this last is no exaggeration. In at least one instance in the start of the battle of the Argonne, boys of the First Gas Regiment set up their Stokes mortars out in No Man's Land to establish a smoke screen behind which the attacking infantry could move. But the First Gas Regiment never forgot the value of gas. On the few occasions that presented themselves they used gas and used it effectively.

Then, too, in order to be in on every problem, they developed the use of high explosive in Livens projectors. This projector, I might say, is the simplest gun known, being a straight drawn steel tube closed at one end, and from 2 ft. 9 in. to 4 ft. in length. It fires a bomb carrying 30 lbs. of liquid gas or high explosive a distance of a mile—a distance that in the future will be increased to at least a mile and a half. They were fired in large numbers simultaneously by being connected up with electric firing batteries, and were exceedingly effective, whether with high explosive or gas. Indeed they provided the method of gas attack most dreaded by the Germans.

For a moment just consider what was done at the attack on Lens in the spring of 1918, an attack participated in by the first two companies of the First Gas Regiment. On that occasion 2500 of these 60-lb. drums, filled with phosgene, were dropped on the enemy at one time, in the middle of the night. The concentration of gas in such attacks is terrific, the detonation of the bursting bombs is terrifying, and the war proved that none but the most highly disciplined soldiers could stand up under the strain of these attacks. But projectors were difficult, slow, and laborious to install. Our efforts in those days, as they will be in the future, were to make these attacks mobile, much easier, and consequently of far greater frequency.

The Gas Regiment in the St. Mihiel battle fired on the Côte des Esparges one hundred of these high explosive bombs at the zero hour on the morning of the attack. That hill, famous for its strength through four years of struggle between the French and Germans, disappeared completely as an enemy stronghold. Nothing remained but torn and broken barbed wire, bits of concrete pill boxes and trenches filled with debris, and a few scattered fragments of clothing.

The gas troops will, in the future, handle all short-range methods of firing gas, smoke, or high explosive. They will deliver the greatest quantities of material possible up to ranges of a mile and a half or a mile and three-quarters. So effective and so efficient are these short-range methods of projection that the No Man's Land of the future will be the width covered by these projectors and mortars. They can't, and never will, compete with the artillery, where range and great accuracy are the most important factors. The efficiency of artillery gas shell, or artillery smoke or high explosive shell is only one-fifth that of the projector. Hence

for economy and efficiency the artillery will be used to fire gas, smoke, high explosive, and incendiary materials only at ranges beyond those reached by the gas troops.

So greatly were these possibilities appreciated in the summer of 1918 that the number of gas troops authorized for use against the Germans was increased from 6 companies to 54. Back of all this, however, was the productive capacity of the United States which insured that those troops would be able to fight day and night, summer, winter, and fall until the war was over. No wonder the German quit—it was time, and he knew it.

COÖPERATION OF CHEMISTS

Interesting as is this discussion of work of the Chemical Warfare in France, it must be dropped now to give a few moments to the discussion of the future relations of the Chemical Warfare Service with the chemists and the chemical industries of the United States.

That relation must be one of whole-hearted coöperation. The Chemical Warfare Service cannot be a success unless the chemists of America back it; unless they are willing to coöperate with it as they did in the war. If they will do that, the United States need have no fear of any future struggles.

WORK DURING THE WAR—Neither the history of the World War nor, indeed, the history of any other war, shows such whole-hearted, enthusiastic coöperation of professional men as was shown by American chemists during the World War. Starting under Dr. Manning, of the Bureau of Mines, at the American University, and at numerous colleges and research institutions throughout the United States in February before the war began, the organization grew until the Research Division alone contained 1200 technical men and 700 service assistants. Its work covered exhaustive research into over 4,000 different materials.

FUTURE RESEARCH IN THE CHEMICAL WARFARE SERVICE—The Government cannot afford, and we do not believe it would be advisable, to attempt an organization of even one-tenth of this size to operate in peace-times under the Chemical Warfare Service. The Chemical Warfare Service must maintain a small research organization. There will be certain lines of investigation that can best be carried on there, or lines which no other institution desires to touch upon. The Chemical Warfare Service must have enough trained men to tell, at least tentatively, whether a new idea, a new substance, or a new method, can probably be successfully adapted to chemical warfare. This will involve, in large measure, a thorough understanding of military problems, military organization, and military methods. We shall expect to be experts in that and to be ready at all times to take up any and every question that you, from the four corners of the United States, may submit to us. We want your ideas—all of them. If something comes to us which has been proven impractical or impossible, we shall courteously tell you why the idea is unusable and will request you to submit others.

The thousand research men that are working throughout the United States as compared with every one that the Chemical Warfare Service can maintain will certainly bring forth 75 to 90 per cent of all the new ideas, materials, and methods for use in chemical warfare. We hope that you will be able to make researches along certain definite lines that the War Department may ask you to pursue. We hope that we shall be able to keep in such close touch, and work in such full coöperation with the chemical industries of the country that the magnificent plant at Edgewood Arsenal may soon be a thing of the past. We do not believe the Government should attempt to manufacture poisonous gases on a huge scale. Many of these gases are now proving useful and even large factors in commercial processes. We hope to keep in such touch with the various chemical industries that we can count upon this one and that one all over the United States to produce within a given number of days on the outbreak of war, a given quantity of gases. We believe that in this way we can build up, more quickly and to a greater ex-

tent than by any other method, the necessary large output of poisonous gases required in a war with a first-class power. When that time comes the great government plants will be abandoned. Our Edgewood plant should be capable for at least five years, with moderate upkeep, of being put into large-scale production without notice. By that time we hope to have worked out with the chemists, the research institutions, and the great chemical industries a plan for utilizing all chemical resources, men, and factories in less than 30 days after the outbreak of war. We believe that if this is done satisfactorily it will be one of the greatest possible guarantees of future peace.

The universal adoption of gas warfare on sea and land and in the air, combined with its persistent quality, will make that nation able to produce and use gas in the largest quantity superior in war to any other nation on the globe. The United States can reach that position and maintain it, and I believe that we are going to get such encouragement from the War Department that we can do it. I feel sure that the Army appreciates the value of chemical warfare, and that it appreciates also the value of the chemists to chemical warfare.

The German had forty years of chemical research and development before the war and he had over two years of swift chemical development in the war before the Americans started in. With a lack of foresight which I trust will never be evidenced again in America, we had done nothing to meet the chemical warfare activities of the Germans. We entered the war, then, in 1917, with as big a handicap as could be imagined, but through the energy and coöperation of the chemists, we not only, in 1918, overcame that lead of forty years and more, but we surpassed it, so that in the end we led Europe not alone in quantity of manufacture, but in research, in training, and in actual gas tactics on the field of battle.

The war taught us conclusively that what may be a tremendous success in the test-tube in the laboratory may be an utter failure in the field. Hydrocyanic acid gas, called "Vincennite" by the French, is a brilliant example. Instantly fatal in high concentrations, it has almost no serious effects in concentrations appreciably below the fatal one. Hydrocyanic acid, being known to be so fatal, was at once seized upon by the Allies, and particularly the French, as a great battle gas; and notwithstanding it was such a complete failure, there are even yet writers on chemical warfare and chemical matters who are not aware of the fact, as shown by the following quotation from a French writer on chemistry in the war. He stated that two French professors discovered a gas "which produced instant mortal action, but which our chiefs, from an excess of humanitarian scruples, which were indeed superfluous, decided not to employ." What an error! It was employed in hundreds of thousands, perhaps millions, of shells and the Germans thought so little of it that in a memorandum to the German armies they stated that the French gas, meaning Vincennite, was harmless, except in the concentrations in which the Germans themselves used it to kill vermin.

TRIBUTES TO INDIVIDUAL CHEMISTS

Right here I want to say a few words of praise and appreciation of the chemists who served with me in France. You all know pretty well the names of those who pointed the way to successful chemical warfare over here. Each of you knows of Walker and McPherson and their coworkers who created at Edgewood Arsenal the greatest chemical plant in the world.

You all know of Burrell, Kohler, W. K. Lewis, Bancroft, Lamb and a score of others who made the American University the greatest research organization ever dreamed of.

You know of Dewey and his five and a quarter million American-made masks; of Dorsey and his Development Division at Cleveland, and scores of others who did work equally as brilliant though in less prominent positions. But how many of you know the story of Major Karl Connell?

Major Connell was not a chemist by profession, but a physician in New York City. Before the war he had won considerable fame by inventing masks for administering anesthetics. As said before, he was not a chemist, but he had a large store of practical chemical knowledge. His name was handed to me in France as that of an energetic, able man who had served several years with the 71st Regiment, National Guard of New York. I cabled for him, and he reported early in November 1917. I had intended to assign him to training; but when, in the first hour of our interview, I had elicited from him a statement as to his work on anesthesia, I showed him models of all masks in use by the Allies and the enemy and told him that we felt that we must eventually have a fighting mask; that while we had adopted the English mask this was simply because it was the best in existence and we believed in using what was available, though at the same time we also believed in starting investigations toward the ideal. After another hour's work with these masks he said he believed he could make a new one embodying the principles of clear vision, comfort, ease of adjustment and fool-proofness—that is, a mask that would be easy to put on and difficult to disarrange and one that could be manufactured economically on a large scale.

I sent him to Paris where in three weeks he produced the first model. Though it was crude he had faith enough in it to try it in a high concentration of chlorine. It leaked a little but the general result was so satisfactory that I sent him to England to complete his work. I consider his first model a marvel, inasmuch as he did it in a strange city, speaking a language foreign to him, and working with a tinsmith who knew no English. His work was continued in England under the most trying circumstances. Doubtters and even scoffers called him everything but a genius and a wise man, but he persevered, and by the first of March 1918 had produced the first all-American mask that was then, and is to-day, a complete protection against all known gases. It embodied the principles of the latest type American mask that was not perfected until nearly eight months later. Its principal features were pressed metal facepiece, sponge rubber to make a gastight fit between the face and the mask, a canister carried on the back of the head, the omission of the mouthpiece and noseclip, and the bringing of the inspired air over the eye-pieces so as to keep them clear.

Sent to the United States in July to take up the question of producing this mask, he was told that the sponge rubber could not be made successfully here and that a mask with a fabric facepiece was being developed which would be superior to his. He dropped his own development and threw his whole soul into the problem of developing what is now the 1919 American mask, the finest mask in the world.

His reward?—no promotion, no medals, not even a citation—only the proud feeling of a man who did all that he could for the cause and who won the love and undying admiration of those who knew him as I knew him.

Then there was G. N. Lewis, of whom I have already spoken, Professor of Chemistry in the University of California, who stepped from the quiet of the university into the maelstrom of war in one of the greatest battles of history, and won in the Chemical Warfare Service a recognition of greatness equal to that which he possesses to-day in the field of chemistry.

Then there was Colonel Zanetti, a teacher of chemistry and a master of the French language, who reported to me in the middle of October 1917. Feeling the urgent need of closer contact with the French, he was at once assigned as Chemical Warfare Service Liaison Officer with them. He did a tremendous work in cementing cordial relations between the French and American Chemical Warfare Services and was allowed to delve deeper into French secrets than any other foreigner living.

Then there was Goss, of Princeton, young, energetic, with a considerable knowledge of the Army gained in the National Guard of New Jersey. Arriving in France about the middle

of October he was assigned as Chemical Advisor and put to work coördinating the chemical information we were getting. Not satisfied with doing only two or three men's work, he proceeded to get into the field—in touch with the British, to hunt Germans in No Man's Land, and eventually to rise to be Chief Gas Officer of the Second Army and one of the best experts on chemical warfare.

There was Colonel Hildebrand, also of the University of California, who progressed so rapidly in the military application of chemical warfare problems that he was put in command of the Proving Ground and the American Army Gas School, a position he filled with the greatest credit to himself and to the United States.

Another was Major Keyes, of the Boston "Tech," who got together in the United States, transported to France, and there put up and operated one of the finest laboratories in all Europe. The work of that laboratory was small in comparison with the work done in American University, but it was vital and would have become vastly more so had the war continued another year. The emergency problems in the field—such as the determination of when the chemicals in the canisters were destroyed, and the opening and determining of the contents of new gas shells—were problems looming bigger and bigger all the time.

Then there was Major Pope, a chemical engineer, whose report on the impossibility of our manufacturing gases in France was timely, correct, and vital. Up to that time certain persons contended that Americans must manufacture their gas in France and must fill their shells there. His lucid report on the impossibility of doing that work stopped all such talk and kept the United States from embarking on an impossibility.

There were many others in the field and in the laboratory or on the proving ground who did all that man could do in their positions and thus contributed powerfully to winning the war. Among them I want to mention just one more name—that of Captain J. E. Mills, of the First Gas Regiment. Joining that regiment in the United States, he was among the first to reach France. Given many opportunities to go into the laboratory or on duty at headquarters, he always begged to stay with the regiment. He stayed there even to his own detriment, so far as promotion was concerned.

He was one of the mainstays of Colonel Atkisson in all his work, giving his entire strength and very nearly his life to the cause. In his loyalty to the regiment regardless of danger and hardships when even more important work under pleasanter conditions was offered him, he typified the best in American manhood. All honor to him and to the unsung thousands like him who suffered and risked all that "Liberty might not perish from the earth."

FUTURE OF CHEMICAL WARFARE

The future of chemical warfare has been pretty well indicated by what has been here said in regard to its use in the past war and its possibilities as the Chemical Warfare Service sees it. Gas is too deadly, too dangerous, too easy to develop and produce in secret for any nation to afford to give it up.

There have been many stories of super-gases invented in America or elsewhere, that even in small quantities would wipe out whole armies. No such gas was produced in the war. No such gas will be produced. A gas may be discovered that will penetrate existing masks, and unless mask development be such as to stop the gas, the result will be disastrous to the army encountering it. It is the business of the Gas Defense side of chemical warfare to prevent just such conditions. It met and solved all such problems in the past war, and I have no doubt that it will meet and solve such problems in the future. If it can't, we are lost—lost, because, while I have the greatest faith in human nature, I would not risk the lust for power and wealth that the future United States might engender in other peoples if the latter felt they had a substance that could wipe out our

Armies as fast as they approached. That all the world is feeling this way about chemical warfare is indicated by the following quotations. The first one is from a statement of the Under-Secretary of War for England, when appealing to Parliament for funds to carry on chemical warfare. He said, in part:

So long as there is any danger of other nations continuing these methods of warfare, research and experiment in chemical warfare must be pursued. Research must not only be directed towards the gases and apparatus likely to be employed in the future, but also towards protection against all possible gases. Training in the use of gas will be confined to appropriate branches, but training in defensive measures will include the whole army.

We must continue our studies of what is known as chemical warfare. No nation has renounced the use of poison gases as the result of the Peace Conference. There are nations whose word we could not respect if they did renounce it. It is essential to study the offensive side of the chemical warfare if we are to be prepared for defense. The great importance of adequate defensive appliances arises from the fact that preparations for the offensive use of gas can be made in peace-time with great secrecy, and may have far-reaching and even fatal results in the early stages of a war.

..... For these reasons it is necessary to make adequate provision for research, experiment, and design in connection with war material. It is equally necessary to avoid overlap, duplication of effort, and the setting up of military institutions for scientific research which can better be done by existing civil institutions. It is our policy to farm out to civil scientific institutions all pure research that can profitably be farmed out, and, generally speaking, to restrict military institutions to applied research and the preliminary design of apparatus.

Another quotation is taken from a statement by General Debeney, Director of the French College of War. This statement first appeared in the United States in the March 20 issue of the *Pittsburgh Dispatch*. Among other things General Debeney said:

Should war begin now, aviation, and especially gas, would play one of the most important parts. The progress of aviation would make the rear of each front, and very far in rear, extremely dangerous, and the progress of chemistry would permit the use of gas on zones of such an extent as cannot be imagined.

Making gas is naturally rapidly done, because all the manufactures of chemical products—still so numerous in Germany—can be requisitioned, but to make airplanes is much slower.

The defense against gas seems to be more difficult than against airplanes. I believe that against airplanes, the anti-aircraft artillery is susceptible of making rapid progress, and perhaps in that very instance gas will be one of the best ways, if with appropriate shells the air can be poisoned all around the attacking airplanes. It would be much more effective to create, for example, a sphere of poisoned air a mile around the airplane, instead of trying to hit the machine directly with bits of the shell.

Finally, the following statement, taken from the *Des Moines Register* of March 22, 1920, probably expresses about as well as any words I have yet seen the position that I believe the average man in the United States takes regarding chemical warfare.

As to poison gas, we would as well accustom ourselves to the thought that it is now a permanent and important part of war. To get hot about that is as futile as to scold the men who invented gunpowder, and is on the same plane. It is ridiculous to declaim about the incidentals of war while ignoring the truth that war itself is the enemy. If America ever again "draws the sword," to use an expression that shows the difference between war of yesterday and war of to-day, America will go in with as much reliance on gas shells as her antagonist can possibly have. Nobody in the military councils at Washington or West Point even dreams of anything else. And if America does not add a few refinements to the use of gas it will be because American wit and ingenuity have broken down, which isn't likely.

VICTORY AND ITS RESPONSIBILITIES

By Charles H. Herty

EDITOR, JOURNAL OF INDUSTRIAL AND ENGINEERING CHEMISTRY

A half decade ago the common lament among men of our ilk was that the science we represent and its applications were not appreciated by our fellow-citizens. We had grown accustomed to and accepted as almost ineradicable the popular conception

of the chemist as a long-haired, bespectacled individual performing some mysterious magic in a carefully secluded workshop. We fretted over the grotesquely distorted reportorial accounts in the press of any matter pertaining to chemistry and our eyes looked in vain for intelligent and sympathetic discussion of the chemist's work on the editorial pages of the American press. Salaries were inordinately low and the general atmosphere surrounding the profession was such as to repel rather than attract the students in our universities.

To-day that state of affairs no longer exists, the American chemist has come into his own. Chemistry is on the lips of everyone, the real personality of the chemist is understood and his work is appreciated at its true value. A victory has been won—a victory over public opinion. What are the evidences of that victory?

In the offices of the SOCIETY'S News Service are thousands of newspaper clippings, the number mounting daily, from every section of the country. The clippings from the news columns show reasonably scientific accuracy, combined with an approved popular style of presentation which makes them readable for the masses of our people, while those from the editorial pages show a sound and far-sighted grasp of matters chemical.

In the bookshops there are large sales of popular books on chemistry. Men like Ellwood Hendrick and Edward Slosson have made splendid contributions. In the Senate gallery lately I happened to be sitting in the section just above the desk of Senator Knox of Pennsylvania. During the "morning hour" while a multitude of minor measures were being considered, I observed the Senator deeply engrossed in reading a book. What book do you suppose it was? Not a work on international law, as might have been expected—no, it was Dr. Slosson's book, "Creative Chemistry," and the Senator was so delighted that he needs must share his pleasure, so he took the book to Senator McNary, evidently with a warm recommendation, for the Senator from Oregon immediately wrote a memorandum of the title and the publisher. Could that have happened five years ago?

Another evidence is the matter of salaries paid young chemists. The head of one of the greatest research laboratories of the country told me just a few days ago that two years ago men just graduated from colleges with the Bachelor's Degree were paid \$18 per week. In that same laboratory they are now paid \$34.

Five years ago but little was heard of industrial chemical research. Now the number of laboratories devoted to such work is legion and constantly increasing. Empiricism in industry is steadily giving way to scientific control and development.

But undoubtedly the strongest evidence of victory won is the sympathetic attitude of the present Congress toward all measures affecting the chemist and his work. I am a thorough believer in the idea that our Congress is truly representative of our people and its present friendly disposition has its roots in a similar feeling on the part of the "folks at home."

Five measures affect favorably our work one of which is now a law; the other four have been passed by the House of Representatives and are now before the Senate, three with favorable reports from Senate Committees and one doubtless soon to receive an equally favorable report.

That measure which is now a law is the National Prohibition Act. In view of the past uniform policy of including a visit to a brewery in the list of plant excursions at our General Meetings some of you may be taking exception to the mention of this act as one affecting favorably the work of the chemist. In explanation, however, let me ask how many of you are aware of the wording of the title of this act? Here it is: "An act to prohibit intoxicating beverages, and to regulate the manufacture, production, use and sale of high-proof spirits for other than beverage purposes, and to insure an ample supply of alcohol and promote

its use in scientific research and in the development of fuel, dye and other lawful industries." That title is the mandate of Congress to the Bureau of Internal Revenue in framing its rules and regulations for the carrying out of the act. And the Bureau has faithfully carried out the expressed purpose of Congress. Both the Congress and the Internal Revenue Bureau have leaned toward the chemical industries in the drafting of the law and in the administrative procedure thereunder. Tax-free "alcohol may be withdrawn * * * for the use of any scientific university or college of learning, any laboratory for use exclusively in scientific research, or for use in any hospital or sanatorium." Pharmaceutical and medicinal manufacturers have been aided by a modification of the act of 1906 which defined tax-free denatured alcohol to be such as "destroys its character as a beverage and renders it unfit for liquid medicinal purposes." The present act requires only that the denaturing "render the alcohol or any compound in which it is authorized to be used unfit for use as an intoxicating beverage." Previously certain formulas for denaturing were set up by the Government and the manufacturer had to utilize whichever of these most nearly suited his needs. Now, however, if a manufacturer finds no suitable formula for denaturing he can demand such from the Government. Surely chemists have abundant grounds to be thankful that Congress in drafting this legislation which gave the knock-out blow to alcohol as a beverage nevertheless kept clearly and sympathetically in mind the importance of alcohol as a chemical reagent. A copy of Regulation No. 61 of the Bureau of Internal Revenue of the Treasury Department should be on the desk of every chemist.

Then there is the Longworth Bill for the safeguarding of our coal-tar chemical industry. You are familiar with its details and you thoroughly understand the character and the source of the opposition it has met. I shall never forget the look of consternation on the faces of two of the leaders of the German dye industry last October when during a conference there was brought into the room a copy of the *Frankfurter Nachrichten* containing the announcement that the Longworth Bill had passed the House of Representatives. That news told them clearly that the American people were determined to make themselves forever independent of any foreign nation for such supplies, and it required no superhuman effort for them to guess which particular nation was in mind. Their gloom, however, was somewhat lightened when I explained to them that the bill had still to pass the Senate. Will it pass that body? I have always believed so, and in confirmation of that belief let me read to you an extract from a telegram I received from Senator Watson, in charge of the bill, just before leaving New York:

I earnestly hope for the passage of the dyestuffs bill as reported from the committee. A widespread propaganda is being engaged in by opponents but I feel sure that the measure that passes the Senate will fully protect the American dye industry and thereby give an impetus to industrial and engineering chemistry in America.

But this was not all of Senator Watson's telegram, he adds: Trust also that the bill for the protection of surgical instruments and chemical glassware will soon become a law in order to stimulate an additional interest in these subjects in the minds of the American youth, particularly those who attend our colleges. I assure you I am doing everything in my power to secure the enactment of these measures.

(Signed) JAS. E. WATSON

Plainly the Senator is in complete accord with the Council of this SOCIETY which voted in favor of the repeal of the duty-free importation clause and which unanimously recognized in this privilege a most dangerous channel for insidious propaganda among those who some day will constitute the chemical personnel of this country. The Bacharach Bill has a deep significance for chemistry.

Another measure affecting directly the work of the chemist is the Nolan Bill, which seeks to strengthen the forces and increase the activities of the Patent Office. For years it was impossible to arouse public interest in this matter, but a few weeks ago a public hearing was held on this bill. The result was its speedy appearance on the House calendar with a favorable committee report. Its passage by the House with only one dissenting vote is a fitting example of the cordial attitude of the Congress.

Of deepest importance, however, is the feeling in Congress toward the Chemical Warfare Service. In the face of the minimizing efforts of those highest in authority in the War Department, the House of Representatives has already voted that this Service shall be an independent unit in the new army. The bill is now being debated in the Senate, and the Senate Committee on Military Affairs has taken an even stronger attitude in behalf of the Service than did the House. Just three days ago I received the following telegram from Senator James W. Wadsworth, Jr., chairman of the Senate Committee on Military Affairs.

You know, of course, that the Army Reorganization Bill already passed by the House of Representatives provides for the maintenance of a separate Chemical Warfare Service in the War Department. The Senate Bill now under discussion in the Senate also provides for a separate Chemical Warfare Service. The Senate Committee on Military Affairs was thoroughly convinced that the Chemical Warfare Service was of such vast importance that it should be allowed to develop its possibilities under its own officers and with the invaluable assistance of the chemists of the country rather than be merged in some other service of the Army in which it would inevitably occupy a secondary position out of all proportion to its real importance. In view of the action of the House of Representatives and similar action by the Senate Committee it is fair to assume that the legislation when finally completed will provide for a separate Chemical Warfare Service.

(Signed) JAMES W. WADSWORTH, JR.

The future of the Chemical Warfare Service is assured. Let us in turn respond to this action of the Congress and give to that Service that loyal support which will insure America's leading the world in chemical warfare.

There is only one regret in this situation, the removal of Major General Sibert from the head command of the Chemical Warfare Service. This action by the authorities of the War Department was a distinct shock to the country. As an aggressive organizer of that Service, General Sibert had won the high regard and affection of the members of the Service and of civilian chemists. He had been unwilling to avail himself of the privilege of retirement at will, granted by Congress in appreciation of his work on the Panama Canal, and had determined to remain on active duty until the future of the Chemical Warfare Service was fully assured in the reorganization of the Army by Congress. From the outset he advocated fearlessly and convincingly the continuation of the Service as an independent unit of the Army, and his views prevailed, though they were in marked opposition to his superiors in the War Department. And he paid the price, and was removed from the head of the Service. Now comes the news of his voluntary retirement. A man who played so conspicuous a part in the construction of the Panama Canal, and who assured to this country the future of the Chemical Warfare Service can well afford to rest upon his laurels. He will carry with him always the best of good wishes from the chemists of America.

It was fortunate that the only logical successor of General Sibert was Lt. Col. Amos A. Fries, whose work as head of this Service with the American Expeditionary Force had demonstrated his fine ability and proved him thoroughly equipped for this important position.

With such evidences of victory over public opinion before us, the question naturally arises: How has this been accomplished? It is needless to say that war activities in the past few years have brought the chemist to the attention of our entire people. Upon the successful outcome of investigations

and their translation into large-scale production depended the lives of our soldiers and the task was worthily met. Then, too, the revelation in the early days of the war of our economic dependence in the matter of dyes and medicinals, of chemical glassware, of optical glass, chemical porcelain, and of scientific instruments cut deep into the very life of many of our most important industries. Dependence does not belong to the American make-up, and the speed with which this dependence was removed both as to quality and quantity of output made a story of amazing wonder. But it was necessary that that story be told and told in language that could be understood in order that the work should receive due recognition. Here it is that the chemist has worked a revolution within himself.

There was a time when the chemist felt that it was no business of his that interpretation of his work should be given in everyday language. His concern was with the day's work in the laboratory and there his responsibilities ended. So, too, he felt that as an organized group he was not called upon to express an opinion on matters of chemical economics. I remember well at the Council Meeting in Seattle in 1915 when the suggestion was made that this SOCIETY should urge legislation which would insure a domestic dye industry there was so much opposition to the SOCIETY's so expressing itself, on the ground that it was an economic rather than a chemical question, that the matter was laid over until the following day. Fortunately the tide turned at that time and vigorous resolutions were passed which marked the beginning of the fight for an American industry.

Since that day this matter of interpretation of chemistry to the public has gone forward by leaps and bounds. Believing that we ourselves were responsible for the neglect on the part of the daily press, prominent chemists made up their minds to meet the press half way, and by their personal efforts furnish material which reporters could make into attractive stories for their papers. This material was greedily accepted by the press. Still there was a lack and this was filled by securing the services of an experienced newspaper man, Mr. John Walker Harrington, who worked daily with the chemist in this matter of interpretation. The officers of the SOCIETY realized that the question of public education as to the real meaning of chemistry to the nation was essentially a function of this SOCIETY. Funds have been appropriated in steadily increasing amounts as the work has progressed. The details of the operation of this News Service have been eagerly sought by representative organizations here and in other lands. The success of the work is due solely to the one fundamental thought that we have striven to promote nothing but the welfare of our country through a better comprehension of chemistry. In an autocracy such as Germany was, chemistry was fostered by its ruler. In a democracy such as America is, we are seeking to foster chemistry through popular education and upon this sure foundation it will last.

Another form of public education which is developing is that of public addresses by chemists to non-technical audiences. I was deeply interested a few weeks ago in reading in a New York paper a lengthy account of an address by our former President, Dr. Julius Stieglitz, before the Fashion Art League of America and the Alliance of Art and Industry on the subject of "The American Dye Industry." Those who know Dr. Stieglitz's delightful method of presentation can readily understand the pleasure and profit of his hearers, but my chief pleasure lay in the fact that Dr. Stieglitz in the midst of his busy life found time and had the disposition to make such an address. He has set an example which should be followed by each of us.

Another powerful factor in public education as to chemistry has been the National Exposition of Chemical Industries. Originating amid predictions of failure, it soon demonstrated its useful public function. In that Exposition the chemist has taken the public into his confidence and furnished the opportunity for public inspection, not only of the products of the industry but

of the crude natural resources from which and the machinery by which these products are manufactured. The character of the attendance at these annual expositions is illustrative of the widespread interest in chemistry and of the eagerness with which people will listen to the story of the chemist when it is attractively told.

What have been the results of this victory over public opinion?

Capital has been attracted to the chemical field in constantly increasing quantities. According to the statistics of the *New York Journal of Commerce* the authorized capital of new chemical, drug, and dye companies during the past five years was as follows:

1915.....	\$ 65,565,000
1916.....	99,244,000
1917.....	146,160,000
1918.....	73,403,000
1919.....	112,173,000

while the total for the first three months of 1920 is \$60,188,000. This great influx of capital has increased the demand for chemists and with this has come increased remuneration for the chemists' services. These conditions have attracted chemistry students in numbers which five years ago would have been incredible. The chief result of this victory, however, is the increased prestige which the chemist has gained. The legitimate feeling of gratification over such an outcome is not based upon complacent self-satisfaction but upon increased opportunity which is afforded for further service.

Such a victory as has been won carries with it distinct and increased responsibilities. It would be a mere platitude to say to you, fellow chemists, that this new atmosphere brings to each of us inspiring cause for even more earnest effort. What I do want to leave in your minds is that in such a sympathetic atmosphere men can work with hope and freshened zeal, in the feeling that their work is being recognized in its true significance. I need not say more about this, because I think we all grasp that point. But, gentlemen, as we work in our laboratories we must show that same spirit of originality, of fearless willingness to break into new lines, to open new paths, that characterized the spirits of our pioneer forefathers. The old fetish of a certain foreign superiority in chemical matters is gone. America's day is coming! America's day in chemistry is coming, but it is not yet here in its full realization. The one responsibility that I want to bring home to you to-day is that of carrying on still further this question of public education. It is the same story I have just been talking about. I have painted the rosy side of the picture, but there is much yet to be done. We can't be satisfied and feel that our work is thoroughly understood in this country when two such institutions as Cornell University and the Massachusetts Institute of Technology formulate plans and offer the facilities of their laboratories, their equipment and their staffs, to the War Department, and have the War Department write back: "We don't need any chemistry in this matter (referring to preparations for war)." Yet that is exactly what happened in the spring of 1917. Of course, soon afterwards they woke up, and then began a general scramble. Now is the time for education and general understanding of chemistry to be completed.

In a much more recent time than three years ago when we went into the war, even within the past six months, there has been put forward by the Secretary of War a proposal to build a great engineering school down in the woods of Virginia, a twenty million dollar institution, and send down there graduates of West Point, who have had fourteen weeks of chemistry, give them a year's training, and let them do the research work for our Army in all the problems of chemistry which pertain to warfare. You laugh, but that is to my mind one of the most suggestive of thoughts, that so intelligent a man as the Secretary should have, at the close of this war, such a hazy idea of what it takes to make a research chemist, and should seriously make that

proposal. And General March says we don't need any Chemical Warfare Service, because there isn't going to be any more chemical warfare. Those are the gentlemen at the head of the War Department. I don't know whether they are educated yet. General Sibert and Colonel Fries have gone strongly the other way and put this thing right, anyway it looks now as if it is right. That is the situation to-day in spite of five years of education.

We talk about chemistry being prolific around New York City. There are hundreds of chemical industries there, and 2,000 chemists in the New York Local Section, and rafts of them over on the Jersey flats, yet when the mayor of that great city appointed his Committee on Defense and announced that he was putting on it representatives of all the interests centered in and around New York there wasn't a chemist within a mile of it. A little while later the governor of New York appointed another war committee consisting of representatives of the various industries. I went through the list carefully to find out which one of the boys was on the committee, and I am still looking for him.

With all that has been said in the papers, in the magazines, before committees of Congress, in both the Senate and the House, in spite of the fact that the President of the United States has twice selected a certain specific industry—the coal-tar chemical industry—and urged upon Congress favorable legislation in its behalf, there was introduced in Congress on the opening day of the session of last year, a bill to protect further that industry, and that bill is not yet a law!

Public opinion is not so organized, not so active in behalf of chemical matters that it will immediately spring up and react favorably upon Congress. What happened the other day when the Appropriations Committee of the House brought in a report cutting the items of the Bureau of Foreign and Domestic Commerce, cutting out the commercial attachés, and limiting the work of the Bureau? What happened? Within twenty-four hours business organizations in every section of the country were pouring telegrams and sending committees into Washington, and that appropriation went back to normal size, and passed. That is what organized sentiment can do. In marked contrast was the action of Congress in severely cutting the appropriations requested by the Bureau of Standards, whereby the splendid work of that important scientific bureau will be badly hampered in the future. Scientific men have not rallied, as did the business organizations, to the support of that bureau which contributes so much to their interests. I believe that Congress represents and desires to represent the people of this country, and yet an industry which has such tremendous ramifications and deep-seated significance to this country from so many standpoints, as testified to by men in the Army, in the Navy, in the Administration, and by workers outside of the industry itself, waits restively for the passage by the Senate of a bill which has been favorably reported by Republicans and Democrats alike with no trace of partisanship about the measure. Why has not this bill become a law? Because public opinion hasn't awakened.

One other matter—at the National Exposition of Chemical Industries there have been shown from year to year the results of the efforts of the railway companies to utilize more and more the chemist in making chemical surveys of natural resources; in other words, the chemists were doing the work of industrial agents. But right now those laboratories are closed. An officer of one of the railroad companies stated recently that he doesn't know whether they can ever get started again, that all records have been lost or destroyed, all specimens are gone. They have nothing to exhibit at this year's Exposition. When an important movement like that can be completely wiped off the boards we haven't yet taught our people fully what chemistry means to them.

Here is another thing. When the expedition was being assembled to go to Paris to represent America in the formulation of the Peace Treaty there were brought together men from nearly every line of American effort. They were going to negotiate a peace with a nation which was primarily a nation of chemists, whose work was so well known to all, for the lack of whose chemical material we had suffered in this country in the early days of the war—and yet, how were the chemists of the country represented on that mission? I don't know Mr. Summers personally. He has never been a member of the AMERICAN CHEMICAL SOCIETY. He has never been at our meetings. He has never published any articles in chemistry journals. He was first heard of down in Washington representing chemical matters on the War Industries Board. We do know him somewhat and somewhat unfavorably in connection with our platinum conservation program for munitions manufacture. Mr. MacDowell I know and am personally very fond of, but he doesn't pose as an organic chemist. He is in the fertilizer business, but he isn't a chemist and doesn't profess to be. As days went along, in came Mr. Baruch. He's been a lot of things, but I never heard him called a chemist. I wrote a little squib once in the JOURNAL, about a new use for the ultramicroscope: to find among the members of the Peace Commission someone who knew anything about chemistry. With the exception of Alonzo E. Taylor, a food chemist, I stick to that statement. If public education as to chemistry had been further advanced such a situation could not have arisen.

Our work affects the national security. It is up to us to make sacrifices, to answer the call of that man right there, the Chief of the Chemical Warfare Service, Colonel Fries, whom we hope soon to call again General Fries, it is up to us to meet him quick with the best we've got. It isn't his work, it's America's work, and the chemists of America must meet that responsibility in every way. I want to quote the words of one of the most prominent industrial dye chemists in Germany, a remark made to a group of members of the Inter-Allied Commission which visited Germany soon after the armistice. Some of you met him over here at the Eighth International Congress, Dr. Duisberg. Speaking about the Peace Commissioners in Paris, he said in words to this effect: "They are going to make a hard treaty for us. We have got to accept it. You beat us on production in this war, but we are going to beat you on production in the next war." "In the next war"—that is our responsibility to this country.

And then remember this. Before the Ways and Means Committee in Washington last spring Mr. Francis P. Garvan, the Alien Property Custodian, was testifying in behalf of the American dye industry. It was just a few days after the first dirigible came to this country from the other side. Mr. Garvan drew a graphic picture of what might be expected within a few years, outlining the possibility of a type of enemy dirigible carrying poisonous gases, flying across the ocean, and wiping out the city of New York in a few minutes. Some said he was overstating the danger. And yet Colonel Wm. N. Hensley, Jr., of the Air Service, gave out only the other day a statement made public by the War Department regarding the scheduled plan of the Germans for Thanksgiving Day, 1918. I'll read it:

* * * the German effort was scheduled to take place about Thanksgiving Day, 1918, and that the L-72, which he described as the "largest airship in the world," was expressly constructed for the raid, measuring 775 feet from tip to tip and equipped with six engines of 260 horsepower each. The L-72 was capable of carrying five tons of high explosives and incendiary material.

Action for every hour and minute of the trip was foreseen. Every possible contingency of weather, fuel exhaustion, damage to ship or machinery failure had been reckoned. Weather charts of the Atlantic were gathered, files of the German Admiralty were combed and the records of the merchant marine were searched.

That was on the schedule for the latter part of November 1918, fortunately just two weeks after the armistice was signed. Mr. Garvan spoke of a possibility, but it has now been disclosed that it was a positive intent scheduled in every detail. The Germans had already planned for this conquest.

Now I want to say a word to you about this same gentleman, Mr. Francis P. Garvan. I think he is the strongest ally that the chemists of this country have. It has been my good fortune in an unofficial capacity to be associated with him for the last three years while he was Assistant Alien Property Custodian, and later Alien Property Custodian, in working out the ramifications of this German network of intrigue, and as president of the Chemical Foundation, in straightening out this patent tangle which has troubled us so much. I received this telegram from him last night:

Please convey to the members of the AMERICAN CHEMICAL SOCIETY my sincere appreciation of the great work performed by them in the recent war. This war was essentially a chemists' war and the initiative, energy, and self-sacrifice on the part of the chemists of this country was, I feel sure, more than any other one thing responsible for the success of America. As a result of this victory the responsibilities of the American chemists have become increasingly greater. We now look to them to make America the leader of this industry in the world. We expect them to excel all other countries in the development of explosives, gases, and the other things necessary in warfare. We expect them to lead the world in the development in industrial chemistry so that the commercial supremacy of our country can be assured. We expect them to excel the Germans in the manufacture of dyestuffs so that never again will America have to depend upon Germany or any other country for these most important products. A great responsibility rests upon the chemists of this country. Long and patient research work and the building of more plants will be necessary so that the American chemists can fulfill the obligations which now rest upon them, responsibilities of our victory in the recent war. The American people are alive to and sympathetic with the work of the chemists of this country. I for one believe that within a very short period America will lead the world in chemical research work. We are only a short way from that goal now. I want to see America the greatest chemical and dyestuff country in the world and I know that every member of your SOCIETY does too.

(Signed) FRANCIS P. GARVAN

Now, gentlemen, I want to read finally one extract in further introduction to you of the personality of Mr. Garvan. It is the conclusion of his testimony before the Senate Finance Committee. I do this because there is a thought in it which to my mind is one of the most inspiring which has been brought forward by a public man, at least as affecting our work.

Gentlemen, Dr. Albert and Bernstorff reported to their government that America could never establish the dye and pharmaceutical industry in this country, as we lacked the moral power for the creation of such an industry; that each party pursued its own selfish interests, but nobody kept the whole in mind; that this problem could only be solved through regard for all points of view, and that the conflicting selfishness of this country rendered that solution impossible. Wrong; wrong; as ever, wrong! Let them await the answer of American patriotism, American sacrifice, and American ability.

We felt that we would like to be a part in the taking of the forces of science developed by them—which they have only turned to the desolation and destruction of mankind—and placing them in the hands of what we believe to be a higher and purer civilization, to see if we cannot, out of the terrible mess of this war, do something constructive with these same scientific forces, do something to direct them into the channels of alleviation and helpfulness to humanity. It is only to give American principles and character a chance that we ask you to hold these people off until we get our education. Then we can meet them without any tariff.

To my mind that puts the goal before us. That elevates our work far above mere commercialism. What in the world does it mean to make dyes, to make sulfuric acid, to make caustic soda? We need them, they are a part of our civilization, and the men who are in the industry are entitled to a just return, but, my fellow chemists, there is a higher mission ahead of us. It is

foreseen in Mr. Garvan's statement, though he is not a chemist. These other things must of course be done and done well, but, oh, my friends, the chemistry of the body is too little known. Think about the fact that the body is a mass of chemical reactions, which when normal mean life, and health, and happiness, but when abnormal signify disease and misery and unhappiness. Yet these are simply chemical changes. This country is spending millions of dollars for drugs on the bare chance that they may happen to be the right thing. We must get these matters on a scientific basis. We must get deeper into the knowledge of how these drugs act. It will take time, and a tremendous amount of research, but surely the goal of American chemists is a noble one, if it can start toward the attainment of the alleviation of the suffering of America. What lies beyond that we do not know, perhaps a prolongation of the average life of mankind, perhaps more happiness through life. There is something deeper in our work than mere commercialism. I am talking about industrial chemistry now, too. I don't know exactly what it is. I can't yet get my thoughts clear about it, but down in my heart I believe that when our people thoroughly understand what we are trying to do, and are thoroughly sympathetic with our aims and aspirations, when full opportunity is given for the highest energy and effort of American chemists, somewhere in that science to which we devote our lives will be found that which is uplifting to life in every way.

COLLOID SYMPOSIUM

SOME PRACTICAL APPLICATIONS OF COLLOIDAL CHEMISTRY

By Jerome Alexander

RIDGEFIELD, CONNECTICUT

"COLLOIDAL" FUEL

What promises to be one of the most far-reaching advances made under the stress of the recent war, when necessity literally was the mother of invention, is the discovery that by means of a suitable "fixateur" or peptizing agent and suitable treatment, very large percentages of cheap tars and finely powdered coal waste may be dispersed in fuel oil with a sufficient degree of permanence to enable the mixture to be stored, piped, atomized, and burned practically like fuel oil itself. Since this new composite fuel will at one stroke relieve the drain on the earth's rapidly diminishing stores of petroleum, as well as lead to the efficient utilization of all kinds of coal waste (culm, screenings, dust), inferior fuels (peat, lignite), and even cellulose waste (slabs, sawdust), it may be hailed as a powerful factor in the conservation of our natural resources and a lasting benefit to mankind.

Realizing the vital importance of the Allies' oil supply in the conduct of naval, military, and manufacturing operations, the German submarines bent every effort to destroy tankers; and Marshal Foch is said to have cabled America: "If you don't keep up your petroleum service, we shall lose the war." While the Allies' navies were dealing with this peril in a most decisive fashion, Lindon W. Bates, head of the Engineering Committee of the Submarine Defense Association, with the assistance in laboratory matters of Dr. S. E. Sheppard and other chemists of the Eastman Kodak Laboratory, courteously opened to him, developed a colloidal fuel, which, by practically doubling the usefulness of every oil cargo, would have of itself materially assisted the defeat of the Hun efforts.

Coal or other combustible solid is prepared for dispersion by being pulverized so that about 95 per cent passes through a 100-mesh, and 85 per cent through a 200-mesh screen. This of course means that by far the greatest weight is in particles hundreds and thousands of times larger than colloidal dimensions. But we should remember that the violent motion of the colloidal

particles causes the Brownian movement of much larger particles, as is the case in milk where the fat globules can be seen in the ultramicroscope oscillating about under the ceaseless bombardment of the casein ultramicrospheres. It has been found that a fluid fuel may be made containing as much as 40 per cent by weight of powdered coal, and mobile pastes containing up to about 75 per cent; and mobile gels may be made both from the liquid and the pastes.

The exact nature of the fixateur is withheld pending the issuance of patents here or abroad, but it is stated that ordinarily between 0.5 and 1.5 per cent of the essential component of the fixateur is used, whereas about 0.1 per cent exercises a noticeable influence. The amount is determined by the nature of the mixture, the components, and the degree of permanence desired. The bulk of the particles does not begin to settle until the period of "life" has passed, the colloidal fuel having a limited "life" which may be regulated to meet requirements—days for power plants, weeks and months for ocean-going vessels and central storage stations. Heat and agitation revivify the liquid fuel, and the paste form may be kept for years.

Being heavier than oil or even water, colloidal fuel compresses in a unit volume the maximum thermal value, thus economizing storage space; and it may be stored under and extinguished by water, thus avoiding evaporation, deterioration, and fire risk. Its operative efficiency is high, for when sprayed into the hot fire box the oil-permeated particles of carbonaceous matter are still further atomized by the sudden gasification of their imbibed oil. It possesses the advantages of fuel oil over coal—absence of smoke, dust, and ash, practical elimination of labor in loading into storage space and in firing, with resultant saving in time for "coaling" and for raising steam. As with oil, a protective smoke screen may readily be produced by over-firing, and the fire is subject to instant control.

SMOKES

While it is an ancient principle of strategy to attack an enemy under cover of natural fog or mist, the recent war resulted in an enormous development of artificial smokes as aids to naval and military offensive and defensive operations. Like the cuttlefish, steamers strove to escape pursuing submarines in colloidal clouds of their own making, and soldiers crept to the attack under the protection of a smoke barrage. In the air, nature's own clouds were favorite hiding places for aeroplanes and dirigibles, and the latter (especially Zeppelins) often produced their own concealing clouds.

In times of peace the treatment of smokes and fumes is mostly confined to their coagulation by the Cottrell process, by which sulfuric acid mist or zinc oxide fumes have been collected as part of the ordinary course of operations, and by which injurious industrial dusts, *i. e.*, smelter and cement fumes, have been converted into sources of profit. There is, however, one case of the commercial production and utilization of smoke that possesses features of interest—the use of the so-called "smudge-pot."

Smudge-pots are burned in orchards, especially in spring or early summer, when weather conditions indicate that the tender blossoms or fruit are apt to be injured by frost. The heat emitted by the pots is apparently a small factor, their efficacy being due to the dense clouds of smoke evolved by the burning smudge oil. This remedy is of course useless in very cold weather, and serves only to prevent what agriculturists term a light frost, which has for its precursors a still, clear atmosphere, and a temperature approximating the freezing point. No frost of this kind occurs on cloudy or windy nights.

The freezing process, in the absence of smoke, seems to proceed as follows: as the temperature drops, moisture deposits like dew upon leaves, fruit, buds, and other surfaces furnishing nuclei, and congeals into ice crystals or "frost," when the slight heat thus set free by condensation is dissipated into the surrounding cold atmosphere, the freezing being also facilitated by the evapora-

tion of part of the deposited water. The smoke, however, furnishes throughout the entire lower atmosphere innumerable nuclei for the deposition of moisture, and the slight but widely distributed heat thus liberated is sufficient to carry the temperature, locally, above the freezing point. Moisture on such small nuclei does not readily evaporate, and the smoke fog furthermore forms a kind of protective blanket which reflects terrestrial heat and prevents its dissipation.

SHOWER-PROOFING OF FABRICS

Ordinary textile fabrics are readily wet by rain, but if coated with a surface film of wax or the like (aluminum stearate), the falling droplets roll off "like water off a duck's back." This water-proofing effect is readily understood if we consider the interfacial surface tensions involved:

- 1—Surface tension water/air, denoted by σ_{WA}
- 2—Surface tension water/fabric, denoted by σ_{WF}
- 3—Surface tension air/fabric, denoted by σ_{AF}

Practically speaking, the surface attraction of the untreated fabric for water is so great (*i. e.*, σ_{WF} is so small!) that both the surface film or skin of the raindrop and the air film on the fabric are burst; the raindrop spreads itself out on the fabric, and is absorbed into its pores. That is, wetting occurs if

$$\sigma_{WF} < \sigma_{WA} + \sigma_{AF} \text{ or } \sigma_{WA} > \sigma_{WF} - \sigma_{AF}.$$

In the case of the treated fabric, σ_{WF} is very much increased; becoming in fact the surface tension water/wax, for surface tension is only skin-deep. Therefore $\sigma_{WF} > \sigma_{WA} + \sigma_{AF}$ or $\sigma_{WA} < \sigma_{WF} - \sigma_{AF}$ and no wetting takes place.

FLOTATION

The understanding of what happens in flotation will be much simplified, if we bear in mind that, notwithstanding the presence of many disturbing and variable factors, there are two main triphasic systems involved. These are:

1—Ore/water/oil, from which the true ore (usually a sulfide) must emerge with a film of oil which enables it to be taken up by the air bubbles and thus floated with the froth, while the gangue is wetted and flowed off at a lower level.

2—Air/water/oil, which yields the bubbles to float the oiled sulfide.

Denoting

- 1—Surface tension water/oil by σ_{WO}
- 2—Surface tension oil/sulfide by σ_{OS}
- 3—Surface tension water/sulfide by σ_{WS}

the oil will then distribute itself over the interface water/sulfide (*i. e.*, the sulfide will be oiled) if $\sigma_{OS} < \sigma_{WO} + \sigma_{WS}$ or $\sigma_{WO} > \sigma_{OS} - \sigma_{WS}$.

In the second system denoting

- 1—Surface tension water/air by σ_{WA}
- 2—Surface tension water/oil by σ_{WO}
- 3—Surface tension air/oil by σ_{AO}

the oil or some of its constituents distribute themselves at the interface water/air because

$$\sigma_{WA} > \sigma_{WO} + \sigma_{AO}$$

75 23 33 (approximate values).

This yields air bubbles with a more or less stable surface film, *i. e.*, a foam or froth. This film is miscible with the oil film on the sulfide, although not necessarily identical with it, because there may be a separation of the constituents of the oil by differential adsorption. The oiled sulfide particles therefore act like oil, and, distributing themselves at the interface water/air, are attached to the air bubbles and lifted to the upper froth layer, if they are not too heavy or are not knocked off.

In the foregoing no account has been taken of complicating factors which are always present and while space will not

¹ It should be borne in mind that the surface tension *decreases*, as the attraction between the phases *increases*. In fact, when the positive surface tension in a two-phase system becomes zero, solution may take place.

permit an exhaustive discussion of them and their results, the most important will be considered.¹

WATER—Here are found dissolved air, crystalloids, and colloids. The dissolved air aids in bubble formation by reducing the internal pressure of the water. For the most part the crystalloids, especially *acid* electrolytes, act beneficially, being adsorbed by and coagulating the fine slimes, clay, etc., which then tend to repel the flotation oil, recalling the old-fashioned household expedient of moistening the mouth and throat with orange or lemon juice before taking castor oil, and thus avoiding the taste. Some electrolytes, especially alkalies (*i. e.*, Na_2CO_3) in small percentages, are powerful defloculators, and by reducing the surface tension water/oil and water/sulfide tend to emulsify the oil, wet the sulfide and deflocculate the slimes. Humic substances, especially when converted into alkali salts, and protective colloids generally, act in like manner—the injurious effects of glue are well known. Colloidal clay or very fine gangue act similarly—"fat" clays will emulsify oils, coal tar, and asphalt.

ORE—Outside of soluble impurities which dissolve in the water, the fineness of grinding is most important. The gangue should be fine enough to flow off, but not so fine as to cause the undesirable effects just referred to above. If the sulfide particles are too large, the air bubbles cannot lift them, or they are easily knocked off; if they are too small, there is a great loss in efficiency, for the surface of the bubbles may be satisfied by a layer of small particles which represents a small weight of sulfide.² The results may also be adversely affected by the surface oxidation of the sulfide.

OIL—This is deserving of the most intensive study, since it is the factor most readily controlled. Moreover, the great and sudden development of the art of flotation has turned the various oil markets topsy-turvy, necessitating a constant search for new oils or combinations not only from financial considerations, but also because of actual shortage in the supply. Since conditions are different at each plant, and since different lots of ore even from the same mine may vary, a careful study is necessary to determine what oil or mixture is best, and also to know how to meet changing conditions. Particularly useful will be a determination of the foam-producing factor as opposed to the sulfide-oiling factor.

LUBRICATION

The consumption of petroleum is increasing so rapidly that a material decrease in the supply, if not actual exhaustion, lurks in the not far distant future. W. B. Hardy in a trenchant paper³ has shown that we may with confidence look to colloid chemistry to aid us in finding the lubricants of the future.

Glass surfaces selectively adsorb from the air rather more of the atmospheric impurities than of the elementary gases and water vapor, yielding a film about $1 \mu\mu$ (1×10^{-7} cm.) thick, which Lord Rayleigh termed "grease" because it has the general properties of an oil.⁴ For this reason a new or raw glass surface has different mechanical properties than a satisfied or neutral surface.

It is not possible to get a raw surface⁵ of glass. One cannot cleave glass; but the "grease" film may be removed by rubbing the surface under water. Soaking is insufficient; actual vigorous

¹ Mechanical agitation and temperature exercise considerable influence. Callow estimated that with four different oils, three oil percentages, two pulp densities, and two temperature changes, there are about 60,000 possible different combinations of conditions.

² The layer of tiny adhering ore particles forms an armadillo-like armor about the air bubbles, which to a large extent protects them from coalescence and destruction.

³ *J. Soc. Chem. Ind.*, **38** (1919), 71.

⁴ This adsorbed layer of "grease" tends to make pipettes and burettes deliver inaccurately, so analysts remove it by oxidation with bichromate.

⁵ Raw surfaces are readily made by cleaving mica, and if immediately put together again, they adhere so strongly that they withstand a powerful lateral pull, and usually tear the surface when finally separated. [J. A.]

rubbing is necessary, preferably with the finger tips until a peculiar clinging feeling is produced: the same which is felt when the edge of a finger bowl is rubbed so as to give out a musical note. A surface cleaned in this way has some remarkable mechanical properties, the most striking being that two cleaned surfaces seize when pressed together with a relatively small force. This may be demonstrated very simply by measuring the tangential force needed to produce slipping. That true seizing occurs is proved by the tearing of the surfaces which takes place in the act of slipping.

The function of lubricants is to keep the applied surfaces in the neutral condition by maintaining a "grease" film on each. Not all fluids, however, can act in this sense as lubricants for any particular surface such as that of glass. Water, ether, alcohol, benzene, and strong ammonia are apparently entirely incapable of maintaining a lubricating film on glass. Seizing occurs just as readily when they are present as it does with cleaned surfaces. Glycerol differs from the fluids mentioned above in the fact that though it will not maintain a lubricating film it does prevent seizing when present in excess. For instance, the maximal tangential force which a pair of cleaned surfaces would support without slipping was measured in grams, 55. Flooding the surfaces with water, benzene, alcohol, etc., left this value unchanged. When a film of glycerol was deposited on the surfaces the force still stood at 55, but it fell to 9 when the surfaces were fully flooded with glycerol.

The expression "film" used above denotes a layer of fluid on the solid surface of the order 10^{-7} cm. in thickness. With a true lubricant the facility for slipping is maximal when a layer of such excessive tenuity separates the solid faces and nothing is gained by increasing the thickness of the layer. Thus with castor oil the weight required just to start one face of glass slipping over another was 10 g. when only the invisible film of fluid mentioned above was present, and it was still 10 g. when the surfaces were flooded with oil.

Some fluids indeed seem to lubricate better in thin than in thick layers; that is to say, to act in the contrary way to glycerol. Acids as a class behave in this way, the solid faces again being of glass.

	PULL IN GRAMS	
	FILM	FLOODED
Acetic acid.....	40	47
Sulfuric acid.....	37	47
Oleic acid.....	10	13

If this result can be fully substantiated it will be an important and striking physical fact likely to throw much light upon the process of lubrication. One broad conclusion emerges from these facts, namely, that lubrication depends wholly upon the chemical constitution of a fluid, and the fact that the true lubricant is able to render slipping easy when a film of only about one molecule deep is present on the solid faces, suggests that the true lubricant is always a fluid which is adsorbed by the solid face. If this be so, then the problem of lubrication is merely a special problem of colloid physics.

Some solids, notably graphite, are lubricants themselves. But the chief function of graphite seems to be as an aid to lubrication by oil. The graphite is disintegrated in the bearing and besides filling up relatively large imperfections, its colloidal particles are adsorbed by the iron, forming in reality a graphite surface which has a lower surface tension against oil than iron has. Practically this means that the oil film adheres more strongly to the graphitized surface and therefore needs greater force or pressure to break it down; *i. e.*, the bearing will stand greater speed and pressure. Acheson's "oidag" consists of a paste of colloidal graphite dispersed in oil. A small percentage is mixed with the lubricating oil and feeds with it through the finest orifices. "Aquadag" is the corresponding water-soluble product, stabilized with tannin.

CERAMIC PROCESSES ASSOCIATED WITH COLLOID PHENOMENA¹

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This paper is intended to present some of the aspects of colloid chemistry which have a bearing upon the technology of clays. Within the scope of this contribution it is not possible to consider

¹ Published by permission of the Director of the Bureau of Standards.

the subject at all completely and attention can be called only to certain of the more important topics.

Clays are mixtures of finely divided hydrated aluminum silicates with granular matter such as quartz, feldspar, mica, etc. These represent all grades of subdivision, from the coarsest to the finest. The property of plasticity, the ability of the substance to be molded when admixed with water and to retain the shape imparted to it, is to be attributed to the dispersed state of the aluminum silicates, principally $Al_2O_3 \cdot 2SiO_2 \cdot 2H_2O$, known under the general term of clay substance. The pure type mineral, known as kaolinite, sometimes found in crystalline form, rarely occurs in clays but has usually been reduced to particles apparently devoid of crystalline structure, of the magnitude of 5μ or smaller, admixed with colloidal material like ferric oxide and organic matter and frequently strained by absorbed salts. Clays are not necessarily plastic in the natural state but may have been indurated by pressure or heat, or set through other agencies so they cannot be made plastic without the aid of grinding. This is particularly the case with materials consisting of hydrated silicates other than $Al_2O_3 \cdot 2SiO_2 \cdot 2H_2O$, such as hallosite, pyrophyllite, indianite, etc.

The colloidal character of clays was recognized as early as 1874 by Schloesing¹ and later more definitely by Rohland² and Cushman,³ and it was agreed that a clay is very plastic, fat or sticky when the colloid matter is in excess or sandy; weak or non-plastic if the granular matter predominates.

The colloidal characteristics of clays are substantiated most effectively by ultramicroscopic examination. Recent work of Jerome Alexander⁴ has shown that the Brownian movement is observed with practically every type of clay suspended in water, varying from the rapid motion of the finest particles to the more sluggish one of the larger particles or aggregates or when hampered by the presence of electrolytes.

CLAY SUSPENSIONS

Clay suspensions in the deflocculated state when filtered through paper will show decided turbidity in the filtrate. Again, such a suspension, after the coarser portion has been allowed to settle out, will give rise to the familiar phenomenon of the Tyndall cone upon passing light through it.

Clay suspensions are affected in a very pronounced manner by various reagents, both electrolytes and non-electrolytes. Probably the first striking phenomenon observed in this respect is that of absorption. Upon adding solutions of salts like NH_4Cl , $BaCl_2$, $Al_2(SO_4)_3$, and $CuSO_4$, an appreciable amount of the basic ion is absorbed but practically none of the acid ion. Thus Sullivan⁵ found that 1 g. kaolin absorbed between 0.0038 and 0.0169 g. of CuO from 50 cc. of a solution containing 2 g. of $CuSO_4$, but was itself slightly dissolved. Cushman⁶ found the absorption of NH_4 from NH_4Cl to be 0.077, of Ba from $BaCl_2$ to be 0.373, and of Al from $Al_2(SO_4)_3$ to be 0.075 per cent after standing three days and using 0.1 *N* solutions.

Deeply colored solutions of metallic salts may thus be de-colored by passage through clay. Organic salt solutions like malachite green or methylene blue, consisting of large, complex molecules are absorbed to a very striking extent, and in fact Ashley⁷ employed the magnitude of absorption of the former as a measure of the colloid content or the plasticity of clay, using a solution containing 3 g. of the dye per liter. This type of absorption follows the general exponential equation with fair agreement.

¹ *Compt. rend.*, 79 (1874), 376, 473.

² *Z. anorg. Chem.*, 31 (1902), 158; 41 (1904), 325.

³ *Trans. Am. Ceram. Soc.*, 6, 65; Bureau of Chemistry, *Bulletins* 85- (1904) and 92 (1905).

⁴ Private communication.

⁵ U. S. Geological Survey, *Bulletin* 312.

⁶ Bureau of Chemistry, *Bulletin* 92.

⁷ U. S. Geological Survey, *Bulletin* 388; Bureau of Standards, *Techno-logic Paper* 23.

Salts as a class tend to coagulate clay while bases deflocculate it, as is common with all dispersed materials. This type of reaction is of considerable technical importance. Thus the action of NaOH, Na₂CO₃ or Na₂SiO₃ in increasing the dispersion of the clay particles brings about a decided decrease in the viscosity of the system and, hence, according to Stokes' law makes possible a sharper and more prompt separation of the granular matter and the clay substance proper, a fact made use of in the washing and purification of kaolins, first proposed by Keppeler.¹ The clay thus treated and separated must later be coagulated with acid or salts to render possible its filter pressing.

Another industrial application of the deflocculation of clays is connected with the so-called casting process in which the constituents of a porcelain or other ceramic body are stirred with water containing about 0.3 per cent of Na₂CO₃ and Na₂SiO₃ in terms of the dry weight of the clay materials, to form a thick but still readily fluid suspension. Here the addition of the alkaline reagents is effective in reducing the water content required to keep the mass in the liquid condition. In fact, the amount of water present is not greater than that found in the same mixture in the plastic state without the use of the alkali. This phenomenon may be readily demonstrated by adding to a plastic mass of kaolin a small amount of Na₂CO₃ or NaOH. It will be noted that the plastic material is suddenly transformed into the fluid state. Upon the addition of acid or a salt like Al₂(SO₄)₃ it will revert to the plastic condition, thus illustrating the effect of deflocculation and coagulation. In casting, the clay suspension is poured into a plaster mold which absorbs a sufficient amount of water to cause the mass to solidify into a layer conforming to the outlines of the mold.

On the other hand, coagulating reagents, acids, and salts are used to increase the plasticity and strength of certain clays and to thicken suspensions of glazes and enamels.

All the phenomena of deflocculation and coagulation occur in phases. Thus the addition of alkali to a clay suspension brings about first a decided viscosity minimum, followed by alternating maxima and minima until finally the phases merge in a definite direction. Similar conditions prevail also in a coagulation series with the phases in reverse sequence. Schulze's statement that the coagulating ions apparently increase in effectiveness proportionately with their valence is confirmed in the work with clays. Certain organic substances like the tannins react upon clay suspensions somewhat like the alkalies, though with less sharply defined phases. They may also assume the function of a protective colloid in that they render the clay less sensitive to the action of electrolytes. Kaolin suspensions are easily affected by the presence of impurities, and impure clays containing soluble sulfates deflocculate only with difficulty or not at all.

The fluidity of clay suspensions is probably the best means of following changes taking place in clay-water systems and for this reason technical viscosimeters have been used frequently for the estimation of the deflocculating or coagulating effect of reagents. While this practice is perhaps justified for suspensions containing a large excess of water it is not warranted for heavier ones, and the criticism of Bingham² that such mixtures show evidence of viscous flow is justified, since the volume of the flow varies directly as the pressure. It would seem then that viscosimeters of the Bingham type should be preferred for measurements of this kind even though the technical instruments suffice to mark the maxima and minima which it is necessary to establish. However, even such determinations of the viscosity or its reciprocal, the fluidity, of clay suspensions suffice to establish the relative plasticity of different clays inasmuch as equal concentrations will show fluidities inversely proportional to the general plastic nature of the materials.

¹ *Z. angew. Chem.*, **22** (1909), 526; Bureau of Standards, *Technologic Paper 23*; Bureau of Mines, *Bulletin 128*.

² Bureau of Standards, *Scientific Paper 278*.

One of the most interesting phenomena observed with clay suspensions is the electrical deposition of the particles by a direct current upon the positive electrode against the flow of water away from it. This is the basis of the invention of Schwerin for removing clay from positively charged particles (iron bearing minerals) and depositing the former upon a metallic belt or revolving drum. The addition of small amounts of sodium hydroxide increases apparently the charge of the clay particles and facilitates their deposition. As a matter of fact the separation is effected far more completely by previous washing treatment with the use of sodium hydroxide for bringing about maximum deflocculation. The principal application of the Schwerin process consists in the deposition of the material upon the moving positive electrode, thus replacing the filter press just as in the electro-osmose filtering apparatus devised by the same worker. It seems that the migration velocity is that of the heavier ions and that the voltage determines the degree to which water has been eliminated.

These several phenomena of clays in the suspended state have been explained as being due primarily to the negative charge of the clay particles, so that the introduction of negative or OH-ions will bring about further dispersion or deflocculation and that of H-ions coagulation. It is apparent, however, that the case is not one of mere electrostatic repulsion or attraction. The presence of salt solutions not only brings about a change of the charges on the particles or neutralization but also a host of possible and complex interactions more or less complete as, for instance, in the addition of sodium carbonate to a clay already carrying absorbed calcium ions, and hydrolysis. Ashley compares these reactions with those of soap in that in either case we are dealing with a weak acid, only slightly soluble, the salts of which are all hydrolyzed and with the exception of those of the alkalies and ammonia but difficultly soluble.

PLASTIC CLAY

Clay in the plastic state likewise possesses some interesting properties most of which still require further elucidation.

The measurement of plasticity itself has not been possible up to the present time except by empirical or indirect means, and it is only recently that progress has been made in this direction.

The simplest mechanical conception of plasticity would be to consider it a case of viscosity according to the relation $d = KW$, where d = deformation, W = load, and K = constant. It seems, however, from the researches of Bingham that this is not the case but that we must differentiate between viscous and plastic flow in that the yield point of the former is zero and of the latter finite. In a diagram correlating pressure with volume of flow the curve of a viscous liquid will pass through the origin and in the case of a plastic substance we have an intercept on the pressure axis. In the case of clays studied by the writer¹ the pressure required to start initial flow through an orifice 0.25 in. in diameter varied from 40 to 75 lbs. per square inch according to the character of the clay. With decreasing water content the pressure required to produce flow increased very rapidly, and on the other hand with increasing amounts of water it dropped, so that the resulting curve was hyperbolic. Atterberg² has made use of the conception of flow and lower plasticity limits. He determined the amount of water required to just cause flow of the clay and also the water content at that stage where it can barely be rolled to form threads. The difference between these values he calls the plasticity number. Kinnison³ has found this factor to be quite characteristic, though he considers that it should be plotted against the water content of the plastic clay and the resulting diagram used for the classification of the different materials.

The plasticity of clay is, without doubt, greatly affected by the

¹ *Trans. Am. Ceram. Soc.*, **16**, 392.

² *International Reports on Pedology*, **1911**.

³ Bureau of Standards, *Technologic Paper 46*.

presence of organic matter, humus, tannins, etc., and it seems to be a fact that acidity is conducive to the greatest development of this property. The aging of clays does indeed improve their plastic working quality and it is believed that this is due in part to the formation of organic acids, caused by bacterial action or chemical processes. The addition of small amounts of reagents like Al_2Cl_6 to kaolins brings about similar results provided the material is stored for some time, but no change is observed with the more impure materials. Tannic acid shows this action in a more marked degree.

Alkalies, on the other hand, are quite active in inhibiting the plastic quality of clays to a very pronounced extent. Electrolytes in general affect clays in that they increase or reduce the water required for the development of the plastic state and also the shrinkage upon drying. This effect depends obviously upon the kind and quantity of the salts already held by the materials and, as a rule, the purer clays are influenced to a greater extent than the more impure ones. Thus, upon adding up to 0.05 per cent of NaCl, $CaCl_2$, and Al_2Cl_6 to Georgia kaolin the volume contraction due to drying was reduced by about 1.0 per cent. Here again the first small additions of the reagent show phases indicated by maxima and minima which gradually disappear as the concentration is increased. With most clays, the salts, chlorides, and sulfates increased the water content and drying contraction.

DRYING SHRINKAGE

In the drying process itself we have the contraction in volume of the plastic clay as a typical property of colloid materials which may show a magnitude of as high as 100 per cent, or as much as the true volume of the clay itself. The drying shrinkage is thus in a measure a criterion of the colloid nature of the clay, being the greater the more pronounced this development is. The presence of electrolytes affects not only the magnitude of the contraction in drying but the capillary flow of the water through the clay as well. Thus it is well known that the addition of sodium chloride in small quantity will accelerate the passage of the water to the surface and thus lessen the strain imposed upon the clay in drying.

Upon drying clay and rewetting, it does not seem to recover its original plasticity at once and this is the more pronounced the higher the temperature to which it is carried. The retempering of the dried clay is associated with evolution of heat. We are dealing therefore with a case analogous to that of a "set" gel, and a change which becomes irreversible at a given temperature, usually at about $500^\circ C.$, unless subjected to treatment with steam at high pressure.

It is interesting to note, likewise, that the drying shrinkage is the greater the more slowly evaporation of the water is allowed to take place, and vice versa.

FIRING OF CLAY

In the firing of clay two points are of special interest during the early periods of the process, the expulsion of the hygroscopic and the chemically combined water. The removal of the former leaves the clay in a state exceedingly sensitive to the presence of water vapor and gases, absorbing them with great avidity. This condition is accentuated after the evolution of most of the chemically combined water, when the material becomes an even better absorbent, taking up eagerly sulfur dioxide and trioxide. In this state it possesses marked catalytic properties, as in the oxidation of sulfur dioxide to trioxide, the formation of ethylene, etc. It is possible that this characteristic of dehydrated clays may find uses in the technical application of a number of chemical reactions. This material shows considerable activity also when ground together with calcium oxide and moistened, producing a cement of good hydraulic setting qualities.

The dehydration is necessarily endothermic and is associated

with a decided increase in molecular volume. Upon further heating of the clay an exothermic reaction takes place at about $900^\circ C.$ which may be associated with an irreversible change of a colloidal nature, or the dissociation of the aluminum silicate, perhaps into $Al_2O_3 \cdot SiO_2$ and silica.

As the firing temperature is raised the external clay volume contracts, especially in the presence of fluxes, due to the effect of surface tension until the heat intensity is sufficient to bring about practically complete closing of the pore space. It is said then that the clay has become vitrified. With increased softening of the mass its lowered viscosity permits the surface-tension effect to become more and more prominent until finally the original shape has been lost and the surfaces curved analogous to the formation of a drop of water from a melting fragment of ice.

In the case of clay products the process, of course, is not allowed to proceed as far as this and the firing is stopped when the desired degree of vitrification has been reached, while with glass it is allowed to continue. At the same time the density of the vitrified or fused silicate decreases with increase in temperature, irrespective of the external contraction.

The relation between temperature, time, and contraction in volume is therefore a very important one and expresses the result of the heat work done. It enables us to represent graphically the progress of condensation or vitrification and is equally important in the study of the firing processes of carbon, alumina, zirconia, thoria, etc.

Although surface tension is probably the most important factor in bringing about the condensation of the substances with which we are here concerned, vapor pressure¹ undoubtedly enters into the case in dealing with substances showing a vapor tension sufficiently high or in the admixture of inert materials with those of a more volatile character. Thus, the comparatively high vapor pressure of magnesia at temperatures considerably below its fusion point is instrumental in bringing about its earlier condensation. Formation of sillimanite from kaolin and alumina may be brought about at about $1500^\circ C.$ by the addition of a small quantity of boric acid, and the cementation of carbon from coal is likewise made possible by the presence of volatile constituents.

In all the silicate reactions at higher temperatures diffusion through the semi-rigid mass plays an important role.

Finally, wherever the chemical composition and the heat treatment permit it the end result is the partial elimination of the colloid phases and their replacement by crystalline identities. Thus, the clay substance decomposes into sillimanite and silica, glass yields calcium silicate, and cement its several calcium silicates and aluminates, but there is invariably present a residuum of an isotropic matrix.

REPORTS

REPORT OF COMMITTEE APPOINTED TO DEFINE THE FIELD COVERED BY RESPECTIVE DIVISIONS AND SECTIONS OF THE AMERICAN CHEMICAL SOCIETY

The Committee Appointed to Define the Field Covered by Respective Divisions and Sections of the AMERICAN CHEMICAL SOCIETY has carried on correspondence with reference to the questions submitted, and now begs to report as follows:

1—It is believed that the designations which have been adopted for the different divisions and sections are themselves sufficient definitions of the field to be covered in each instance.

2—It is recommended that the established procedure of the SOCIETY shall be to give authors their choice of the division or section in which they are to present their papers whenever it is possible to do so. The Secretary of the SOCIETY shall be empowered to assign the papers to other divisions or sections in case the paper is obviously out of place or when in his opinion it will be to the advantage of the division or to the program in general so to assign it.

3—When submitting the titles of papers authors must indi-

¹ E. Podszus, *Sprechsaal-Arch.*, No. 1, 1912.

cate something of the discussion to be given, which might influence the proper placement of the paper. The title only in some cases is not sufficient indication, since a given subject may be discussed from any one of several angles, each one of which might place it before a different division of the SOCIETY.

Authors are required to state whether it is their bona fide intention to be present at the meeting so that papers to be read for them by others or to be presented by title can be properly indicated on the program.

4—The committee recommends that the secretaries of the divisions and sections adhere strictly to the vote of the Council that no papers shall be presented at the meeting unless their titles appear in printed form on the final program.

The committee recommends that the Council discuss the desirability of extending the rule now in force in the Division of Industrial and Engineering Chemistry, with respect to abstracts, to all other divisions and sections of the SOCIETY. It has been suggested that all authors be required to submit a brief abstract of their paper to the Secretary of the division or section before which they wish to appear, supplying their abstract at the time their title is submitted for the program. In case of misplacements of papers the division or section secretary would forward abstract and title to the General Secretary who could then properly place the paper on the final program. It is recognized that under such a plan the closing date for papers on the given program would be a few days earlier than at present, but it is believed that the advantage to be gained would more than offset this slight disadvantage.

H. E. HOWE, *Chairman*

COMMITTEE ON GUARANTEED REAGENTS AND STANDARD APPARATUS

A note was published in the December number of the *Journal of Industrial and Engineering Chemistry* requesting members of the SOCIETY to inform the committee of specific instances of unsatisfactory deliveries of chemicals or apparatus. There was little response to this request for collaboration, but the committee has reason to believe from work done by its members and from correspondence with others that deliveries of unsatisfactory reagents and apparatus are frequently made. The committee has assisted some members of the SOCIETY to obtain satisfactory chemicals for certain purposes.

In discussions in regard to apparatus the committee hears many complaints about delay in the testing and certification of apparatus at the Bureau of Standards. This question was considered from various points of view. Members of the committee visited the Bureau, meeting the Director and observing the testing of some kinds of apparatus. The committee believes that steps have been taken which will improve the conditions governing this work. With the present tendency to decrease appropriations for the scientific work of the government departments and the difficulty in obtaining persons to do the work at the salaries which can be paid, the committee feels that this question is one on which the SOCIETY may well continue to cooperate with the Bureau of Standards.

The committee was invited to a conference with members of the Bureau of Standards who have been studying the question of suitable glass tubing for making lamp-blown apparatus. It was felt that the project was still at a stage where it could be handled best by the Bureau of Standards. The committee believes that the practical problem of securing a reliable uniform supply of such tubing is one which is of fundamental importance if we are to have wholly American-made apparatus. The question is an economic and business matter rather more than it is technical.

Work has begun on the obvious task of preparing specifications for reagents. Manufacturers have given full details of their specifications and tests for the three acids and ammonia which were taken up for consideration. A number of complete analyses have been furnished from different laboratories and results are available of tests covering six or eight years in one laboratory. The committee has data to show what has been delivered in the past on orders for these reagents. Complete specifications have not been drawn up by the committee for any of these reagents. It was felt that a report of tentative specifications presented merely for discussion at this time would have little value.

The multiplicity of sizes and shapes of the ordinary laboratory apparatus has been a cause for complaint from dealers, manufacturers, and users. The committee has gathered some data and made some tentative selections of sizes and shapes for some of the common articles. These selections have not yet been submitted to manufacturers, and it does not seem worth while to publish them before they have been considered by the manufacturers. The Association of Scientific Apparatus Makers

of the United States has a committee on Standardization of Chemical Apparatus with which your committee has been cooperating.

It is believed that all the lines of work which have been discussed in this report might profitably be continued during the coming year.

W. D. BIGELOW, *Chairman*

COMMITTEE ON COÖPERATION BETWEEN UNIVERSITIES AND INDUSTRIES

It is the unanimous opinion of your committee that it is desirable that such students as can spend five years in their training should add a year of graduate study at the close of the work leading to the Bachelor's Degree, rather than continue a fifth year in work covered by a fixed curriculum. The training of such students in methods of research is considered more important than an increase in the number of formal courses of an undergraduate character. Some advanced courses should, however, be taken by such students during the fifth year. It is further the opinion of our committee that four years is too short a time for the thorough training of a professional chemist.

Professor Ellery has reported that an arrangement has been made by which advanced students in chemistry at Union College are permitted to attend the colloquia held by workers in the research laboratory of the General Electric Company. Such an arrangement is evidently very useful and it is hoped that the research laboratories of other companies may follow the example which has been set.

W. A. NOYES, *Chairman*

REPORT OF THE COMMITTEE ON OCCUPATIONAL DISEASES IN THE CHEMICAL TRADES

Since the last annual report (Buffalo Meeting, April 1919) some matters of interest have been the concern of your Committee on Occupational Diseases in the Chemical Trades which promise to be of importance.

(1) EYE PROTECTION—A representative of the committee (Dr. R. B. Moore or Dr. C. L. Parsons) has attended all the conferences of the General Advisory Committee on Industrial Safety Codes held under the auspices of the Bureau of Standards at Washington. A plan of procedure, after discussions and much correspondence, has been agreed upon. Specifications for goggles for different purposes have been drawn up and discussed, and will soon be ready for issuance. The specifications may be had, on application, from the Bureau of Standards.

(2) INFLUENZA AND POISONOUS GASES—Data on the probable prophylactic action of very dilute chlorine against influenza have been collected and a summary of the facts published in the *Journal of Industrial and Engineering Chemistry*, 12 (1920), 293.

Immunity from influenza of workers in bromine plants is certainly indicated from the experiences in the largest producers in the United States. Just the contrary appears from data secured from smaller producers and users. It is to be hoped that an investigation of this matter may be undertaken.

In connection with this it may be mentioned that Dr. Louis I. Dublin, Statistician of the Metropolitan Life Insurance Company, has secured other interesting data on the immunity against influenza of people in illuminating gas works. Dr. A. Gregor¹ has found the number of cases of influenza among cordite and tin workers in fumes to be one-sixth to one-third of those not subject to fumes. Dr. F. Shufflebotham² found one-twenty-fifth to one-fifteenth the number of cases of influenza in poison gas factories. Dr. Gregor's further studies³ indicate that the immunizing action of nitrogen dioxide and sulfur dioxide is due to the production of an acid medium in the upper respiratory passages which is inimical to microbial growth. An influenza outbreak was stamped out at a military camp by sulfur dioxide.

Mr. H. N. Jordan, Semet-Solvay Company, Syracuse, has found that a liberal and deep application of lanoline to the interior of the nose prevents the sensitive condition of the mucous membrane from irritating gases and germ infection which causes colds, etc.

(3) WOOD ALCOHOL—By invitation the chairman addressed the New York Academy of Medicine upon "Some Chemical Aspects of the Wood Alcohol Problem." The paper in full will appear in the *New York Medical Journal*. Certain phases of the subject are dealt with, especially some relative to prohibition, in a paper to be presented at the St. Louis meeting of our SOCIETY. The chairman has conferred with and assisted officials of the Society for the Prevention of Blindness. At least 90 per cent of the producers of refined methyl hydroxide have agreed

¹ *Brit. Med. J.*, March 1 (1919), 242.

² *Ibid.*, April 1919, 478.

³ *Ibid.*, October 1919, 523.

to eliminate the word "alcohol" entirely from their labels, using "methanol" instead. Their advertisements now so read. It is to be hoped that every chemist will endeavor to render the designations "wood alcohol" and "methyl alcohol" obsolete.

No material changes in state laws have been made since 1912. This has been learned by correspondence with all the secretaries of the states. Administration of the present laws will conserve the public welfare and work little hardship upon the producers.

(4) DERMATITIS AMONG CHEMICAL WORKERS—Dr. E. R. Hayhurst, Ohio State Department of Health, reports to the committee numerous complaints of dermatitis by workers in rubber plants, seemingly due to the use of hexamethylenetetramine as an accelerator.

Dr. W. S. Landis has directed attention, in Bulletin No. 10 of the American Cyanamid Company, to the fact that "liquor and cyanamide are quite antagonistic." Heavy drinkers are unable to work in an atmosphere containing cyanamide. The dust seems to produce a skin irritation resembling eczema with those who imbibe mildly. This does not seem to have been the case in certain European plants, according to Dr. C. L. Parsons, and is now apparently of passing interest only in America.

A note published in the *Journal of Industrial and Engineering Chemistry*, 11 (1919), 797, on development of skin diseases in working with certain lubricants created not a little correspondence and did some good.

More publicity of an educational nature in regard to occupational rashes is deemed advisable.

(5) OCCUPATIONAL DISEASES AND DYE INDUSTRIES—Dr. T. H. Cone, National Aniline and Chemical Company, presented a valuable paper at the Eighth Annual Safety Congress, entitled, "Dangers in the Manufacture of Dyes." The risks "are largely centered about the manufacture of intermediates." The explosion risks are the greatest, the dyes themselves being the dangerous element. "Be extremely critical toward dyes which are to be handled in the form of dry powders, unless they are well supplied with sulfonic acid groups."

In connection with the above, particular attention is directed to a most valuable report by Dr. Alice Hamilton, entitled "Hygienic Control of the Anilin Dye Industry in Europe," which has been reviewed and commented on by the chairman of the committee in the April number of the *Journal of Industrial and Engineering Chemistry*.

(6) COÖPERATION WITH NATIONAL SAFETY COUNCIL—The National Safety Council has now nearly 4,000 members, including almost all the important industries doing safety work. The chairman continues his membership for the CHEMICAL SOCIETY as the National Safety Council has become a clearing house for safety experiences. While many of these are specifically accidental in character and might not appear to come within the scope of the activities of this committee, yet many are distinctively occupational and deal directly with prevention of diseases, acute and chronic, growing out of, or arising from, the chemical trades. The *National Safety News* has become a valuable medium for the dissemination of information on every phase of industrial development. The *Safety Practice Pamphlets* distributed to members are alone worth the annual dues. Attention of chemical manufacturers, not already familiar with this excellent agency, is especially directed to the National Safety Council by the committee.

(7) INSTITUTE FOR INDUSTRIAL HYGIENE—On November 14, 1919, a serious conference on Industrial Hygiene was held at the request and under the auspices of the Rockefeller Foundation at the Yale Club, New York City. Copies of the personnel list and digest of the important discussions may be had from Mr. E. R. Embree, Secretary of the Foundation, 61 Broadway. The contribution of your chairman, who was one of the guests, is given *in extenso*. This has later been published in the *Journal of Commerce* (New York, February 23, 1920) and will appear also in *Modern Medicine* (Chicago). The need of education and sane publicity, adequate social workers of qualified knowledge, and the necessity for research, with the coöperation of the chemist but the direction of a preventive medicine welfarer, were pointed out by your representative. The necessity for a program of magnitude calling for large financial support was agreed upon in principle, although there were the expected personal differences as to details by the experts present.

The trustees subsequently decided not to undertake the project, due to no lack of appreciation and sympathy, but for fear "that any studies or activities which in any way affected the industrial field would be subject to some prejudice and misunderstanding if carried on by one of the large foundations." General Schereschewsky advocated it as a Federal function, perhaps as a part of the United States Public Health Service. The subject is one of fundamental importance, not only because its purpose is humanitarian, but because it is good business to

have workers healthy. Our SOCIETY should, and no doubt will, coöperate with other agencies in the furtherance of a project so commendable.

(8) FINANCIAL SUPPORT—It is patent to anyone giving the matter consideration that the work of our committee has not been systematic. Although something of distinct value has been accomplished in the way of research, in legislation, and in publicity, it could not be organized on a satisfactory basis for the very good reason that it has had no financial support. The time has arrived when the Council needs to give the matter serious consideration. We cannot get away from the responsibility.

The mephitic odor of the laboratory or the "works" covers a maze of mystery in the lay mind and is held responsible for numerous maladies. The word "chemical" is sinister. Where chemical processes are in operation, especially when fumes or vapors, even though the latter be only steam, are seen to escape from the works, there grows up a feeling of suspicion, often with reason, and all kinds of sickness are attributed to these "poisonous substances." If the chairman may judge from numerous letters and many conferences, even the members of the medical profession are not free from these sensations and confessions. And well they may be in many instances, for even though the action of many chemicals in varying degrees of concentration be known, the insidious effects of many more are absolutely closed books. They require most diligent study and painstaking research.

In this connection there is an urgent need for publication of an immense amount of valuable research carried on by the Chemical Warfare Service of the Army and Navy, which now appears to have little chance to reach the light of day. The data on the physiological factors and changes involved in fatigue are of immense value. The alterations in gas masks for use in chemical industries in peace-times constitute an important problem. A consideration of the few new facts collected bearing upon the seeming influence of certain toxic gases, referred to elsewhere in this report, opens a vista comparable to the hopeful days of the Pneumatic Institute, out of which came inhalation anesthesia, gaseous disinfection, and foundational knowledge of bacterial changes.

CHAS. BASKERVILLE, *Chairman*

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REPORT OF THE COMMITTEE TO FORMULATE SPECIFICATIONS FOR THE CONSTRUCTION OF A POLARISCOPE FOR LABORATORY USE

Before the committee was appointed there had been considerable correspondence among sugar chemists regarding the desiderata for a polariscope for ordinary laboratory use. The correspondents included the sugar chemists of the United States, Hawaii and Cuba and there were several conferences in the various places among the men who could arrange to meet. The correspondence regarding this matter was available to the committee and greatly facilitated our work. It has not been possible for our committee to arrange a meeting which all members could attend and most of our consultation has been by correspondence. There has been much personal conference among individual members of the committee however, and one conference was arranged at which five members of the committee were present. We have also had the advantage of the coöperation of manufacturers of optical instruments in this country, one of whom has made a polariscope during the last year which complies with the requirements of the committee.

GENERAL CONSTRUCTION

The general construction of the saccharimeter outlined in these specifications should be as simple and substantial as possible. All parts of the instrument should be easily accessible and the number of bolts and screws for holding the parts in place should be reduced to the necessary minimum.

So far as possible the instrument should have smooth, plain surfaces and be without unnecessary ornamentation. An irregular ornamented surface affords grooves and crevices for the accumulation of dirt and is not easily cleaned.

The instrument should meet the requirements of exposure to a humid tropical climate and must be constructed to withstand corrosion.

The construction of the saccharimeter, so far as possible, should be of such a type that repair parts can be furnished separately, thus obviating the expense, the delay, and the danger of shipping the entire instrument. Wherever this is not practicable manufacturers should undertake to make repairs in a satisfactory manner without undue delay.

HEIGHT

The standard height of most saccharimeters from table to center of field eyepiece is between 32 and 34 cm. This height is convenient for manipulation with the elbow of the operator resting upon the table and has found most general approval.

MOUNTING

The saccharimeter should be mounted upon a rigid trestle support and not upon a tripod. Instruments mounted upon tripods are unstable and easily turned out of alignment, the result being an error in the zero point.

The base of the trestle should be a solid piece of metal at least 2 cm. thick, the bottom edge of which can rest at all points upon the table. A base elevated above the table by supporting knobs or projections lacks rigidity and has the disadvantage of permitting cover glasses and other objects to escape underneath.

As many chemists prefer to fasten their instruments to the table, the base of the trestle should be provided with slots or screw holes to facilitate this.

LAMP SUPPORT

The lamp end of the trestle should be designed to accommodate a strong removable bracket for the convenience of those who may wish to use it as a lamp support, thereby keeping lamp and instrument always in undisturbed alignment.

The holder of the lamp must be placed at the proper focal distance and should be adjustable. Bracket and holder should be designed so as to prevent transmission of heat to the polarizer of the instrument.

For rooms of constant temperature the lamp should be in a separate room.

TROUGH

The trough, or tube holder, should be of solid metal, in one piece, and sufficiently thick to prevent denting or bending under ordinary conditions of usage. The diameter of the trough at the top should be about 3 cm. and should be adjusted exactly to fit the end pieces of the observation tubes. The cross-section of the trough should be semi-circular in shape. A wedge-shaped trough does not give the necessary support to inversion tubes or other tubes of unstable equilibrium.

The length of trough should be 42 cm. This length is necessary for the accommodation of the control tube which is used for verifying the accuracy of the scale. The short 20 cm. trough does not permit the use of the control tube and is also inadequate for the polarization of sweet waters and other dilute solutions.

The base of the trough should be supported to the frame of the trestle and there should be a 2 mm. space between its ends and the rest of the instrument. This clearance allows the escape of any liquid which may be spilled in the trough and protects against warping of the trough and transmission of heat to the optical parts when polarizations are made at high temperature. The base of the trough must be parallel with the optical axis of the instrument.

The trough should be made removable for the accommodation of other forms of tube supports or baths that may be needed in special cases. Owing to the corrosive action of solutions, which may be spilled inside of the trough, the screws for fastening the trough should be on the outside.

TROUGH COVER

The trough should be provided with a hinged cover for excluding light. The cover should be long enough to cover the 2 mm. space between trough and splash-glass holders, and should fold back to a horizontal position where it can be used in case of need as a receptacle for tubes.

The hinges of the cover should not be riveted. Many chemists find the trough cover an encumbrance, and for the convenience of such it should be easily removable.

For the convenience of those who use continuous, or side-filled, polarization tubes, a slotted cover should be provided as an optional accessory.

SPLASH GLASSES

The splash glasses at the ends of the trough, for protecting prisms and wedges against dust and drops of liquid, should be mounted in holders which can be quickly removed, cleaned, and replaced without the use of tools. Slip holders with a tension spring are most generally preferred, and they should be designed to prevent sticking.

The two holders should be as near alike as possible, at least 1.5 cm. deep and so constructed that glasses can easily be removed for cleaning. For ease of replacement when damaged, the splash glasses should be of the same size as the standard polariscope tube cover-glass.

QUARTZ WEDGES

For a commercial saccharimeter all chemists prefer the compensating single-wedge system. The wedge should be of sufficient length to give a range of scale from -35 to 115 sugar degrees.

If quartz of sufficient optical purity to give this lower range cannot be secured, a dextro quartz plate should be provided as an accessory for use in invert polarization.

The driving mechanism of the wedge should consist of a vertical rod supported to the front of the trestle frame and provided

at the bottom with a milled head about 7 cm. from the table and convenient for operation with either hand.

The spiral rack and pinion with which the driving rod connects should operate smoothly and without lost motion.

SCALE

The scale should be etched upon ground glass and read by transmitted light obtained from the light source of the instrument. The design of the instrument should be such that the scale can be illuminated, when continuous or control tubes are in upright position in the trough. The range of scale should be from -35 to 115 . This upper limit is necessary for those who wish to determine purities without diluting below 20° Brix.

The scale should have an adjustable double vernier, for plus and minus degrees, and should easily be read to 0.05° to which end the magnifying power of the reading microscope should be amply large. The error of scale graduation should nowhere exceed 0.05° .

The adjusting screw for moving the vernier to the zero point of the scale should operate positively in either direction. In some instruments a spring is designed to act when the adjusting screw is withdrawn. The objection to such a spring is its liability to stick and not to operate as intended.

Before shipment, the scale of each instrument should be carefully standardized at suitable intervals throughout its entire range and the standardization values should be incorporated permanently in some way upon a plate attached to the instrument.

PROTECTION CASE

Scale and wedges should be enclosed in a tight protection case to prevent deposition of dust or spattering with drops of liquid. The covering of the case should be easy to take off, when it is desired to gain access to scale or wedge, by the removal of a few fairly large-size screws. The rim of the protection case should have a covered aperture for inserting the key of the adjusting screw.

Whenever desired the front of the case should be provided with a small thermometer having a range of 10° to 40° C. and with its bulb near the quartz wedge. The thermometer should be arranged so that it can be read in a darkened compartment by light obtained from the lamp which illuminates the instrument.

SCREEN

The protection case should be designed to accommodate a removable screen to protect the eye which is not in use from the glare of the lamp. The screen should have a diameter of about 15 cm. at the level of the two oculars.

ANALYZER

While the analyzer is one of the parts which should require least attention, there are occasions when it needs to be adjusted. It should be made fairly accessible and be provided with simple means for firmly securing the adjustment.

LIGHT FILTER

The light filter should be placed between the polarizer and light source of the instrument and should be so supported that it can be quickly thrown into the field or out without disturbing the position of the instrument.

The standard bichromate cell should consist of a glass tube 3 cm. long encased in a metal jacket with threaded ends to accommodate the screw caps for holding the glasses. The cell should have a sufficient diameter so as not to require refilling because of air bubbles during an ordinary campaign (or more than twice a year).

Many chemists desire a lightly ground glass over the aperture at the lamp end of the instrument to equalize the light. Such a glass, if properly tinted, might serve the double purpose of light filter and equalizer. As a matter of convenience the instrument should be equipped with a light filter consisting of a glass plate of the same depth of color and absorptive power as the standard light filter.

OCULARS

The oculars in front of the instrument for reading field and scale should focus with a screw motion. The sliding eyepiece is objectionable, owing to the ease with which it is pushed out of adjustment by the face of the observer.

The distance from center of field eyepiece to center of scale eyepiece varies in present instruments from about 3 to 6 cm. For convenience and rapidity in reading, the interval between the two eyepieces should lie within these limits.

FIELD

American chemists with few exceptions prefer the customary double field with vertical semi-circular halves. The field should be of good size, sharply defined, and not obscured with the rim or halo of extraneous light, which results from improper optical construction.

POLARIZER

The preferences as to polarizer are divided between the Lippich and Jellet-Cornu prisms. Many chemists, while admitting certain advantages of the Lippich polarizer, complain of its frequent disintegration along the sharp edge of the half-prism upon which the telescope is focussed, the result being an imperfect or shattered field. The disruption of the half-prism may result from a jar of the instrument or it may take place from no apparent cause. More saccharimeters are made unserviceable for this reason than for any other. The difficulty of repairing the damage, owing to the extreme fragility of the parts, renders the Lippich polarizer less suited for localities which are far distant from repair shops. Many chemists, on the other hand, who admit the greater stability of a modified Jellet-Cornu prism, complain of its lower degrees of sensitiveness owing to the pronounced dividing line of the field, the result either of too thick a film of balsam between the halves of the upper part of the prism or of imperfect alignment of the polarizer. The defects peculiar to each type of prism can largely be overcome by careful manufacture. There are many stable Lippich polarizers and many Jellet-Cornu prisms that are satisfactory in sensibility.

If manufacturers can make repairs rapidly and can furnish extra interchangeable half-prisms of easy adjustment, the usefulness of the Lippich polarizer would be much widened and the majority of chemists in fairly accessible localities would probably then prefer it. For remote tropical countries where repairs are difficult and time-consuming a modification of the Jellet-Cornu prism would probably be the better type. For these reasons the type of polarizer should in great measure be left optional with the purchaser. In their manufacture of polarizing prisms manufacturers should take every precaution to insure stability and to prevent drying out and cracking of the films of balsam cement.

A very serious complaint from tropical countries is the infection of the polarizer, analyzer, and other optical parts of the instrument by molds, the mycelia of which grow over the prisms, corroding their surface and obscuring the field. Efforts to prevent infection by enclosing the parts more adequately have not proved successful. The best means of preventing the growth of molds seems to be a construction that permits of easy accessibility and removal of parts for cleaning and for placing in desiccators during periods when the saccharimeter is not used.

MOUNTING OF PRISMS—Wax, as a mounting material for prisms, has proved objectionable in warm climates on account of its softening. A mounting in cork and plaster is said to be the most satisfactory.

HALF-SHADOW ANGLE—The fixed half-shadow angle of the polarizer in most saccharimeters varies from 5 to 9 angular degrees, the choice of angle by different manufacturers seeming to depend somewhat upon the length and pitch of the quartz wedge. It is probable that for general commercial purposes the half-shadow angle should fall within this range. The sensibility is greater but the intensity at the end-point is less with the smaller half-shadow angle. Recent improvements in electric stereopticon lamps with concentrated filament and high candle power make it possible for manufacturers to adapt saccharimeters to a lower half-shadow angle than was formerly the case. For a normal weight of 26 grams the fixed half-shadow angle should have a magnitude of at least 7° for the average class of sugar factory raw products. The angle may be smaller than this for colorless products. The angle may also be reduced for raw products with instruments which are adapted to a normal weight of 20 or 16.29 grams.

Chemists who work constantly with dark-colored sirups and molasses prefer a polarizer with a rather wide half-shadow angle. It would, therefore, be a distinct advantage if manufacturers could supply interchangeable polarizing prisms—one with a medium half-shadow angle between 5 and 8 angular degrees and another with a higher half-shadow angle between 9 and 12 degrees.

Polarizing prisms should be mounted in metal holders which can be easily removed and inserted and the adjustment of which can be quickly and securely fixed.

The sleeve, or cover, which protects the polarizer should be easy to take off by the removal of a few fairly large-sized screws.

The standard temperature for the calibration of saccharimeters shall be 20° C. For laboratories working at a temperature materially different from this, correction of polarizations to 20° C. may be made at discretion by any of the following methods:

- 1—By the use of a table of temperature corrections for each particular product.
- 2—By changing the normal weight.
- 3—By changing the capacity of normal flask.
- 4—By changing length of normal tubes.
- 5—By having a scale calibrated by the manufacturer so that it is correct for the temperature desired.

With the exception of the first method these methods of correction are strictly applicable only to products which contain no other optically active constituent than sucrose. For general sugar house and food products containing several sugars, in case constant temperature polarization at 20° C. is not permissible, Method 1 gives results which are nearest to those obtained at the standard temperature.

STANDARD OR NORMAL WEIGHT

A large majority of chemists believe that the present is a most suitable time to abandon the inaccuracies and inconveniences of previous national standards and to agree upon a saccharimetric scale which shall be accurate, convenient, and so far as possible international. From opinions expressed by leading chemists in America, England and France, it is apparently more easy to secure general agreement, in these three countries at least, upon the so-called international normal weight of 20 grams proposed in 1896 by Sidersky and Pellet. According to this standard the 100 degree point of the saccharimeter scale shall indicate the polarization of 20 grams of chemically pure dry sucrose dissolved in distilled water to 100 metric cc. and polarized in a 20 cm. tube, the temperature of solution and instrument to be 20° C. and the illumination to be white light filtered through a solution of potassium bichromate of such concentration that the percentage content of the solution multiplied by the length of the layer of solution in centimeters is equal to nine.

While many chemists believe that all saccharimeters hereafter manufactured for American use should be graduated solely according to this proposed international standard, it is the opinion of this committee that pending international agreement upon the question manufacturers of saccharimeters should standardize their instruments according to the scale desired by the individual purchaser.

In the adoption of an international saccharimetric standard this committee believes that—in order to avoid the numerous unfortunate changes which in the past have characterized previous standards, and in order to have absolute accuracy and uniformity in different parts of the world—the percentage content, specific gravity, refractive index and angular rotation of the normal sugar solution, and the angular rotation, in terms of sodium and mercury monochromatic light, of the quartz plate, which shall read 100 upon the saccharimeter scale, should be established by carefully conducted experiments in the governmental laboratories of the different countries; and that from the results thus found international agreement shall be reached in regard to the final values, upon which manufacturers shall base the standardization of their instruments. The U. S. Bureau of Standards has already completed investigations upon the rotation values and other constants of the 26 and 20 gram normal sugar solutions.

It is not intended that the establishment of an international normal weight shall throw out of use the large number of saccharimeters which are at present doing excellent service. The transition is to take place gradually as in the change from Mohr to metric cc. Old instruments as they wear out are to be replaced by instruments with the new scale. Owners can also have their old instruments rescaled but this should be done only when the accommodation of scale, polarizer, lenses, etc., secures an equal or greater accuracy in reading. If an old instrument is not rescaled it should at least be standardized and the weight of pure sucrose necessary to read 100 upon its scale be engraved or stamped upon a plate attached to the instrument. Correction of instruments by the adjustment of an incorrect scale to a correctly standardized quartz plate is open to criticism, as a scale thus adjusted is accurate only for the reading of the plate and a considerable error may be introduced at other points of the scale, especially when readings have to be corrected for dilution.

W. D. BIGELOW, *Chairman*

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**REPORT OF THE COMMITTEE ON ENDOWMENT OF THE
AMERICAN CHEMICAL SOCIETY**

The committee, I. K. Phelps, *Chairman*, submitted a report which, in abstract, was as follows:

There is every reason why the AMERICAN CHEMICAL SOCIETY should be endowed since it shares with the universities the common purpose of the advancement of learning.

The endowment should be general rather than specific, for the SOCIETY is a living thing with changing needs. For instance, just now it may seem desirable to provide for improvements and extensions in *Chemical Abstracts*, but in the course of time the plan may be revived, which Ernest Solvay developed before the war, of an abstract journal in various languages to care generally for the chemists of the world. If this plan should ever be carried out, it might involve a duplication of effort to continue a line of work here which to-day is greatly needed.

SUGGESTED USES FOR ENDOWMENT

Conditions are now favorable for American journals to assume the leadership formerly held by German journals. The SOCIETY publishes three journals, all ably edited; but if the budget could allow adequate appropriations for the *Journal of the American Chemical Society* and *Chemical Abstracts*, and permit the *Journal of Industrial and Engineering Chemistry* to profit by its own earnings, all could be brought up to a better standard.

We have also to face the problem of publishing monographs and compendia. The urgency for the compilation and publication of these is extreme, but the funds are lacking.

If the Secretary's office could act as a clearing house for chemical information, coordinated with the National Research Council and the government activities, it could accomplish much in collating and spreading reliable chemical information. A traveling assistant secretary, as suggested by Mr. Little, could be added to that office with much profit.

Under present conditions, it will probably become necessary to raise the dues of the SOCIETY before long, thus cutting off from its benefits chiefly students and young chemists in the formative period. Such an increase in cost would, therefore, be a tax on progress.

While the more immediate purpose of the endowment is for the spread of knowledge rather than for its initial achievement, it would be unfortunate to forbid the authorities of the SOCIETY to use its funds for research if the need should arise.

PROPOSED PLAN OF SECURING ENDOWMENT

It is proposed that the present Endowment Committee be discharged after the completion of the work upon the plans for securing endowment; that two new committees be appointed by the President; the one (hereafter to be referred to as the Endowment Collection Committee) to carry out such plans of collect-

ing endowment as shall be approved, the other (hereafter to be referred to as the Endowment Finance Committee) to receive and invest such moneys as are collected for endowment. This latter committee should have a banker included in its numbers.

To start the project, it is suggested that the Advisory Committee select definite objects, upon which the Endowment Collection Committee can make a campaign; that the appeal of this committee be for a Permanent Endowment Fund, the income only of which shall be available for expenditure at any time and the objects for which this income shall be expended to be determined by the Advisory Committee, the Directors or a new committee constituted for the purpose; that at the present time an attempt be made to secure one million dollars for this fund; that the Endowment Collection Committee consist of a chairman, who shall be appointed by the President and empowered to associate with himself such other helpers as may be approved by the President.

The following plans for use of the Endowment Collection Committee are suggested:

1—Direct appeal for contributions, under the advice of the President, to the end the appeals may not be overdone.

2—Appeals for bequests of definite sums by will from chemists, from chemical manufacturers, and from others.

3—Appeals for the purchase of Endowment Life Membership, to cost such a sum as shall by its interest during the life of the member defray his dues to the SOCIETY and at his death be transferred to the General Endowment Fund together with any excess in income which may be derived from the Endowment Life Membership Fund. This fund should be under the same management and control as the General Endowment Fund, and entirely separate and distinct from the present Life Membership Fund.

ORIGINAL PAPERS

LOW TEMPERATURE DISTILLATION OF SUB-BITUMINOUS COAL

By H. K. Benson and R. E. Canfield

LABORATORY OF INDUSTRIAL CHEMISTRY, UNIVERSITY OF WASHINGTON, SEATTLE, WASHINGTON

Received December 29, 1919

The results obtained from the low temperature distillation of a non-commercial lignite coal¹ in the state of Washington appeared of sufficient value to warrant further investigation on a semi-commercial scale. Such work is now under way with somewhat different apparatus on another lignite coal. In connection with the mining of sub-bituminous coals it has frequently been found unprofitable to work certain veins on account of their high ash content or other unmerchantable properties. For instance, the Bagley vein at Coal Creek, Newcastle, Washington, is described as being "too dirty" for the production of a commercial fuel and although of sufficient size is not worked. It seemed of interest, therefore, to ascertain the suitability of this material for distillation purposes.

DESCRIPTION OF SAMPLE

The coal is black in color, but the streak is brown-black as the powder shades off to brown. It has a rather dull luster, is massive in texture, without joints, and has a conchoidal fracture. The proximate analysis is given in Table 1.

TABLE 1—PROXIMATE ANALYSIS OF NEWCASTLE COAL (PER CENT)

	Laboratory Sample		Sample Reported	
	As Received	Air-Dry	U. S. Geol. Sur., Bull. 474 As Received	Air-Dry
Moisture.....	12.1	5.8	7.1	5.9
Volatiles.....	36.8	39.4	37.8	38.1
Fixed Carbon.....	40.7	43.6	42.9	43.1
Ash.....	10.41	11.15	12.2	12.9
Sulfur.....	0.34	0.36	0.34	0.38
Nitrogen.....	1.37	1.47	2.44	2.61
B. t. u.....	10410.0	11150.0	10410.0	11300.0

¹ THIS JOURNAL, 9 (1917), 946.

APPARATUS AND PROCEDURE

The methods of procedure and the apparatus were with a few minor modifications the same as previously described.

PRODUCTS OF DISTILLATION

The yield of raw products at the different distillation temperatures is given in Table 2.

Temperature C.	Total Distillate Per cent	Dry Tar Per cent	Aqueous Solution Per cent	Coke Per cent	Gas Cu. Ft. per Ton
150	6.9	..	6.9	90.2	3
200	9.2	0.5	8.7	85.1	8
250	17.1	1.9	15.2	74.1	22
300	18.3	2.8	15.5	70.6	2070
350	19.8	3.5	16.3	67.2	3540
400	18.9	3.2	15.7	66.1	4850
450	18.7	2.8	15.9	65.3	5990
500	17.7	2.3	15.4	64.2	6910
550	17.1	1.6	15.5	63.1	7670
600	16.1	1.0	15.1	62.2	8310

GAS

A wet test meter was used for measuring the gas the yields being indicated in Fig. I. The gas samples were analyzed in a Morehead gas apparatus and the percentage composition is given in Table 3.

TABLE 3—PERCENTAGE COMPOSITION OF GASES

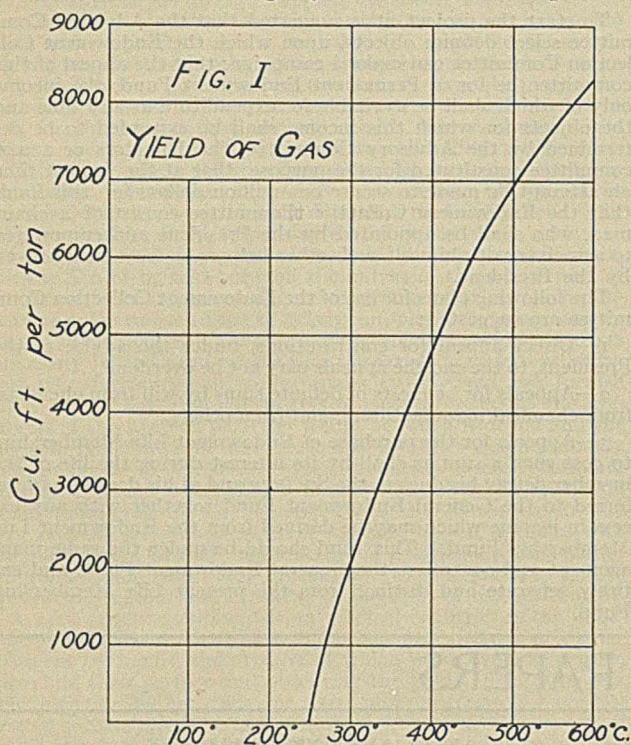
° C.	CO ₂	H ₂	O ₂	CO	H ₂	CH ₄	N ₂
250	45.1	3.3	1.4	7.1	0.3	8.6	34.2
300	33.2	3.8	0.8	8.3	0.9	10.7	42.3
350	20.4	4.9	0.4	9.6	8.1	23.6	33.0
400	18.9	5.0	0.4	11.8	9.8	21.2	32.9
450	17.3	4.9	0.4	13.3	11.5	20.2	32.4
500	16.4	5.1	0.4	14.5	13.0	18.4	32.2
550	14.1	5.1	0.4	15.6	15.1	17.8	31.
600	12.6	5.3	0.4	16.8	15.9	17.2	31.8

The calorific values calculated by Lunge's method are plotted against the temperatures of distillation in Fig. II.

COKE RESIDUE

The coke obtained up to 500° C. was of a dull black color and retained more or less the size and form

of the original particles. From this point to 600° C. the particles possessed more of a metallic or silvery luster and swelled slightly in size. Its composition



is given in Table 4, which also shows the yields per ton of coal. In Fig. III is shown the increase in calorific value with increase of temperature. The thermal values of the coke were obtained by means of the Emerson bomb calorimeter.

TABLE 4—YIELD AND COMPOSITION OF COKE

Temp. °C.	Coke Lbs.	Volatiles Per cent	Ash Per cent	Nitrogen Per cent	B. t. u.
150	1804	38.0	14.3	2.9	11,800
200	1702	31.8	15.1	2.7	12,210
250	1482	26.7	17.5	2.5	12,500
300	1412	22.5	18.2	1.8	12,690
350	1344	19.4	19.3	1.4	12,790
400	1322	16.2	19.5	1.1	12,870
450	1306	13.1	19.9	0.9	12,930
500	1284	9.9	20.1	0.7	12,980
550	1262	6.8	20.4	0.6	13,020
600	1244	4.1	20.7	0.5	13,040

AMMONIACAL LIQUOR AND CYANIDES

The total distillate was well washed and the washings tested for ammonia and cyanides. Most of the ammonia was found to be held by the tar before it reached the acid wash bottle, while the cyanogen content seemed to be nearly all carried over to its respective wash bottle. The cyanide precipitated out upon the addition of ferric alum even at the lower temperatures. As this precipitate was intermixed with impurities, as noted by the color, no significance would attach to the amounts obtained at the various temperatures, but it was estimated that approximately 1.25 lbs. of cyanogen may be obtained at 600° C. per ton of coal. The yield of ammonium sulfate in pounds per ton of coal is given in Fig. IV.

TAR OILS

All of the water was extracted by calcium chloride from the crude distillate and the dry tar obtained was red-brown in color and semi-solid at ordinary tempera-

tures. Its specific gravity ranged from 0.975 to 1.033. The oils were distilled in small glass retorts and the percentage yields are given in Table 5, including the hard and soft paraffin separated by a modification¹ of Eisenlohr's method.² About half a gram of the paraffin oil was weighed to the nearest milligram in a tared 7-in. test-tube and warmed slightly.

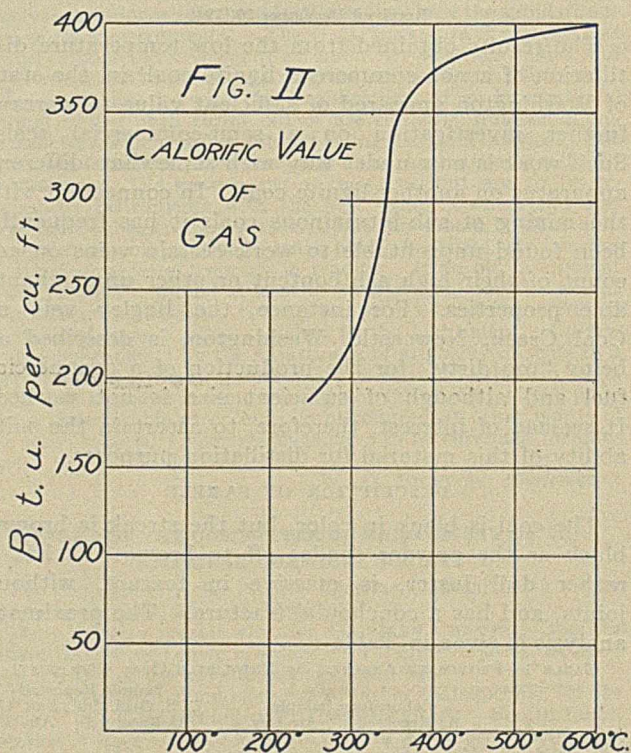
TABLE 5—PERCENTAGE COMPOSITION OF OILS

Temp. °C.	To 150° C. Light Oil	150°–300° C. Medium Oil	Above 300° C. Paraffin Oil	Hard Paraffin	Soft Paraffin	Coke	Loss
250	10.3	42.1	33.1	3.9	1.3	9.6	4.3
300	10.7	42.5	33.4	5.0	1.8	10.0	3.3
350	11.3	41.9	33.9	5.5	2.1	9.7	3.2
400	10.5	41.7	33.2	5.2	1.9	10.3	4.3
450	9.3	41.4	31.4	3.4	1.5	13.6	4.0
500	7.8	41.5	39.8	2.1	1.4	15.9	5.0
550	5.9	41.2	29.2	0.9	0.7	17.6	5.9
600	3.9	40.9	27.8	0.6	0.5	21.1	6.3

TABLE 6—YIELD OF OILS IN POUNDS PER TON OF COAL

Temp. °C.	Raw Tar	Light Oil	Medium Oil	Paraffin Oil	Hard Paraffin	Soft Paraffin
250	38	3.9	16.0	12.6	1.5	0.5
300	56	6.0	23.8	18.7	2.8	1.0
350	70	7.9	29.3	23.7	3.8	1.5
400	64	6.7	26.7	21.2	3.3	1.2
450	56	5.2	23.2	17.6	1.9	0.8
500	46	3.6	19.1	13.7	1.0	0.6
550	32	1.9	13.2	9.3	0.3	0.2
600	20	0.8	8.2	5.6	0.1	0.1

Twenty cc. each of ethyl ether and ethyl alcohol were added and the mixture cooled to -20° C. in a salt and ice bath. The flaky mass was filtered through a 4-in. funnel surrounded by freezing mixture, the filtrate being evaporated and the soft paraffin determined by freezing from an ether-alcohol (1 : 2) mixture after evaporating upon the water bath. Any admixture of impurities was removed by dissolving the paraffin in the filter by pouring boiling ethyl alcohol through it and collecting the filtrate in a tared beaker,

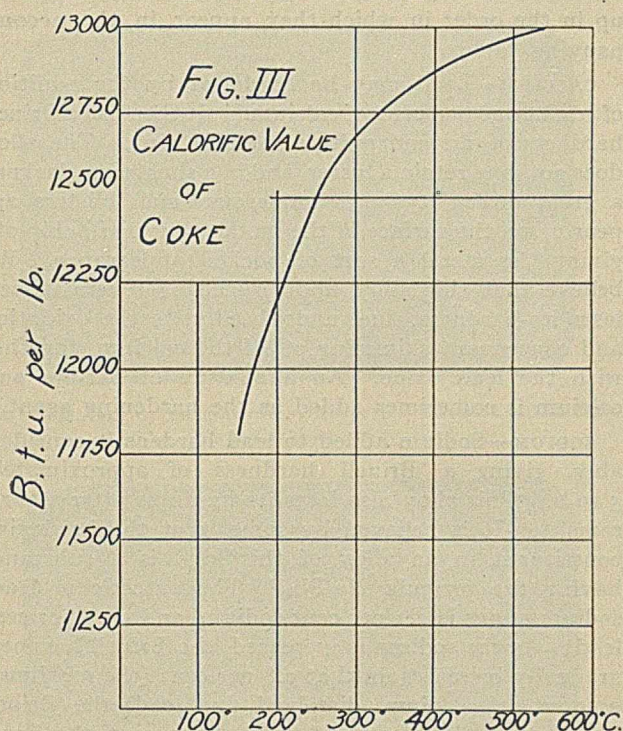


the alcohol evaporated and the hard paraffin (m. p. 45° to 55° C.) weighed.

¹ School of Mines and Metallurgy, Univ. of Missouri, *Bulletin* [4], 3, 55.
² *Z. angew. Chem.*, 1897, 300, 332.

PROPERTIES OF TAR OILS

The light oils were washed with sodium hydroxide and sulfuric acid, 20 per cent being soluble in alkali.



The washed oils were then treated with concentrated nitric and sulfuric acids in a reflux condenser, whereupon nitro derivatives were obtained to some extent, but the limited amount of oils obtained made it impractical to ascertain the yield. Phenols also were isolated upon the addition of bromine water to the cresols obtained from the alkali washings. The specific gravities of the various fractions are recorded in Table 7.

TABLE 7—SPECIFIC GRAVITY OF TAR FRACTIONS

Temp. ° C.	Light Oil	Medium Oil	Paraffin Oil
250	0.807	0.905	0.939
300	0.815	0.908	0.950
350	0.812	0.915	0.957
400	0.815	0.926	0.968
450	0.816	0.935	0.966
500	0.816	0.938	0.978
550	0.818	0.946	0.983
600	0.821	0.955	0.985

DISCUSSION

The most striking feature of the investigation is the well-defined decomposition point between 350° C. and 400° C., approximating closely the same temperature as in the previous study, *viz.*, 380° C. This marks a maximum in the yield of tar oils, and an abrupt rise in the quantities of hydrogen and methane, the change of composition in the gas being evident from the shape of the curve (Fig. II). At this point also occurs a decrease in paraffins, suggesting the possibility of the cracking of the oils.

Unlike the previous study, the presence of cyclic compounds in the light oil was proved by the formation of nitro derivatives and phenols. The light oils were distilled¹ and very small amounts of benzene (to 95° C.) and toluene (95° to 120° C.), and a considerable amount of solvent naphtha (120° to 200° C.) were

¹ *Met. & Chem. Eng.*, 17 (1917), 551.

recovered. The light oils soluble in sodium hydroxide constitute the cresols and unsaturated acids. The medium oil is made up in part of naphthalene, anthracene, and the higher paraffins, and is semi-solid at ordinary temperatures. Soft pitch is contained only in tars obtained at 500° C. and above.

CONCLUSIONS

I—The maximum yield of tar oils is obtained at 350° C.

II—About 3.5 per cent of the coal may be obtained as raw oils.

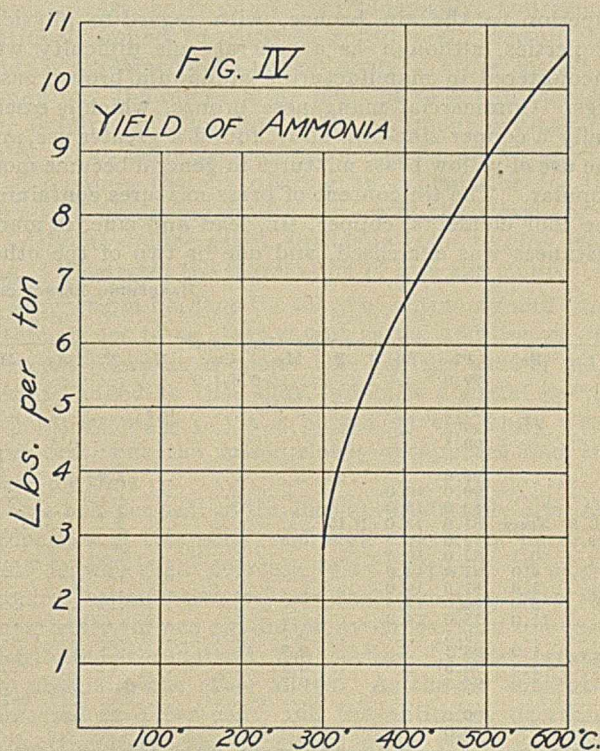
III—These raw oils are a mixture of coal tar and petroleum-like oil, with the former predominating.

IV—The yield of light oils decreases rapidly as the temperature increases, that of the paraffin oils less rapidly, while the yield of the medium oils remains fairly constant.

V—About 5.3 lbs. of paraffin wax per ton of coal may be obtained at 350° C.

VI—The gas given off up to 600° C. is small in volume and low in heat value, but relatively high in illuminants.

VII—Appreciable amounts of ammonia may be obtained from the tar water and also small amounts of cyanides.



VIII—The specific gravities of the tar oils and their respective fractions increase correspondingly with increase in the temperature of distillation.

IX—This coal, when subjected to destructive distillation, has a temperature of marked decomposition which corresponds to a sudden increase in the amounts of hydrogen and methane evolved and a corresponding increase in the quantity of oil distilled which is generally the maximum point, and, as in most low-grade

coal, this temperature lies between 350° and 450° C.

X—The residue at 350° has a calorific value of 12,790 B. t. u., which is an increase of 22.8 per cent over the coal as mined, and of 14.7 per cent over the dry coal.

HARDENING EFFECTS OF VARIOUS ELEMENTS UPON LEAD

By Carl O. Thieme

MICHIGAN SMELTING AND REFINING COMPANY, DETROIT, MICHIGAN

Received November 22, 1919

During the war, the high price and the threatened famine in tin, due to labor conditions, shortage of ship bottoms, and possibly to the submarine warfare, caused the manufacturers of this country to search and experiment with other metals and alloys that could be used in place of those containing tin. Of course, the greater portion of this work was done on tin-base bearing metals, but the conservation of tin was aided by the substitution of other metal for tin, in brass and bronze alloys.

It is quite well known that the composition of gun metal, 88-10-0-2, was modified to 88-8-0-4, with results that were just as satisfactory. In other cases, tin was entirely omitted from bronzes, and aluminum substituted, producing alloys that were decidedly superior to the tin bronze, with regard to physical properties, although as a general rule difficulty was encountered in manufacturing aluminum bronze castings. Commercial manganese bronze, which is essentially a copper-zinc-iron alloy, found a greater use, and the use of yellow brass mixtures in general became more popular. The tin content of brass mixtures containing the four elements, copper, tin, lead and zinc, in many instances was decreased, and one or two of the other

show the hardening effect of the various elements upon lead.

The hardening effect of the elements will be taken up in the order in which they appear in the accompanying table.

CALCIUM—Lead may be hardened by the addition of about one per cent of calcium.¹ It then has a Brinell hardness of 15, and quite a metallic ring. The alloy does not long retain a luster, and in a short time darkens a great deal. It shows numerous small blisters appearing on the surface of the metal, which will chip off, giving the metal a sort of pocked appearance. We believe that this alloy, upon remelting several times, loses its hardness, due, undoubtedly, to the oxidation and consequent skimming off of the calcium, together with the lead oxide. An admixture of barium and calcium is sometimes added as the hardening agent.

SODIUM—Sodium added to lead hardens it considerably, giving a Brinell hardness of approximately 5 to 6. This alloy also loses its hardness after several remelts. It is believed by some that the hardening constituent in an alloy of this kind is a substance having the formula Na₂Pb₅.² The eutectic of lead-sodium alloys is 2.5 per cent sodium, and 97.5 per cent lead. As the sodium is increased, the Brinell hardness gradually increases until at 0.8 per cent (the maximum amount of sodium dissolved by lead) the Brinell hardness is 8. From 0.8 to 2.5 per cent the hardness does not increase, but falls off with sodium values higher than 2.5 per cent.

ARSENIC—Arsenic hardens lead and increases the fusibility of the alloy. The quantity of arsenic added is generally under one per cent, and probably not more than 0.5 per cent. On account of the increased fusibility, this metal is used to advantage in making lead

HARDENING EFFECT OF VARIOUS ELEMENTS ON LEAD

COMPOSITION (PER CENT)											HARDNESS			Fig. No.	REMARKS
Cu	Sn	Pb	Sb	P	Mg	Ca	Na	Ni	As	Hg	Magnified Hammer Scleroscope	Brinell Test			
...	...	99.0	0.8-1.0	14.0	15.0	...	An admixture of barium and calcium, sometimes added as hardening agent	
...	...	99.5	0.5	5-6*	...		
...	...	99.2	0.8	7.5-8*	...		
...	...	99.5	0.5	7.5-8.5*	...	Known as shot lead, also added to antimonial lead for bullets	
...	...	98.0	2.0	Hardness not determined	
...	...	90.0	10.0	10-11	14.0	1	Popularly known as antimonial lead	
...	...	82.0	18.0	14.0	16.0	2	Metallic packings—filling and hammer metal	
0.8	Trace	81.0	18.0	0.15	24.0	3		
...	...	82.0	17.5	..	0.5	17.5	..	Experimental alloy	
...	0.5	82.0	17.5	15.5	..		
...	4.0	82.0	14.0	10-12	18.0	4		
...	5.0	81.0	14.0	11-13	19.0	..		
...	8.0	80.0	12.0	14-15	20-21	5		
...	11.0	75.0	14.0	15-16	22.0	6		
...	..	99.3	0.7	12.0	15.0	7	Experimental alloy	
...	1.0	98.3	0.7	12.0	..	Experimental alloy	
...	2.5	96.8	0.7	10.5	..	Experimental alloy	
...	5.0	94.3	0.7	12.0	..	Experimental alloy	
...	0-7.0	4-9*	..		
...	..	100.0	4-4.5*	..	Cast lead	
...	..	100.0	5-5.5*	..	Cold hammered lead	

* Hardness numbers followed by asterisk, 100 kilogram load and 10 mm. ball; all others, 500 kilogram load and 10 mm. ball.

metals increased, producing alloys that were cheaper and gave just as satisfactory results for the purposes for which they were used.

As stated above, the conservation of tin was due largely to the use of lead-base and leaded white metals. It is not my intent to enter into a discussion of the values of lead-base bearing metals at this time, for there are many articles, pro and con, published on this subject, but to present the following data which will

shot, for the time taken in solidifying gives the metal better opportunity to form a spherical drop.

NICKEL—Nickel, when added to lead, has a decided hardening effect, but on account of the great difference in their melting points, the use of this alloy is rather out of the question. The component metals must be

¹ Chem. & Met. Eng., Sept. 28 (1918), p. 253.

² C. H. Desch, "Metallography," 2nd Ed., 1913; C. H. Mathewson, Z. anorg. Chem., 50 (1906), 171.

melted separately, and mixed in the molten condition. An alloy of 2 per cent nickel and 98 per cent lead has a good appearance and is quite resonant.



FIG. 1—MATRIX OF LEAD-ANTIMONY EUTECTIC. EXCESS LEAD SHOWING AS CRYSTALLITES

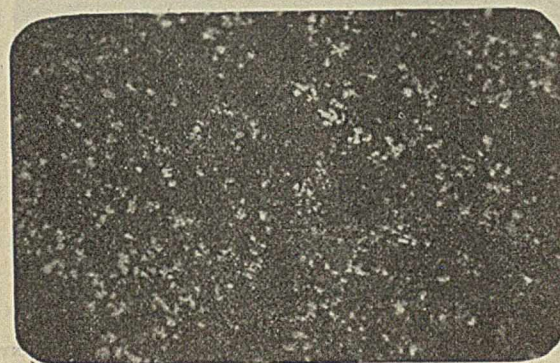


FIG. 2—MATRIX OF LEAD-ANTIMONY EUTECTIC. EXCESS ANTIMONY CRYSTALS DISTRIBUTED UNIFORMLY THROUGHOUT THE SECTION

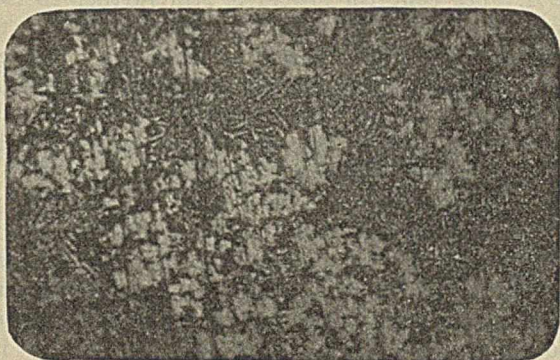


FIG. 3—MATRIX OF LEAD-ANTIMONY EUTECTIC. FREE ANTIMONY CRYSTALS IN EXCESS IN WHICH IS DISSOLVED COMPOUND Cu_3Sb

ANTIMONY—Antimony has long been used to harden lead, and has a wide use commercially as filling metal and hammer metal, and in some instances as an anti-friction metal. When the alloy contains more than 18 or 20 per cent antimony, it becomes too brittle for practical use. The hardness is proportional to the antimony content, and the increase is due to the formation of a eutectic having the composition 87 per cent lead and 13 per cent antimony. The hardness is increased approximately one number (Brinell) for each

¹ All photomicrographs shown here are taken at 100 magnifications and are etched with a dilute solution of nitric acid in ethyl alcohol.

per cent of antimony added up to the eutectic point, when the proportionate increase of hardness in the alloy does not hold true. The hardness of a mixture of 10 per cent antimony and 90 per cent lead is approximately 14 Brinell, which corresponds to the calcium-lead alloy (Fig. 1).

An alloy of antimony and lead, of the composition 87 per cent lead and 13 per cent antimony, shows neither lead nor antimony in excess, and is entirely composed of the eutectic. When the antimony content is increased to 18 per cent the alloy shows a hardness of 16 Brinell. The grain of this metal, however, varies greatly and is controlled only with considerable difficulty by pouring into a water-cooled mold. In order to prevent segregation, it is necessary to pour this metal as near the melting point as possible. The mixture is quite resonant, and, unlike the calcium and the sodium hardened lead alloys, does not lose its hardness upon remelting. It is brittle and fractures easily. Fig. 2 shows structure of an alloy of the composition 82-18.

The addition of one per cent phosphor copper to the alloy just mentioned has a decided hardening effect, showing a Brinell hardness of 24.0. However, this alloy is more brittle than the antimony-lead mixture, and loses its luster more quickly. It is extremely hard to cast, as it pours very sluggishly, and segregation is bound to occur in spite of all precautions. This is explained by the fact that, in order to cast a sound bar, the temperature must be raised to increase the fluidity, and flotation of the antimony crystals results. The grain is finer than the 82-18 antimony-lead mixture, thereby offering an explanation for the increased hardness, and a fracture of the metal shows a finely crystalline grain, similar in color to gray iron. Fig. 3 illustrates the segregation of free antimony.

Magnesium hardens the alloy of antimony and lead, although not to as great extent as the addition of one per cent phosphor copper. One-half per cent of magnesium added to this alloy produces a metal having a hardness of 17.5. It is almost silvery white when first cast, but the luster slowly disappears and the alloy darkens.

One-half per cent of tin does not noticeably alter the properties of this alloy, although a series of hardness tests shows a slight decrease to 15.5 Brinell. A microscopical examination reveals the formation of a few imperfectly formed tin-antimony cubes, very unevenly distributed throughout the section. These crystals are absent in the other alloys. As the tin content is increased to 4 per cent, and the antimony decreased proportionately, the resultant alloy gives a Brinell hardness of 17.5 to 18.0 (Fig. 4). The addition of 5 per cent tin produces an alloy of 19.0 Brinell hardness. On increasing the tin content still further to 8 per cent, the structure of the metal is changed, and as the composition nears the hypothetical ternary tin-lead-antimony eutectic (none formed) we find a fine grained alloy. This is an ideal alloy of the three metals, due to the fact that it retains its luster better than the majority of lead-base metals of this series, and readily takes a high polish. Its hardness is 20.0 (Fig. 5).

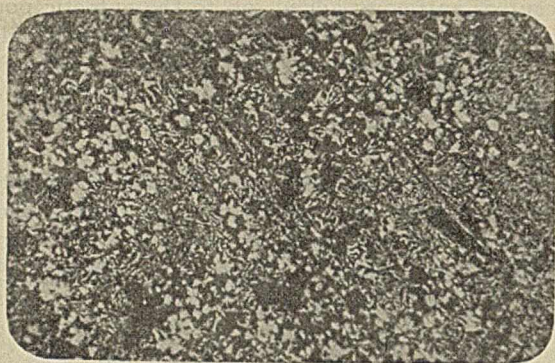


FIG. 4—MATRIX OF LEAD-ANTIMONY EUTECTIC. IMPERFECTLY DEVELOPED TIN-ANTIMONY GAMMA CONSTITUENT AND A SMALL PERCENTAGE OF EXCESS LEAD

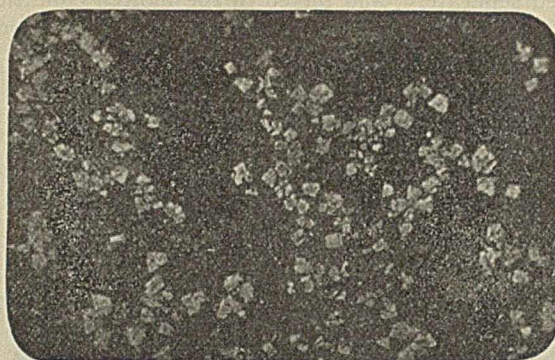


FIG. 6—LEAD-ANTIMONY EUTECTIC MATRIX IN WHICH IS IMBEDDED TIN-ANTIMONY GAMMA CONSTITUENT

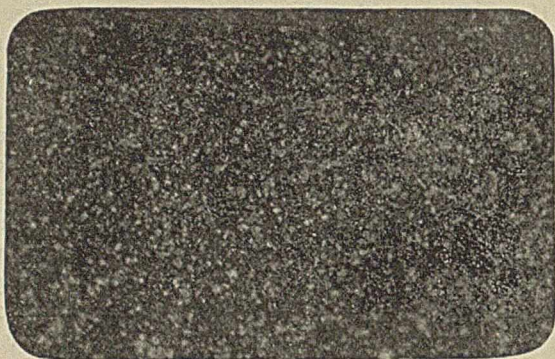


FIG. 5—LEAD-ANTIMONY EUTECTIC WITH A FLOTATION OF TIN-ANTIMONY GAMMA CONSTITUENT AND TIN CRYSTALS



FIG. 7—MATRIX OF LEAD IN WHICH IS IMBEDDED Mg_2Pb CONSTITUENT AS PRIMARY CRYSTALLITES

An 11 per cent tin alloy of the same series gives a hardness of 21.5 to 22.0 Brinell (Fig. 6). It will be noticed in the tin-lead-antimony alloys that, as the tin is increased 1 per cent, the hardness is increased half a number in each instance. Most bearing metals of the lead-base series should not contain more than 10 per cent tin unless the antimony is increased proportionately. However, the alloy then becomes brittle, as in the case of the binary alloy of lead and antimony. Above 10 per cent tin, the addition of tin to lead-antimony alloys does not exert as great a hardening effect as is true when smaller percentages are added.

MAGNESIUM—Magnesium¹ hardens lead when added in small amounts, usually one per cent or under. An alloy containing one-half per cent magnesium has a Brinell hardness of 15.0. Difficulty is encountered, however, in alloying these metals. The alloys were made by introducing small pieces of magnesium into the molten lead until the former gradually melted and dissolved. After the magnesium is alloyed with the lead it does not oxidize rapidly, and, therefore, retains its luster and its hardness upon remelting. The writer believes the hardening constituent in this alloy to be a substance having the composition Mg_2Pb , which Kurnakoff and Stepanoff found to solidify at $551^\circ C$.² The photomicrograph (Fig. 7), although considerably scratched, illustrates the formation of this material.

¹ *Metall u. Erz*, n. s., 7 (1919), 79; abstracted in *J. Inst. Metals*, 21 (1919), 435.

² Grube, *Z. anorg. Chem.*, 44 (1905), 117; 46 (1905), 177.

The addition of one per cent tin to this mixture softens the alloy, giving a Brinell hardness of 12.0, while 2.5 per cent tin lowers it still further to 10.5. Between 2.5 and 5 per cent tin there is a limit to the falling hardness, and a mixture containing 5 per cent tin again raises the hardness to 12.0. An alloy of this composition would be rather expensive, as compared with the alloys previously mentioned, which give a greater hardness at a lower cost.

MERCURY—A recent investigation of lead alloys, with mercury, sodium, and tin, by J. Goebel,¹ shows that mercury, when added to lead, increases its hardness from 4 to 9 Brinell. Any amount of mercury up to a maximum of 7 per cent increases the hardness proportionately.

The hardness of cast lead and cold hammered lead is given at the foot of the table, to allow of comparison in degree of hardness.

THE SOLUBILITY OF MONO- AND DIAMMONIUM PHOSPHATE

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In connection with certain experimental work recently carried on in this laboratory, more definite information than could be found in the literature was required concerning the solubility of the two commercial

¹ *Z. Ver. deut. Ing.*, May 10, 1919; abstracted in *THIS JOURNAL*, 11 (1919), 1065.

phosphates of ammonia. It therefore became necessary to prepare solubility curves for these two salts which would be of sufficient precision for the work in hand. The temperature ranges in which we were interested were, for the mono-salt, from about zero to the boiling point of the saturated solution, and from 0° to about 70° C. for the di-salt. These solubility determinations were made with care, and with as great precision as was possible with the equipment in a technical laboratory. While we make no claims for the highest precision, we feel that the data are of sufficient value to warrant their general distribution.

Dammer, Abegg, and the *Chemiker Kalendar* state that the di-salt, $(\text{NH}_4)_2\text{HPO}_4$, is soluble in four parts of cold water, and that the mono-salt, $(\text{NH}_4)\text{H}_2\text{PO}_4$, is less soluble than the di-salt. Comey adds that the mono-salt is soluble in 5 parts of cold water. Olsen¹ gives the same figure for the di-salt, but for the mono-salt he gives the solubility as 171 parts per hundred of water at 0° C. and 260 parts per hundred of water at 31° C.; that is, the mono-salt is much more soluble than the di-salt. The new edition of Seidell's "Solubilities" (1919) gives a value for the solubility of the di-salt, attributed to Greenish and Smith, of 131 g. $(\text{NH}_4)_2\text{HPO}_4$ in 100 g. water at 15° C.

THE SOLUBILITY OF MONOAMMONIUM PHOSPHATE, $(\text{NH}_4)\text{H}_2\text{PO}_4$

PREPARATION—The salt was prepared by recrystallization of commercial mono-salt, "Ammono-Phos," and analyzed as follows:

Per cent NH_3 = 14.80; Per cent P_2O_5 = 61.57; Ratio = 0.240.
Calculated for $(\text{NH}_4)\text{H}_2\text{PO}_4$:

Per cent NH_3 = 14.80; Per cent P_2O_5 = 61.72; Ratio = 0.240.

(By "Ratio" we mean the per cent ammonia divided by the per cent P_2O_5 . For the mono-salt the "ratio" is 0.240, and for the di-salt 0.480.)

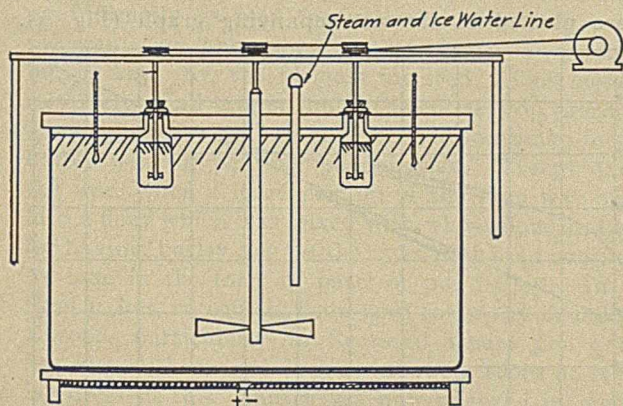


FIG. 1

METHOD—The apparatus used is shown diagrammatically in the accompanying sketch (Fig. 1). The temperature of the thermostatic bath was controlled by the introduction of steam or ice water, with an electrically heated hot plate as an auxiliary for the higher temperatures. For the very highest temperatures calcium chloride brine was used in the bath. This thermostatic bath was of large size and the water was kept in vigorous motion by a motor-driven propeller. Although hand

controlled, no difficulty was experienced in maintaining the temperature constant to within 0.5° C., which was as precise regulation as was desired.

To each of two glass bottles of about 250 cc. capacity there were added water and an amount of the salt largely in excess of the requirement for a saturated solution at the temperature in question. One of these bottles was cooled to about 10° below the desired temperature, while the other was heated to about the same distance above it. The bottles were now placed in the bath, the agitators introduced, and vigorous agitation at constant temperature maintained for one hour. At the end of an hour the agitators were stopped and samples of the clear liquid withdrawn in the special weighing pipette shown in the sketch

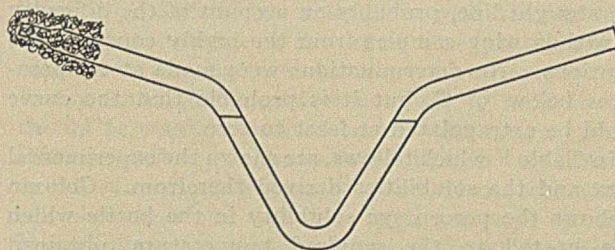


FIG. 2

(Fig. 2), care being taken that the cotton filter on the end of the pipette was tight, preventing the introduction of crystals with the liquid. The agitators were again inserted and the stirring at constant temperature continued while the samples were being analyzed for ammonia by distillation. At the time of withdrawal of the sample a final temperature reading was taken in the bottles themselves by means of a carefully standardized thermometer. This temperature was considered the temperature of the determination.

If the two portions agreed sufficiently well, it was assumed that equilibrium had been reached; if they did not agree, two more samples were withdrawn. This was continued until satisfactory agreement was secured. This agreement was rarely obtained in less than 2 hrs.; almost invariably the bottle with the rising gradient reached equilibrium more rapidly than the bottle with the falling gradient. From the ammonia determinations, the amount of ammonium phosphate present in the sample, and hence the solubility, was obtained. This method of calculation is applicable, since the mono-salt is entirely stable at solution temperatures. An analysis of the material remaining in the bottles at the close of the series showed that the "ratio" had not changed at all during the experiments.

The method of procedure outlined was employed for all the determinations up to and including that made at 90° C. In the case of the measurement at 102° both bottles were brought to equilibrium from below. The last point, which is that of the boiling saturated solution, 110.5° C., was obtained by withdrawing the samples from a saturated solution during vigorous ebullition.

All thermometers used were checked by comparison with a standard thermometer. Where necessary, suitable stem exposure corrections were applied. We be-

¹ Van Nostrand's "Chemical Annual," 1918.

lieve that the temperature measurements reported are certainly correct within $\pm 0.5^\circ \text{C}$.

EXPERIMENTAL DATA—When the solubility data are calculated as percentage solubility, that is, as grams of salt dissolved in 100 g. of the saturated solution, and these results are plotted (Fig. 3), we find that the points up to and including 90°C . lie in a straight line, none of the determinations lying off the straight line by an amount greater than the experimental error. From this straight line we derive the equation for the solubility of the mono-salt over the temperature range of 5° to 90°C . as follows:

Solubility of Monoammonium Phosphate (g. in 100 g. of saturated solution between 5° - 90°) = $18.0 + 0.455t$

The two high temperature points fall a little below the straight line, probably on account of the difficulty in withdrawing samples from the highly concentrated solutions. No determinations were made at temperatures below 5°C . but it is probable that the curve could be extrapolated at least to zero.

In Table I, which follows, are shown the experimental data and the solubilities derived therefrom. Column 1 shows the percentage solubility in the bottle which was heated to the required temperature, obtained by dividing the observed percentage of ammonia by 14.80 (the per cent of ammonia in monoammonium phosphate). Column 2 gives the percentage solubility in the bottle which was cooled to the desired temperature, and Column 3 gives the mean of Columns 1 and 2. Column 4 gives the percentage solubility as calculated by use of the above formula, and Column 5 gives the values of Column 4 recalculated as grams salt dissolved in 100 g. water.

TABLE I—SOLUBILITY OF MONOAMMONIUM PHOSPHATE, $(\text{NH}_4)_2\text{HPO}_4$

Temp. ° C.	Grams Monoammonium Phosphate				100 G. Water (5)
	—100 G. of Saturated Solution— (1)	(2)	(3)	(4)	
4.8	20.5	20.3	20.4	20.2	25.3
18.3	25.9	26.6	26.3	26.3	35.7
30.0	31.4	31.7	31.6	31.7	46.4
40.0	36.1	36.3	36.2	36.2	56.7
50.0	40.8	40.8	40.8	40.8	69.1
69.0	49.7	49.8	49.8	49.4	97.7
90.0	59.3	58.8	59.1	58.9	143.0
102.0	63.2	..	63.2	64.4	181.0
110.5	67.3	..	67.3	68.3	215.0

THE SOLUBILITY OF DIAMMONIUM PHOSPHATE, $(\text{NH}_4)_2\text{HPO}_4$

PREPARATION—The diammonium phosphate used in these experiments was prepared by ammoniating at a temperature above 80°C . a nearly saturated solution of mono-salt, cooling the mixture and filtering out the di-salt crystals. The air-dried crystals analyzed as follows:

Per cent NH_3 = 25.8; Per cent P_2O_5 = 53.9; Ratio = 0.479
 Calculated for $(\text{NH}_4)_2\text{HPO}_4$:
 Per cent NH_3 = 25.8; Per cent P_2O_5 = 53.8; Ratio = 0.480

METHOD—While the mono-salt is very stable at solution temperatures, the di-salt is much hydrolyzed, and precautions have to be taken, particularly at higher temperatures, to minimize ammonia losses. In the apparatus used by us it was not possible to entirely eliminate this loss, but its amount was determined in each experiment and its effect on the results as a whole largely eliminated by proper weighting of the experimental results.

The experimental procedure was practically the same as that used for the mono-salt, except that two samples were withdrawn from each bottle instead of one, as formerly. One sample from each bottle was analyzed for ammonia, and, if these showed satisfactory agreement, the other pair were analyzed for P_2O_5 by precipitation of the double magnesium salt. From the two analyses it was possible to arrive at the amount of decomposition which had taken place.

EXPERIMENTAL DATA—Table II gives the experimental data. The column headings are all self-explanatory, except possibly the last, which is intended to show the per cent of the dissolved phosphates of ammonia which was present as di-salt, as calculated from the "mean ratio." This column is of value in furnishing information as to the weighting of the data in deriving the solubility formula.

TABLE II—THE SOLUBILITY OF DIAMMONIUM PHOSPHATE $(\text{NH}_4)_2\text{HPO}_4$

Temp. ° C.	—Per cent NH_3 —			—Per cent P_2O_5 —			Ratio (Mean)	Per cent Di-salt from Ratio
	Up	Down	Mean	Up	Down	Mean		
0	7.69	7.62	7.66	16.28	16.28	16.28	0.470	96
10	9.89	9.85	9.87	20.70	20.61	20.66	0.478	99
20	10.54	10.46	10.50	21.94	21.89	21.92	0.480	100
30	10.87	10.89	10.88	23.00	23.00	23.00	0.473	97
40	11.45	11.46	11.46	24.47	24.55	24.51	0.467	95
50	12.06	12.20	12.13	25.41	25.34	25.38	0.478	99
60	12.58	12.55	12.57	26.80	26.90	26.85	0.468	95
70	13.07	13.22	13.15	27.58	28.01	27.80	0.473	97

Due to the decomposition which has taken place in some of the samples, there is sometimes a little doubt as to just how the percentage solubility should be calculated. In Table III is shown in Column 1 the solubility as calculated by dividing the mean ammonia percentage by 25.78, the theoretical percentage of ammonia in diammonium phosphate. Column 2 contains the solubility calculated in a similar manner from the P_2O_5 content of the solution, and Column 3 is the mean of Columns 1 and 2. The values of Column 3 are plotted in the accompanying graph (Fig. 3).

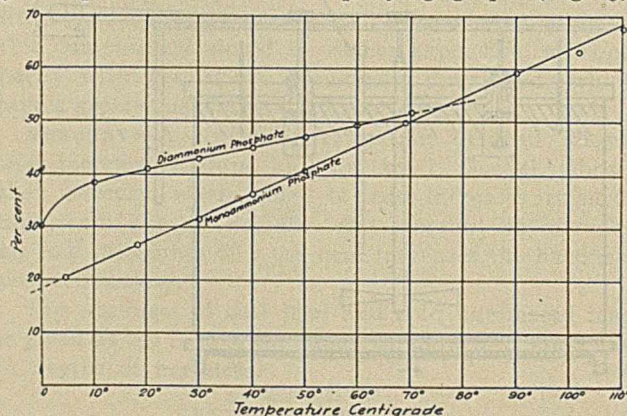


FIG. 3—THE SOLUBILITY OF MONO- AND DIAMMONIUM PHOSPHATES

As will be seen, the values between 10° and 70°C . lie in a straight line, and we derive therefrom the solubility formula:

Solubility Diammonium Phosphate (g. in 100 g. of the saturated solution between 10° - 70°C .) = $36.5 + 0.213t$

The values of Column 4 are derived by calculation from this formula. Column 5 contains the values of Column 4, calculated as grams diammonium phosphate dissolved in 100 g. water.

TABLE III—SOLUBILITY OF DIAMMONIUM PHOSPHATE

Temp. ° C.	Grams Diammonium Phosphate				100 G. Water
	—100 G. of Saturated Solution—				
	(1)	(2)	(3)	(4)	(5)
0	29.7	30.3	30.0
10	38.3	38.4	38.4	38.6	62.8
20	40.7	40.8	40.8	40.8	69.0
30	42.2	42.8	42.5	42.9	75.2
40	44.4	45.6	45.0	45.0	81.8
50	47.0	47.2	47.1	47.2	89.2
60	48.8	49.9	49.4	49.3	97.3
70	51.0	51.8	51.4	51.4	106.0

SUMMARY

Solubility determinations made on the two commercially important phosphates of ammonia permit the derivation of the following solubility equations, representing the grams of the salt dissolved in 100 g. of the saturated solution between the temperature limits given:

$$\text{Solubility Monoammonium Phosphate} \begin{matrix} 5^{\circ} \text{ C.} \\ 90^{\circ} \text{ C.} \end{matrix} = 18.0 + 0.455t.$$

$$\text{Solubility Diammonium Phosphate} \begin{matrix} 10^{\circ} \text{ C.} \\ 70^{\circ} \text{ C.} \end{matrix} = 36.5 + 0.213t.$$

PEARL BARLEY: ITS MANUFACTURE AND COMPOSITION

By J. A. LeClerc and C. D. Garby

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Received November 8, 1919

The barley crop of the United States approximates 200,000,000 bu. yearly, most of it being grown in North Dakota, California, Minnesota, South Dakota, Wisconsin, Kansas, and Iowa. The average annual yield per acre is about 25 bu. Heretofore fully one-third of the crop, or somewhat over 60,000,000 bu., has gone into malt for brewing and distilling, and a large amount is still malted for utilization in the manufacture of malt extract. About one-half of the crop is used directly as feed for animals, and from 2 to 3 per cent, or approximately 4,000,000 or 5,000,000 bu., is kept by the farmers for seed. This leaves a relatively small proportion of the entire crop (normally about 3,000,000 bu.) to go directly into human food as barley flour or pot and pearl barley. Except during the war, when a large portion of the crop was milled into a flour which was mixed with wheat flour and used for baking, barley has for the most part been consumed by man in the form of pearl or pot barley. In this form it has, of course, been used for many decades in Europe, particularly in Scotland where pot barley has met with special favor. About a score of manufacturers in this country are now engaged in making pearl barley, the total output of which before the war was 500,000 100-lb. sacks. The present paper contains the results of a study of the method of manufacture of pearl barley and of the chemical composition of the various products of the pearling operations carried out by the Plant Chemical Laboratory.

PEARL AND POT BARLEY

According to McGill,¹ the reduction in weight in producing pot barleys is 46 per cent. Such barley contains from 0.89 to 2.44 per cent of ash. The loss in making pearl barley is greater, as is evident from the

¹ Lab. Inland Rev. Dept., Ottawa, Canada, *Bulletin* 329.

fact that McGill finds that pearl barley contains from 0.58 to 1.27 per cent ash. Tibbles¹ states that Scotch barley is not decorticated as extensively as pearl barley. Church² states that from 100 lbs. of barley only 63 lbs. of pot barley, or about 32 lbs. of pearl barley, are obtained. Doubtless this pearl barley is the product resembling the fifth pearl barley of commerce. Gill³ states that when the integuments of the barley are removed and the product rounded and polished the result is pearl barley.

Sometimes in the process of manufacturing pearl barley, sulfur dioxide is used to whiten the final product, and talc or similar substances are added to brighten and give it a "pearl-like" appearance. The amount of insoluble ash in normal pearl barley, which has not been subjected to the treatment with talc or other mineral substances, is less than 0.10 per cent. According to Lverage and Hawley⁴ any sample of pearl barley containing a greater quantity of insoluble ash should be regarded as adulterated with mineral facing. Pluecker and Flebbs⁵ have shown that the use of sulfur or talc is not for the purpose of preventing the growth of microorganisms, as has been claimed by some manufacturers, but is simply a commercial practice.

MANUFACTURE

The various steps to which barley is subjected in the pearling process are indicated in Fig. 1. The barley as first received is passed into storage bins from which it travels to automatic scales. The weighed grain is sent to the separator where it is partially cleaned by screens. From the separator it goes to the reel,

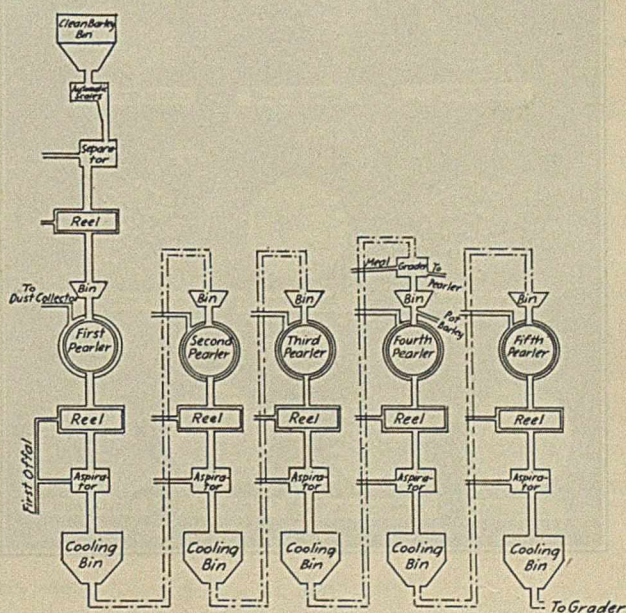


FIG. 1—DIAGRAM SHOWING BARLEY PEARLING PROCESS

which cleans it more thoroughly. The cleaning is completed by sieves and aspirators, after which the barley is ready for the pearling process. The grain

¹ "Foods, Their Origin and Manufacture," p. 471.² "Food," p. 74.³ "Bread Maker," p. 75.⁴ *J. Soc. Chem. Ind.*, 34 (1915), 203.⁵ *Z. Nahr. Genussm.*, 28 (1914), 28.

is carried from the cleaning devices, which differ in various mills, to the small bin over the first pearler.

The pearling machine used in the preparation of pearl barley generally consists of a large, vertical, cylindrical abrasive stone surrounded by a metal sieve, and resembles an ordinary grindstone in shape and position. The whole is enclosed in a dustproof metal jacket. The stone, usually of corundum, carborundum, or emery, may be as much as 4 ft. in diameter, and is made to revolve from 300 to 400 times per minute. The sieve surrounding this stone revolves about one-tenth as fast in the opposite direction. The barley, introduced automatically from a small bin or hopper, is subjected to a scouring action between the stone and the sieve. This abrasion continues for about 2 min., and the finer dust particles produced are removed from the enclosing jacket by means of dust collectors. The partly pearled barley and ofal are then ejected, and a new charge of clean barley introduced. The entire process is automatic and practically continuous.

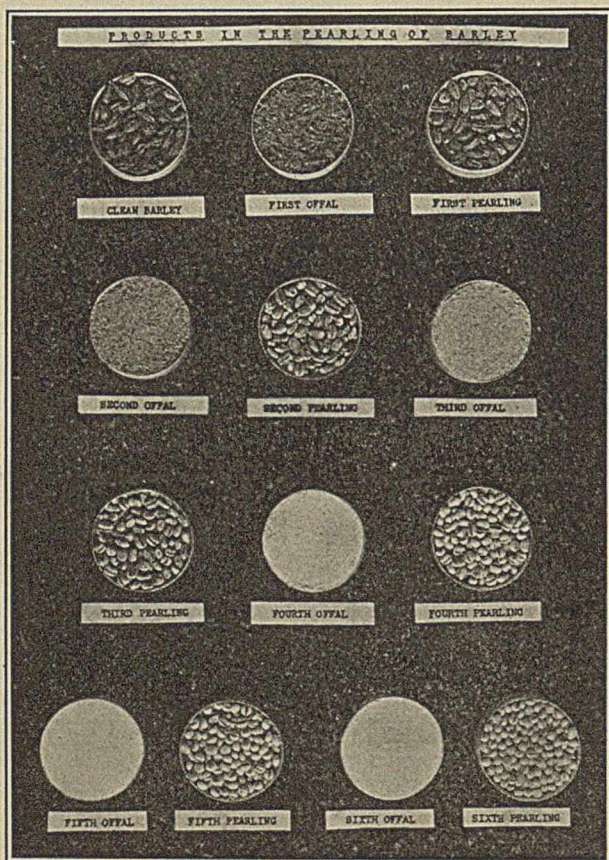


FIG. 2

From the first pearler the mixture of ofal and barley is sent to a reel which removes the ofal or husk. From the reel the barley enters an aspirator where the remaining fine particles are removed. After cooling, the barley is ready for the second pearling.

The second operation, which is identical in process with the first, removes the next remaining layer of the grain, and the product thus obtained is likewise sent to a reel for the separation of the ofal and the dust

particles. The pearling is generally repeated from 4 to 6 times in order to remove by attrition all the outer coats of the barley grain.

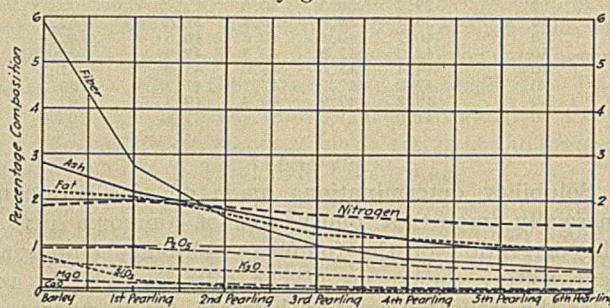


FIG. 3—COMPOSITION OF PEARLED BARLEY

After passing through the third process the partly pearled barley is often sent to a grader where it is assorted (Fig. 1). The large kernels are subjected to further attrition, thus producing a pearl barley of special type, the grain of which is rather large and well rounded. The smaller broken particles are collected and ordinarily used for feed. If the pearling process is stopped at the stage where only the outer cuticle is removed a "pot" barley (French, *Orge Mondé*) results. This is sometimes called Scotch barley, and is also known by various trade names. When the attrition is carried on still further a smaller type of pearl barley, which is generally separated into grades according to size, is produced. The different pot and pearl barleys and ofal are shown in Fig. 2.

EXPERIMENTAL WORK

SAMPLES—Two sets of samples were obtained from a large cereal factory, one in 1916 and the other in 1917. In each case samples of the original barley, as well as of the products resulting from each pearling process, were collected. That is, the samples represent the original barley, the pearled or partly pearled barley, and the ofal from each of the 6 pearling operations.

METHODS OF ANALYSIS—After grinding the samples so that they would pass through at least a 1-mm. sieve, they were analyzed for moisture, ash, protein, fat, and

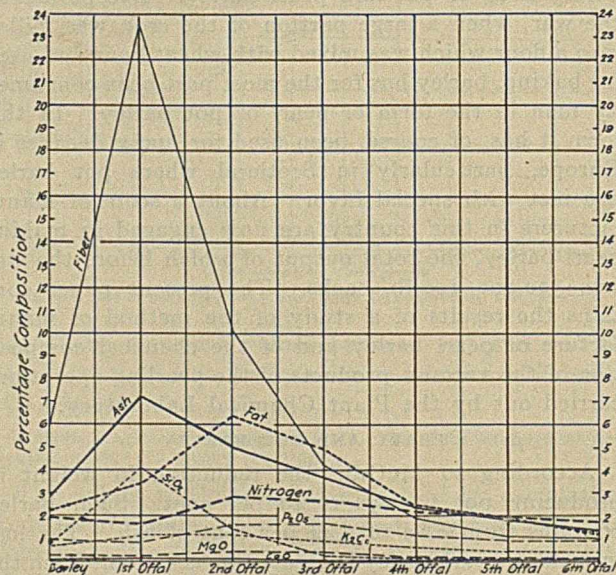


FIG. 4—COMPOSITION OF OFFAL

fiber, according to the methods of the Association of Official Agricultural Chemists. The constituents of the ash were determined as follows: P₂O₅ and K₂O according to the official methods of the Association of

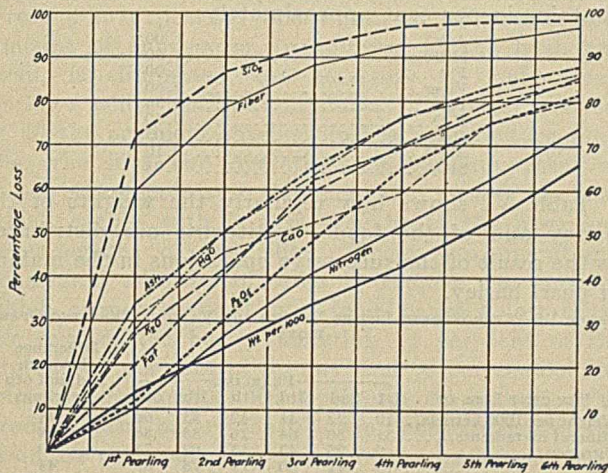


FIG. 5—PERCENTAGE LOSS OF CONSTITUENTS DURING PEARLING PROCESS

Official Agricultural Chemists; the silica also by the official method, except that it was not separated from carbon and sand, the amounts of which were so small as to be negligible. The calcium and magnesium were determined according to the McCrudden method,¹ with slight change in that it was not found necessary to boil the calcium solution during any part of the determination.

TABLE I—PERCENTAGE COMPOSITION ON DRY BASIS OF PEARL BARLEY AND OFFAL (5 Operations)

DESCRIPTION	P. C. No.	Wt. per 1000 Kernels G.	Constituents								
			Ash	Protein (N X 6.25)	Fat	Fiber	P ₂ O ₅	K ₂ O	CaO	MgO	SiO ₂
Original barley	18408	28.32	2.81	12.06	2.22	5.89	0.962	0.654	0.074	0.265	0.785
1st offal	18410	7.25	10.37	3.45	23.50	0.842	1.170	0.117	0.284	4.080	
1st pearling	18411	24.37	2.15	12.56	2.07	2.76	0.984	0.553	0.059	0.221	0.257
2nd offal	18413	5.05	18.00	6.43	10.17	1.632	1.180	0.119	0.438	1.470	
2nd pearling	18414	21.44	1.80	11.69	1.73	1.67	0.881	0.501	0.052	0.173	0.138
3rd offal	18416	3.73	17.12	4.98	4.28	1.691	0.977	0.092	0.448	0.259	
3rd pearling	18417	18.72	1.44	10.75	1.26	1.04	0.744	0.400	0.053	0.146	0.069
4th offal	18419	2.42	13.81	2.43	1.93	1.180	0.609	0.070	0.282	0.210	
4th pearling	18420	16.18	1.15	10.13	1.16	0.75	0.606	0.340	0.052	0.108	0.038
5th offal	18422	1.86	12.31	1.90	1.15	0.923	0.496	0.063	0.186	0.098	
5th pearling	18421	13.20	0.96	9.69	1.02	0.65	0.506	0.276	0.039	0.102	0.029

RESULTS OF ANALYSIS—As has been stated already, from 4 to 6 pearling operations are necessary to secure a well-rounded, white, pearl barley. Tables I and II

TABLE II—LOSSES DUE TO PEARLING (5 Operations)

DESCRIPTION	P. C. No.	Wt. per 1000 Kernels G.	Constituents (Per cent)								
			Ash	Protein	Fat	Fiber	P ₂ O ₅	K ₂ O	CaO	MgO	SiO ₂
1st pearling	18411	11.5	30.0	8.0	17.6	58.6	9.4	25.2	29.7	26.0	71.1
2nd pearling	18414	22.1	49.2	25.0	39.7	77.9	28.7	40.4	45.8	49.0	86.3
3rd pearling	18417	30.9	63.6	38.5	60.1	87.8	46.4	57.6	50.0	61.9	93.9
4th pearling	18420	38.1	74.5	48.3	67.8	92.1	60.9	67.9	56.7	78.5	97.0
5th pearling	18421	48.1	81.7	58.7	76.1	94.3	72.6	78.1	73.0	80.0	98.3
1st pearling	...	13.7	34.1	10.3	19.8	59.8	12.0	27.2	31.1	28.3	71.8
2nd pearling	...	24.3	50.6	26.8	40.9	78.6	30.7	42.0	45.0	50.5	86.7
3rd pearling	...	34.2	65.3	41.2	62.4	88.3	48.7	59.5	52.7	63.4	92.8
4th pearling	...	42.8	76.4	51.9	70.0	92.7	64.9	70.2	59.4	76.6	97.2
5th pearling	...	53.0	83.5	62.7	78.2	94.8	75.3	80.2	75.7	81.9	98.2

¹ A complete charge from one of the pearling machines, consisting of from 50 to 100 lbs. of mixed barley and offal, was secured from each pearling operation. The pearl barley was separated from the offal, and the percentage loss calculated.

(P. C. 18408 and 18422) show the results of the analysis of pearl barley and the products of pearling obtained

TABLE III—PERCENTAGE COMPOSITION ON DRY BASIS OF PEARL BARLEY AND OFFAL (6 Operations)

DESCRIPTION	P. C. No.	Wt. per 1000 Kernels G.	CONSTITUENTS						
			Ash	Protein (N X 6.25)	Fat	Fiber	P ₂ O ₅	K ₂ O	CaO
Original barley	17575	42.50	2.82	13.19	2.09	5.08	1.02	0.66	0.062
1st offal	17577	6.89	10.56	2.77	23.74	0.97	1.12	0.095	
1st pearling	17576	39.66	2.17	12.94	2.02	2.36	1.07	0.59	0.048
2nd offal	17579	5.13	19.56	6.48	9.25	1.92	1.15	0.097	
2nd pearling	17578	34.10	1.80	12.56	1.47	1.37	0.94	0.50	0.043
3rd offal	17581	3.93	16.38	4.92	4.81	1.81	1.03	0.076	
3rd pearling	17580	30.40	1.45	10.25	1.19	0.91	0.81	0.41	0.035
4th offal	17583	2.68	14.38	2.58	2.06	1.37	0.70	0.952	
4th pearling	17582	24.52	1.20	10.50	0.91	0.73	0.68	0.34	0.033
5th offal	17585	2.13	13.45	2.04	1.35	1.14	0.58	0.047	
5th pearling	17584	20.13	1.06	10.37	0.94	0.58	0.62	0.32	0.030
6th offal	17585-y	1.67	12.50	1.67	1.22	1.00	0.47	0.038	
6th pearling	17585-x	14.25	0.98	9.44	0.89	0.44	0.56	0.30	0.035

through 5 operations. Tables III and IV (P. C. 17575 and 17585) give similar results on products obtained from 6 pearling operations. These results are also graphically represented by the curves shown in Figs. 3, 4, and 5.

TABLE IV—LOSSES DUE TO PEARLING (6 Operations) Calculated from Weight per 1000 Kernels

DESCRIPTION	Wt. per 1000 Kernels G.	Constituents (Per cent)						
		Ash	Protein (N X 6.25)	Fat	Fiber	P ₂ O ₅	K ₂ O	CaO
1st pearling	6.7	28.0	8.5	9.6	56.7	2.2	17.0	24.2
2nd pearling	19.8	48.9	23.7	43.5	78.4	26.2	39.8	45.1
3rd pearling	28.4	63.1	44.6	59.3	87.2	43.4	55.3	58.0
4th pearling	42.3	75.5	54.1	74.9	91.7	61.7	70.3	69.3
5th pearling	52.7	82.2	62.7	78.7	94.6	71.4	77.2	77.4
6th pearling	66.5	88.4	74.4	85.7	97.1	81.8	85.1	80.6

DISCUSSION OF RESULTS—From the chemical and physical study of the products of pearling, it is obvious that the offal resulting from the first pearling process (P. C. 18410 and 17577) is composed almost entirely of the hulls of the barley (Fig. 2). This offal has relatively little food value for man, both because of its extremely high content of fibrous material and of silica (23.6 per cent on an average for fiber and 4.1 per cent for the silica), and because of its physical

TABLE V—PERCENTAGE OF MINERAL CONSTITUENTS IN ASH

DESCRIPTION	Total Ash	Constituents				
		P ₂ O ₅	K ₂ O	CaO	MgO	SiO ₂
Original barley	2.82	36.2	23.4	2.2
1st offal	2.81	34.2	23.3	2.6	9.4	27.9
1st pearling	6.89	14.1	16.2	1.4
2nd offal	7.25	11.6	16.1	1.6	3.9	56.3
1st pearling	2.17	49.3	27.2	2.2
2nd offal	2.15	45.8	25.7	2.8	10.3	11.8
2nd pearling	5.13	37.4	22.4	1.9
3rd offal	5.05	32.3	23.4	2.3	8.7	29.1
3rd pearling	1.80	52.2	27.8	2.4
4th offal	1.80	49.2	27.8	2.9	9.6	7.7
4th pearling	3.93	46.1	26.2	1.9
5th offal	3.73	45.3	26.2	2.5	12.0	6.9
5th pearling	1.45	56.0	28.4	2.4
6th offal	1.44	51.6	27.7	3.7	10.1	4.8
6th pearling	2.68	51.1	26.1	1.9
7th offal	2.42	48.8	25.2	2.9	11.6	8.7
7th pearling	1.20	56.6	28.3	2.7
8th offal	1.15	53.6	29.6	4.6	9.4	3.4
8th pearling	2.13	53.5	27.2	2.2
9th offal	1.86	49.6	26.7	3.3	10.0	5.2
9th pearling	1.06	58.5	30.2	2.8
10th offal	0.96	52.7	28.7	4.0	10.6	3.0
10th pearling	1.67	59.9	28.1	2.3
11th offal
11th pearling	0.98	57.1	30.6	3.6

characteristics. There is no doubt, however, but that there is some value as animal feed in the offal, even from the first pearling, because it contains an average of approximately 10.50 per cent of protein (N X 6.25) and an average of 3.11 per cent fat. The offal from the second-pearling (P. C. 18413 and 17579) contains over 18.75 per cent protein, 6.45 per cent fat, 9.70 per cent of fiber, and less than 1.5 per cent

¹ J. Biol. Chem., 7 (1909), 98; 10 (1911), 187.

SiO₂. The high content of P₂O₅ (over 32 per cent of the ash (Table V)), together with the high percentage of fat and protein, indicates that this offal is made up to a marked degree of the aleurone layer, or the so-called protein layer, a substance rich in protein, fat, and mineral ingredients. The second offal contains less than half as much fiber and about one-third as much SiO₂ as does the first offal. In addition, it contains twice as much fat and almost twice as much protein. A significant fact is the difference in ash constituents. The second-pearling offal contains less ash than does the first offal, but at the same time it contains twice as much P₂O₅, besides being much poorer in silica. All this shows that while at least two processes of pearling are essential for the production of an edible product, even this second pearling operation removes an appreciable amount of food constituents.

The third offal is also rich in protein, fat, ash, and P₂O₅, as well as in fiber. That this fiber is composed chiefly of the bran coating, however, and not of the hulls may be assumed from the relatively low silica content, and from the high P₂O₅ content of the ash of this product.

The edible product obtained from the third pearling operation, or the pot barley, is almost entirely free from the aleurone layer, as is evident from the low percentage of fat, fiber, and ash (Nos. 18417 and 17580 in Tables I and III). The fourth and fifth pot barleys are not only whiter, but are practically free from all bran material. They are still poorer in fat, fiber, and minerals than the pot barley from the third operation. The fifth pearl barley contains about one per cent each of ash and fat, and 0.6 per cent fiber. The ash contains a very high amount of P₂O₅ and a very low amount of SiO₂. The fourth offal contains about 2.5 per cent each of ash and fat, about 2.35 per cent nitrogen, and 2 per cent fiber, while the fifth offal still has about 2 per cent ash, nitrogen, and fat, and about 1.25 per cent fiber.

From Table V it is evident that the amount of SiO₂ in the ash in the first offal is over 56 per cent, and that it decreases rapidly, the offal after the second operation containing but a small amount of silica. The ash of the third offal is composed of only about 8 per cent SiO₂, while that of the fifth contains only 5 per cent. On the other hand, the percentage of P₂O₅ gradually increases from 11 per cent in the first offal to over 50 per cent in the fifth. The high P₂O₅ content and the low percentage of SiO₂ may well be considered a criterion of the quality of the product.

Tables I and III also show that the second-process pot barley (P. C. 18414 and 17578), which is practically free from hulls and constitutes the first product of the pearling process fit for human consumption, has, on the water-free basis, the following average percentage composition:

Ash.....	1.80
Nitrogen.....	1.94
Fat.....	1.60
Fiber.....	1.52
P ₂ O ₅	0.91
K ₂ O.....	0.50
CaO.....	0.048
MgO.....	0.17
SiO ₂	0.138

This is essentially the same as the composition of hull-less barley. With each process of pearling the percentage of nitrogen, fat, fiber, and the ash constituents gradually decreases. The fifth-pearling barley contains in per cent approximately:

Ash.....	1.00
Nitrogen.....	1.60
Fat.....	1.00
Fiber.....	0.60
P ₂ O ₅	0.56
K ₂ O.....	0.30
CaO.....	0.035
MgO.....	0.10
SiO ₂	0.03

Table VI shows how regularly the weights of the barley and of the food constituents are distributed as the result of the successive operations in the making of pearl barley.

TABLE VI—APPROXIMATE LOSSES DUE TO THE VARIOUS PEARLING OPERATIONS

PER CENT LOSS IN	PEARLING						Difference between 2nd and 6th Pearlings
	1st	2nd	3rd	4th	5th	6th	
Weight per 1000 kernels..	10	22	31	42	53	66	44
Mineral ingredients.....	31	50	64	76	83	88	38
Nitrogen.....	9	25	43	53	62	74	49
Fat.....	15	41	60	72	78	85	44
Fiber.....	57	78	88	91	94	97	19
P ₂ O ₅	7	28	45	62	73	83	55
K ₂ O.....	22	40	57	70	78	85	45
CaO.....	27	45	55	64	76	80	35
MgO.....	28	50	63	76	82	..	32
SiO ₂	72	86	93	97	98	..	12

Assuming that 5,000,000 bu. of barley are now being yearly converted into pearl barley, the figures given in Table VII represent the weight of barley material and of the food constituents removed as the results of the two essential pearling operations and of the four subsequent processes. These figures mean that if all these 5,000,000 bu. of barley were passed through the 6 operations of the pearl barley process, as against the 2 operations necessary to make pot barley, 52,000 tons of barley material, containing over 6,700 tons of protein, over 1,000 tons of fat, and 1,100 tons of mineral ingredients, would be removed.

TABLE VII—APPROXIMATE AMOUNT OF BARLEY AND OF BARLEY CONSTITUENTS REMOVED BY PEARLING (IN TONS)

LOSS IN	Weight in Original Barley	Weight in Pearlings						Diff. in Weight between 2nd and 6th Pearlings
		1st	2nd	3rd	4th	5th	6th	
Weight.....	120,000	12,000	26,400	37,200	50,400	63,600	79,200	52,800
Mineral ingredients	3,000	930	1,500	1,920	2,280	2,490	2,640	1,140
Protein.....	13,650	1,229	3,412	5,896	7,234	8,463	10,159	6,747
Fat.....	2,330	350	955	1,374	1,677	1,817	1,979	1,025
Fiber.....	5,920	3,374	4,620	5,215	5,385	5,563	5,740	1,124
P ₂ O ₅	1,068	75	299	481	662	780	886	587
K ₂ O.....	702	154	281	400	491	547	597	316
CaO.....	74	20	33	41	47	56	59	26
MgO.....	288	80	144	181	220	236	..	92
SiO ₂	847	593	707	765	798	806	..	100

The figures in Table V show, moreover, that the first 2 pearlings are sufficient to remove practically all of the hull- and silica-containing portions of the barley, or the portions of the barley which are recognized as being unfit for human food. This is evident from the fact that 78 per cent of the fiber and over 85 per cent of the silica are removed in the course of these first 2 pearling operations. It is interesting to observe that the offal from the sixth pearling has essentially the same composition as the pot barley resulting from the second pearling process (Table III, P. C. Nos. 17585-y and 17578). This indicates that the offal from the sixth pearling might properly be used as a barley flour.

SUMMARY

Pot barley results from the gradual elimination of the outer layers of the barley (chiefly the husk and bran) which is accomplished in from 2 to 3 pearlings. From 5 to 6 pearling operations, in which a certain amount of endosperm, in addition to the husk and bran, is eliminated, are necessary to make pearl barley a white pearl-like product. Chemical analyses of all the products obtained in the manufacture show that the first two operations of pearling, resulting in pot barley, consist in the removal of most of the husk which carries with it three-fourths of the fiber and seven-eighths of the silica. These two operations, considered essential in order to remove the portions which cannot be used as a food, cause a loss of 22 per cent of the barley material, 25 per cent of the protein, 41 per cent of the fat, and 50 per cent of the mineral constituents. In continuing the operations to produce pearl barley the following constituents of the grain are removed: 65 per cent of the barley material; 74 per cent of the protein; from 80 to 85 per cent of the fat, P_2O_5 , K_2O , CaO , and MgO ; and from 97 to 98 per cent of the fiber and SiO_2 . Assuming that 5,000,000 bu. of barley are being pearled yearly, 52,000 tons of barley material, consisting of over 1,000 tons each of fat and mineral ingredients and 6,700 tons of protein, are removed.

1000, and 10,000 spores per gram. At the end of 5.5 mos. incubation at room temperature the contents of each flask were analyzed for sucrose by direct polarization and modified Clerget, and for reducing sugars and moisture. It has already been shown¹ that the most satisfactory criterion of deterioration of sugar is the gain in per cent of reducing sugars. In order to summarize the results as briefly as possible, there is given in Table I the increase over check of the averages of closely agreeing triplicate determinations of reducing sugars. The abbreviation M. R. stands for moisture ratio, which value is derived as follows: $M. R. = \frac{\text{Moisture}}{100 - \text{Polarization}}$; *Asp. n.* is the abbreviation for *Aspergillus niger*, while *Asp. S. B.* and *Pen.* represent *Aspergillus Sydowi* Bainier, and *Penicillium expansum*.

TABLE I—SUMMARY SHOWING THE INFLUENCE OF AMOUNT OF MOLD INOCULUM ON THE DETERIORATION OF SUGARS WITH FILMS OF KNOWN CONCENTRATION. INCREASE IN PER CENT REDUCING SUGARS OVER CHECK

No. Spores per Gram	CONCENTRATION											
	A M. R. = 0.14			B M. R. = 0.16			C M. R. = 0.18			D M. R. = 0.24		
	<i>Asp. n.</i>	<i>Asp. S. B.</i>	<i>Pen.</i>	<i>Asp. n.</i>	<i>Asp. S. B.</i>	<i>Pen.</i>	<i>Asp. n.</i>	<i>Asp. S. B.</i>	<i>Pen.</i>	<i>Asp. n.</i>	<i>Asp. S. B.</i>	<i>Pen.</i>
100	0.09	0.01	..	0.09	0.07	..	0.10	0.10
1000	..	0.02	0.02	..	0.05	0.04	0.12	0.22	0.17	0.04	0.18	0.18
10,000	0.04	0.21	0.11	0.03	0.12	0.11	0.27	0.31	0.28	0.12	0.27	0.28

EFFECT OF VARYING THE AMOUNT OF INOCULUM AND CONCENTRATION ON THE DETERIORATION OF SUGAR BY MOLDS¹

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In a previous paper² it was shown that a decrease in concentration of films of known concentration in laboratory-made sugars was responsible for an increase in deterioration when heavily inoculated with mold spores. The industrial application of this conclusion is determined by two important variable factors, namely, the concentration of the films surrounding the sugar crystals, and the degree of infection. Therefore, a further investigation of the influence of these factors was considered necessary.

The method of procedure was identical with that outlined in the previous article, except that the incubation period was 5.5 mo. instead of one month. A series of sugars with films of known composition was made in the laboratory by coating large crystals of sterilized sugar with sterilized blackstrap molasses and 60° Brix sugar sirup in definite proportions and purging in the centrifugal, a method previously employed with success. Blackstrap molasses, $\frac{5}{6}$ blackstrap + $\frac{1}{6}$ sirup, $\frac{4}{6}$ blackstrap + $\frac{2}{6}$ sirup, and $\frac{3}{6}$ blackstrap + $\frac{3}{6}$ sirup when arranged in order of increasing moisture ratio are designated as Concentrations A, B, C, and D, respectively. These sugars were inoculated with *Aspergillus niger*, *Aspergillus Sydowi* Bainier and *Penicillium expansum*, at the rate of 100,

It will be seen from this table that in every instance but one an increase in the number of spores per gram caused an increase in per cent of reducing sugars over check. This held true not only at every concentration employed, varying in moisture ratio from 0.14 to 0.24, but likewise for every organism used at any single concentration. This fact is very significant and indicates conclusively that an increase in degree of inoculation of mold spores at any definite concentration is responsible for an increase in deterioration of sugar. This corroborates our previous work where solutions varying from 10 to 70 per cent were employed,² as well as the results obtained in the experiment just concluded,³ where an inoculation of 100,000 spores per gram at each of the above-mentioned concentrations was employed. A closer scrutiny of the results presented in Table I reveals the fact that the increase over check of reducing sugars with an inoculation of 100 spores per gram is insignificant at practically all concentrations. The same is true of an inoculation of 1000 spores in the two higher concentrations, namely, A and B. This is of practical importance in defining the limits at which deterioration occurs, since in plantation granulated sugars the moisture ratio may be said ordinarily to be below 0.18. It is generally considered that good Cuban raw sugar likewise should have its moisture ratio below 0.25 to 0.33. Thus, it might be inferred from the foregoing data that where the moisture ratio is below 0.18, mold infection of less than about 5,000 spores per gram would cause slight, if any, deterioration. As a rule we have rarely found sugars which had more than 250 mold spores per gram, al-

¹ Read before the Louisiana Section of the American Chemical Society, November 21, 1919.

² THIS JOURNAL, 12 (1920), 256.

¹ Louisiana Bulletin 166.

² J. Agr. Res., 18 (1920), 537.

³ THIS JOURNAL, Loc. cit.

though no quantitative survey has been carried out as exhaustively as might have been desirable.

When the two lower concentrations C and D are considered, we find that there is evidence of deteriorative activity with 100 spores per gram, while with more than 1000 spores the deterioration is quite appreciable. It would appear, therefore, that for safety (from the standpoint of mold infection) a sugar having a moisture ratio of 0.17 to 0.18 would have to contain less than 100 mold spores per gram. On the other hand, a mold infection of more than 10,000 spores per gram will cause a deterioration in sugars with moisture ratios varying from 0.14 to 0.24. It may be noted in this connection that in the previous investigation an inoculation of 100,000 spores per gram was responsible in one month for a deterioration in sugars with moisture ratios varying from 0.08 to 0.20.

An interesting fact which corroborates all our previous work with sugars, as well as solutions, is that at any definite concentration and with an equal number of spores per gram *Aspergillus* Sydowi Bainier is more effective than *Penicillium expansum*, which in turn is more effective than *Aspergillus niger* in deteriorative activity.

TABLE II—SUMMARY SHOWING THE INFLUENCE OF CONCENTRATION OF FILM ON DETERIORATION OF SUGARS BY MOLDS

Concentration	Moisture Ratio	—Per cent Gain in Reducing Sugars over Check—								
		100 Spores per G.			1000 Spores per G.			10,000 Spores per G.		
		Asp. n.	Asp. S. B.	Pen.	Asp. n.	Asp. S. B.	Pen.	Asp. n.	Asp. S. B.	Pen.
A.....	0.14	0.02	0.02	0.04	0.21	0.11
B.....	0.16	..	0.09	0.01	..	0.05	0.04	0.03	0.12	0.11
C.....	0.18	..	0.09	0.07	0.12	0.22	0.17	0.27	0.31	0.28
D.....	0.24	..	0.10	0.10	0.04	0.18	0.18	0.12	0.27	0.28

In Table II is presented a summary of results so arranged as to show the influence of concentration of film on the deterioration of sugars by molds. It must be stated at the outset, however, that where the increasing increments of moisture are so slight as in the present instance, and especially when dealing with the activity of microorganisms over such a long incubation period, it is hardly to be expected that the differences will be very sharply defined. However, it will be observed that as a rule an increase in moisture ratio (which actually signifies a decrease in concentration) is responsible for an increase in deterioration with any single inoculation. It is not necessary to repeat here what was stated in the discussion of Table I concerning the limiting effects of concentration with any definite inoculum. Suffice it to say that this work fits in very closely with our preceding investigations and proves quite conclusively that with a high mold infection, deterioration takes place in sugars with moisture ratios below 0.14, or, according to the previous experiment, at 0.08. This substantiates the claim previously made that "the factor of safety for sugars well infected with fungi would appear to be lower than is generally supposed," and defines more clearly what such limits must be. In other words, knowing the number of molds present in any sugar, it may be predicted (from the standpoint of mold infection alone) what deterioration may be

TABLE III—COMPARISON IN CONTENT OF REDUCING SUGARS OF INOCULATED SUGARS (WITH FILMS OF CONCENTRATION D) AFTER 1 AND 5.5 MOS. INCUBATION, RESPECTIVELY

Incubation	Check	Asp. n.	Gain over Check	Asp. S. B.	Gain over Check	Pen.	Gain over Check
After 1 mo.....	0.23	0.44	0.21	1.10	0.87	0.77	0.54
After 5.5 mo.....	0.23	0.53	0.30	1.46	1.23	0.87	0.64
Gain in reducing sugars, per cent...	0.00	0.09	..	0.36	..	0.10	..

expected in a storage period of about 5 mo. with a sugar of known moisture ratio. It is to be assumed that at the present time sugars are not stored for such a long period of time, but the differences obtained upon a long incubation period are not so much greater as to invalidate the above generalizations, as will be seen from Table III, which gives a comparison in the reducing sugar content of inoculated sugars with films of Concentration D (moisture ratio = 0.24) after 1 and 5.5 mo., respectively. It will be readily seen that the differences in incubation as represented by the gain in per cent of reducing sugars are indeed slight when compared with the initial gain over check in one month.

Next to the elimination of deterioration, the most important commercial consideration is prediction of the keeping quality of a sugar. Table IV is a tentative plan based on the results obtained in all our investigations which will give some conception of the deterioration to be expected from a definite number of molds in sugars of known moisture ratio. It must be clearly understood that this plan is advanced with considerable diffidence, and that its value rests on further verification. Furthermore, it is of importance to note that in the above table mold infection only has been considered. We have data which are concerned with deterioration due to bacterial infection and unquestionably the bacterial flora would seriously influence the deterioration of sugars as shown in the important researches of previous investigators. However, since individual molds, such as *Aspergillus* Sydowi Bainier, *Penicillium expansum*, and *Aspergillus niger*, are vastly more efficient in their deteriorative activity than any bacteria that have come to our attention, and since the first-named mold is to be found in practically all sugars, it may be that the above table will prove of some value to those who are ready to take cognizance of the molds which are undoubtedly causing large

TABLE IV—DETERIORATION TO BE EXPECTED FROM A DEFINITE NUMBER OF MOLDS IN SUGARS OF KNOWN MOISTURE RATIO

No. of mold spores per g.	Moisture Ratio = $\frac{\text{Moisture}}{100 - \text{Polarization}}$						
	0.08	0.14	0.16	0.18	0.20	0.24	Over 0.24
0-100.....	—	—	±	+	+	+	+
100-1000.....	—	±	±	+	+	+	+
1000-10,000.....	±	+	+	+	+	+	+
10,000-100,000.....	+	+	+	+	+	+	+
+ Deterioration							
— No deterioration							
± Slight, if any deterioration							

economic losses in the sugar industry. In Table IV it will be seen that the facts previously discussed have been so arranged that one may tell at a glance what deterioration, if any, might be expected. It was not deemed necessary to carry out the work in moisture ratios beyond 0.24, because it is generally conceded that sugars having a moisture ratio above 0.30 are

susceptible to deterioration. Browne,¹ Owen,² and others have advanced much valuable evidence on this point.

This paper may be said to round out one phase of the problem of sugar deterioration, namely, that concerned with the importance of mold infection, which we have carried through to completion, from a survey of mold species in sugar and their deteriorative activities in sugars and solutions to a study of the effect of varying the amount of inoculum and concentration on deterioration.

The writer wishes to thank Messrs. D. F. Stanfill, Jr., and R. S. Hays for their help with the chemical analyses and the Station staff for their assistance, and is indebted to Mr. W. L. Owen for his kindness in reading the manuscript.

SUMMARY

1—An increase in number of mold spores inoculated into sugars (with films of varying concentration) is responsible for an increase in deterioration.

2—A decrease in concentration of the films surrounding the sugar crystals is responsible for an increase in deterioration.

3—A table is presented showing the deterioration which may be expected from a definite number of molds in sugars of known moisture ratio.

4—At moisture ratios of less than 0.18 there is little, if any, deterioration with a mold infection of less than 5,000 spores per gram. More than this number of spores induces deterioration. At moisture ratios above 0.18, deterioration occurs with upwards of 100 spores per gram.

5—At any definite concentration and with an equal number of spores per gram *Aspergillus* Sydowii Bainier is more effective than *Penicillium expansum* or *Aspergillus niger* in its deteriorative activity.

ACIDITY AND ACIDIMETRY OF SOILS.³ II—INVESTIGATION OF ACID SOILS BY MEANS OF THE HYDROGEN ELECTRODE

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INTRODUCTION

Although the literature upon the subject of soil acidity is voluminous, the use of the hydrogen electrode in soil investigations has been rather limited.

Gillespie⁴ made use of the hydrogen electrode for determining the hydrogen ion concentration of a mixture of soil with pure water; but, for reasons which will develop in the investigations given herewith, the presence of a conducting medium was found to be desirable. Sharp and Hoagland⁵ studied the hydrogen

concentrations of suspensions of soils in pure water under various conditions, effects of natural salts and bases upon hydrogen ion concentrations of soil suspensions, and made titrations with various bases.

The purpose of the present investigation is to study: (1) the speed of reactions between neutral salt solutions and soils; (2) the speed of reactions in the presence of a base; (3) the change in hydrogen ion concentration with change of amount of base and with time, and the change in conductivity of soil solutions.

APPARATUS

Preliminary experiments were conducted with an apparatus similar to that described by Hildebrand,¹ and some experiments described elsewhere with soil solutions were carried out, but for use with solutions in contact with the soil it was found to be rather unsatisfactory. For all work reported, unless otherwise stated, a high-grade potentiometer (Leeds and Northrup, No. 28952) was used with a gas cell especially designed for the work.

GAS CELL—Preliminary experiments showed that for uniform results it was necessary to have a gas cell which could be agitated continuously, as apparently the agitation produced by the entering gas was not sufficient. After a number of trials the gas cell shown in Fig. 1 was designed for this work. It is cylindrical in shape, 3.3 cm. in diameter and 16 cm. in length,

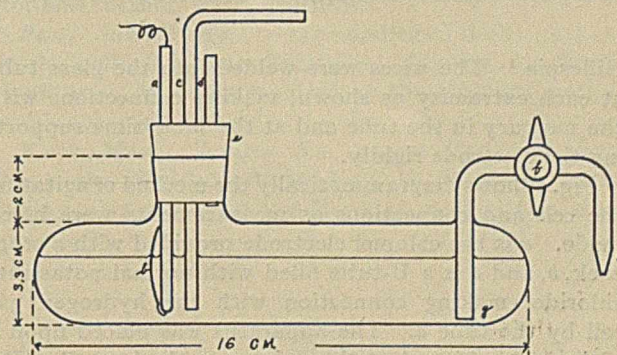


FIG. 1

the ends being rounded off. At one end an opening is provided of a size to carry a No. 4 rubber stopper, through which pass the electrical connection *a* to the platinum plate *b* to serve as the hydrogen electrode, the tube *c* for the ingress of hydrogen gas, and tube *d* for the outlet. Tubes *c* and *d* have capillary tubes sealed into the ends to regulate the flow of hydrogen. To make connections with the calomel half cell a glass tube, *g*, is provided at the further end of the gas cell provided with a stopcock, *f*, and a constricted tip, *h*. This tube reaches to within a few mm. of the bottom of the gas cell and as it did not readily clog with soil was found to be very satisfactory. The gas cell was designed to be of 100 cc. capacity and to be filled half full of liquid, leaving room for 50 cc. of gas. The reason for this arrangement will develop later.

The hydrogen electrode *b* is a rectangular piece of sheet platinum, 1.2 x 2.4 cm., with pieces of platinum wire welded to each end, and is similar to that used by

¹ THIS JOURNAL, 10 (1918), 178.

² Louisiana Bulletin 162.

³ This is a thesis submitted to the faculty of the University of Illinois in partial fulfillment of the requirements for the degree of Doctor of Philosophy. Acknowledgment is made of many helpful suggestions and criticisms received from Prof. C. G. Hopkins and H. A. Noyes.

⁴ J. Wash. Acad. Sci., 6 (1916), 7.

⁵ J. Agr. Res., 7 (1916), 124.

¹ J. Am. Chem. Soc., 35 (1913), 847.

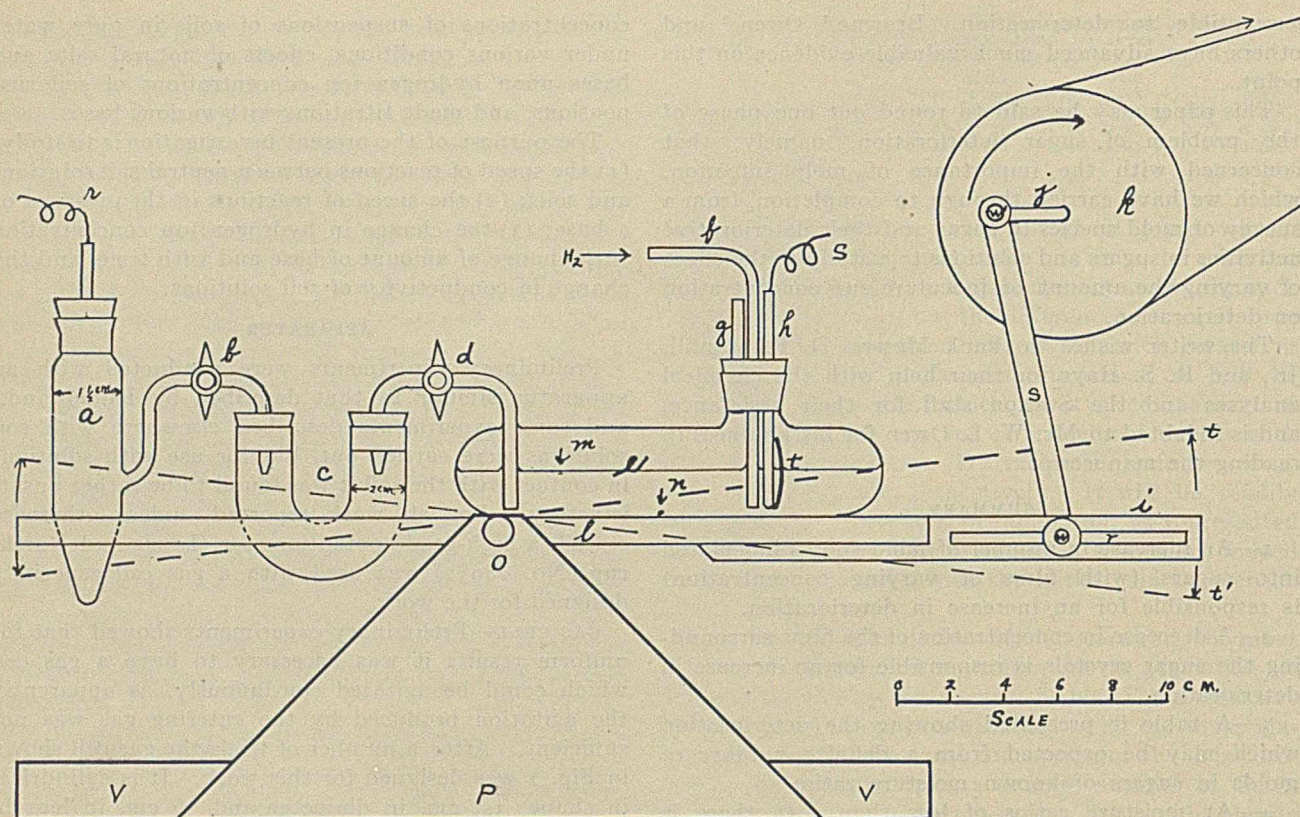


FIG. 2.

Gillespie.¹ The wires were welded into the glass tube at each extremity as shown, making connections with the mercury in the tube and at the same time supporting the electrode rigidly.

Fig. 2 shows diagrammatically the method of agitating the cell and connections as measurements were being made. *a* is the calomel electrode provided with a stopcock, *b*, and *c* is a U-tube filled with normal potassium chloride, making connection with the hydrogen gas cell by the tube *d*. The apparatus was placed upon a tilting table hinged at the point *o* to the base *P*. The table is rocked by means of the adjustable arm *s* and crank *j* attached by suitable gears and pulleys to a constant source of power. In building up the shaking apparatus free use was made of the parts of one of the popular metal construction shapes to teach children to make their own toys.

It was found by experiment that by giving the table 72 complete oscillations per minute through a total angle of 6° to 7° the liquid in the gas cell was thoroughly agitated; that the rubber stopper was not wetted, but that the soil was kept thoroughly mixed with the solution while the coarser particles of sand in the soil would tend to collect in a nodal point *n* in the bottom of the cell near the center. At this speed the hydrogen bubbles rose to about the point *m* before they broke, causing the cell to be quickly freed of air. Except for very light peat soils there was no tendency for the soil to ascend into the tube *d*.

The arrangement of the different parts of the apparatus as used in making measurements is shown diagrammatically in Fig. 3. The apparatus is designed to carry

two gas cells, marked E. M. F., so that duplicate measurements may be made without changing cells.

The use of the U-tube for making connections between the gas cells and calomel electrode was found to be a most satisfactory arrangement. The resistance of the chain is slightly increased, but diffusion is markedly reduced. During several weeks of continuous use, the calomel electrode changed less than 0.0005 volt. The gas cell is claimed to have the following advantages:

- (1) large capacity, (2) absence of dead air space, (3) ease of cleaning, (4) all-glass contact with liquid, (5) freedom from clogging, (6) ease of manipulation, (7) adaptability to thorough agitation, (8) minimum possibility of diffusion.

CALOMEL ELECTRODE—Calomel was prepared by treating pure mercury with dilute nitric acid, precipitating with hydrochloric acid, washing twenty times with potassium chloride, and finally shaking with normal potassium chloride and pure mercury, as recommended by Ellis¹ for obtaining an electrode of constant potential. The electrodes were prepared with this gray mixture of mercury, calomel, and normal potassium chloride by first putting into the bottom of the carefully cleaned electrode cell a small amount of pure mercury to cover the platinum connection. No difficulty was experienced in getting electrodes to check within 0.0005 volt. The stopcock upon the tube leading from the calomel electrode was well greased and was opened only when readings were being taken. Connection was made to the U-tube *c* (Fig. 2) through a rubber stopper well driven home, making this side of the system practically gastight.

¹ *Loc. cit.*

¹ *J. Am. Chem. Soc.*, 38 (1916), 737.

HYDROGEN ELECTRODE—The platinum electrode was prepared by plating as a cathode in a one per cent solution of platinum chloride, containing a small amount (about 0.05 per cent) of lead acetate to cause the platinum black to adhere. Attempts were made to use a solution of pure platinic chloride as recommended by Ellis, but this proved unsatisfactory as the platinum black invariably washed off within a few minutes after being placed in the cell.

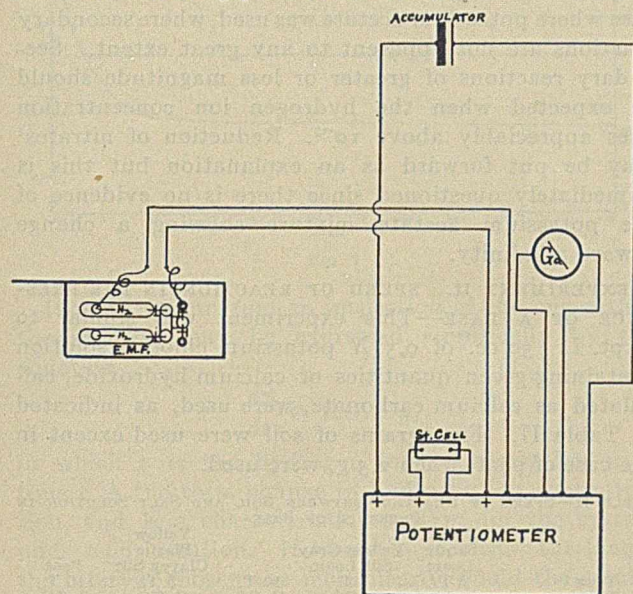


FIG. 3

In plating a platinum anode was used. The strength of the current was varied from time to time but no variation in the potentials of the electrode or in the time required to become saturated was noted from this cause. If the evolution of hydrogen was too lively some of the platinum black was loosened. The plating was continued from 1 to 2 hrs., or until a good heavy deposit of platinum black was formed. The electrodes were placed in distilled water and given a final wash just before using.

HYDROGEN GENERATOR—Hydrogen was prepared electrolytically as needed, using potassium hydroxide and nickel electrodes, as shown in Fig. 4. Two salt-mouth bottles, *G* and *G'*, were used. *E* and *E'* are the nickel electrodes, *M* and *M'* are glass partitions running almost to the bottom of the cells to reduce diffusion as much as possible. *P* and *P'* are safety valves made of test-tubes. The outlets *O* and *O'* were closed while hydrogen was being taken off. By using two cells in parallel and electrolyzing with a current of 10 amperes, about 150 cc. of hydrogen per minute were obtained. The usual general precautions were taken in washing and purifying the gas.

PROCEDURE

To fill the tube *g* of the gas cell (Fig. 1) the cell was partly filled with liquid, a stopper inserted in the opening *e*, and pressure exerted, so that, when the stopcock *f* was opened, the liquid would fill the tube *g* and flow out at *h*. Care was taken to thoroughly wet the stopcock *f* by loosening and turning. All measurements were made with this stopcock closed.

After the gas cell was filled with soil and solution and placed as shown in Fig. 2 the table was oscillated and hydrogen run in at *f* for 4 or 5 min.; the tube *g* was closed and rocking continued for 4 min.; hydrogen was again run in for 4 min. to drive out the last traces of air; *g* was again closed, the stopcock *b* opened, and readings were taken immediately.

The procedure varied somewhat with the number of readings to be taken, type of experiment, etc. If readings were to cover a period of half an hour or more, after the first large volume of hydrogen was run in, it was found advisable to allow a small amount to bubble through to overcome any diffusion which might take place through the rubber connections.

DESCRIPTION OF SOILS INVESTIGATED

Yellow-Gray Silt Loam, Sample No. 1—An acid subsoil collected from the southern part of the state of Illinois. This soil gave a lime requirement by the Hopkins method¹ of 4.2 tons and by the Veitch method² 5.6 tons calculated as calcium carbonate.³ No carbonates were present as determined by the Marr⁴ method.

Black Peaty Loam, Sample No. 1-281⁵—Collected by C. G. Hopkins near Bolton, N. C. Limestone requirement by the Hopkins method was 3.445 tons.

Black Clay Loam, Sample No. 1-284—Collected by C. G. Hopkins near Bolton and Byrdsville. Some leaf mold was present. Limestone requirement by the Hopkins method was 4.982 tons.

Peat, Sample No. 1-241—Labeled deep peat or muck, collected near Titusville, Florida, by C. G. Hopkins. Limestone required by the Hopkins method was 3.267 tons.

Black Muck, Sample No. 1-242—Collected from Wauchula, Florida, collected by C. G. Hopkins. Limestone requirement by the Hopkins method was 4.056 tons.

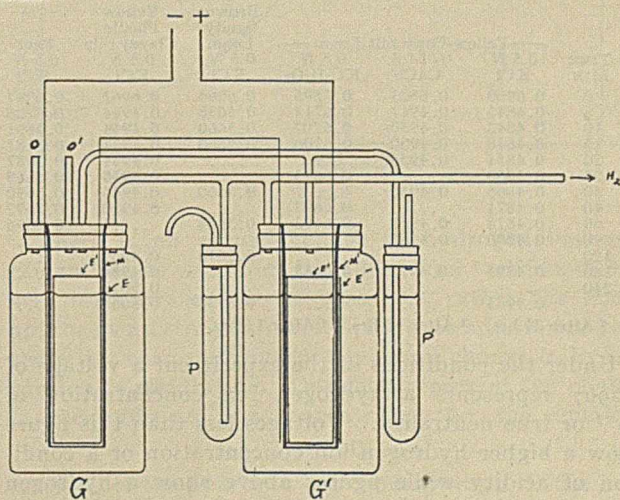


FIG. 4

Yellow Silt Clay, Sample No. 2660—Collected from Clay County, Illinois. A silty clay, stiff and plastic.

¹ U. S. Dept. of Agr., Bureau of Chem., *Bulletin* 107 Revised (1908), 20.

² *J. Am. Chem. Soc.*, 24 (1902), 1120.

³ All figures are based upon an acre of soil 6 $\frac{2}{3}$ in. in depth, calculated to weigh 2,000,000 lbs., except peats which are calculated to weigh 1,000,000 lbs.

⁴ *J. Agr. Sci.*, [II], 3, 155.

⁵ Numbers refer to the University of Illinois Soil Survey Numbers.

Subsoil sample. Limestone required by the Hopkins method was 5.08 tons.

Gray Clayey Silt, Sample No. 2968—Collected from Jackson County, Illinois. A compact, impervious subsoil containing iron blotches and concretions. Limestone required by the Hopkins method was 7.92 tons.

Gray Plastic Clay, Sample No. 3957—Collected from Winebago County, Illinois. A subsoil sample containing clayey sand. Limestone required by the Hopkins method was 2.6 tons.

Yellow Plastic Clayey Silt, Sample No. 3556—Collected from Clay County, Illinois. A subsoil sample. Limestone required by the Hopkins method was 4.67 tons.

Yellow Silt Loam, Sample No. 4068—Collected from La Salle County, Illinois. This was subsurface sample having a lime requirement by the Hopkins method of 2.89 tons.

Brown Sandy Loam, Sample No. 6316—This was subsurface sample having a limestone requirement by the Hopkins method of 1.65 tons.

EXPERIMENTAL

EXPERIMENT I. SPEED OF REACTION BETWEEN AN ACID SOIL AND A NEUTRAL SALT—This experiment was planned to observe the speed of change in hydrogen ion concentration of a neutral salt solution when shaken together with an acid soil.

The gas cell was filled with 50 cc. of 0.5 *N* salt solution and after the potential had become constant, which was usually within 10 min., 5 g. of soil (2.5 g. in the case of peat soil) were quickly introduced and readings were taken at stated intervals.

TABLE I—SPEED OF REACTION BETWEEN ACID SOIL AND A NEUTRAL SALT SOLUTION

(Readings are given in volts. Temperature 25° C.)

TIME MIN.	Yellow-Gray Silt Loam			Brown Sandy Loam	Yellow Plastic Clayey Silt	Peat
	0.5 <i>N</i> KCl	1/4 Mol. CaCl ₂	0.5 <i>N</i> K ₂ C ₂ H ₃ O ₂	0.5 <i>N</i> KCl	0.5 <i>N</i> KCl	0.5 <i>N</i> KCl
0	0.6969	0.6823	0.7295	0.6966	0.6967	0.6967
5	0.4843	0.4947	0.6715	0.5658	0.4944	0.5706
10	0.4843	0.4950	0.6707	0.5660	0.4944	0.5691
15	0.4848	0.4950	0.6706	0.5660	0.4944	0.5687
20	0.4854	0.4950	0.4944	0.5687
25	0.4860	0.6693	0.4946	0.5688
30	0.4865	0.4950	0.5660	0.4946	0.5690
40	0.4871	0.6693	0.4950	0.5692
60	0.4878	0.4957	0.6693	0.5664	0.5696
90	0.4890	0.4963
120	0.5670	0.4969
180	0.4895	0.4972	0.6688	0.4984
240	0.5010
	0.4963 ¹	0.5733 ²	0.5063 ³

¹ After 24 hrs. ² After 48 hrs. ³ After 12 hrs.

Under the conditions of the experiment a voltage of 0.6967 represents a hydrogen ion concentration of 10^{-7} or true neutrality. Voltages less than this figure show a higher hydrogen ion concentration or a condition of acidity while figures above show a hydrogen ion concentration of less than 10^{-7} or an alkaline condition.

In Table I it will be noted that the lowest readings were obtained within 5 min. in all cases but two, the yellow-gray silt loam with potassium acetate, and peat with potassium chloride. The potassium acetate solution showed a marked alkalinity before the soil was added, therefore we should expect it to act more as a base. It will be discussed in connection with the

next experiment. The peat did not wet readily which may account for its behavior. After the low readings were obtained there was a gradual increase in the voltages, corresponding to a decrease in the hydrogen ion concentration.

It is evident that the main reaction between the salt solution and the soil reaches an equilibrium very quickly. Secondary reactions are indicated by an increase in voltage after a lapse of time, except in the case where potassium acetate was used, where secondary reactions are not apparent to any great extent. Secondary reactions of greater or less magnitude should be expected when the hydrogen ion concentration rises appreciably above 10^{-7} . Reduction of nitrates¹ may be put forward as an explanation but this is immediately questioned since there is no evidence of the potassium acetate mixture showing a change toward alkalinity.

EXPERIMENT II. SPEED OF REACTION IN THE PRESENCE OF A BASE—This experiment was similar to Expt. I. 50 cc. of 0.5 *N* potassium chloride solution containing given quantities of calcium hydroxide, calculated as calcium carbonate, were used, as indicated in Table II. Five grams of soil were used except in the case of peat, when 2.5 g. were used.

TABLE II—SPEED OF REACTION BETWEEN SOIL AND SALT SOLUTION IN PRESENCE OF BASE

TIME MIN.	Brown Sandy Loam	Yellow-Gray Silt Loam	10 T.	Yellow Plastic Clayey Silt	Peat
	2 T. CaCO ₃	4 T. CaCO ₃	10 T. CaCO ₃	5 T. CaCO ₃	10 T. CaCO ₃
0	0.9543	0.9715	0.9961	0.9803	0.9961
5	0.7783	0.7483	0.8505	0.6562	0.8345
10	0.7565	0.6923	0.8410	0.6302	0.7642
15	0.7440	0.6467	0.8363	0.6228	0.7370
20	0.7313	0.6161	0.8356	0.6199	0.7173
25	0.7269	0.6037	0.8337	0.6170	0.7053
30	0.7232	0.5978	0.8326	0.6148	0.6951
40	0.7165	0.5935	0.8298	0.6111	0.6809
60	0.7113	0.5897	0.8273	0.6072	0.6672
90	0.5855	0.8262
120	0.7066	0.5987	0.6499
180	0.5820	0.8206	0.5951	0.6432
	0.7022 ¹	0.8186 ²	0.5933 ³	0.6394 ³

¹ After 48 hrs. ² After 5 hrs. ³ After 4 hrs.

The change in the hydrogen ion concentration was very rapid at first, but continually rose as long as the experiments were conducted. The plotted curves are all similar in character and of the general type shown in Fig. 5, which is for the yellow-gray silt loam with four tons of lime.

Although equilibrium is not reached for a considerable period under the conditions of the experiment, the greater portion of the reaction takes place within a few minutes. After the neutral point is reached the hydrogen ion concentration continues to increase at the same relative rate without change. This phenomenon explains the condition noted by MacIntire² who distinguishes between immediate and continued lime requirement and Truog³ who makes the division into active and latent soil acidity. Both investigators added an excess of lime to the soil while in certain of the above experiments the lime requirements were not satisfied. Apparently the reaction is in each case an equilibrium reaction. Such being the case, any division

¹ An attempt was made to use potassium nitrate but it was noted that even in a neutral solution reduction of the nitrate to ammonia took place to an extent which could be noted with organic indicators.

² Tenn. Expt. Sta., *Bulletin* 107 (1914), 193.

³ *This Journal*, 8 (1916), 341.

as indicated by them is an arbitrary one, and the results will not necessarily bear any relation to the total lime requirements.

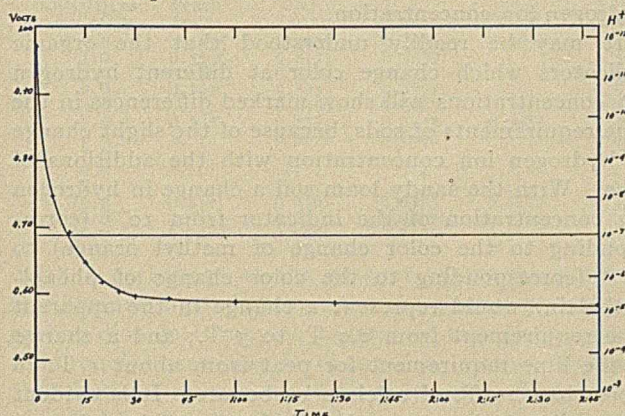


FIG. 5

It would seem from an inspection of the curve that the relation may be expressed empirically by the equation

$$\frac{dx}{dt} = K(A-x),$$

in which x is the hydrogen ion concentration with time t , A the total change in hydrogen ion concentration, and K a characteristic constant for the system under consideration. It would appear that the longer the interval chosen the more nearly would the results approach the maximum lime absorption, provided side reactions are not considered. It is quite probable that under field conditions the reactions will be far slower than the above experiment would indicate. The conditions in the field are often such that reactions cannot take place rapidly, while the converse is true in the laboratory. In this experiment a salt solution was used with calcium hydroxide, a condition, certainly, where reaction may take place with utmost speed.

This experiment would seem to throw some light upon the action of soil with potassium acetate in Expt. I. Potassium acetate is basic in character and even at the end of 180 hrs. the mixture with an acid soil contained only a slight excess of hydrogen ions.

EXPERIMENT III. STUDY OF CHANGE IN HYDROGEN ION CONCENTRATION OF NEUTRAL SALT SOLUTION CONTAINING VARYING AMOUNTS OF BASE—50 cc. of a 0.5 N neutral salt solution containing the desired amount of base were placed in the gas cell and readings were taken at the end of 20 min. The gas cell was rocked continuously as in the former experiments.

In the light of Expt. II, it can be readily understood that duplicates are somewhat difficult to obtain unless the manipulation is the same, and the time element eliminated. The speed of the reaction should depend somewhat upon the base used and the neutral salt with which it is combined. Considering these facts it is rather remarkable that the results obtained as shown in Table III should be of the same order. The experiments were all conducted upon yellow-gray silt loam.

TABLE III—STUDY OF CHANGE IN HYDROGEN ION CONCENTRATION OF A NEUTRAL SALT SOLUTION TO WHICH VARIOUS AMOUNTS OF BASE HAVE BEEN ADDED WHEN PLACED IN CONTACT WITH SOIL.

(Results given in volts. Soil is yellow-gray silt loam. 5 g. were taken in each case)

Base Equivalent to T. CaCO ₃	0.5 N KCl + Ca(OH) ₂	0.5 N NaCl + Ca(OH) ₂	0.5 N CaCl ₂ + Ca(OH) ₂	0.5 N K ₂ SO ₄ + Ca(OH) ₂	0.5 N KCl + KOH
0.0	0.4843	0.4995	0.4966	0.5245	0.4843
1.0	0.5099	0.5118	0.5105	0.5345	0.5119
2.0	0.5247	0.5180	0.5259	0.5527	0.5269
3.0	0.5428	0.5575	0.5475	0.5679	0.5505
4.0	0.6019	0.6125	0.6215	0.6169	0.6136
4.2	0.6187
4.4	0.6342
4.6	0.6405
4.8	0.6651
5.0	0.6868	0.6890	0.6894	0.6700	0.6888
5.2	0.6943
5.4	0.7059
5.6	0.7211
5.8	0.7232
6.0	0.7387	0.7414	0.7070	0.7330	0.7391
7.0	0.7726	0.7743
8.0	0.7942
10.0	0.8529	0.8335	0.7990	0.8443	0.8819

The salt solutions in contact with the soil (Series 0.0 T. CaCO₃) in Table III show considerable difference in potential, but all show about the same neutral point, *i. e.*, 5.0 T. CaCO₃. From this point the variation in the readings becomes rather wide in both directions.

The general form of the curves is shown by the type curve, Fig. 6. Two points regarding this curve may be discussed. It will be noted that the curve in the slope inclines more toward the vertical as the neutral point is reached and after this point is passed the slope inclines away from the vertical, approaching the slope at the lower end of the curve.

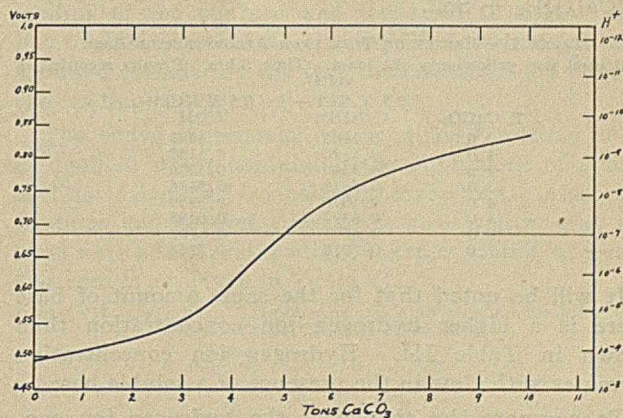


FIG. 6

There is no abrupt change in hydrogen ion concentration, as was noted by Hildebrand,¹ when a strong acid is neutralized by a base, but rather the curve approaches a straight line, *i. e.*, for each addition of base there is a corresponding nearly equal change in hydrogen ion concentration. One may represent the change in the hydrogen ion concentration by the equation

$$\log C = KB + K_1$$

where C is the hydrogen ion concentration, B the base added, and K and K_1 characteristic constants.

The systems investigated above have all the characteristics of mixtures with a high reserve acidity,² *i. e.*, the hydrogen ion concentration suffers very little change with comparatively large additions of base. This would be expected if the acid is comparatively insoluble.

¹ *Loc. cit.*

² Washburn. *J. Am. Chem. Soc.*, 30 (1908), 37.

Referring again to Table III it will be noted that after the neutral point is reached the hydrogen ion concentration for the system $\text{CaCl}_2 + \text{Ca}(\text{OH})_2 + \text{Soil}$ shows a higher hydrogen concentration than the system $\text{KCl} + \text{KOH} + \text{Soil}$ for the same equivalents of base. This may be accounted for by the precipitating action of calcium hydroxide. The difference in conductivity adds weight to this view.

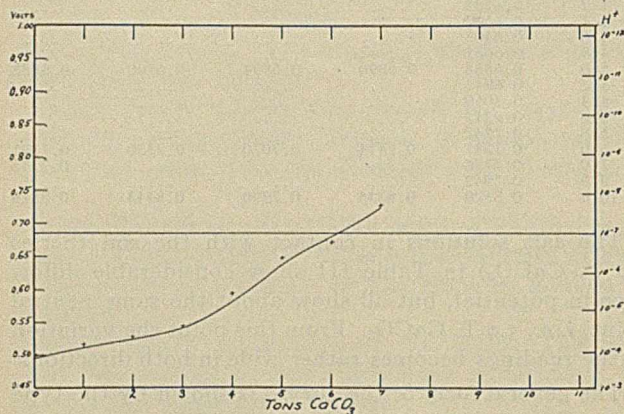


FIG. 7

When the base is allowed to act for a longer period of time there is in general a depression of the whole curve toward the acid side as is shown by Table IV and Fig. 7 which is the graph of the system $0.5\text{ N KCl} + \text{Ca}(\text{OH})_2 + \text{Soil}$.

TABLE IV—EFFECT OF TIME UPON ABSORPTION OF BASE
(Soil used was yellow-gray silt loam. Time, 3 hrs. Results recorded in volts)

T. CaCO_3	$0.5\text{ N KCl} + \text{Ca}(\text{OH})_2$	$0.5\text{ N KCl} + \text{KOH}$
0.0	0.4963	0.4997
1.0	0.5120	0.5157
2.0	0.5249	0.5276
3.0	0.5418	0.5445
4.0	0.5820	0.5959
5.0	0.6397	0.6488
6.0	0.6797	0.6702
7.0	0.7217	0.7268

It will be noted that for the same amount of base there is a higher hydrogen ion concentration than shown in Table III. Hydrogen ion concentration increases with time in the presence of a soluble base.

To compare the change of the hydrogen ion concentration of solutions in contact with different soils, a number of soils were chosen and similar determinations made as with the yellow-gray silt loam in Table IV. All the soils were shaken for 3 hrs. with 50 cc. of 0.5 N potassium chloride solution containing the required amount of lime. In each case 5 g. of soil were used, except in the case of the peat soils, of which 2.5 g. were taken. The results are given in Table V.

Graphs of some of the above readings (Fig. 8) show nearly straight line functions. Certain of the soils tested, Nos. 1, 2, 3, 4, 6, and 11, show straight line functions for all measurements taken, while the others show more or less distinct changes in slope at two points, one being on the acid side, *i. e.*, where the H^+ concentration is greater than 10^{-7} , and the other on the basic side. The same is true of the yellow-gray silt loam (Fig. 7). At no point in these graphs is there an abrupt change in slope, as would be expected if we were neutralizing a strong acid, but instead the

neutral point is reached at an angle depending upon the character of the soil. It requires comparatively large additions of base to produce a marked change in hydrogen ion concentration.

It may be readily understood that the organic indicators which change color at different hydrogen ion concentrations will show marked differences in the lime requirements of soils, because of the slight change in hydrogen ion concentration with the additions of lime. With the sandy loam soil a change in hydrogen ion concentration of the indicator from 10^{-5} (corresponding to the color change of methyl orange) to 10^{-8} (corresponding to the color change of phenolphthalein) would represent a change in the apparent lime requirement from 0.0 T. to 3 T.; and a change in the lime requirement for peat from about 1 T. to more than 20 T. of calcium carbonate. It is evident that soil solutions may be distinctly acid to litmus while alkaline to methyl orange.

The slight changes in hydrogen ion concentration with addition of base will account for the great variation in results obtained for the lime requirement of soils by the different methods proposed. The greater the slope of the curve the less will be the differences observed. With sandy soils it would be expected that the results obtained by various methods would approach each other, greater variations would be observed with other inorganic soils, while with soils high in organic matter it would be expected that the widest differences would be noted.

The time factor becomes important, as is shown in Tables I and II. The longer the base acts the higher is the hydrogen ion concentration at all initial concentrations of base. The temperature undoubtedly should receive consideration, for the speed of reaction is increased, salts are hydrolyzed to a greater extent,¹ and water of hydration may be decreased with increase in temperature.

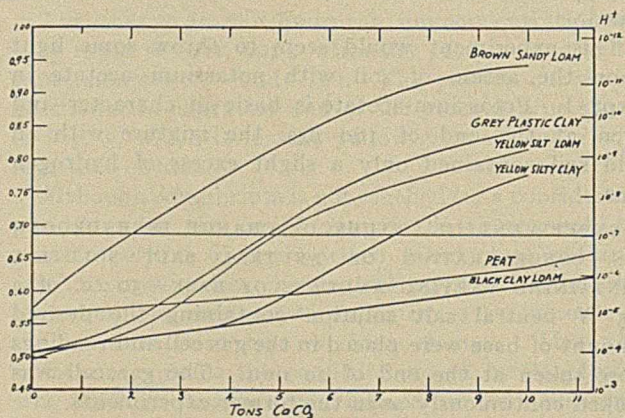


FIG. 8

We should expect that an acid soil would take up larger quantities of calcium from calcium carbonate than from calcium chloride, or sodium from sodium acetate than from sodium chloride as has been repeatedly shown to be the case. This may be explained at least partially by differences in hydrolysis of the salts and the differences in the ionization of the acid.

¹ Landolt and Börnstein, "Physikalisch Chemische Tabellen."

TABLE V—CHANGE IN HYDROGEN ION CONCENTRATION OF SALT SOLUTIONS IN CONTACT WITH SOILS
(Results recorded in volts)

	(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)	(11)
Ca(OH) ₂ as T. CaCO ₃	Black Peaty Loam	Black Clay Loam	Peat	Black Muck	Yellow Silty Clay	Gray Clayey Silt	Gray Plastic Clay	Yellow Plastic Clayey Silt	Grayish Yellow Clayey Silt	Yellow Silt Loam	Brown Sandy Loam
0.0	0.5290	0.5089	0.5654	0.4995	0.4979	0.4707	0.5072	0.4997	0.5034	0.4984	0.5753
1.0	0.5396	0.5171	0.5699	0.5184	0.4831	0.5271	0.5065	0.5126	0.5177	0.6366
2.0	0.5487	0.5257	0.5757	0.5199	0.4922	0.5526	0.5227	0.5386	0.6887
3.0	0.5629	0.5352	0.5828	0.5311	0.5001	0.6077	0.5275	0.5338	0.5780	0.7340
4.0	0.5463	0.5705	0.5485	0.5076	0.6408	0.5538	0.6364	0.7796
5.0	0.5953	0.5571	0.5948	0.5413	0.5942	0.5178	0.6974	0.5719	0.6955	0.8240
6.0	0.5679	0.6006	0.6338	0.5206	0.7485	0.6233	0.6502	0.7297	0.8596
7.0	0.6755	0.6748
8.0	0.7164	0.7216
9.0	0.7372
10.0	0.6391	0.6105	0.6373	0.5822	0.7770	0.5820	0.8483	0.7658	0.7737	0.8178	0.9446
15.0	0.6783	0.6470	0.6699	0.6198
20.0	0.7267	0.7006	0.7153	0.6684

With a carbonate as the reagent, since the point of equilibrium depends also upon the partial pressure of carbon dioxide, it may be understood that the apparent lime requirement may be changed by simply changing the partial pressure of carbon dioxide. The results reported by Ames and Schellenberger,¹ by the Hutchinson-MacLennan² and the vacuum methods, are in line with this argument.

The placing of the soil under artificial conditions with respect to temperature, the use of powerful reagents and the extremely finely divided material employed in most of the methods suggested will certainly affect the speed of the reaction. Furthermore, the equilibrium is without doubt a metastable one. The present researches may not be said to be free from these faults.

that the above statement is true in regard to the aluminates.

In anticipation of criticism from the use of neutral salt solution in the potential readings throughout these investigations the following experiment was planned.

It may be reasoned that if slightly soluble acids are present in acid soils it should be possible to measure the hydrogen ion concentration from such a water solution, if the acid ionizes at all, by means of the hydrogen electrode and other indicators. This has been done by Gillespie,¹ but no attempts have been made to follow the change in hydrogen ion concentration upon the addition of a base. In repeating Gillespie's experiment, using yellow-gray silt loam, it was found that after shaking 5 g. of the soil with 50 cc. of distilled water for one-half hour, the potential reading of the mixture was depressed to 0.565 volt, but there is question whether equilibrium had yet been attained considering the fact that neutral salts depress the reading to about 0.5 volt.

TABLE VI—CONDUCTIVITY OF SOIL SOLUTION CONTAINING VARIOUS QUANTITIES OF BASE, IN CONTACT WITH ACID SOIL
(Base Calculated as Tons of CaCO₃ per Acre
Results = Specific Conductivity × 10⁴)

Base Calc. as T. CaCO ₃	Yellow-Gray Silt Loam. Base Ca(OH) ₂			Yellow-Gray Silt Loam. Base KOH			Black Peaty Loam Base Ca(OH) ₂			Black Clay Loam. Base Ca(OH) ₂ Standing 1 Hr.
	Standing 1 Hr.	Standing 18 Hrs.	Shaken 3 Hrs.	Standing 1 Hr.	Standing 18 Hrs.	Shaken 3 Hrs.	Standing 1 Hr.	Shaken 3 Hrs.		
0.0	1.016	1.016	0.913	1.016	1.016	0.913	2.341	2.837	3.813	
1.0	1.089	1.129	0.916	1.276	1.203	1.132	2.825	3.388	4.015	
2.0	1.220	1.129	0.994	1.733	1.618	3.050	3.720	
3.0	1.638	1.464	1.146	2.520	2.376	1.968	3.468	4.013	4.498	
4.0	1.906	1.980	1.495	3.748	3.245	2.402	3.744	4.296	
4.4	2.033	2.120	1.691	4.637	3.748	
4.8	2.226	2.248	1.826	5.100	4.122	
5.2	2.226	2.276	1.980	5.864	4.620	
5.6	2.346	2.365	2.048	7.318	5.352	
6.0	2.480	2.352	2.074	9.300	5.978	4.888	4.140	4.767	
7.0	3.390	2.799	2.310	14.250	8.718	4.440	4.968	5.408	
10.0	10.170	4.842	3.427	43.580	29.330	25.420	6.055	5.457	5.820	

In soils in contact with solutions we have to deal with solid phases whose reaction velocities are necessarily very slow. The substances which produce the acid phenomena when salt solutions are added to acid soils are undoubtedly only slightly soluble and the products formed may pass into similar solid phases. Even substances which are assumed to be soluble may be held in the colloidal condition. Kahlenburg and Lincoln³ claim that the silicic acid of spring waters is present in the colloidal condition, and the fact that aluminum salts are hydrolyzed to a marked extent while the solubility of aluminum hydroxide is very slight may be taken as an indication that it is held in solution in part as a peptized colloid. The work of Mahin, Ingraham, and Stewart⁴ seems to be evidence

The above experiment was modified by using 50 cc. of distilled water containing the equivalent of 4 tons of lime calculated as calcium carbonate. After the readings had become constant 5 g. of yellow-gray silt loam were added and readings taken at stated intervals. The results are given in Table VII.

TABLE VII—CHANGE IN POTENTIAL IN AN ACID SOIL, ON SHAKING WITH SOLUTION OF CALCIUM HYDROXIDE

Time Minutes	Volt
0	0.9723
30	0.7056
60	0.6772
90	0.6580
240	0.6529
420	0.6416

Equilibrium evidently had not been reached after 7 hrs. of continuous shaking. In performing the experiment some difficulty was encountered because of the high resistance of the chain, but the readings are typical. It will be noted that the reaction is much slower than in the presence of a neutral salt as may be expected from a consideration of the number of ions present in each case.

The salt solutions used throughout this research, unless otherwise stated, were 0.5 N, while the calcium hydroxide solution used above was 0.008 N, or an approximate ratio of 500:8 at the beginning of the reaction. While the neutral salt concentration remained practically unchanged the calcium hydroxide concentration, and consequently the hydroxyl ion concentration, became progressively less.

¹ THIS JOURNAL, 8 (1916), 243.

² Chem. News, 110 (1914), No. 2854, 61.

³ J. Phys. Chem., 2 (1898), 88.

⁴ J. Am. Chem. Soc., 35 (1913), 30.

¹ Loc. cit.

The above experiment brings out another fact of importance. After the base has been neutralized the hydrogen ion concentration continues to rise slowly. The substance producing the action comes to an equilibrium very slowly with water, and 6 hrs. after the lime was neutralized hydrogen ions were still being thrown into the solution.

EXPERIMENT IV. CONDUCTIVITY OF SOIL SOLUTIONS—To test the question of the insolubility of the products formed when a base is added to an acid soil, some conductivity experiments were conducted as a preliminary investigation.

Five grams of soil were shaken with 75 cc. of distilled water containing the required amount of base and conductivity measurements made upon the recently shaken mixture of soil and solution with a plunge electrode having a constant of 0.0305. All readings were taken at $25^{\circ} \pm 1^{\circ}$.

From an inspection of Table VI, the following deductions may be made:

The specific conductance increases with each addition of base, but the increase is much greater with potassium hydroxide than with calcium hydroxide. The difference is far too great to be accounted for by the difference in conductivity of potassium and calcium ions.¹ Either the calcium salts formed are less soluble or they must ionize to a far less extent than the potassium salts. The former view is substantiated by the difference noted in the soluble base left in solution when a soil is treated with water containing an excess of base. From the standpoint of the absorption theory it may be argued that calcium hydroxide is absorbed to a greater extent than potassium hydroxide, but this theory can hardly be substantiated in face of the fact that potassium and calcium hydroxide seem to have practically equivalent power to neutralize the acid of the soil noted elsewhere in this paper.

The specific conductivity decreases with time. This is true in every case except with the black peaty loam, where this may be accounted for by the increase in soluble material brought into solution. It will be noted that the conductivity of the water solution in contact with this soil increased after shaking, indicating that equilibrium had not been reached. If this is taken into account, the conductivity of the solution in contact with this loam also decreases with time. It would seem that the reactions are progressive and that equilibrium is not reached for a considerable time period. This is directly in line with the change in hydrogen ion concentration determinations discussed elsewhere in this paper.

As the acid-producing substances present in the soil are extremely insoluble, as shown by the fact that the pure water extract shows little acidity with ordinary indicators, it would be expected that reactions would be exceedingly slow, especially near the equilibrium point. It will be noted in this connection that

¹ For method of calculating conductance for calcium and sodium ions see Bates, *J. Am. Chem. Soc.*, 35 (1913), 534; and Washburn, "Principles of Physical Chemistry," New York, 1915, p. 214, for calculated values, the equivalent conductance of calcium and potassium ions at infinite dilution being 51 and 63.3, respectively.

there was a much more marked change in conductivity with time at the higher concentrations of base.

The rather slow increase in conductivity would point to precipitation effects which are more marked where calcium is used as the base. Acid substances which are highly insoluble would show high reserve acidity, a condition which is quite evident in the soils investigated.

SUMMARY

1—A new hydrogen electrode cell has been designed which has proved satisfactory for certain types of soil investigation.

2—When an acid soil is added to a neutral salt solution the hydrogen ion concentration of the solution reaches a maximum almost immediately if the soil is wet thoroughly by the solution, but secondary reactions later cause a decrease in hydrogen ion concentration of the solution.

3—When an acid soil is added to a neutral salt solution containing a free base the base is neutralized rapidly, as indicated by the change in hydrogen ion concentration of the solution, following closely the law for equilibrium reactions, but the hydrogen ion concentration of the solution continues to rise for an unknown period.

4—There is no sharp break in the progress of base absorption by an acid soil which will warrant any arbitrary division, such as active and latent acidity, or immediate and eventual lime requirement.

5—The changes in $\log C_H$ approach nearly straight line functions with progressive addition of base in the presence of a neutral salt solution.

6—Different indicators will give differences in lime requirement for soils depending upon the slope of the $\log C_H$ curve. The greatest differences may be expected with soils high in organic matter.

7—A general explanation is given of the different results obtained by different methods for determining soil acidity.

8—When a base is added to an acid soil comparatively insoluble products are formed. Calcium produces a product less soluble than does potassium.

9—The specific conductance of a water solution of an acid soil to which a base has been added increases with each addition of base, but the increase is greater with potassium than with calcium hydroxide, which is far too great to be accounted for by the difference in conductivity of potassium and calcium.

10—Calcium and potassium hydroxide have practically equivalent power to neutralize the acid of an acid soil.

11—The specific conductivity of a pure water solution containing a base in contact with an acid soil decreases with time.

12—An acid soil shows high reserve acidity.

13—The reaction between a water solution of a base and an acid soil is much slower than in the presence of a neutral salt.

14—The absorption of bases by acid soils is due largely to relatively insoluble acids.

THE TRUE TANNING VALUE OF VEGETABLE TANNING MATERIALS¹

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During the past century an enormous amount of energy has been expended in efforts to devise a method for determining the tannin content, or rather the true tanning value, of vegetable materials. Numerous methods have been proposed,² but without any indication as to the correctness of the results obtained. In fact the methods now in general use, both here and in Europe, were made official without any knowledge as to their accuracy, but solely because they are of such nature that different analysts have comparatively little difficulty in getting concordant results. Since tanning materials are usually sold on a tannin basis, these methods have proved of very great value in enabling buyer and seller to agree as to price, but investigators who have blindly accepted the results as reliable have sometimes been led into serious error. In this paper we present what we believe to be the first successful method for determining the true tanning value of vegetable materials.

PRACTICAL DEFINITION OF TANNIN

Work on the chemistry of the tannins is still so far from complete that no rigid chemical definition of them as a class can be given, but their extensive use, especially in the leather industry, has necessitated defining them in terms of some property of practical value. It has therefore become customary to apply the name *tannin* to that portion of the water-soluble matter of certain vegetable materials which will precipitate gelatin from solution and which will form compounds with hide fiber which are resistant to washing. The remaining portion of the soluble matter is called *non-tannin*.

NEW METHOD

PRINCIPLE OF THE METHOD—The method aims, of course, to determine exactly what is called for in the definition. A convenient amount of the tanning material is shaken with a definite amount of purified hide powder until all tannin has been removed from solution. This point is determined by filtering off a portion of the residual liquor and adding drop by drop, avoiding a large excess, a solution containing 10 g. of gelatin and 100 g. of sodium chloride per liter; if the solution becomes turbid or a precipitate forms, it shows that all tannin has not been removed from solution, in which case the mixture must be discarded and the test repeated, using less of the tanning material or shaking for a longer time, until the solution after filtration gives no visible reaction with the gelatin-salt reagent. The tanned powder is then washed free from soluble matter, including the non-tannin removed from solution by the hide powder, which is responsible for the large errors in the methods now in use. It is then carefully dried and analyzed for tannin as in the regular procedure for

vegetable-tanned leathers, and from this figure the percentage of tannin in the original material may readily be calculated.

SENSITIVITY OF GELATIN-SALT TEST AND ITS EFFECT UPON THE METHOD—The gelatin-salt test appears to be the most satisfactory one known for determining whether or not a tan liquor has been completely detannized. In the present official methods, it is assumed that the hide powder completely detannizes the liquor because the non-tannin filtrate remains clear upon addition of the gelatin-salt reagent. This reagent, however, gives negative results with extremely dilute solutions of tannin. Another fact tending to make it difficult to detect the presence of small amounts of free tannin in the liquor after shaking with hide powder is that these liquors are always turbid and must be filtered, and it is a known fact that filter paper will remove tannin from solution to some extent. It was therefore essential to determine the sensitivity of the test for the conditions under which the analyses were made.

If the time of shaking, volume of tan liquor, and quantity of hide powder are kept constant, the less the concentration of the liquor the greater will be the fraction of tannin removed by the hide powder. This made it desirable in checking the accuracy of the work to make several sets of analyses for each tanning material examined, using a different concentration of liquor in each case. The most concentrated solution of each material noted in Table I was used to test the sensitivity of the gelatin-salt reagent by diluting to greater and greater extent and *then* filtering and testing with the reagent under the same conditions as employed in the actual analyses. The reagent was added a drop at a time to 10 cc. of the clear filtrate in a test-tube. The dilutions at which the test failed, expressed in cc. of tan liquor diluted to 100 cc., were as follows: quebracho 1, hemlock bark 2, oak bark 2, larch bark 2, chestnut wood 0.5, osage orange 2, sumac 1, and gambier 4. It therefore follows, since the detannized filtrates from all these liquors gave a negative test with the gelatin-salt reagent, that more than 95 per cent of the tannin of the gambier liquor and more than 97 per cent of the tannin of every other liquor were removed from solution by the hide powder. But the analyses were also carried out with much more dilute liquors, from which the hide powder should remove an even greater percentage of the tannin. Since the results obtained for any one material, using several dilutions, checked so closely, we may reasonably conclude that the hide powder removed *practically* all of the tannin from all solutions which gave no test with the gelatin-salt reagent after the shaking process.

HIDE POWDER—The hide powder used in this work is a highly purified product known as American Standard and is prepared by the Standard Mfg. Co. of Ridgway, Pa., especially for use in the analysis of tanning materials. Since this hide powder is of variable composition due to changes in water content, it was necessary to base our calculations upon the protein it contained, to which we shall hereafter refer simply as hide substance. Von Schroeder¹ found hide substance to

¹ Presented before the Section of Leather Chemistry at the 59th Meeting of the American Chemical Society, St. Louis, Mo., April 14, 1920

² For a review of work done since 1803, see Procter's "Leather Industries' Laboratory Book" (Spon, 1908), pp. 168-176.

¹ Procter, *Loc. cit.*

contain 17.8 per cent of nitrogen, which figure is generally accepted. The per cent of nitrogen determined by the Kjeldahl method, multiplied by 5.62, should therefore give the per cent of hide substance. The hide powder used in this work showed by analysis

	Per cent
Water.....	12.29
Ash.....	0.36
Fat (chloroform extract).....	0.82
Hide substance (N X 5.62).....	86.50
TOTAL.....	99.97

from which it would appear that the factor 5.62 is correct.

MATERIALS EXAMINED—Eight materials were selected for examination which are fairly extensively used and which show great differences in properties, especially in so-called astringency. The solid quebracho extract and the four liquid extracts of oak bark, larch bark, chestnut wood, and osage orange are typical samples of the best of these materials now on the American market. The gambier is the ordinary pasty product from the East Indies; the sumac, consisting of ground leaves and small twigs, is from a reputable dealer in Palermo; and the hemlock bark came from the forests of Wisconsin.

PROCEDURE—The extracts were simply dissolved in hot water, cooled slowly, and made up to the mark. The bark and sumac were finely ground and leached by percolation, only the extracted portions being used after making up to definite volume. In each test, 12 g. of hide powder (of known hide substance content) were put into a wide-mouth, rubber-stoppered, half-pint bottle, the tanning material dissolved in 200 cc. of solution was added, and the whole was shaken in a rotating box for 6 hrs.

The amount of material that could be used was limited by the amount of tannin that the hide powder was capable of taking up in 6 hrs. On the other hand it was desirable not to use too little, since the less the amount of tannin fixed per unit of hide substance, the less the accuracy of the method, since the tannin was determined by difference. Whenever the liquor after the 6-hr. shaking gave a turbidity or precipitate with the gelatin-salt reagent, the test was repeated with less material. The smallest amount used for any material represents the least we were able to use and still get reproducible results.

The tanned powder was washed by shaking with 200 cc. of water for 30 min., squeezing through a suitable cloth, and repeating the washing operation until the wash water showed no color and gave no test with ferric chloride solution. Except for the osage orange and chestnut wood extracts, which are unusual in several respects, not more than 12 washings were required to free the powders from non-tannin, which shows that the line of demarcation between tannin and non-tannin is fairly sharp for the commoner materials. The wash water continued to extract coloring matter from the powders tanned with osage orange until after the fiftieth washing, while as many as 25 washings were required to free the powders tanned with chestnut wood from soluble matter producing a dark color with ferric chloride. All wash water was tested with the gelatin-salt reagent, but in every case the test was

negative. This method of washing was found to be more efficient than using running water on a suction filter, probably because the mechanical agitation assists the diffusion of soluble matter from the interior of the hide fibers.

The washed powders were dried at room temperature for 24 hrs. or longer and then analyzed for water, ash, fat, and hide substance. The difference between 100 and the sum of these percentages was taken as the per cent of tannin in the leather. The parts of tannin per 100 parts of hide substance in the leather divided by parts of tanning material used per 100 parts of hide substance gave the fraction of tannin in the original material.

The results for the eight materials examined are given in Table I.

TABLE I
Percentage Analysis of
Tanned Hide Powder

MATERIAL	Material Grams per Liter	Percentage Analysis of Tanned Hide Powder			Hide Substance (N X 5.62)	Tannin (by Difference)	Per 100 G. of Hide Substance		Tannin in Material Per Cent
		Water	Ash	Fat			Tannin Found Grams	Material Used Grams	
Quebracho.....	18.8	11.56	0.14	0.35	74.92	13.03	17.39	36.8	47.26
Quebracho.....	18.8	11.42	0.08	0.35	75.05	13.10	17.46	36.8	47.45
Quebracho.....	11.5	13.81	0.03	0.30	77.53	8.33	10.74	22.6	47.52
Hemlock Bark.....	150.0	9.94	0.12	0.24	76.54	13.16	17.19	287.9	5.97
Hemlock Bark.....	100.0	10.73	0.13	0.28	79.39	9.47	11.93	191.9	6.22
Hemlock Bark.....	75.0	12.76	0.05	0.28	79.53	7.38	9.28	147.1	6.31
Oak Bark.....	67.5	12.54	0.07	0.12	74.76	12.51	16.73	131.6	12.71
Oak Bark.....	45.0	11.19	0.09	0.24	79.36	9.12	11.49	87.6	13.12
Oak Bark.....	25.0	13.53	0.05	0.34	81.00	5.08	6.27	48.9	12.82
Larch Bark.....	67.5	12.59	0.09	0.13	75.61	11.58	15.32	131.6	11.64
Larch Bark.....	45.0	13.65	0.09	0.30	77.90	8.06	10.35	87.7	11.80
Larch Bark.....	25.0	16.52	0.08	0.25	78.65	4.50	5.72	48.9	11.70
Chestnut Wood.....	67.5	12.43	0.11	0.05	75.76	11.65	15.38	131.6	11.69
Chestnut Wood.....	45.0	12.82	0.13	0.19	78.54	8.32	10.59	87.7	12.08
Chestnut Wood.....	37.5	12.05	0.10	0.21	80.74	6.90	8.55	71.7	11.92
Sumac.....	93.8	11.39	0.16	0.36	74.92	13.17	17.58	179.3	9.80
Sumac.....	62.5	12.26	0.23	0.31	78.38	8.82	11.25	119.5	9.41
Sumac.....	37.5	11.75	0.12	0.37	81.97	5.79	7.06	73.5	9.61
Osage Orange.....	48.8	12.82	0.13	0.17	77.35	9.53	12.32	95.0	12.97
Osage Orange.....	32.5	12.83	0.09	0.25	80.09	6.74	8.42	63.3	13.30
Osage Orange.....	26.3	12.43	0.12	0.25	81.43	5.77	7.09	51.2	13.85
Gambier.....	50.0	12.08	0.18	0.19	81.44	6.11	7.50	97.4	7.70
Gambier.....	49.5	11.77	0.26	0.28	81.70	5.99	7.33	94.7	7.74
Gambier.....	29.0	13.06	0.14	0.35	82.74	3.71	4.48	56.5	7.93

COMPARISON WITH A. L. C. A. METHOD

Practically all tanning materials are bought and sold in this country on the basis of tannin content as determined by the official method of the American Leather Chemists Association. The principle of the method is similar to that of the new method as regards the shaking of a solution of the tanning material with hide powder, but differs in that the drop in concentration of the liquor upon shaking is taken as the measure of its tannin content, while the tanned powder is simply discarded. The details of the A. L. C. A.¹ method follow:

The hide powder is specially prepared by giving it a light chrome tannage with chrome alum, washing it practically free from soluble matter, and squeezing it until it contains not less than 71 nor more than 74 per cent of water. The solution of tanning material for analysis must contain not less than 0.375 nor more than 0.425 g. of tannin per 100 cc., as found by this method. To 200 cc. of this solution is added such an amount of the wet hide powder as contains not less than 12.2 nor more than 12.8 g. of dry hide powder and the whole is shaken for 10 min. The limits defined are to some extent arbitrary, but it has been found necessary to set limits of some sort in order to get concordant results. The detanned solution is separated from the powder by squeezing through linen and is then filtered through

¹ *J. Am. Leather Chem. Assoc.*, 14 (1919), 654.

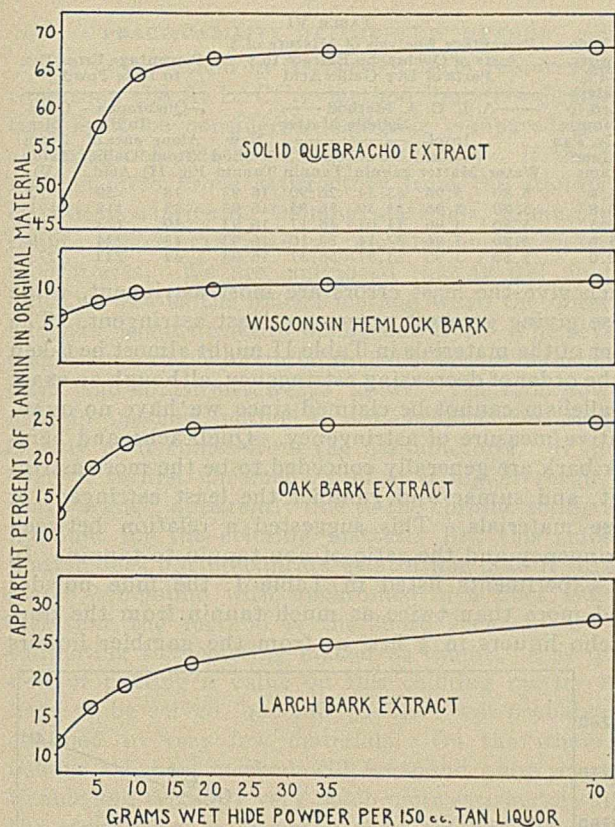


FIG. 1

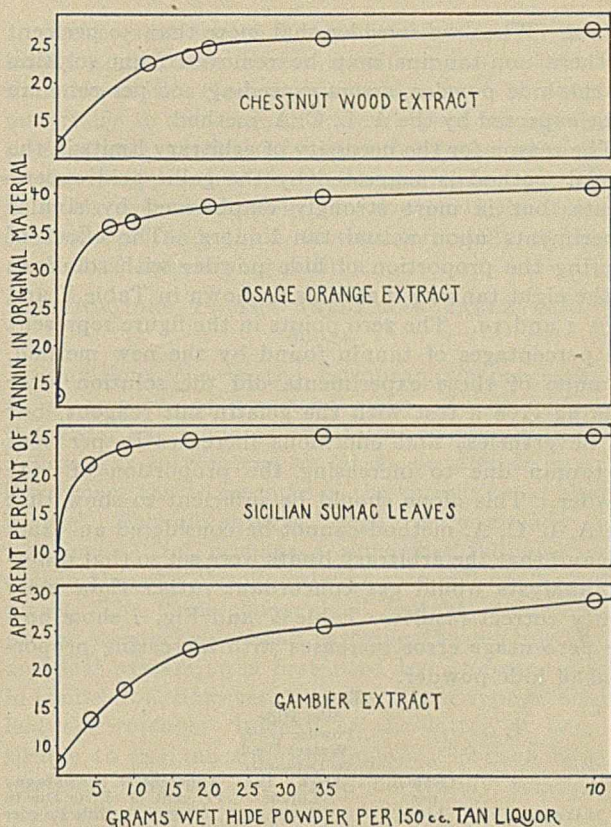


FIG. 1a

paper, after the addition of kaolin, the solution being returned until the filtrate is quite clear. The amount of residue from an aliquot portion of this filtrate, after correcting for the water introduced by the hide powder, is taken as a measure of the non-tannin in the original material. The difference between the total soluble matter and the non-tannin is called tannin.

Several outstanding sources of error in this method were emphasized by the authors in an earlier paper.¹ The most serious defect is that it does not take into account the fact that hide powder removes non-tannin of certain kinds, such as gallic acid, pyrocatechol, etc., from solution to an extent depending upon the concentration. These substances do not make leather and are readily washed out. This explains the fact that where tanners try to keep a rigid cost account of tanning materials, they find no such amount of tannin in the leather as would be expected from the amount of tannin used, as determined by the A. L. C. A. method. The analyses of the eight materials by the A. L. C. A. method are given in Table II along with a calculation of the percentage errors involved in this method, made upon the assumption that the results obtained by the new method are correct.

TABLE II
Percentage Analysis of Material
A. L. C. A. Method

MATERIAL	Soluble Matter				New Method Tannin	Percentage Error in A. L. C. A. Method
	Water	Insoluble Matter	Non-tannin	Tannin		
Quebracho.....	17.87	7.16	6.96	68.01	47.41	43
Hemlock Bark...	8.90	74.33	6.71	10.06	6.17	63
Oak Bark.....	52.66	3.68	19.46	24.20	12.88	88
Larch Bark.....	51.08	5.88	20.90	22.14	11.71	89
Chestnut Wood..	58.90	1.50	13.80	25.80	11.90	117
Sumac.....	9.25	47.20	17.99	25.56	9.61	166
Osage Orange...	46.05	3.45	10.63	39.87	13.37	198
Gambier.....	51.12	5.36	18.57	24.95	7.79	220

¹ J. Am. Leather Chem. Assoc., 13 (1918), 429.

Although the enormous errors in the A. L. C. A. method are nothing short of sensational, we are convinced that they are not at all exaggerated. The extent of these errors is less surprising, however, when the behavior of certain non-tannins, like gallic acid, in the presence of hide powder is studied. A series of solutions of gallic acid of different strengths was prepared and treated by the A. L. C. A. method as though they were tan liquors of the required strength. The results for non-tannin should have been 100 per cent in every case, if the method were reliable, but the actual data in Table III show that a very large proportion of the acid is taken up by the hide powder.

TABLE III—RESULTS OF TREATMENT OF PURE GALLIC ACID SOLUTIONS BY THE A. L. C. A. METHOD

Using 47 g. of wet hide powder (73% water) to 200 cc. of solution

Gallic Acid Grams per Liter	Non-tannin Per cent	Tannin Per cent
8.88	54.0	46.0
4.44	47.1	52.9
2.22	43.8	56.2
1.11	40.4	59.6

TABLE IV—EFFECT OF ALTERING PROPORTION OF HIDE POWDER UPON AMOUNT OF GALLIC ACID REMOVED FROM A 0.888 PER CENT SOLUTION

Using the principle of the A. L. C. A. method

Wet Hide Powder (73% water) G. per 200 cc.	Non-tannin Per cent	Tannin Per cent
5	91.8	8.2
10	86.0	14.0
25	69.6	30.4
50	52.1	47.9
75	43.7	56.3

Table IV shows that the amount of gallic acid removed from solution is very largely dependent upon the proportion of hide powder employed. Our results by the new method for gambier indicate that this sample contains more than 4 times as much non-tannin as

tannin. When we consider that more than 50 per cent of these non-tannins may be removed from solution by the hide powder, errors exceeding 200 per cent are to be expected by the A. L. C. A. method.

The reason for the necessity of arbitrary limits in the official method is suggested by the gallic acid experiments, but is more strongly emphasized by similar experiments upon actual tan liquors. The effect of altering the proportion of hide powder with solutions of the eight tanning materials is shown in Table V and Figs. 1 and 1a. The zero points in the figure represent the percentages of tannin found by the new method. In none of these experiments did the solution after shaking give a test with the gelatin-salt reagent, but we nevertheless find enormous increases in per cent of tannin due to increasing the proportion of hide powder. This alone should be sufficient to show that the A. L. C. A. method cannot be considered an exact one and that the arbitrary limits were set so that different analysts would get concordant rather than more nearly correct results. Table V and Fig. 2 show how the percentage error increases with increasing proportions of hide powder.

TABLE V

MATERIAL	Grams per Liter	Wet Hide Powder (73% Water) Used to Detannize 150 Cc. Tan Liquor Grams	Apparent Per cent of Tannin	Percentage Error Due to Hide Powder
Quebracho.....	3	70.0	68.18	44
		35.0	67.56	43
		20.0	66.61	40
		10.0	64.36	36
		5.0	57.56	21
Hemlock Bark.....	20	70.0	10.98	78
		35.0	10.60	72
		20.0	9.76	58
		10.0	9.35	52
		5.0	7.98	29
Oak Bark.....	4.11	70.0	25.02	94
		35.0	24.59	91
		17.5	24.01	86
		8.8	22.09	72
		4.4	18.77	46
Larch Bark.....	4.37	70.0	28.10	140
		35.0	24.52	109
		17.5	21.97	88
		8.8	19.10	63
		4.4	16.24	39
Chestnut Wood.....	15	70.0	26.87	126
		35.0	25.80	117
		20.0	24.59	107
		17.5	23.52	98
		12.0	22.49	89
Sumac.....	4	70.0	24.98	160
		35.0	25.05	161
		17.5	24.47	155
		8.8	23.45	144
		4.4	21.45	123
Osage Orange.....	8	70.0	40.48	203
		35.0	39.47	195
		20.0	38.21	186
		10.0	36.27	171
		7.0	35.67	167
Gambier.....	4.58	70.0	29.04	273
		35.0	25.60	229
		17.5	22.56	190
		8.8	17.22	121
		4.4	13.38	72

TABLE VI

Percentage Analysis of Mixture of 5 Parts of Quebracho Extract to 9 Parts of Dry Gallic Acid

Wet Hide Powder (73% Water) Used to Detannize 150 Cc. Tan Liquor Grams	A. L. C. A. Method				New Method Tannin	Percentage Error Due to Hide Powder		
	Soluble Matter		Non-tannin	Tannin		Quebracho Alone (from Fig. II)	Gambier (from Table V)	
	Water	Insoluble Matter						
4.4	5.80	3.96	63.34	26.90	16.93	18	59	72
8.8	5.80	3.96	53.39	36.85	16.93	33	118	121
17.5	5.80	3.96	44.07	46.17	16.93	39	173	190
35.0	5.80	3.96	37.14	53.10	16.93	43	214	229
70.0	5.80	3.96	33.87	56.37	16.93	44	233	273

which give the least errors are most astringent, while those giving greatest errors are least astringent. The order of the materials in Table II might almost be taken as the order of decreasing astringency, although an exact parallelism cannot be claimed since we have no quantitative measure of astringency. Quebracho and hemlock bark are generally conceded to be the most astringent, and sumac and gambier the least astringent of these materials. This suggested a relation between astringency and the ratio of non-tannin to tannin. In the experiments listed in Table I, the hide powder fixed more than twice as much tannin from the quebracho liquors in 3 hrs. as from the gambier liquors

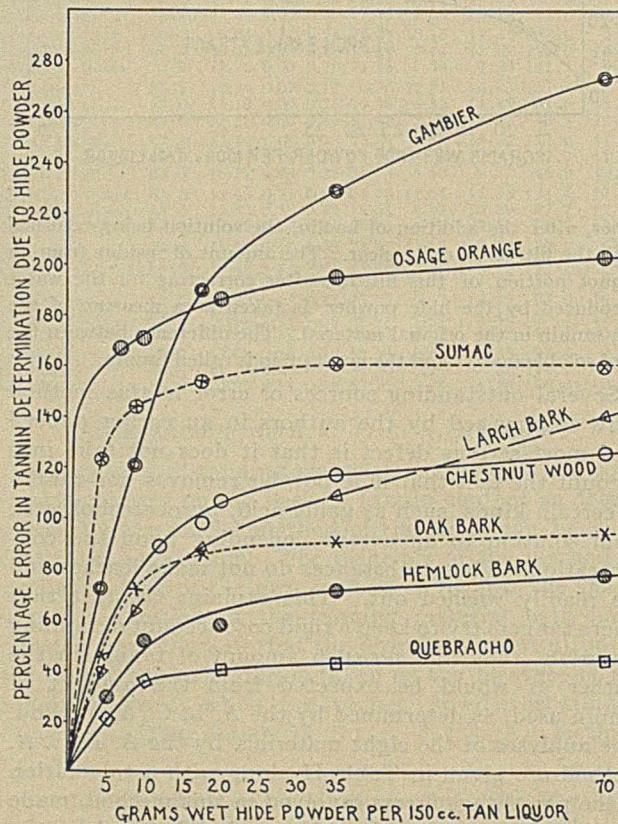


FIG. 2

As might be expected, the greatest errors in the Official method are obtained with those materials containing the greatest proportion of non-tannin. Quebracho, having least non-tannin, gives the smallest error. However, if the quebracho is mixed with gallic acid to make the proportion of non-tannin to tannin about the same as in the case of the gambier, it gives errors nearly as great as in the case of the gambier. This is shown in Table VI.

Comparison of the two methods has brought out at least one fact of practical significance: Those materials

in 6 hrs. But, when enough gallic acid was added to the stronger quebracho liquors to give them the same proportion of non-tannin to tannin as in the gambier, the hide powder did not remove anywhere nearly all the tannin in 6 hrs. Upon addition of the gelatin-salt reagent to the liquors after shaking, huge precipitates were formed, suggesting a great reduction in astringency. That the effect was only one of slowing up, the tanning action was proved by the fact that the hide powder was able to detannize the solution completely in 24 hrs.

PRACTICABILITY OF THE NEW METHOD

The new method in its present form requires more time than the A. L. C. A. method, but this disadvantage must be considered negligible compared to the advantage of greatly increased accuracy. If the new method is to prove satisfactory from the standpoint of setting a price on tanning materials, it must give results which are readily reproducible in different laboratories. We are convinced that it will do this quite as well as the official method when the different analysts become used to manipulating it. The several results for any one material in Table I were determined days, and sometimes weeks, apart. The hide powder used in the new method is not chromed, but is used exactly as it comes from the manufacturer. If, however, it is first chromed, it gives a higher result for osage orange, apparently due to the chrome acting as a mordant for the coloring matter. But this coloring matter does not precipitate gelatin nor does it form a stable compound with unchromed hide fiber and we feel that the method is the more accurate for not estimating this coloring matter as tannin. The question of putting a value on this coloring matter may have to be solved, but this problem will probably be confined to very few materials. On the whole we believe the new method will be found quite as practicable and certainly very much more satisfactory from the standpoint of accuracy than the present official method. What has been said concerning the A. L. C. A. method applies equally well to the European methods since they differ only in detail.

SUMMARY

A new method of tannin analysis is described which we believe determines exactly what is called for in the generally accepted definition of tannin from a practical viewpoint, namely, that portion of the water-soluble matter of certain vegetable materials which will precipitate gelatin from solution and which will form compounds with hide fiber which are resistant to washing.

The analyses of eight common tanning materials by the new method and by the official method of the American Leather Chemists Association indicate that the latter method is in error to the extent of from 43 to 220 per cent.

The new method gives reproducible results and is considered entirely practicable.

PRACTICAL METHODS FOR THE DETERMINATION OF RADIUM. III—ALPHA-RAY METHOD, GAMMA-RAY METHOD, MISCELLANEOUS¹

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In two previous papers² the writer has described a form of interchangeable electroscope, the details of its construction, and its use in connection with the determination of radium by the emanation method.

¹ Published with permission of the Director of the U. S. Bureau of Mines.

² THIS JOURNAL, 7 (1915), 406, 1024.

In the present paper, it is proposed, first, to discuss briefly some more recent modifications in the construction of the electroscope, which have contributed materially to the ease and accuracy of its manipulation; second, to discuss rather fully the application of the α -ray method to the determination of radium in solids without reference to any particular form of electroscope; and, third, to consider briefly the γ -ray method of determining radium.

MODIFICATIONS IN THE INTERCHANGEABLE ELECTROSCOPE

Conditions due to the European war necessitated an electroscope of American make. In devising one, the writer had economy and accuracy equally in mind, and sought to combine as far as possible in a single instrument the advantages of the various types already in use. The advantages sought have already been mentioned,¹ but perhaps a somewhat fuller explanation in the way of acknowledgments will not be out of place. The "interchangeable" feature, enabling the use of a detachable head carrying the reading device and leaf system, was first used by Professor Ebler,² in connection, however, with the Exner type of double-leaf electroscope. Instead of the latter, it was desirable to use the simpler single-leaf Wilson type so modified that the leaf could be readily removed if replacements were necessary.

The highly advantageous feature of having the reading microscope firmly fixed to the head so there can be no chance of accidentally changing the relative positions of leaf and micrometer scale of the microscope was adapted from the Wulf³ electrometer, thus providing rigidity while preserving complete visibility of the leaf system—an advantage not possessed by the Wulf instrument.

The most far-reaching change in the instrument, since its earlier description, consists in the substitution of amber or amberoid insulation instead of sealing-wax for both insulators (points *g* and *d* in the original).⁴ This substitution made it necessary, however, to provide a new means of rendering the emanation chamber gastight. This has been accomplished without the use of any binding material by an ingenious arrangement, due to Mr. P. F. Elzi, of the Sachs-Lawlor Company, Denver. Fig. 1 illustrates its construction. The brass collar *a* is threaded into the lower chamber (not shown) at *b* and made gastight by means of a lead washer, *c*. The collar *a* is hollow and provided with an interior projecting shoulder, *d*, on which rubber washers rest at *f* and *f'* above and below. On *f* and *f'* rest amberoid discs, *g* and *g'*, pierced by the rods *h* and *h'*, both of which are threaded into the connector *i*. The holes through *g* and *g'* are made tight by small rubber washers *j* and *j'*. *h* terminates above in a shoulder, *k*, resting on the washer *j* and a tip *l* to make electrical contact with the spring *s* attached to the leaf system above. Below, *h'* is provided with a similar shoulder, *k'*, which rests

¹ Part I, *Loc. cit.*

² *Chem. Kalendar*, 1914, Part II, 367.

³ *Physik. Z.*, 8 (1907), 246, 527.

⁴ Part I, *Loc. cit.* Fig. 3

on the washer *j'* and leads below to the electrode *e*. By screwing *h* and *h'* into the common connector *i*, tension is applied on the washers *f* and *f'* and *j* and *j'*, thus giving gastightness to the lower chamber without any binding material. This simple device has proved eminently satisfactory, usually holds gastight for a year or more, and can be readily tightened or, if necessary, taken apart to renew the washers. Much labor in recalibrating the instrument is avoided in this way.

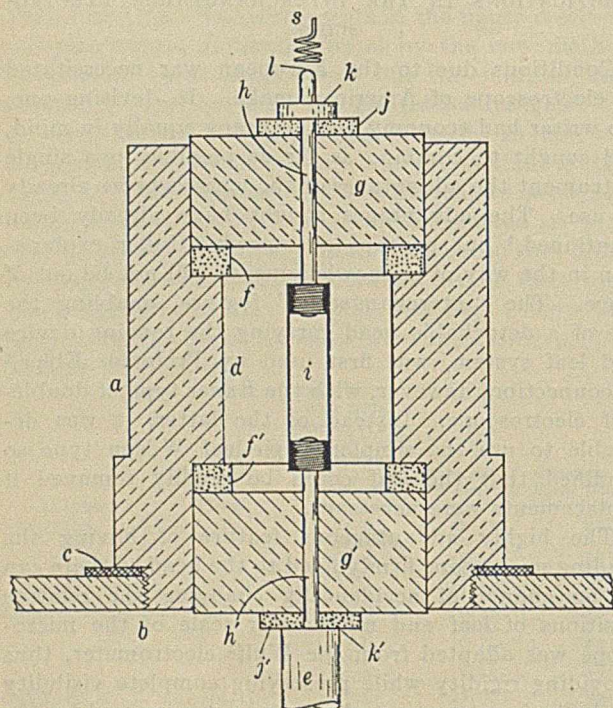


FIG. 1—COLLAR OF ELECTROSCOPE WITH AMBEROID INSULATORS MADE GASTIGHT WITHOUT CEMENT

Further changes have consisted in replacing the two rubber-connected glass stopcocks with metal stopcocks brazed into the chamber. The microscope holder has also been made adjustable by means of a heavy milled-head screw (Fig. 2). In the new construction the front metal plate carrying the microscope fits into the head cylinder by means of a V-groove carrying three tightening screws, by loosening which the plate can be rotated to bring the microscope to view the leaf at any desired part of its arc.

The changes mentioned above have materially added to the usefulness and accuracy of the interchangeable electroscopes.

THE ALPHA-RAY METHOD OF ESTIMATING RADIUM IN SOLIDS

In its simplest form, this method consists in exposing a solid powder covering a plate of definite surface and depth in the discharge chamber of an electroscopes, which might be of the type illustrated by the middle chamber shown in Fig. 2. By comparing the rates of discharge of the leaf as viewed through a microscope provided with a micrometer scale, first using a solid with known uranium or radium content and later the unknown, a more or less approximate idea is obtained of the radium or uranium content of the latter.

This method, as practically applied to radioactive ores, has been described by Moore and Kithil¹ and briefly commented on by Parsons, Moore, Lind, and Schaefer.² Its refinement for scientific measurements, with reference to the penetration of layers of various depths by α -rays, has been carefully worked out by McCoy.³ Owing to its sensitiveness, simplicity, and ease of manipulation, this method has been more generally used than any of the methods of measuring radioactivity. α -Ray instruments are now very generally used in prospecting, ore sorting and milling, and for certain purposes even in the control of radium plant operations. For qualitative and very roughly quantitative purposes the α -activity furnishes a quick and satisfactory method. Attempts to extend its use for more quantitative purposes are beset with many pitfalls and uncertainties which are discussed in this paper. To illustrate the fundamental difficulty in its use for quantitative purposes, complaint has been made that the results of α -ray activity measurements frequently do not check even roughly with other results, such as the determination of uranium by chemical methods, or the direct determination of radium by the emanation method. These discrepancies have been especially marked for carnotite ores from different localities, as can be seen from Table I.

TABLE I—SHOWING THE VARIABILITY OF DIFFERENT CARNOTITES IN α -RAY ACTIVITY PER URANIUM CONTENT

SAMPLE	Per cent U_3O_8 (By Analysis)	α -Activity (Arbitrary Units)	α -Activity Per cent U_3O_8	Em. power Per cent	Ra U	α -Activity Per cent U_3O_8 Corrected
1...	1.52	0.087	0.0572	20.4	102.7	0.0638
2...	1.60	0.064	0.0400	50.5	94.9	0.0582
3...	2.07	0.139	0.0671	29.6	101.5	0.0804
4...	3.16	0.172	0.0544	39.7	119.2	0.0631
5...	4.78	0.207	0.0433	33.90	98.8	0.0542
6...	9.12	0.259	0.0284	30.4	84.1	0.0382
7...	23.40	0.733	0.0313	45.8	72.4	0.0508
8...	33.20	1.206	0.0363	16.2	107.8	0.0381

In Table I, the samples of carnotite represent all grades from a low to a high percentage of U_3O_8 , and also come from various localities in Colorado and Utah.

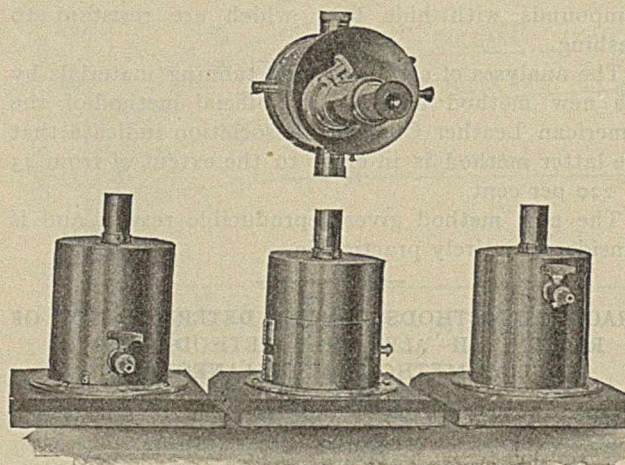


FIG. 2

If the α -ray method, as usually applied, were really applicable to all carnotites, then the ratio of surface activity to the percentage of U_3O_8 given in the fourth

¹ U. S. Bureau of Mines, *Bulletin 70* (1913), 64.
² *Ibid.*, 104 (1916), 87.
³ *Phys. Rev.*, 1 (1913), 393.

column should be constant. Evidently this is not even approximately true, and one can see at once how far astray one might be led in choosing any of these different samples as standards for other α -ray comparisons.

In seeking the reason for these deviations, three principal causes present themselves:

1—Variation in the amount of the loss of radium emanation by gaseous diffusion from the ore; the percentage loss is usually referred to as the "emanating power." This entails the loss of α -radiation, not only from the emanation itself, but also from the succeeding α -ray members, RaA, C, and F. The actual percentage loss for the ores under consideration is reported in the fifth column of Table I, and, as has already been pointed out by Lind and Whittemore,¹ is not only unusually high for carnotite, but also quite variable.

2—A second possible source of error is the variability of the radium-uranium ratio in carnotite. As also reported by Lind and Whittemore,² this variation is confined to *small* specimens of carnotite which can vary considerably from the normal value of the radium-uranium ratio. This variation is reported in the sixth column of Table I in terms of per cent, placing the normal (pitchblende) ratio equal to 100 per cent. In the last column the values of Column 4 have been corrected according to the data in Columns 5 and 6; that is, the discrepancies introduced by the variation of α -radiation, due to "emanating power" and abnormal ratio of radium to uranium, have been eliminated. The improvement in the degree of constancy of activity compared with the uranium content can be seen to be very slight. Evidently there must be another source of error more far-reaching in its influence than either of the first two.

3—This source of error lies in the position of the radioactive material in the individual grains and also to some extent in the nature of the gangue material. Since the α -ray can penetrate material of the density of silica very slightly—only about 0.03 mm., or 0.001 in.—it is evident that the effective rays come only from near the surface, and any variation of the position of the active material in different samples with respect to the surface would have a great influence on the α -ray activity observed. It is also evident that fine grinding would not obviate the difficulty materially, as it would be impossible to produce comminution below 0.001 inch. McCoy's method of correcting for the absorption could be applied, but is hardly within the powers of the ordinary operator, does not obviate the difficulties due to 1 and 2, and involves time and labor to a prohibitive degree for ordinary practical purposes. In short, any efforts to make the α -ray method really accurate involve more labor than a direct radium determination by the emanation method. There is one favorable factor, however, to the advantage of the α -ray comparison, even for carnotite, namely, that specimens from the same mine or claim

do not usually show such wide deviation in the ratio $\frac{\text{activity}}{\text{per cent U}_3\text{O}_8}$ as those reported in Table I.

With reference to carnotite in general, the conclusion must be drawn that the α -ray method can by no means take the place of a direct radium determination, nor of a uranium analysis, for purposes of sale or for scientific or commercial control. Qualitative and very roughly quantitative results only can be expected. It is not desired that the foregoing shall discourage the use of the α -ray method. Its application is so simple and its results so quickly derived that it will continue to be of great service in obtaining preliminary estimates of the content of radioactive materials as well as invaluable in their detection. It is merely meant to emphasize that it cannot be relied on for final quantitative measurements.

PITCHBLENDE—With reference to pitchblende, the case is decidedly more favorable for the use of the α -ray method. Since the emanation loss is small and varies only slightly in pitchblende, the first difficulty is practically eliminated. The radium-uranium ratio also appears to be constant for all different samples of pitchblende,¹ which eliminates the second difficulty. From the results reported in Table II, the third difficulty also appears to be slight, perhaps due to the primary nature of pitchblende as compared with carnotite, a secondary mineral. At any rate, pitchblendes of different grades can be compared with a fair degree of accuracy.

TABLE II—SHOWING THE RELATIVE CONSTANCY OF THE RATIO $\frac{\text{PER CENT U}_3\text{O}_8}{\alpha\text{-ACTIVITY}}$ IN PITCHBLENDENES

SAMPLE	Per cent U ₃ O ₈ (By Analysis)	α -Activity (Arbitrary Units)	Per cent U ₃ O ₈ α -Activity
1.....	4.15	0.150	27.7
2.....	8.02	0.296	27.0
3.....	10.55	0.388	29.2
4.....	16.4	0.576	28.5
5.....	17.3	0.572	30.2
6.....	24.7	0.931	26.6
7.....	25.3	0.973	26.0

While it would hardly be advisable to use the method where great accuracy is desired, for many purposes it can be employed with convenience and satisfaction.

SULFATES—One of the intermediate products in the production of radium by most of the different processes consists in a precipitated (Ra)BaSO₄. The writer has found by experience that if the radium content is not too high to preclude the possibility of using the α -ray method, the results approximate fairly closely to the radium content, as may be seen from Table III.

TABLE III—SHOWING THE APPROXIMATE CONSTANCY OF $\frac{\text{RADIUM}}{\alpha\text{-ACTIVITY}}$ IN CRUDE (Ra)BaSO₄

SAMPLE	Mg. Ra per Kilo (Emanation Method)	α -Activity (Arbitrary Units)	Radium α -Activity
P ₁	1.41	0.71	1.98
P ₂	2.15	1.20	1.79
40.....	1.32	0.73	1.80
70.....	2.18	1.28	1.70

While the results for the sulfates are not so good as with pitchblende, they show that for a quick measurement preliminary to the employment of the emanation method the α -ray method may be used with benefit. The α -ray method has also been used very

¹ *J. Am. Chem. Soc.*, **36** (1914), 2066.

² *Loc. cit.*

¹ Heimann and Marckwald, *Jahrb. Radioakt. Elektronik.*, **10** (1913), 299.

successfully in the determination of other radioactive substances in small quantity for scientific purposes; but since it is the object of the present paper to deal only with the practical methods of measuring radium itself, they do not require consideration here.

THE GAMMA-RAY METHOD OF MEASURING RADIUM

Radium itself emits α -rays only, but both RaB and RaC emit γ -rays which present the most convenient means of measuring radium in quantities above 0.1 mg. and of not less than 0.05 per cent purity. Since RaB and RaC are products resulting from the decay of radium emanation, a gas, it is necessary to confine radium preparations in a closed vessel to prevent the escape of gas before the measurement is made. The measurement may be carried out after the vessel has been closed for a month or more, in which case the γ -radiation will have reached a constant maximum and no time correction will be necessary; the γ -radiation then having become directly proportional to the quantity of radium present, as is the case of all standard tubes of radium salt which have been sealed for more than a month. If the measurement is made prior to this period, a correction must be made for the unelapsed time. This correction is readily made by reference to the Kolowrat table, just as described in Part II¹ for the emanation method.

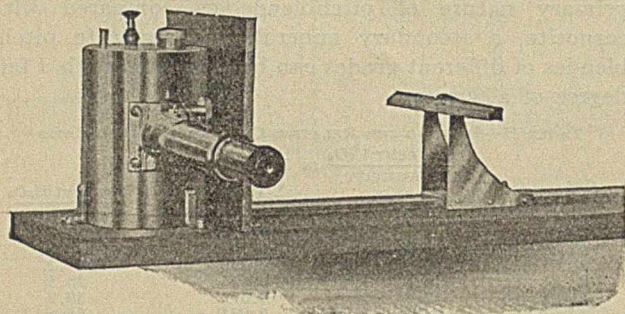


FIG. 3

In order that the accumulation of emanation shall have taken place over a definite period of time, the starting point must be rendered exact by sealing the radium salt in a glass tube as soon after crystallization as possible. It is first necessary, however, to dry the salt thoroughly by raising the temperature for 20 min. or more to 250° C. or higher. Otherwise, decomposition by the α -rays of any water remaining, even in the form of water of crystallization, would generate a dangerous gas pressure in the limited volume of the tube. If the zero period is indefinite or not known, a series of measurements must be made at different intervals, from which the final maximum value can be calculated by comparisons with the Kolowrat table.

The measurement itself consists simply of a comparison of the rates of discharge produced by a tube with known radium content and that of the unknown, each being placed successively in the same fixed position at a suitable distance from the discharge chamber. Almost any type of electro-scope may be used by placing a lead screen, one-eighth to one-

fourth inch thick, between the instrument and the tube containing the radium. A type of γ -ray electro-scope is shown in Fig. 3.

An accuracy of about 1 per cent can be readily attained with ordinary precautions by the use of the simple aluminum or gold-leaf electro-scope.

Radium salts are bought and sold in the United States almost entirely on the certificate of the Bureau of Standards. The measurements are made electro-scopically by the γ -ray method, using standards that have been compared with the International standard in Paris. Every radium laboratory should have at least one secondary standard that has been certified by the Bureau of Standards.

Radium emanation, which is now quite largely used therapeutically instead of radium itself, may be measured by the γ -ray method exactly as radium, and is expressed in equivalent units, one curie being the amount of emanation in equilibrium with one gram of radium element. It is necessary only to allow the emanation to remain in a closed vessel for 4 hrs. to arrive at maximum γ -radiation before making the measurement. In making this measurement, one additional correction is necessary. On account of the short life of radium emanation (3.85 days half period) RaC lags behind in decaying by 0.8 per cent; and since it is RaC, not emanation, which furnishes the principal γ -rays, this correction must be deducted from the γ -ray indication to give the true quantity of emanation. If one wishes to know simply the γ -radiation and not the actual quantity of radium emanation, this correction is not necessary.

The writer is indebted to the Denver Fire Clay Company for Figs. 2 and 3, and to the Sachs-Lawlor Company of Denver for permission to describe Mr. Elzi's arrangement shown in Fig. 1. The electroscopes described are made by the Sachs-Lawlor Company and distributed by the Denver Fire Clay Company.

THE DISTRIBUTION OF CERTAIN CHEMICAL CONSTANTS OF WOOD OVER ITS PROXIMATE CONSTITUENTS

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INTRODUCTORY

In a previous paper,¹ the author has proposed a scheme for separating wood into a number of proximate groups. In addition to the groups designated, certain organic radicals (such as CH₃O and CH₂CO) are known to occur in woods. Any given species is characterized by definite contents of these radicals which have accordingly become quite generally recognized as constants for that particular wood. They found no place in the proposed scheme for the reason that the quantitative relations between these so-called constants and the proximate groups were not known and it appeared likely that their inclusion would produce an overlapping of constituents. The purpose of

¹ *Loc. cit.*

¹ THIS JOURNAL, 11 (1919), 556.

the present investigation has been to learn the relations existing between the more important constants and proximate groups. Experiments have, therefore, been conducted to ascertain the distribution of (1) furfural-yielding groups, (2) acetic-yielding groups, and (3) methoxy groups, over the two chief proximate groups—cellulose and lignin.

The three above-mentioned constants have been considerably investigated. The pioneer work of DeChalmot¹ on the furfural-yielding complex and of Benedikt and Bamberger² on the methoxy groups has resulted in quantitative data for the most important woods and fibrous products and considerable information as to the relation of these data to the life history of the plant. Cross and Bevan³ have studied the production of acetic acid from the lignocelluloses by various reactions of decomposition. Of these reactions, acid hydrolysis may probably be safely taken as a measure of the acetic-yielding groups preëxistent in the woods, which according to these authors are not acetyl groups, CH_3CO , but acetic residue groups CH_2CO .⁴ Schorger⁵ has recently reported values for all three of these constants for a number of American woods.

The yields of acetic acid by hydrolysis and methoxy groups by the Zeisel method have a considerable industrial significance as measures, respectively, of the maximum commercial yields of acetic acid and wood alcohol obtainable by destructive distillation. The fact that the full yield of these substances is never realized industrially, due to losses by secondary reactions,⁶ detracts from the practical value of their determination. On the other hand, it affords an incentive for the improvement of the manufacturing processes in the direction of a closer approach to theoretical yields. The furfural yield is not a measure of valuable products in the wood, but its determination in finished products has been used to give information as to the source of raw materials used in their manufacture.⁷

While precise information is lacking as to the relation of these constants to cellulose and lignin, certain opinions have found considerable support. The methoxy group is generally recognized as being closely connected with the lignin complex and the methoxy determination has been proposed as a measure of the lignin content.⁸ The furfural-yielding groups have been found to be partly associated with the cellulose and partly with the lignin. W. E. Cross⁹ has concluded from his experiments that the groups yielding acetic acid by hydrolysis are contained in the lignin and absent from the cellulose portion of woods.

The extent to which any of these radicals remain attached to either the cellulose or lignin, when lignocellulose is subjected to reactions of decomposition,

must necessarily depend very much upon the nature and intensity of these reactions. The experiments herein described were conducted to ascertain the distribution of the constants under certain specified conditions, namely, those of the analytical determinations of cellulose and lignin. The results may therefore be taken to indicate whether the constants are to be regarded as groupings attached to and subordinate to these chief proximate groups, or as possessing an independent existence and themselves entitled to rank as proximate groups.

EXPERIMENTAL

The experiments were confined to redwood (*Sequoia sempervirens*). A quantity of sawdust was obtained by making a number of cross-sectional cuts through a redwood slab. The mixture of coarse and fine sawdust was sifted through a 50-mesh screen and the selected fine material used for the various determinations. The samples were analyzed in the air-dry condition with a moisture content of 11.62 per cent.

PREPARATION OF EXTRACTED WOOD—Portions of 2 g. were weighed into alundum thimbles and extracted in a Soxhlet apparatus for 6 hrs. with benzene, then for a further 6 hrs. with 95 per cent alcohol. The substance was then transferred to Gooch crucibles, containing as a filtering medium disks of mercerized cotton cloth, and washed with distilled water. Material so prepared is regarded as purified lignocellulose free from extractives.

PREPARATION OF CELLULOSE—Two grams of the wood, extracted as above, were subjected to the alternate action of chlorine and hot 3 per cent sodium sulfite solution in accordance with the procedure of Sieber and Walter.¹ The moistened material was placed in a Gooch crucible and the gas drawn through it by suction for 4 periods of 20, 15, 15, and 10 min. After each chlorination the residue was treated with half saturated sulfurous acid, washed with hot water, digested in 3 per cent sodium sulfite solution for 45 min., filtered off, and washed thoroughly with hot water. After the final treatment the residue was dried for 16 hrs. at 100° C.

PREPARATION OF LIGNIN—Two grams of the wood were extracted as before, partly dried at 60° C. and placed in a 750 cc. Erlenmeyer flask. 20 cc. of 72 per cent sulfuric acid were added and the flask rotated so as to bring all parts of the sawdust in contact with the acid. It was then allowed to stand 3.5 hrs. Fifty cc. of cold water were added, followed by 500 cc. of hot water, the mixture filtered on a Gooch crucible containing a cloth filtering disk, washed and dried for 16 hrs. at 100° C.

ANALYTICAL METHODS FOR DETERMINING THE CONSTANTS

FURFURAL-YIELDING GROUPS—These were determined by distillation of the material with 12 per cent hydrochloric acid (sp. gr. 1.06) and precipitation of the furfural in the distillate with phloroglucine, according to the method described by Schorger for pentosans.² No correction was made for methyl furfural, the total precipitate being calculated to furfural.

¹ *Papier-Fabr.*, 11, 1179; *Chem. Abs.*, 8 (1914), 1202.

² *THIS JOURNAL*, 9 (1917), 558.

¹ *Am. Chem. J.*, 16 (1894), 218; Cross and Bevan, "Cellulose," p. 181.

² *Monatsh.*, 11 (1890), 260; "Cellulose," p. 188.

³ "Cellulose," p. 191.

⁴ Cross and Bevan, "Researches on Cellulose," 3, 103.

⁵ *THIS JOURNAL*, 9 (1917), 560.

⁶ Palmer, *Ibid.*, 7 (1915), 633; Schorger, *Ibid.*, 9 (1917), 556.

⁷ Tollens, *J. Soc. Chem. Ind.*, 26 (1907), 987.

⁸ Benedikt and Bamberger, *Loc. cit.*

⁹ *Ber.*, 43, 1526.

ACETIC-YIELDING GROUPS—The material was hydrolyzed by heating for 3 hrs. under a water-cooled reflux condenser with 100 cc. of 2.5 per cent sulfuric acid. The mixture was quickly filtered into a 750 cc. flask to avoid the introduction of carbon dioxide and washed with CO₂-free water to a total volume of about 200 cc. The acetic acid in the solution was then distilled off and determined by means of the following apparatus:

The 750 cc. flask was placed in a paraffin bath and closed by a rubber stopper containing a glass stopcocked funnel tube and a delivery tube. The funnel tube served to introduce CO₂-free water during the distillation and was protected by a soda lime tube attached to its open end by means of a rubber stopper. The delivery tube bent downward and terminated in a vertical condenser. This in turn was connected by a rubber stopper to a suction filter flask which served as a receiver for the distillate.

Suction was applied to the receiver and the paraffin bath raised to and maintained at 85° C. Higher temperatures were avoided as tending to the formation of formic acid from the hexose carbohydrates present. When most of the liquid had distilled over from the flask, 100 cc. of CO₂-free water were introduced and distilled over, after which the procedure was repeated with a second 100 cc. The total distillate was then removed and titrated with standard sodium hydroxide solution using phenolphthalein as indicator. The results, representing "total volatile acids," were expressed as acetic acid.

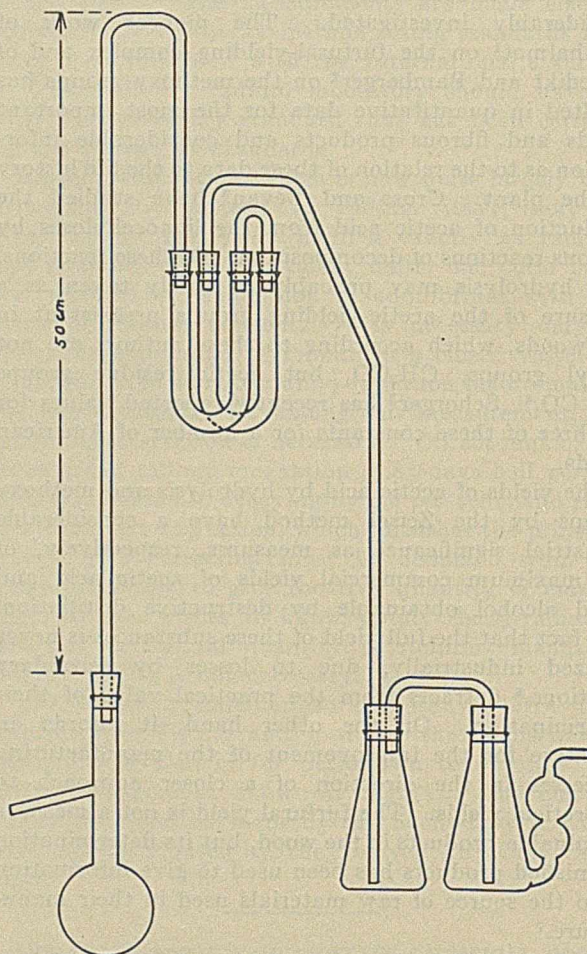
To obtain the actual acetic acid it was necessary to correct for the formic acid invariably present. A few cc. of silver nitrate solution were added to the solution after titration and the mixture left upon the steam bath for several hours. The silver formate was thereby decomposed, yielding an amount of metallic silver corresponding to the formic acid present. The precipitated silver was filtered off, washed, dissolved in dilute nitric acid, precipitated as silver chloride, filtered off, washed, ignited, and weighed. From the weight of silver chloride so obtained the amount of acetic acid equivalent to the formic acid was calculated by multiplying by the conversion factor 0.4. This result, deducted from the total acidity as found above, gave the actual acetic acid present.

METHOXY GROUPS—Methoxy groups were determined by the Zeisel method.¹ The apparatus and procedure here described have been found to give consistent results.

The decomposition flask was a small distillation flask (of about 130 cc. capacity) having its side tube connected with a carbon dioxide generator. In the mouth of the flask was placed a perforated cork carrying a delivery tube. This delivery tube ran vertically for 50 cm. and so acted to some extent as a reflux condenser and fractionating column. It then made two right-angled bends and, descending, was connected at its lower extremity with two U-tubes in series. The first of these contained distilled water and a few milligrams of phosphorus, the second distilled water only.

¹ *Monatsh.*, 6 (1885), 989; Meyer-Tingle, "Determination of Radicals in Carbon Compounds," John Wiley & Sons, Inc., p. 38.

The U-tubes were placed in a beaker of water arranged to be heated by a Bunsen burner. From the second U-tube, a delivery tube ran to the absorption apparatus which consisted of an Erlenmeyer flask and a Fresenius nitrogen bulb. The general arrangement and dimensions of the apparatus are shown in the accompanying figure. The paraffin and water baths and the carbon dioxide generator are omitted.



In the first and second absorption flasks were placed 35 and 15 cc., respectively, of freshly filtered alcoholic silver nitrate solution. (The solution was prepared by dissolving 10 g. of solid silver nitrate in 25 cc. of water and adding 225 cc. of 95 per cent alcohol.) 0.3 g. of material was placed in the decomposition flask and 15 cc. of hydriodic acid (1.70 sp. gr.) were added. The mixture was heated to 130° C. on a paraffin bath and the temperature maintained at 130° to 140° C., while a slow stream of carbon dioxide was passed through the apparatus. The beaker of water surrounding the U-tubes was kept at a temperature of 50° to 60° C. throughout the process. The operation was continued until the precipitated silver iodide in the absorption apparatus settled out, leaving a clear supernatant liquid, indicating the completion of the reaction.

The apparatus was then disconnected and the contents of the absorption flasks rinsed into a 600 cc. beaker. Water sufficient to make about 500 cc. was added and the whole evaporated on the steam bath to a volume of 150 to 200 cc. in order to expel the

alcohol. A few drops of nitric acid were added, the solution again diluted to about 500 cc. and allowed to stand on the steam bath for about one-half hour for the silver iodide to settle. The precipitate was then collected in a tared asbestos Gooch crucible, washed, dried at 130° C. for 2 hrs., and weighed. CH₃O was calculated from the weight of silver iodide by multiplying by the factor 0.132.

By the above methods the furfural yield, acetic yield on hydrolysis, and methoxy yield were determined upon the raw wood, extracted wood, cellulose, and lignin. The results are shown in the table.

DISTRIBUTION OF GROUPINGS IN REDWOOD
Expressed in Percentages of Air-Dried Wood (11.62 Per cent Moisture)

	Furfural Yield	Total Volatile Acids by Hydrolysis	Formic Acid Expressed as Acetic	Actual Acetic Acid by Difference	Methoxy Group
Raw Wood.....	5.73	0.94	0.16	0.78	5.46
	5.95	1.03	0.16	0.87	5.74
AVERAGE.....	5.84			0.83	5.60
Extracted Wood.....	5.77	0.75	0.09	0.66	5.33
	5.65	1.03	0.06	0.97	5.35
AVERAGE.....	5.71			0.82	5.34
Cellulose.....	2.36	0.75	0.21	0.54	0.35
	2.69	0.66	0.10	0.56	0.35
AVERAGE.....	2.52			0.55	0.35
Lignin.....	0.24	0.09	0.09	5.49
	0.23	0.09	0.09	5.78
AVERAGE.....	0.24			0.09	5.63

Determinations were also made of the furfural yield of the filtrates and washings from the cellulose determination. The combined solutions from four determinations or 8 g. of wood were evaporated to somewhat less than 500 cc., placed in a 500 cc. volumetric flask, and diluted to the mark. 125 cc. aliquots corresponding to 2 g. of original wood were placed in the distilling flasks and mixed with 30 cc. of concentrated hydrochloric acid. The determination was then carried out as usual. Three determinations gave 2.30, 2.08, and 2.08 per cent of furfural in the chlorination liquors, the results being referred to the weight of air-dried wood.

Determinations of the methoxy group were made upon portions of lignin that had been hydrolyzed by boiling with 2.5 per cent sulfuric acid for 3 hrs. after being separated by the usual treatment with 72 per cent sulfuric acid. The methoxy content based on the original wood was 4.08 and 3.88 per cent. There is accordingly an average loss of 1.65 per cent of methoxy during the hydrolysis. Separate experiments have shown that the weight of lignin after the treatment is about 2 per cent less than when prepared in the usual manner. From the above results it is evident that the loss in weight of lignin is due chiefly to the splitting off of the methoxy group.

DISCUSSION

The foregoing results indicate that none of the three radicals investigated is to be regarded as existing entirely independent of the proximate constituents cellulose and lignin. Apparently they are so related to these substances that, when lignocellulose is attacked by hydrolytic reactions, they tend to remain at least partially joined to the cellulose or the lignin or to both.

No method is known whereby lignocellulose may be separated so as to yield both cellulose and lignin unchanged, but in every case one of them is obtained as a residue while the other is converted into alteration products and removed. It is therefore unsafe to draw

the conclusion that radicals found with either cellulose or lignin, or both, exist similarly associated with them in the original lignocellulose complex. The radical is rather to be regarded as occupying an intermediate position between these chief proximate constituents, and remaining with the residue according to the extent that it resists the treatment employed. The same portion that remains with the cellulose under the conditions of one type of decomposition may remain with the lignin under another set of conditions.

A comparison of the data for raw and extracted wood shows that none of the three constants examined is materially affected by treatment with non-hydrolyzing solvents.

About half of the furfural-yielding complex remains in the cellulose residue. Most of the remaining portion is found in the chlorination liquors. As was pointed out in a previous paper¹ this last portion is probably due to xylan or other true pentosan existing as such in the wood, while the non-hydrolyzable furfural-yielding material contained in the cellulose is probably oxycellulose. The amount of furfural-yielding material remaining with the lignin is almost negligible.

The amount of acetic acid present in redwood is small and its determination is rendered uncertain by the presence of formic acid either preëxisting or produced by the action of the hydrolyzing acid upon hexoses. No general conclusions can therefore be drawn in regard to its distribution but it appears to be associated chiefly with the cellulose constituent. With the lignin there is very little. The combined amount found with the lignin and cellulose is slightly less than the total amount in the wood, indicating that part of the acetic-yielding groups separates independently. In view of the small quantities involved the results are not conclusive. In the case of redwood, and probably also in the case of other coniferous woods, which yield only small amounts of acetic acid on hydrolysis, it would appear that acetic acid should not be considered as an independent proximate group but as a radical already largely accounted for in the cellulose and lignin residues. This conclusion probably does not apply to hardwoods, where the acetic yield is much higher.

The methoxy groups are found quantitatively in the lignin constituent, directly confirming the frequent assertion that methoxy is a purely lignin characteristic. This occurs only when lignin is separated by the method described and subsequent hydrolysis avoided. If this is not done there is considerable splitting off of the methoxy groups.

These facts have an important bearing upon the proximate analysis of wood. A summative analysis of a wood may be made by drying, extracting with benzene and alcohol, and determining the cellulose and lignin in the manner previously described. It appears from the foregoing results that, when this is done, the cellulose and lignin constituents account for all of the methoxy group, nearly all of the acetic-yielding groups, and about half of the furfural-yielding groups. Except for the furfural due to hydrolyzable pentosans found

¹ Dore, THIS JOURNAL, 12 (1920), 264.

in the chlorination washings, these constants are not to be regarded as proximate constituents but rather as radicals connected with the constituents cellulose and lignin. With the exception noted, these constants may be ignored in the summative analysis of coniferous woods.

SUMMARY

I—A study is made of the distribution of the groups contained in redwood which yield furfural, acetic acid, and methoxy, with the object of learning their relation to the constituents cellulose and lignin.

II—About half of the furfural-yielding groups are associated with the cellulose, but only a small amount with the lignin. The portion present as true pentosan is hydrolyzed and removed during chlorination.

III—The acetic-yielding groups are partly associated with the cellulose, much less so with the lignin. A small amount appears to be detached from either. The results are not conclusive in view of the small amount present in redwood and the analytical difficulties. In the case of coniferous woods acetic acid probably need not be considered as a proximate group.

IV—The methoxy groups are wholly associated with the lignin. They may be partially split off from it by acid hydrolysis.

V—In the summative analysis of coniferous woods, all of the acetic-yielding and methoxy groups and part of the furfural-yielding groups may be disregarded as already accounted for in the cellulose and lignin. The furfural-yielding substances contained in the chlorination washings and representing hydrolyzed pentosans should be estimated.

THE PROXIMATE ANALYSIS OF CONIFEROUS WOODS

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INTRODUCTORY

A method has been recently proposed by the author for the summative proximate analysis of woods.^{1*} Later investigations have shown the advisability of certain modifications of the original scheme which are considered in detail in this paper.

The proposed system for the analysis of wood effected a separation of the wood substance into the following fractions: loss on drying, benzene extract, alcohol extract, water-soluble, soluble in one per cent sodium hydroxide, cellulose, and lignin. Full information as to the composition of these fractions was lacking, but it was believed that the wood constituents were separated into logical groups based upon chemical similarity and economic value.

The scheme was found to account for nearly all of the material of coniferous woods in that the sum of all constituents varied from 96 to 97 per cent for the three conifers examined. With the hardwoods the method was unsuccessful, as 10 to 17 per cent of the constituents remained unaccounted for. The available data seem to indicate that the chemistry of the hardwoods is an entirely separate problem. The woods of the broad-leaved trees have accordingly been omitted

from consideration in this investigation and the conclusions are applicable to the conifers only.

DEFECTS IN PROPOSED SCHEME

(1) ITS FAILURE TO GIVE CORRECT RESULTS FOR CELLULOSE AND LIGNIN—It was noted in the original paper¹ that when digestion in one per cent sodium hydroxide was used less cellulose and lignin were obtained than when this treatment was omitted. In a subsequent paper² it was shown that the diminished yield of cellulose was due to partial destruction of true cellulose instead of to greater purity of the product. Accordingly the results for cellulose are incorrect. No direct data are available for lignin, but it has been shown in the preceding paper that lignin readily loses a portion of its methoxy groups when it undergoes acid hydrolysis. Possibly the same effect is produced by alkalis.

(2) THE VAGUE SIGNIFICANCE OF THE WATER-SOLUBLE AND ALKALI-SOLUBLE FRACTIONS—In the absence of detailed studies of the substances contained in these portions, it was believed that they consisted of substances corresponding approximately to König's "proto" and "hemi" forms of cellulose and lignin.³ It is apparent from subsequent studies,² however, that they contain also degradation or hydrolytic products of true cellulose and true lignin. The numerical expression of these fractions has therefore no value as an analytical statement of wood constituents.

(3) THE FAILURE TO EXPRESS THE HEMICELLULOSES—Schorger's studies of mannan⁴ and galactan⁵ have shown that these carbohydrates are found in all conifers, sometimes in considerable quantity. Xylan is well known as a constituent of woods. It is therefore evident that a complete analysis of a wood should take these hemicelluloses into consideration. In the proposed scheme the water- and alkali-soluble portions only partially accounted for these bodies together with other substances.

(4) ORGANIC RADICALS—The original scheme took no account of organic radicals present—the so-called "wood constants." The preceding paper has shown that, with the exception of the furfural-yielding substances soluble in the chlorination washings, these radicals may be disregarded as proximate constituents of coniferous woods.⁶ The original scheme is defective, however, in that it ignores this soluble furfural-yielding substance.

The remedies for the above defects are quite evident. The cellulose and lignin determinations should be made reliable by omission of the alkaline hydrolysis. The water- and alkali-soluble fractions would disappear, leaving the hemicelluloses entirely unaccounted for. It would then be necessary to give the hemicelluloses a separate place in the scheme, and methods would be required for their estimation either collectively or individually. The soluble furfural-yielding substance (probably the pentosan, xylan) would be included in the hemicelluloses.

The present investigation is an attempt to revise the original scheme in accordance with the above suggestions. Methods have been applied for the determination of the hemicelluloses. The revised procedure has then been checked by making these and

* Numbers refer to Reference, page 479.

the other determinations required for a complete summative analysis on the three coniferous woods formerly employed.

EXPERIMENTAL

The woods examined were redwood (*Sequoia sempervirens*), yellow pine (*Pinus ponderosa*), and sugar pine (*Pinus lambertiana*). The original material was in the form of slabs. In the case of the redwood it represented mostly sapwood with a small amount of the outer portion of heartwood. In the case of the pines it represented sapwood only.

The samples were in the form of fine sawdust obtained by making a number of cross-sectional cuts of the slabs and screening the resultant material through a sieve having 50 meshes to the linear inch. As considerable coarse material was rejected, the samples cannot be regarded as strictly representative of the original material. That, however, is of no importance to the purpose of this study which was to determine the applicability of the analytical methods to the woods employed.

ANALYTICAL METHODS

LOSS ON DRYING—Two grams of the wood were dried at 100° C. to constant weight.

BENZENE EXTRACT—Two grams of material dried as above were placed in an alundum thimble and extracted in a Soxhlet apparatus for 6 hrs. with benzene. The solvent was then evaporated and the residual extract dried for 1 hr. at 100° C., and weighed.

ALCOHOL EXTRACT—The residue from the above treatment was extracted for 6 hrs. with 95 per cent alcohol. The solvent was evaporated and the residue dried and weighed as before.

CELLULOSE—Two grams of material after extraction with benzene and alcohol as above were transferred to a Gooch crucible containing a cloth filtering disk and washed with distilled water. The cellulose was determined by the Sieber and Walter method as described in the preceding paper and elsewhere.⁷ The residues were tested for lignin by digestion in 72 per cent sulfuric acid and when appreciable amounts were found, the lignin was filtered off and weighed, and the cellulose corrected for it.

LIGNIN—Two grams of material after extraction with benzene and alcohol were air-dried at low temperature (60° C.) and transferred to a 750 cc. flask. 20 cc. of 72 per cent sulfuric acid were added and the mixture allowed to stand at room temperature for 3.5 hrs. 50 cc. of cold water were added, then 500 cc. of hot water. The residue was filtered off on a tared Gooch crucible, washed thoroughly with hot water, dried, and weighed.

HEMICELLULOSES—The hemicelluloses are apparently contained quantitatively in the chlorination washings,² and it was thought at first that these solutions might be utilized for their determination. They were found to be unsatisfactory for this purpose except in the case of the soluble pentosans. The presence of lignin derivatives interferes seriously with either the collective estimation of the hemicelluloses by reducing sugar methods or the separate mannan and galactan determinations.

SOLUBLE PENTOSANS—The chlorination filtrates and washings from four cellulose determinations representing 8 g. of original material were evaporated to a little less than 500 cc., transferred to a 500 cc. volumetric flask and made up to volume. Aliquot parts of 125 cc. each were placed in the distilling flask of the pentosan apparatus, 30 cc. of concentrated hydrochloric acid added, and the furfural distilled off in the usual manner. The furfural was precipitated as phloroglucide, and the precipitate filtered off, washed, dried, and weighed. The results were calculated to xylan by means of Krober's tables.

MANNAN—The method used was that described by Schorger.⁸ Ten grams of the wood were hydrolyzed by boiling for 3.5 hrs. with 150 cc. of hydrochloric acid (sp. gr. 1.025) in an Erlenmeyer flask under a reflux condenser. The mixture was then filtered and the residue rinsed into a beaker and digested with 100 cc. of water for a few minutes on the hot plate. The solution was poured through the filter and the residue again rinsed back into the beaker and the digestion repeated. This process was continued until about 500 cc. of filtrate were obtained. The filtrate was neutralized with sodium hydroxide and the solution made slightly acid with acetic acid, after which it was evaporated to 150 cc. It was filtered into a glass-stoppered Erlenmeyer flask, and a mixture of 10 cc. of phenylhydrazine and 20 cc. of water acidified with glacial acetic acid was added. The mannose hydrazone was in every case immediately precipitated. The whole was allowed to stand for 2 hrs. with frequent shaking and then filtered on a Gooch crucible fitted with a filtering disk of mercerized cotton cloth. It was washed several times with cold water and then with acetone, dried at 100° C. and weighed. The weight of residue was calculated to mannan by multiplying by the factor 0.6.

GALACTAN—Five grams of the material were placed in a 100 cc. beaker, and 60 cc. of nitric acid (sp. gr. 1.15) were added. The beaker was placed in a water bath and the liquid evaporated to about 20 cc., care being taken not to allow the temperature of the water bath to exceed 87° C. The mixture was then diluted to about 75 cc. with hot water, and filtered. The residual cellulose was washed until the filtrate came through practically colorless. A total volume of about 250 cc. was generally thus obtained. The filtrate and washings were evaporated on the water bath at 87° C. to a volume of about 10 cc. The residue was set aside for several days to allow the mucic acid to separate out. Large crystals (possibly oxalic acid) always formed at first, then a day or two later fine flakes of mucic acid separated out. At this point the mixture was stirred vigorously to facilitate the precipitation. About 24 hrs. after the mucic acid appeared the mixture was diluted with 20 cc. of cold water. The larger crystals redissolved leaving the mucic acid unaffected. After a further 24 hrs. standing, the mucic acid was filtered off on a tared asbestos Gooch crucible and washed with about 50 cc. of water, 60 cc. of alcohol, and several times with ether. It was then dried at 100° C. for 3 hrs. and weighed. Galactan was calculated by multiplying the weight of residue by the factor 1.2.

RESULTS

By the foregoing methods the following results were obtained for the three coniferous woods examined. The figures are in every case averages of two or more determinations. In the first three columns are given the results on air-dry material as found by analysis; in the second three, the same results are recalculated to the oven-dry basis.

SAMPLE NO.....	ANALYSES OF WOODS (Results in Percentages)					
	—AIR-DRY BASIS—			—OVEN-DRY BASIS—		
	1	2	3	1	2	3
	Red-wood	Yellow Pine	Sugar Pine	Red-wood	Yellow Pine	Sugar Pine
Loss on drying at 100° C.	11.62	8.98	9.84	0.34	2.22	2.84
Benzene extract.....	0.30	2.02	2.56	4.39	1.49	1.90
Alcohol extract.....	3.88	1.36	1.71	54.89	57.72	59.18
Cellulose.....	48.51	52.54	53.36	34.50	29.47	29.50
Lignin.....	30.49	26.82	26.60	3.67	3.49	1.86
Soluble pentosans (as xylan).....	3.24	3.18	1.68	3.21	6.37	6.63
Mannan.....	2.84	5.80	5.98	0.50	0.78	0.50
Galactan.....	0.44	0.71	0.45	101.32	101.41	102.18
TOTAL.....	101.32	101.41	102.18	101.50	101.54	102.41

DISCUSSION OF METHODS

CELLULOSE—It has been previously shown that the method here practiced (omission of preliminary hydrolysis) gives the maximum yield of cellulose free from hemicelluloses and lignin. The process therefore conforms to the requirements of an accurate cellulose method and the product to the proposed definition of a true cellulose derived from lignified material.²

LIGNIN—The term "lignin" has not yet been defined with sufficient precision to permit a comparison of the product with exact specifications. Lignin is generally understood to denote the non-cellulose portion of the wood tissue proper and this conception implies its freedom from extractives, hemicelluloses, and cellulose. The most accurate method for lignin is therefore the one that yields the greatest amount of the substance with the highest purity according to the above standards.

It is believed that there is sufficient evidence to justify the claim that the method used conforms to these requirements. Both acid and alkaline hydrolysis result in diminished yield of product and it has been shown that this is due (at least in the former case) to a partial splitting off of the methoxy groups which are characteristic portions of the lignin complex. Accordingly, only by the complete omission of hydrolysis is the full yield of lignin obtainable.

Inasmuch as the lignin disappears completely on being submitted to the alternate action of chlorine gas and sodium sulfite solution, it is free from cellulose which would otherwise remain as a residue. On hydrolyzing with 2.5 per cent sulfuric acid no reducing sugars were obtained, so the material is free from hemicelluloses. The preliminary treatment with benzene and alcohol precludes the possibility of the presence of extractives. The residue may be therefore considered "pure" in accordance with the requirements outlined.

SOLUBLE PENTOSANS—Dilute alkaline solutions have been recommended for extracting xylan or wood gum from the hard woods.⁹ It has been asserted, however, that the wood gum of coniferous woods is much less readily soluble in alkaline solvents than that from the wood of the broad-leaved trees.¹⁰ For this reason it

appeared to be desirable to use the chlorination liquors for the determination of soluble pentosans.

MANNAN—The method gave results that were in good agreement with each other and consistent with those given by Schorger¹¹ for the corresponding woods. Small portions of the residue, recrystallized by alcohol, gave a melting point of 188° C.

GALACTAN—In their paper on the galactan of the western larch, Schorger and Smith¹² report only traces of soluble galactans in the coniferous woods examined. In their experiments 100 g. of the material were digested several times in hot water and the resulting solution evaporated nearly to dryness, and treated with nitric acid in the usual manner. Other investigators,¹³ however, have reported the presence of galactan in sulfite waste liquors, indicating that galactans are contained in some of the paper-making woods. It therefore seemed probable that galactans are present in not readily soluble form. Accordingly no attempt was made in the present investigation to extract the galactan as such from the wood by means of solvents. Instead the procedure of attacking the wood directly with nitric acid was decided upon as the best means of giving the full galactan content of the wood.

The evaporation of the wood substance with nitric acid results in the production of a large amount of insoluble oxycellulose which interferes with the separation of the mucic acid. This difficulty was overcome by the procedure devised by Miyake¹⁴ in which the mixture, after evaporation, is diluted, and the solution containing the mucic acid is filtered off from the insoluble matter and again evaporated.

The author has pointed out the uncertainty of yield of mucic acid by the usual process when only small amounts of galactan are present.¹⁵ Schorger and Smith¹⁶ have shown that much better results are obtained if the temperature is not allowed at any time to exceed 87° C., instead of maintaining the bath at 94° to 96° C., as prescribed in the official method for galactan.¹⁷ The use of the lower temperature has accordingly been adopted.

Considerable difficulty was encountered at first in obtaining a crystallization of the mucic acid after evaporation. This appeared to be due chiefly to the interfering action of oxalic acid or other substances contained in the solution. When the latter was evaporated to the usual volume of 20 cc., no separation of mucic acid occurred even after several days' standing. By evaporating to a smaller bulk clear crystals separated out after one or two days. These crystals on standing increased in size and a day or two later the characteristic mucic acid flakes were precipitated. After the last precipitation was judged to be complete, the larger crystals were easily removed by diluting the solution to the usual volume of 30 cc. The amounts of residue were always too small to permit of a melting-point determination but the appearance of the precipitate was such as to leave little doubt that it was actually mucic acid.

Considering the nature and difficulties of the method, the agreement of results may be considered satisfactory. Redwood gave 0.40 and 0.47 per cent; yellow

pine gave 0.96, 0.66, 0.43, and 0.80 per cent; sugar pine gave 0.55 and 0.35 per cent of galactan. It is believed that the described procedure gave approximately correct results. A critical study of the whole galactan method is still much needed, however.

DISCUSSION OF RESULTS

It is believed that analyses of coniferous woods made by the methods described account for all important constituents of those woods. The sum is in every case slightly over 100 per cent but not more so than might be expected from the character of the methods employed. A variation of several tenths of a per cent between duplicate determinations is usual. The cellulose and lignin are probably always slightly over-estimated on account of the practical impossibility of getting complete contact between all particles of the material and the attacking reagents. This difficulty could be partly overcome by better mechanical condition of the wood.

The possibility of overlapping of values due to certain constituents being included in more than one determination has received careful consideration throughout these investigations. It is believed that such double estimations have been largely avoided. Most of the proximate groups can be shown to be free from any of the constituents contained in the other groups. It has already been pointed out that this is true in the case of cellulose and lignin. Mannan and galactan determinations depend upon reactions too specific to admit of the inclusion of other constituents.

By improving the mechanical condition of the sample in respect to fineness, better results should be obtained in all the determinations. Some of the determinations, particularly that of galactan, could probably be made more accurate and reliable. These changes would no doubt give a considerably better summation and a consequently more satisfactory accounting of the constituents of the coniferous woods.

SUMMARY

1—An improved procedure is described for the summative analysis of coniferous woods. Methods are given for the estimation of the following constituents: loss on drying, benzene extract, alcohol extract, cellulose, lignin, soluble pentosans, mannan, and galactan.

2—By the omission of preliminary hydrolysis, more reliable results are obtained in the cellulose and lignin determinations than by methods previously used.

3—Soluble pentosans are determined in the chlorination liquors, mannan and galactan on separate portions of the original material. Improvements in the galactan determination are described.

4—Complete analyses of redwood, yellow pine, and sugar pine are carried out by these methods. A summation of slightly over 100 per cent is obtained in every case.

5—The results indicate that overlapping of the proximate groups, *i. e.*, partial inclusion of any constituent in more than one group, has been largely avoided.

6—Analyses by the proposed scheme probably

account for all important constituents of coniferous woods.

REFERENCES

- 1—THIS JOURNAL, 11 (1919), 556.
- 2—*Ibid.*, 12 (1920), 264.
- 3—*Z. Nahr. Genussm.*, 28 (1914), 177.
- 4—THIS JOURNAL, 9 (1917), 748.
- 5—Schorger and Smith, *Ibid.*, 8 (1916), 494.
- 6—Dore, preceding paper, p. 472.
- 7—Sieber and Walter, *Chem. Abs.*, 8 (1914), 1202; Johnsen and Hovey, *Paper*, [23] 21 (1918), 36; *J. Soc. Chem. Ind.*, 37 (1918), 1321.
- 8—THIS JOURNAL, 9 (1917), 748.
- 9—Cross and Bevan, "Cellulose," p. 187.
- 10—Klason, Cross and Bevan, "Researches on Cellulose," 3, 105; Schorger, THIS JOURNAL, 9 (1917), 562.
- 11—*Ibid.*, 9 (1917), 749.
- 12—*Ibid.*, 8 (1916), 499.
- 13—Lindsey and Tollens, *Ann.*, 267, 341; Klason, *Loc. cit.*; Hagglund, *Biochem. Z.*, 70 (1915), 416.
- 14—*Chem. Abs.*, 6 (1912), 2958; 8 (1914), 2007.
- 15—THIS JOURNAL, 7 (1915), 721.
- 16—*Ibid.*, 8 (1916), 498 (footnote).
- 17—U. S. Department of Agriculture, *Bulletin* 107, 55.

THE DETECTION OF ARSENIC IN SULFUR

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A certain sample of sulfur was reported to contain arsenic. It had been tested by a method first devised by Schaeppi¹ and later accepted by many authorities.² We found that the sulfur contained no trace of arsenic, but that when it was tested by Schaeppi's method, a decided qualitative test for arsenic was obtained. On further investigation it was found that this method contains a fundamental error which makes it useless as a test for small quantities of arsenic.

The method as described by Schaeppi consists essentially in first digesting the sulfur with dilute nitric acid for the purpose of removing calcium chloride, sulfate, and sulfide. The sulfur washed free from acid is now treated with dilute ammonia solution for 15 min. at 70° to 80° C. Schaeppi assumes that any arsenic present in the sulfur must exist as sulfide, mainly arsenious sulfide, and will be dissolved by the ammonia. The arsenic is then estimated indirectly through determination of the amount of sulfide by precipitation with silver nitrate in neutral solution.

"For very rough estimations, it is sufficient to treat 10 g. of finely ground sulfur with nitric acid, to extract with ammonia, and to add silver nitrate. From the intensity of the color or the quantity of the precipitate of silver sulfide, it may be judged whether the sulfur is approximately free from arsenic or is strongly contaminated."³

Schaeppi endeavored to test the accuracy of his method by dissolving known amounts of pure arsenious sulfide in ammonia solution, carefully neutralizing with nitric acid, and titrating with decinormal silver nitrate using chromate as indicator. His results showed fairly good agreement between the quantities of arsenic taken and those found. However, his results would have

¹ *Chem. Ind.*, 4 (1881), 409.

² Crooks, "Select Methods of Chemical Analysis," 4th ed., 416; Lunge, "Technical Methods of Chemical Analysis," 1 (1908), 267; Lunge, "The Technical Chemist's Handbook," 1916, 107.

³ Crooks, *Loc. cit.*

been conclusive only if the arsenious sulfide had been added to the sulfur and the mixture tested by his method. Any other sulfide if present in the ammonia solution would also be precipitated by the silver nitrate. As a matter of fact, we found that sulfur continuously reacts with dilute ammonia at 70° to 80° C. to form sulfides, so that even arsenic-free sulfur will give a qualitative test for arsenic by this method.

EXPERIMENTAL

The sulfur used in these experiments was obtained from Texas deposits¹ and was found to be absolutely free from arsenic when tested by the Gutzeit method as described later in this paper.

Ten grams of a representative sample, powdered to pass a 160-mesh, were digested with 25 cc. of hot water, to which 0.4 cc. concentrated nitric acid was added. It was then washed free from acid and sulfides and digested with 25 cc. water and 0.5 cc. ammonium hydroxide (sp. gr. 0.90) at a temperature of 70° to 80° C. for 15 min.

The filtrate was somewhat tinged with a bright yellow color and gave a heavy dark brown precipitate on the addition of silver nitrate. The sulfur was now washed clean from alkali and sulfides and the extraction repeated with ammonia solution as before. Again the filtrate gave a heavy precipitate on the addition of silver nitrate.

The extraction was repeated a third and a fourth time, washing the sulfur clean from alkali and sulfur in each case and the same results were obtained. The precipitate with silver nitrate in the last extract seemed as heavy as that in the first.

A sample of flowers of sulfur was tested in the same way, the extraction with dilute ammonium hydroxide being repeated three times. The results were the same as in the case of the crude sulfur.

To determine whether the ammonium hydroxide solution really contained arsenic or not, the first extract from 10 g. of crude sulfur was acidified with hydrochloric acid, a crystal of potassium chlorate added, and the solution boiled until all the chlorate was decomposed. This solution was now tested by Gutzeit's method for arsenic and none was detected.

Further, 8 g. of crude sulfur were extracted with dilute ammonia at 70° to 80° C. The extract was acidified with strong nitric acid and evaporated to dryness on a water bath. A few cc. of concentrated nitric acid were added and was again evaporated to dryness. The residue was dissolved in water and tested by Gutzeit's method for arsenic with negative results. In a check test, 0.0004 g. of arsenic under similar conditions in the Gutzeit apparatus gave a well-marked arsenic stain.

These experiments indicate that sulfur reacts with dilute ammonium hydroxide at 70° to 80° C. to form ammonium sulfides. This is substantiated by the well-known fact that sulfur readily dissolves in hot solutions of the strong alkalies to form polysulfides.

¹ From the recently developed deposits of the Texas Gulf Sulfur Co., at Gulf, Matagorda Co., Texas. The three great deposits of sulfur which are being worked commercially in the country (Union Sulfur Co., Freeport Sulphur Co., Texas Gulf Sulfur Co.) are all free from arsenic.

A sample of arsenic-free sulfur would, therefore, continue indefinitely to give a test for arsenic by Schaeppi's method.

TESTING THE SULFUR FOR ARSENIC BY GUTZEIT'S METHOD

To prepare the solution for the test, Scott¹ recommends the following procedure:

Ten grams of the material (brimstone) are treated with 30 cc. of carbon tetrachloride mixture (3 parts CCl_4 + 2 parts Br) and after standing for 10 min. 25 cc. strong nitric acid are added in small portions (a watch glass covering the beaker during the intervals of addition). The mixture is taken to dryness on a steam bath, water is added, and the evaporation repeated. Arsenic is now determined on the residue by the Gutzeit method for arsenic.

In our experience 25 cc. of strong nitric acid were insufficient to oxidize the sulfur bromide even when added drop by drop from a pipette; 10 to 15 cc. more were needed, depending on the length of time the solution was allowed to stand on the water bath. It was found advisable to warm the sulfur bromide slightly before adding the nitric acid from a dropping pipette. The final evaporations were carried out directly over a flame, instead of on a water bath, until sulfur trioxide fumes appeared.

By the action of the bromine and nitric acid the sulfur is completely oxidized to sulfuric acid. The carbon tetrachloride is added to moderate the violence of the reaction and to prevent the precipitation of sulfur. Any excess of these reagents is expelled by the final evaporations. The acid solution remaining in the flask was now tested for arsenic by Gutzeit's method, details of which may be found in any standard textbook on chemical analyses.

The crude sulfur used in the work on Schaeppi's method was tested in this way with the following results:

1—Ten g. of sulfur gave no test for arsenic. At the completion of the test 1 cc. of 0.001 *N* As_2O_3 solution (0.0004 g. As) was added to the solution remaining in the Gutzeit apparatus and a strong test for arsenic was obtained in a few minutes. This proved that no substance was present which would prevent the arsenic test from showing, had arsenic been present.

2—Ten g. of crude sulfur to which 0.0004 g. As were added gave a distinct test for arsenic, quite comparable with the stain obtained when a solution containing an equal quantity of arsenic was tested directly in the Gutzeit apparatus.

SUMMARY

I—It has been found that sulfur reacts somewhat with dilute ammonium hydroxide at 70° to 80° C. to form sulfides.

II—Schaeppi's method of testing for arsenic in sulfur has been shown to contain a fundamental error due to this interaction of sulfur with dilute ammonium hydroxide.

III—The method of testing for arsenic in sulfur which consists in oxidizing the sulfur with bromine and nitric acid and testing the residue by Gutzeit's method for arsenic has been proved to be reliable.

¹ *Loc. cit.*, 415.

BUTYL ALCOHOL AS A MEDIUM FOR THE DETERMINATION OF SAPONIFICATION NUMBERS¹

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In a recent article² it was shown that normal butyl alcohol is an excellent medium in which to conduct the quantitative saponification of esters. This suggested its use in the determination of the saponification numbers of fixed oils, fats, and waxes. The method of Koettstorfer³ has, with little modification, been generally adopted.⁴ That difficulties are met with is shown by the number of modifications that have been proposed. The chief trouble has been in getting some of the fats and waxes into perfect solution in the ethyl alcoholic potash.

Smithan⁵ adds 20 cc. of ether, and Henriques⁶ uses petroleum ether to aid solution in his cold saponification method.

Lewkowitsch⁷ advises heating fats which are difficult to saponify under pressure, using copper bottles with screw caps.

Kossel and Obremueller treat the sample with absolute alcohol in which metallic sodium has been dissolved.

Wool fat has given much trouble. Lewkowitsch says "wool wax must be boiled with an excess of 0.5 *N* alcoholic potash for a least 24 hrs." Fabrian⁸ has completely saponified it in an open capsule, on the water bath, with 2 *N* caustic soda, repeatedly boiling it almost to dryness to get rid of the water, and taking up the residue each time with strong alcohol.

Winkler⁹ recommended the use of propyl alcohol, since it is a better solvent for fats and has a higher boiling point. He tried twelve oils, fats, and waxes, and obtained good results.

Normal butyl alcohol, boiling at a higher temperature than either ethyl or propyl alcohol, makes possible the use of higher temperatures for the saponification. Its longer hydrocarbon chain increases its solvent power for fats, waxes, etc. From the work of Anderson and Pierce¹⁰ it appears that at 25° the saponification velocities of an acetate in methyl, ethyl, and isoamyl alcohols are in the ratio 1 : 7 : 15. From this saponification might be expected to proceed much more rapidly in normal butyl than in ethyl alcohol.

The simple method of Koettstorfer gives excellent results for the more common oils and fats and, by various modifications, special cases can be taken care of, but it is desirable to have a simple method which will be applicable to a wide variety of materials with-

out extra precautions. The official directions read "boil for 30 min. or until the fat is completely saponified." The operator is thus required to observe and to decide when the "fat is completely saponified," since much longer time is required for some fats than for others.

The plan of the present study was to put a number of fats, oils, and waxes through identically the same treatment, making the manipulation as simple as possible in the hope of finding a simple routine method with wide application. For comparison, runs were made using ethyl alcoholic soda in the same volume as when butyl alcohol was used. It is the common practice to use considerably larger proportions of ethyl alcohol than this, so as to effect complete solution, and thereby excellent results are obtained. The comparison is simply between butyl and ethyl alcohol under these special conditions.

MATERIALS

The ethyl alcohol for making the standard solution was purified by treating with caustic potash and silver nitrate, and distilling. The normal butyl alcohol was used as obtained in the market. In each case, the alcoholic alkali was prepared by adding metallic sodium to the alcohol. The oils, fats, and waxes were the best commercial samples obtainable, rancid and discolored specimens being rejected. Those which appeared to contain water were dried at 110°. They were selected according to the groups given in Allen's "Commercial Organic Analysis."

EXPERIMENTAL PROCEDURE

One-gram samples were taken. The fats were weighed into 100-cc. round bottom Jena flasks, five of which, making a series, were fastened to a rack so that they could be lowered and raised simultaneously. They fitted into five openings in a steam bath and were provided with 3 ft. air condensers.

The oils were weighed into the saponification flasks from small Erlenmeyer flasks fitted with dropping pipettes. The waxes were cut into small cubes, while the butter, woolfat, and lanolin were weighed in small glass capsules.

To the material in the flask were added 10 cc. of the standard 0.5 *N* alcoholic alkali. The flask was placed immediately on the steam bath and heated exactly one hour when it was removed, 50 cc. carbon dioxide-free water added, and the excess alkali titrated with 0.5 *N* hydrochloric acid. In case the unsaponified material was high melting the mixture was kept hot during the titration.

As the butyl alcohol was nearly anhydrous, 0.5 cc. of water was added to each flask containing this alcohol, half an hour after the heating had begun, to provide for the saponification of the butyl ester, the formation of which is the first thing that takes place.

RESULTS

The results are given below in tables, the figures in each line representing simultaneous experiments. "E" designates the series in ethyl alcohol and "B" those in normal butyl alcohol. Under "Difference" is placed the percentage by which the average of the ethyl al-

¹ Our thanks are due to Dr. G. S. Jamieson, in charge of Oil, Fat, and Wax Laboratory, U. S. Bureau of Chemistry, for several helpful criticisms. The experimental work was carried out at Washington and Jefferson College by A. M. Pardee and R. L. Hasche.

² THIS JOURNAL, 12 (1920), 129.

³ Z. anal. Chem., 18 (1879), 199.

⁴ Official and Provisional Methods of Analysis, U. S. Dept. of Agr., Bulletin 107 (1911), 137; THIS JOURNAL, 11 (1919), 1165.

⁵ Analyst, 18 (1893), 193.

⁶ Z. angew. Chem., 1895, 721.

⁷ "Chemical Technology of Oils, Fats and Waxes," 1 (1909), 85.

⁸ Z. angew. Chem., 1898, 268.

⁹ Ibid., 24 (1911), 636.

¹⁰ J. Phys. Chem., 22 (1918), 51.

cohol figures falls short of the butyl. Since it has been customary to give saponification numbers in terms of KOH, the results obtained with the sodium alcoholates have been calculated to this basis and are so put down in the tables.

TABLE I—SAPONIFICATION NUMBERS IN ETHYL AND BUTYL ALCOHOLS

		OILS						PER-CENT-AGE DIFFERENCE
		E	B	E	B	E	B	
Castor	E	180.0	180.6	180.7	180.2	179.9	180.3	
	B	179.1	181.5	181.9	182.0	181.5	181.2	0.5
Coconut	E	247.1	248.7	249.1	...	248.3	...	
	B	250.0	249.6	249.8	250.8	...	250.0	0.7
Cod-liver	E	179.7	178.7	179.5	178.7	178.9	179.1	
	B	191.0	190.3	190.7	190.2	190.4	190.5	6.0
Cottonseed	E	190.3	189.6	190.2	190.2	...	190.1	
	B	193.2	187.6	193.5	189.3	193.5	191.4	0.7
Croton	E	204.8	206.1	206.4	206.1	205.1	205.7	
	B	225.7	225.4	224.7	225.1	225.1	225.3	8.7
Linseed	E	186.3	187.6	189.0	185.5	180.0	185.6	
	B	198.0	198.8	197.4	199.4	198.9	198.5	6.5
Neat's-foot	E	112.3	109.8	109.5	111.8	114.1	111.5	
	B	132.5	133.5	130.7	132.1	...	131.7	15.3
Palm	E	198.2	196.9	196.7	197.0	...	197.2	
	B	204.1	206.5	205.8	204.3	204.4	205.2	3.9
Peach Kernel	E	189.9	190.6	189.1	190.6	...	190.0	
	B	191.1	190.9	189.4	191.8	189.7	190.6	0.3
Rape Seed	E	169.0	167.3	168.0	168.7	168.3	168.3	
	B	175.9	176.5	174.1	175.2	174.5	175.2	4.0
Sesame	E	191.4	190.1	190.1	188.8	...	190.5	
	B	191.4	191.7	190.5	191.8	191.6	191.4	0.4
Sperm	E	133.4	132.1	134.2	134.0	134.1	133.5	
	B	147.7	148.6	147.7	147.3	148.7	148.0	9.7
Sperm, adulterated	E	136.7	138.1	137.7	138.7	139.0	138.5	
	B	151.4	152.7	153.4	151.1	151.3	152.0	8.9
WAXES								
Beeswax, yellow	E	82.4	82.3	80.6	83.8	81.5	82.1	
	B	91.4	91.4	92.2	90.7	91.3	91.4	10.2
Beeswax, white	E	79.9	74.7	70.9	78.5	76.9	76.2	
	B	94.4	94.7	96.6	96.4	97.2	95.9	20.5
Spermaceti	E	114.9	116.2	114.3	115.2	
	B	126.0	125.2	126.3	125.3	126.2	125.8	8.5
FATS								
Butter	E	227.9	219.3	223.3	219.4	221.0	220.5	
	B	232.4	230.6	229.4	231.5	228.6	230.5	4.3
Japan Wax	E	218.0	214.4	215.6	216.8	219.0	216.7	
	B	225.3	228.9	226.9	225.2	...	226.6	4.1
Tallow	E	185.8	182.6	181.8	184.5	183.6	183.7	
	B	197.1	198.7	199.0	198.3	...	198.3	7.4
Wool Fat, Anhydrous	E	101.3	98.2	98.1	99.2	
	B	(120.0)	111.6	112.5	114.2	113.2	112.9	12.1
Lanolin	E	56.4	51.0	48.3	49.1	48.1	50.6	33.4
	E ¹	70.0	70.5	70.6	70.7	...	70.4	
	B	76.5	74.6	77.3	76.4	75.2	76.0	7.3
	E	84.7	77.7	84.3	84.5	...	83.8	
	B	95.6	95.6	95.4	99.3	97.9	96.8	13.3
	E	67.8	66.8	67.7	66.9	...	65.5	
	B	79.5	80.0	82.4	78.6	79.4	80.0	18.0

¹ With absolute alcohol and sodium, according to Allen's "Commercial Analysis," II, 495.

It appears from these runs that substantially the same values are obtained in ethyl and butyl alcohols for the more readily saponified oils and fats, though the values in butyl alcohol are always somewhat higher. For substances that are more difficult to saponify, considerably higher and more reliable values are obtained in butyl alcohol than in the same volume of ethyl alcohol. Doubtless the ethyl alcohol values would have come up to the higher figures had larger amounts of alcohol been employed, or had the heating been continued till complete saponification had been effected. The figures obtained in butyl alcohol agree well with the accepted values. Our sample of neat's-foot oil must have been adulterated. The low results with tallow in ethyl alcohol are surprising. They were repeated with a different sample of tallow which gave higher figures but those for the two alcohols were in the same ratio.

In order to get some idea of relative rates of saponification in the two solvents, the following experiments were run. The procedure was exactly as in the foregoing except that the flasks were removed at the intervals noted.

TABLE II—RELATIVE RATES OF SAPONIFICATION IN ETHYL AND BUTYL ALCOHOLS

Time, Min.	5	10	15	20	25	30	40	45	50	60
Cod-liver Oil:										
E	140.1	...	153.8	152.7	...	180.6	...	185.7
B	190.2	...	190.4	190.3	190.3
Castor Oil:										
E	171.1	173.3	178.0	177.3	177.7
B	147.5	166.0	177.5	...	180.4	180.5
Spermaceti:										
E	...	56.5	113.2	82.7	115.3	115.3
B	104.7	105.9	109.8	...	126.7	126.7

It appears from these results that the saponification of cod-liver oil is complete in 5 min. in normal butyl alcohol but requires considerably more time in ethyl. Castor oil is saponified rapidly in both, while spermaceti is acted on less rapidly in both but much more rapidly in butyl than in ethyl.

CONCLUSION

Normal butyl alcohol is found to be superior to ethyl as a medium for quantitative saponification, as by its use satisfactory results are more readily obtained and special precautions are less often required.

A NEW METHOD FOR THE DETERMINATION OF SULFUR IN OILS¹

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It is well known that crude petroleum from whatever source contains more or less sulfur in combination. The lubricating oils and other products obtained from the crude oil must also contain sulfur unless special methods of refining are applied. In addition to their original sulfur content, the oils may be contaminated by sulfonic acids² which are formed when the oils are treated with concentrated sulfuric acid during refining, and are not afterwards completely washed out.

Little is known about the relation between the sulfur content and certain of the properties of oils. The chief reason for this may be the inconvenience, if not also the inaccuracy, of nearly every method that has been suggested for the determination of this element. Specifications have little to say about it. A limiting value may be set for fuel oils on account of the corrosive effect of the products of combustion. Transformer oils must contain a minimum amount. The test with polished copper, which is frequently called for, may be regarded as a demand for a low content of sulfur, be-

¹ Published by permission of the Director of the Bureau of Standards.

² Although spoken of as sulfonic acids, it is certain that they are, at least in part, the acid esters of sulfuric acid and, therefore, analogous to ethyl sulfuric acid. The true sulfonic acids cannot be hydrolyzed by digestion with mineral acids, but this action takes place readily with the esters, and the sulfuric acid is recovered. When true sulfonic acids, e. g., benzenesulfonic acid, is broken down by regulated fusion with caustic alkali, the sulfite of the metal is formed. A heavy red oil (from F. R. Baxter, Vacuum Oil Co.), known to contain "sulfonic acids," gave no reaction for sulfuric acid when digested with water, but a slight reaction when so treated with 1:1 hydrochloric acid. When sealed up with concentrated hydrochloric acid and heated inside a steam bath, it gave a strong reaction for sulfuric acid.

cause it is commonly believed that this element will always blacken the metal,¹ and will, therefore, have a bad effect upon bearings.

It is believed by some that "sulfur compounds" are the cause of the tendency of an oil to form emulsions with water.² This may be true if the compounds are "sulfonic acids."

It is well established that when an oil is heated in the air, the amount of asphaltic matter formed is increased by the presence of sulfur or sulfur dioxide.³ It might also be expected that the "gumming" of an oil at ordinary temperatures will be affected by the presence of sulfur, unless it is due only to the loss of the more volatile constituents.

Sulfur is known to have a beneficial effect in the flotation of certain ores. This is referred to by E. C. Bingham⁴ in discussing the reason for using it in cutting oils.

For about eleven years the author has worked, as opportunity offered, on a variety of methods for the determination of sulfur in petroleum oils. Not only the published methods but others that seemed promising were tried. Because the subject first came up in connection with transformer oils, the aim was to find an accurate and convenient method which could be used with at least 2 or 3 g. of oil. With the bomb calorimeter, one gram is all that can be burned completely. No more can be used when the oil is burned in a bottle of oxygen, according to the Graefe method, and even much less is almost certain to cause the bursting of Carius tubes. Other methods are too slow, inconvenient, dangerous, or unreliable. Even combustion in the bomb is believed to give slightly low results. Analyses made by the method described in this paper appear to prove this.

Some years ago it was found that concordant results for sulfur could be obtained by digesting the oil in a Kjeldahl flask with concentrated nitric acid. From time to time a little crystallized potassium permanganate was added. This quickly decomposed, the acid became filled with suspended manganese peroxide, and the oxidation was materially hastened. Permanganate was chosen rather than chlorate because of this formation of a solid peroxide, instead of gaseous oxides. Chlorate was used about this time by Gilpin and Schneeberger⁵ in the analysis of fractions from California crudes. They obtained results that checked those by the Sauer method. Although digestion with nitric acid usually gives concordant figures, some of our recent work shows that a considerable part of the sulfur may be lost.

In 1911 the author worked out a procedure for the

¹ This is not true. A topped Mexican crude, which contained 4.05 per cent of sulfur, barely tarnished a strip of copper which was heated in it for 2 hrs. at 290° to 295° C. At room temperature there was barely any action in several days.

² C. W. Stratford, private communication. A few experiments in collaboration with G. A. Kramer indicate that a high content of sulfur does not always cause an oil to emulsify readily.

³ Holde and Eickmann, *Mitt. kgl. Materialprüfungsamt*, 1907, 145; *Z. angew. Chem.*, 20 (1907), 1263, 1923; Waters, Bureau of Standards, *Tech. Paper* 4 (1911), 9; *THIS JOURNAL*, 3 (1911), 814; Southcombe, *J. Soc. Chem. Ind.*, 30 (1911), 261.

⁴ Unpublished paper.

⁵ *Am. Chem. J.*, 50 (1913) 66.

determination of total sulfur in rubber goods.¹ The rubber is first decomposed to a considerable extent by digesting it with concentrated nitric acid saturated with bromine. It is then completely burned by heating with a mixture of soda and saltpeter. The decomposition of oils by means of nitric acid and permanganate requires many hours, and the disappearance of the oily drops does not indicate with certainty the end of the reaction. For this reason it was decided to try the method used for rubber, though with little hope of success. A topped Mexican crude, in which the sulfur content was found by nitric acid digestion to be 2.21, 2.17, and 2.27 per cent, was used. Three analyses by the method for rubber gave 4.06, 4.03, and 4.05 per cent of sulfur. Similar data from another oil showed that more than half of the sulfur was lost by the nitric acid method. This loss was traced to the formation of a nitro-compound which contained considerable sulfur. This nitro-compound was thrown down when the digestion flask was washed out after all of the oil had, apparently, been decomposed. It was soluble to some extent in water and dilute nitric acid, could be extracted but incompletely by ether, and was slightly volatile with water vapor when heated on the steam bath. Although a clear yellow oil, it formed a very dark brown salt when dissolved in a solution of soda. The success of the new method for the determination of sulfur appears to depend upon the formation of nitro-compounds which react with soda, and are therefore not driven off during the subsequent fusion with soda and saltpeter. An oil which yielded 1.83 per cent of sulfur showed only 1.37 per cent when the preliminary treatment with nitric acid and bromine was omitted.

DESCRIPTION OF METHOD

Weigh from 0.5 to 1.5 g. of oil into a 100 cc. porcelain crucible, add 5 cc. of concentrated nitric acid saturated with bromine, and at once cover with a watch glass. If there is danger of losing sulfur, as with a vulcanized oil or a rather volatile one, each sample should be treated with acid as soon as it is weighed out, and not left until the series of weighings is finished. It is best to weigh such oils in a covered crucible. Place on the steam bath, but not in direct contact with the steam, and digest for 30 min. or more, with an occasional gentle swirling of the crucible. If the mixture does not threaten to foam up to the cover, the crucible may now be heated directly by the steam for 2 or 3 hrs.

Remove the crucible from the bath and, when its contents are cold, raise the watch glass slightly and add cautiously in small portions, 10 or 12 g. of anhydrous sodium carbonate. This is conveniently measured in a 15 cc. crucible, rather than weighed. The first portions of the soda usually remain on top of the oily layer, so that the crucible must be tilted or swirled slightly to start the reaction. When about half of the soda has been added it is safe to rinse the watch glass with 1 or 2 cc. of water and remove it permanently. Add the rest of the soda and thoroughly mix the contents of the crucible with a stout glass rod (3 mm.

¹ Waters and Tuttle, Bureau of Standards, *Scientific Paper* 174; *THIS JOURNAL*, 3 (1911), 734.

thick and about 12 cm. long). The resulting pasty mass is spread around on the sides and one-half to two-thirds of the way to the top. If the right amount of water is used the mass will adhere to the crucible. The rod is not removed. Dry on the steam bath, or in an air bath at about 100° C., place the crucible in an inclined position on a wire triangle and start the ignition over a low flame. When more than a gram of oil has been taken, there is a tendency for it to burn too briskly, but this is controllable by judicious use of the stirring rod, which scrapes the burning portion away from the rest. When part of the mass is burned white, a fresh portion is worked into it, and so on until all of the organic matter is destroyed. It is necessary to hold the edge of the crucible with tongs. Towards the last half of the operation the flame should be increased somewhat, but it is never necessary to heat the crucible to redness. With care a crucible can be used for at least ten or twelve analyses.

According to the amount of oil taken the mixture of sodium salts resulting from the combustion varies from pasty to granular. After cooling, place the crucible on its side in a 400 cc. beaker with about 150 cc. of water. Cover and digest on the steam bath. When the salts have dissolved, remove and rinse out the crucible. Filter the solution to remove specks of carbon chips from the glass rod, etc. Acidify with concentrated hydrochloric acid, using Congo red paper as indicator, and add about 2 cc. of acid in excess. The reaction proceeds more safely if nearly the required amount of acid is placed in a covered beaker and the solution filtered directly into it. The total amount of carbonate and nitrite to be neutralized will depend upon how much oil was taken. Heat the acidified solution on the steam bath, add 10 cc. of a 10 per cent solution of barium chloride and digest until the sulfate is all thrown down. If time permits, the barium sulfate should be allowed to stand over night before it is filtered off, ignited, and weighed.

A blank should be run with the reagents. In doing this an ignition is to be made just as if oil were present, because the flame may contain some sulfur. The reagents used in making the analyses upon which this method is based gave only traces of barium sulfate when mixed in the usual amounts without ignition; including this, the correction amounted to 0.03 per cent of sulfur, based on one gram of oil. Most of this barium sulfate came down over night.

In this work, as in the routine analysis of rubber goods in this laboratory, a petroleum ether flame is used for the ignition. Gasoline can be used, but it is necessary to set the vessel which contains it in a beaker of warm water. A simple gasoline gas generator was described in 1913.¹ In place of the special form there described, a gas-washing bottle, containing a layer of glass beads to break up the bubbles, or even a wide-mouthed bottle with a good cork and two glass tubes, one of which extends to the bottom, can be used. A current of air blown through the petroleum ether carries with it enough vapor to supply a small burner of the Meker type. When first lighted the flame is

apt to be smoky, but this ceases when the ether becomes chilled by evaporation. The smokiness is no great drawback because at the start of the ignition the flame should be so low as not to touch the crucible.

It is a merit of the method that a considerable number of determinations can be started and completed at the same time. Individual attention is needed chiefly during the ignition, which takes from 15 to 25 min. Rubber is so completely broken down that much less time is required. With oil there is so little change that most of it is driven off in clouds of vapor, and it seems that only a small part of the sulfur could possibly be held back. But the concordance of the results is strong, presumptive evidence that there is very little or no loss. In all, twelve oils were analyzed and found to contain from 0.29 to 4.05 per cent of sulfur. In three cases duplicate analyses differed by 0.06, 0.05, and 0.04 per cent; twice the differences were 0.03 and 0.01 per cent; and twice exact agreement was obtained. In three instances the discrepancy was 0.02 per cent.

In some recent work done in cooperation with the sub-committee on sulfur, Committee D-2, American Society for Testing Materials, three oils were analyzed in nine laboratories, including that of the author. The results varied widely and, in every case but one, the method just described gave the highest figures. The other members of the sub-committee used various methods: the bomb calorimeter, Sauer's method, Conradson's (burning in a lamp), and burning in oxygen at atmospheric pressure (Graefe's method). Table I gives the highest and lowest figures¹ obtained in the other laboratories, by all the methods. The results of all the analyses by the new procedure are also given. These have been corrected, by small amounts, for the sulfur in the reagents, in the flame and in the air. The correction calculated for one gram of oil was 0.03 per cent.

TABLE I—COMPARISON OF RESULTS OBTAINED BY DIFFERENT METHODS

OIL	PERCENTAGES OF SULFUR						Av.
	By Other Laboratories		By New Method				
Mexican (topped crude with same vol. mineral seal).....	1.27 to 1.98			1.83	1.82		
Vulcanized 300 oil.....	0.80 to 1.22	1.31	1.37	1.32	1.32	1.33	Av. 1.33
Heavy red oil.....	0.39 to 0.53	0.56	0.55	0.59	0.58		Av. 0.57

The vulcanized oil contained much hydrogen sulfide and probably also free sulfur. The heavy red oil contained "sulfonic acids."

In view of all the above figures it seems fair to claim that the present method is sufficiently accurate for the analysis of oils no more volatile than the "300" oils. It has not yet been applied to kerosene or gasoline. The analysis of six fuel oils, both light and heavy, which were obtained for this purpose from the Bureau of Mines Experiment Station, Pittsburgh, Pa., has been begun. The few results so far obtained seem to show that the new method gives somewhat higher sulfur than the bomb calorimeter.

It is admitted that the conditions under which the barium sulfate is precipitated are far from ideal, because of the occlusion of sodium salts. Of these the

¹ Taken from the report of the Chairman, F. R. Baxter, Vacuum Oil Co., and used by his permission.

sulfate tends to low results, while the nitrate has the opposite effect. Only by chance will these errors cancel one another.¹ A few determinations made with double the usual amounts of reagents seem to show that the net error due to occlusion is small.

The present paper is a preliminary one, and it is intended to publish a more detailed discussion of the whole subject later on.

SUMMARY

The desirability of investigating the possible effect of sulfur compounds on the properties of lubricating oils is pointed out.

The disadvantages of the methods now in general use for the determination of sulfur are indicated.

A new procedure, which gives results at least as accurate as those by the bomb calorimeter, is described.

DETERMINATION OF MINUTE QUANTITIES OF OIL IN SULFUR

By L. S. Bushnell and H. Smith Clark

FREEMPORT SULPHUR COMPANY, FREEMPORT, TEXAS

Received November 25, 1919

The presence of very small amounts of oil in sulfur is objected to by manufacturers of certain sulfur products. The sulfur may contain carbonaceous residues from burned oil, and the method here described is not intended to include these.

The estimation of oil by simple extraction as ordinarily made with a volatile solvent—such as sulfuric ether or petroleum ether—will not suffice in the case of sulfur, because, while the sulfur is only slightly soluble, there is usually such a small amount of oil present (from 0.001 per cent to 0.32 per cent, or more) that the extract contains considerably more sulfur than oil. In this method extraction is made as usual, and the solvent purified from sulfur by boiling it in a Wiley continuous extractor in which has been placed a roll of copper foil.

APPARATUS

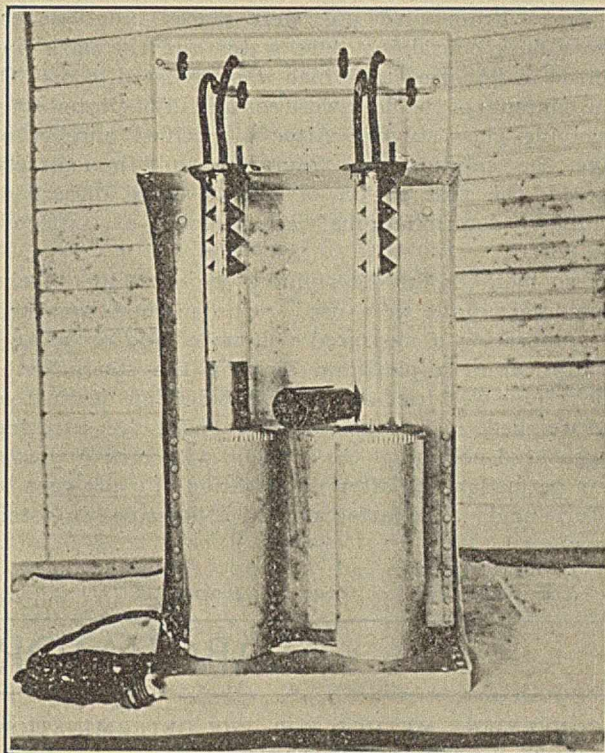
A sheet of copper foil 4 in. \times 15 in. is rolled up in such fashion that no two points of its surface come in contact. It is desirable that the copper sheet be rolled uniformly so that there will be a space of about three-sixteenths inch between any one turn of the roll and the next turn. The sheet may be cut so that at one corner a tab three-eighths inch square projects. A hole is punched in this tab so that the coil may be lifted from the Wiley tube with a wire hook. The end with this tab should be rolled first, and the tab project from one end of the coil.

A 100 cc. volumetric flask; a thin glass beaker, tall form, without lip, 300 cc. capacity; and a Wiley continuous extractor, without crucible or thimble, complete the required apparatus.

PROCEDURE

Fifty grams of the pulverized sulfur (or a smaller quantity, if the oil content is known to be high) are weighed and transferred to the 100 cc. flask. On this are poured about 50 cc. of redistilled petroleum ether;

the flask is thoroughly shaken every half hour¹ for several hours, and allowed to settle. The petroleum ether is decanted through a filter into a Wiley tube, and a second smaller quantity of the solvent is added to the flask, shaken, settled, and filtered, as before. The washing is continued in this way until the sulfur and the filter paper are free of oil, and the Wiley tube contains about 175 cc. of solvent, or enough to fill the tube above the top of the copper roll or coil.



The copper coil is cleaned with dilute nitric acid, washed with water, then with alcohol, dried with ether, and placed in the Wiley tube. The petroleum ether is then boiled until all of the sulfur has been deposited on the copper as copper sulfide.

The solvent is transferred to the tall beaker, filtering if necessary to eliminate the copper sulfide, which will doubtless partially flake off. The coil, the Wiley tube, and the filter are washed with small quantities of petroleum ether, the combined filtrate and washings gently evaporated to dryness at very low heat, and the residue weighed.

NOTE ON THE SOLUBILITY OF BENZIDINE SULFATE IN WATER

By C. S. Bisson and A. W. Christie

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Received September 9, 1919

One of the methods for the determination of sulfates which has found considerable application consists in precipitation as benzidine sulfate, which may be quantitatively titrated with standard alkali or potassium permanganate. In most of the articles describing such methods, the benzidine sulfate is said to be insoluble in water. A review of the literature

¹ Allen and Johnston, *J. Am. Chem. Soc.*, **32** (1910), 588; Johnston and Adams, *Ibid.*, **33** (1911), 829.

failed to reveal any definite data on its solubility except a statement by Muller¹ which gives it as from 0.01 to 0.03 per cent at 25° C. We have determined its solubility at different temperatures by agitating an excess of benzidine sulfate with pure water in a constant temperature thermostat.

The benzidine sulfate was prepared by adding an excess of dilute sulfuric acid to an aqueous solution of Kahlbaum's benzidine. The precipitated sulfate was filtered and washed with distilled water till free of acid. From 3 to 5 g. were placed in a 500 cc. Pyrex flask containing pure water and the flask immersed in a thermostat which was regulated to 0.5° C. The contents of the flask were continually agitated by a revolving glass stirrer. After a specified number of days the solution was removed and immediately filtered, the first 20 cc. of filtrate being discarded. Solubility determinations were made at 0°, 25°, 50°, and 80° C.

Two methods for determining the solubility of the benzidine sulfate were used. One method consisted in evaporating a measured volume of 100 or 200 cc. to dryness in a platinum dish on the steam bath. The residue was dried at 100° C., cooled in a desiccator, and weighed. The other method consisted in titrating a measured volume of the solution with 0.05 *N* potassium permanganate after the addition of 5 per cent of sulfuric acid. The latter method is described in detail elsewhere.²

¹ *Ber.*, **35** (1902), 1587.

² A. W. Christie and C. S. Bisson, *THIS JOURNAL*, **12** (1920), 171.

The solubility of benzidine sulfate in water as determined by the above methods is given in the accompanying table.

SOLUBILITY OF BENZIDINE SULFATE IN WATER AT VARIOUS TEMPERATURES

Temperature ° C.	Determination	
	By Weight G. per Liter	By Titration G. per Liter
0	0.049	0.048
25	0.098	0.096
50	0.141	0.149
80	0.290	0.252

The two methods gave concordant results at all the temperatures except 80° C. The solutions for the determination of solubility at 0° and 25° C. were stirred in the thermostat for four days. Those at 50° and 80° C. remained in the thermostat only 24 hrs., since it was found that after several days the solutions became badly discolored, indicating that the benzidine sulfate had undergone decomposition. The solution obtained at 80° C. was slightly discolored even after 24 hrs., as was also the residue obtained on evaporation. This is probably due to the partial oxidation of the benzidine at the higher temperature, as evidenced by the low figure for solubility obtained by titration. The residue obtained from the 50° solution showed only a very slight discoloration and the results may be considered to give the approximate solubility at this temperature.

From a consideration of these results it is evident that in washing the benzidine sulfate in a quantitative determination of sulfate, the minimum amount of cold water should be used.

LABORATORY AND PLANT

A CONVENIENT METHOD FOR THE DETERMINATION OF WATER IN PETROLEUM AND OTHER ORGANIC EMULSIONS¹

By E. W. Dean and D. D. Stark

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Received January 15, 1920

INTRODUCTION

One of the problems frequently confronting the petroleum chemist is the determination of emulsified water. Numerous methods have been devised, several of which have been described in a previous publication² issued by the Bureau of Mines. Allen and Jacobs discuss in that paper the advantages and limitations of various types of procedure and recommend as most desirable the scheme of distilling in a special electrically heated still. This type of still involves heating the entire outer surface of a 250 cc. distilling flask and its use permits the "breaking" of troublesome froth that is usually formed when a viscous petroleum emulsion is heated. Certain difficulties in the construction and operation of this still have led to discontinuing its use in favor of the more common method of diluting the emulsion before distillation with a solvent immiscible with water. The present

paper reports the work of the Bureau in modifying this latter method to obviate certain of its admitted disadvantages.

DISTILLATION IN THE PRESENCE OF AN IMMISCIBLE SOLVENT

There are various conditions under which it is desirable to determine water in petroleum emulsions and no one method is best in all cases. The method of gravity separation¹ is a convenient one and is satisfactorily reliable for emulsions that are not too viscous or that contain water in not too fine a state of subdivision. For the estimation of extremely small percentages of water, in such products as transformer oils, special methods are necessary which are too delicate for ordinary needs. The distillation method is without doubt most generally applicable, highly reliable, and sufficiently accurate for all usual requirements. The use of a solvent not miscible with water offers several advantages, the most important of which is prevention of frothing. Its principle disadvantages concern details of operation, and the modified procedure described in the present paper has been found to obviate most of these difficulties.

This method of determining water has been used in a

¹ Published by the permission of the Director of the U. S. Bureau of Mines.

² See Bibliography, p. 490.

¹ A common procedure is to place a measured volume of oil in a suitable graduated tube, dilute with naphtha, and spin in a centrifuge. The water is driven to the bottom of the graduated tube and its volume can be read directly.

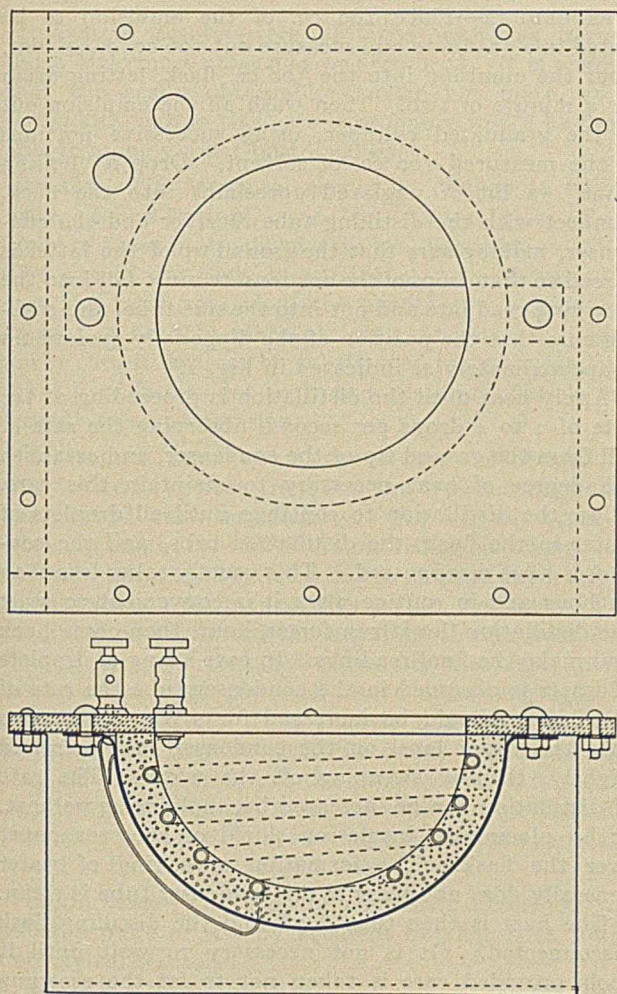


FIG. 1

variety of modifications and discussed in a number of published articles.¹ The following description covers the procedure commonly employed in petroleum laboratories.

A measured volume of the oil to be tested is placed in a glass or metal distilling flask and a suitable volume of solvent, generally petroleum naphtha of proper distilling range or a mixture of xylene and benzene, is added. The flask is connected with a condenser which delivers into some sort of a graduated receiver, heat is applied, and the distillation continued until the volume of water no longer increases. This volume is then read and the percentages of water in the original emulsion calculated. The principal objections and difficulties are:

1—Drops of water sometimes stick in the cool end of the condenser and are not easy to remove quantitatively.

2—The distillation requires a certain amount of attention and the distillate must be examined from time to time to determine when all the water has been driven over.

3—When emulsions of high-water content are tested it is sometimes necessary to add several successive portions of solvent to the contents of the distilling flask before all the water is driven over.

¹ See Bibliography, p. 490.

4—If a solvent such as xylene-benzene mixture is used there is an error due to the fact that an appreciable amount of water is dissolved in the upper layer of the distillate. Results must be corrected for this error or, preferably, the solvent must be saturated with water before adding it to the emulsion.

BUREAU OF MINES METHOD

The experimental work incident to the development of the method described in the present paper brought out a number of facts, which, though interesting, do not seem of sufficient general importance to warrant a detailed discussion. The results of the work are, therefore, reported in the form of a definite recommendation concerning apparatus and procedure. The specific recommendations are discussed in a subsequent portion of the paper. The following description covers the method as employed by the Bureau for all ordinary types of petroleum emulsions.

APPARATUS—The apparatus consists of a source of heat, a 500 cc. short-necked distilling flask, a special device combining the functions of distilling tube and receiver (hereafter referred to as "distilling tube receiver") and a condenser.

The source of heat may be an alcohol or gas burner, but the authors prefer a special type of electric heater, similar to that described by one of us in a previous article.¹ The form of the heater is, however, slightly different, as indicated in Fig. 1. The resistance elements now in service (with 110 volt alternating current) are composed of 23 ft. of No. 27 gauge nickel-chromium wire, having an approximate resistance of 72 ohms and an approximate capacity of 165 watts. These heaters are used without regulating rheostats, the capacity indicated having been found just sufficient to maintain the desired rate of distillation.

The 500 cc. round-bottomed distilling flask is a readily obtainable stock article. The authors have found Pyrex flasks particularly satisfactory.

The "distilling tube receiver" (Fig. 2) is usually made² by attaching a proper side tube to a stock type of tapered graduated test-tube. It is the one essential part of the apparatus that must be specially constructed and the authors have been assured that the work is simple and that the article ought not to be expensive when put on the market.

The condenser, readily obtainable from chemical

¹ E. W. Dean, "A Convenient Electric Heater for Use in the Analytical Distillation of Gasoline," *THIS JOURNAL*, 10 (1918), 823.

² The authors hereby acknowledge their indebtedness for aid in the design of this device to F. E. Donath, glass blower in the Pittsburgh Experiment Station of the Bureau of Mines.

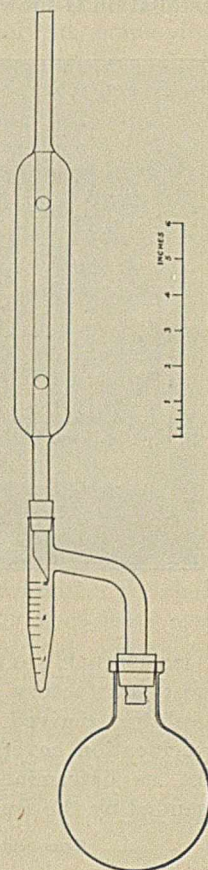


FIG. 2

supply houses, is about 15 in. long over all, and has an 8-in. sealed-on water jacket. The outside diameter of the inner tube is about $1\frac{15}{32}$ inch. Before being put into service the tip should be ground diagonally, as indicated in the figure.

The general method of assembling the glass parts of the apparatus is indicated diagrammatically in Fig. 2. Cork stoppers are used for the two junctions and if a good grade of cork is employed it is unnecessary to lute these joints.

For its own work the Bureau of Mines has developed a 4-unit electrically heated installation which has proven exceedingly satisfactory. A photograph of this installation is shown in Fig. 3.¹

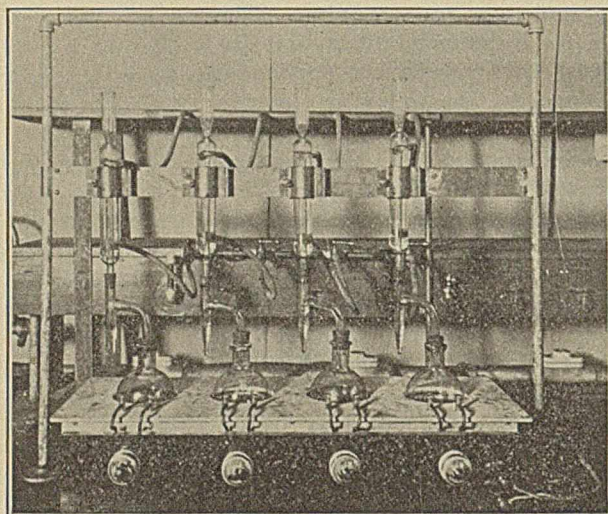


Fig. 3

IMMISCIBLE SOLVENT—The authors have found the most satisfactory solvent to be a so-called cleaner's naphtha derived from petroleum. The following specification covers the distillation range of this product, as determined by the standard method recommended by the Bureau:²

5 per cent mark not below 90° C. (194° F.)
nor above 100° C. (212° F.)
90 per cent mark not above 195° C. (383° F.)

A mixture of 80 per cent of commercial xylene and 20 per cent of commercial (so-called 90 per cent) benzene has also been found satisfactory. This mixture should not be saturated with water before using.³

¹ The authors acknowledge their indebtedness to W. A. Jacobs, Assistant Chemical Engineer, and Harry Buss, Chief Instrument Maker, of the Pittsburgh Experiment Station of the Bureau, for the design and construction of this highly satisfactory installation.

² E. W. Dean, "Motor Gasoline, Properties, Laboratory Methods of Testing, and Practical Specifications," Bureau of Mines, *Technical Paper* 214 (1919), 18.

³ The commercial hydrocarbon products derived from the distillation of coal appear to dissolve water in slight but appreciable degree. With the original distillation method the full amount of solvent was distilled and collected in the receiver in contact with the water layer. A convenient means of compensating for the amount of water necessarily dissolved was to saturate the solvent before using. With the modified method herein described the volume of solvent in contact with water in the receiving graduate is of the order of 10 cc. or less and the error introduced through solution of water by this quantity is negligible. If the solvent is saturated before using, the amount of water collected in the receiver is always in excess of that actually contained by the oil tested.

PROCEDURE—Place 100 cc. of the emulsion to be tested in a graduate and measure out 100 cc. of solvent. Pour the emulsion into the 500 cc. flask, letting drain for a minute or two. Then wash all the emulsion out of the graduated cylinder, using successive portions of the measured 100 cc. of solvent. Drop a "boiling stone" (a bit of unglazed porcelain) into the flask. Connect with the distilling tube receiver and the condenser, making sure that the ground tip of the latter is placed so that drops of the condensate must fall into the receiving graduate and not into the side tube, and making sure that the position of the diagonally ground tip is approximately as indicated in Fig. 2.¹

Apply heat until the distillation is proceeding at the rate of 2 to 4 drops per second, observing the rate of fall from the ground tip of the condenser, and establish the degree of heat necessary to maintain this rate. Allow the distillation to continue until all droplets of water in the flask, the distillation tube, and the condenser have disappeared. This usually takes less than an hour and in routine work it is convenient to start the distillation, let it run for an hour, then come back and make the final readings. In case a ring of droplets of water is observed in the condenser tube the rate of distillation should be increased for a few minutes so that the vapor level on the condenser tube is raised slightly, thereby sweeping off the water. The rate of distillation can be increased by applying more heat, or by placing an insulating shield of asbestos paper over the flask. The formation of a ring of water generally does not occur if the condenser tube is clean.

The heat is then turned off and the distilling flask disconnected. (It is not necessary to wait until it cools provided care is taken not to let the escaping vapor ignite.) A drop of water sometimes hangs on the ground tip of the condenser; this, however, practically always falls off when the graduated receiver is detached from the condenser.

The volume of water collected in the graduated receiver is then read, taking care that the tube is held in a vertical position. The receiver is emptied and dried, and is ready for another determination. The flask is usually set aside to cool and a previously cleaned and dried one used for the next determination.

DISCUSSION OF DETAILS OF THE ABOVE PROCEDURE

SOURCE OF HEAT—The authors have tried electric heaters of the design described, and gas burners.² The former are by far the more satisfactory but are not essential to the accuracy of the method. Perfectly

¹ When determinations are made in an atmosphere of high humidity it is advisable to take certain precautions designed to prevent any extraneous water from entering the receiving graduate. A protecting shield such as a circle of blotting paper may be slipped over the condenser tube and held in place between the cooling jacket and the cork stopper connecting the condenser and distilling tube receiver. This obviates any possibility of water dripping from the condenser and passing through a cork that is not absolutely tight. Another more essential precaution for operations in an atmosphere of high humidity is to place a loose plug of absorbent cotton in the upper (open) end of the condenser tube, thereby preventing free access of air which might be deprived of moisture with a resulting increase in the volume of water apparently separated from the oil. A properly trapped calcium chloride tube can, of course, be used instead of a plug of cotton, but the latter is regarded as adequate protection for all cases except those requiring extreme accuracy.

² If a flame heater is employed it seems advisable to have the flask bottom protected by a piece of wire gauze.

satisfactory results were obtained with gas burners but a little more attention was required. It is possible that the easily obtainable electric hot plate would serve, although in this case it probably would be desirable to employ a flat-bottomed flask.

FLASK—The authors generally employ a short-neck, round-bottom, 500 cc. Pyrex glass flask. None of these particular details are, however, essential. The size of the flask, in particular, may be varied in case the total volume of the charge of emulsion and solvent is notably more or less than 200 cc. Metal flasks can, if desired, be substituted for glass, and the scheme of using flat-bottomed distilling vessels has already received mention.

DISTILLING TUBE RECEIVER—Numerous designs for this device have been tried and the type recommended has proved most satisfactory. For certain types of work it can undoubtedly be modified to advantage and occasion has already arisen in the Bureau laboratories to substitute a plain tube with a glass stopcock. This modification permitted the handling of emulsions containing large percentages of water, the water being drawn off through the cock into a measuring cylinder from time to time. It is entirely possible that other modifications may be desirable under conditions that may be encountered and the present device is recommended as simply the most convenient type that has been used with the types of emulsions that are generally tested in the Bureau laboratories.

CONDENSER—The type of condenser described was selected more or less by accident but has been found entirely satisfactory.

SOLVENT—A considerable variety of solvents have been tried and the two recommended above were found most satisfactory. The desirable properties are those which insure "smoothness" in distillation and a vapor temperature slightly above the boiling point of water. The solvent must have a certain percentage of constituents boiling below 100° C., but this percentage must not be too great. For instance, ordinary motor gasoline, with about 20 per cent boiling below 100° C., was notably less satisfactory than the special naphtha that is described in a previous connection. The final boiling point of the solvent apparently should not be too high as was evidenced by the fact that unsatisfactory results were obtained by the use of a solvent made by blending kerosene with ordinary motor gasoline in sufficient proportion to reduce the portion distilling below 100° C. to between 5 per cent and 10 per cent.

The solvent must be immiscible with water but should, under ordinary conditions at least, be miscible with the oil or other non-aqueous constituent of the emulsion. The application of this latter requirement is illustrated later when the results of tests on coal tar are discussed. The degree of solubility of water in the solvent is of some importance, though such errors are minimized by the fact that the quantity of non-aqueous distillate in the receiving tube is never more than 10 or 12 cc. Water is almost perfectly insoluble in the petroleum naphtha. It is probable, however, that many

laboratories will find it more convenient to prepare the xylene-benzene mixture than to obtain a special grade of petroleum naphtha. Water is slightly soluble in coal-tar distillates and in addition there is sometimes a tendency for the distillate to be cloudy, due to the holding back of minute particles of water. Permitting this cloud to settle has never, however, added as much as 0.01 cc. to the volume of the water layer.

PROCEDURE—The use of a charge of 100 cc. of emulsion and 100 cc. of solvent has proved satisfactory for oils containing between 0.5 and 10.0 per cent of water. If an accurate determination of water content of less than 0.5 per cent is desired it is necessary to use 200 cc. or more of both emulsion and solvent, employing, of course, a correspondingly larger distilling flask. If the emulsion to be tested contains more than 10 per cent of water the quantity taken should be some suitable fraction of 100 cc. According to the authors' experience it has not seemed necessary to change either the quantity of solvent or the size of the distilling flask when this method of handling emulsions of high water content is used.

The procedure recommended for measuring out the emulsion is a convenient one but not essential to the method. In some cases a pipette can probably be used to advantage. The use of a boiling stone has proved necessary and a number of determinations have been spoiled through failure to remember this detail. If it is omitted boiling sometimes begins explosively and oil is blown out through the top of the condenser.

RELIABILITY AND ACCURACY OF METHOD

The method has been tried with a number of emulsions prepared by mixing known amounts of water with anhydrous oils. Results have almost invariably checked the theoretical water content within ± 0.1 cc., which apparently represents the normal magnitude of error. The applicability of the method for the separation of water from refractory emulsions has been questioned and tests have been made to settle this point. Water was determined according to the procedure described. The flasks were allowed to cool somewhat, then connected with an ordinary distilling tube and condenser and the distillation continued until a vapor temperature of 250° C. was reached. In no case was any additional water separated or observed, thereby indicating that distillation at the temperature existing in the presence of the solvent was adequate to "break" the emulsion.

APPLICABILITY TO SUBSTANCES OTHER THAN PETROLEUM

The method was tried with several samples of grease and yielded results that were considered highly satisfactory by the operator who had had considerable experience with the conventional methods for determining the water content of greases. It was also tried with several samples of coal tar. The naphtha solvent proved useless for this type of emulsion, the tars in question being quantitatively insoluble in petroleum distillate. The xylene-benzene solvent, however, was satisfactory and the results of the determinations were

apparently as reliable as when petroleum emulsions were tested.¹

The authors believe that the method herein described is applicable to a wide variety of organic emulsions, possibly solid as well as liquid, and recommend its trial in all laboratories not fully satisfied with the methods at present in use. While it is not believed that results may necessarily be more accurate than those now obtainable, an increase in convenience and rapidity is anticipated.

PREVIOUS WORK ALONG SIMILAR LINES

On account of the simplicity of the modified method the authors have found it difficult to believe that their work could be new. The literature has been searched diligently, however, and it has been found that practically all the distillation methods for water determination have involved the use of apparatus of the conventional type and arrangement. The methods of Besson and Gray approach most closely the procedure recommended in the present paper. The basic idea of the authors' method was conceived after reading a description of the apparatus of Besson. Neither his method nor that of Gray is, however, believed to be as practical as that of the authors. The apparatus is not as simple and convenient and no attempt is apparently made to make the distillation continuous by refluxing the solvent into the flask.

SUMMARY

General experience has indicated that the method of distilling in the presence of an immiscible solvent is the most reliable and most generally satisfactory scheme of estimating the water content of organic emulsions, particularly petroleum emulsions.

The Pittsburgh Petroleum Laboratory of the Bureau of Mines has developed modifications of this method that add considerably to its convenience.

A description of the apparatus and procedure employed by the Bureau appears in the present paper.

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¹ Coal-tar emulsions are commonly believed to be more difficult to break up than petroleum emulsions and with the samples of tar tested particular care was taken to prove that no water remained after distillation in the presence of xylene-benzene mixture. The scheme of subsequently distilling the residue was employed and no water was discovered when the vapor temperature was carried as high as 250° C.

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HEAT BALANCE OF A DISTILLATION PLANT FOR THE RECOVERY OF FATTY ACIDS FROM COTTONSEED FOOTS

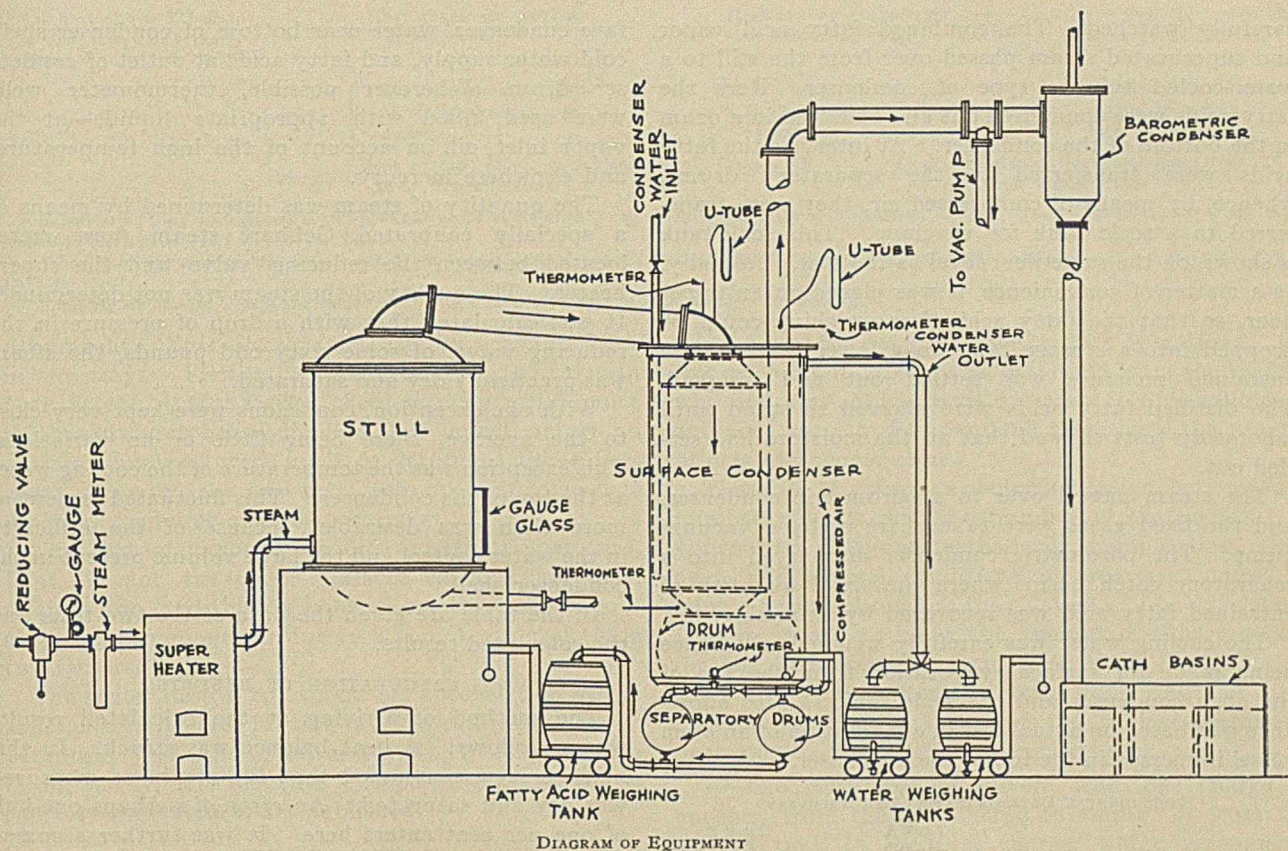
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Some years ago the writer was engaged in development work along lines of fatty acid distillation. Before this could be undertaken it was necessary to know the value of the heat of vaporization of fatty acids under operating conditions, *i. e.*, at the temperature and reduced pressure usually employed in distillation. A search of the literature of this subject failed to yield the desired information and the determination of these values was, therefore, undertaken by the writer. The results are given here in the hope that they may be of value to the profession.

The heat of vaporization can be calculated from the vapor pressure curve of the fatty acids, and the latter can be determined in the laboratory. As a commercial fatty acid distillation plant was available, however, advantage was taken of this fact to make a large-scale determination. This was preferred, as it was thought that it would not be possible to reproduce working conditions on a laboratory scale. One of the most important of these factors is the effect of the pitch in the still bottom. As it accumulates it becomes necessary to raise the distillation temperature sometimes



as much as seventy-five degrees or more. It was also believed that any variation in composition of the fatty acids or other factors would be averaged in a larger-scale determination.

The fatty acids were obtained from foots from the refining of crude cottonseed oil. The crude oil consists principally of a mixture of the glycerides of palmitic, linoleic and oleic acids containing also small quantities of glycerides of arachidic and possibly stearic acids.¹ In addition to these glycerides there are also present the glycerides of all these acids in various stages of decomposition.

Crude cottonseed oil is usually refined with caustic soda, whereby the partially and wholly decomposed glycerides are separated out as a soap (foots). This soap is only partially saponified and contains considerable quantities of neutral oil, together with most of the coloring, nitrogenous and mucilaginous matter that was present in the crude oil. The preponderant constituent of the foots is probably a soda soap of oleic acid, inasmuch as olein, the glyceride of oleic acid, is largely present in cottonseed oil and is most readily broken down. The composition of the foots will vary considerably, depending to a great extent on the amount of neutral oil retained in refining.

The foots may be killed and acidulated, *i. e.*, the saponification may be completed by boiling with sufficient alkali and the resulting soap treated with mineral acid to liberate the fatty acids; or the foots may be first treated with a mineral acid to free it from alkali, and then saponified by the Twitchell or

¹ Lewkowitsch, "Chemical Technology and Analysis of Oils, Fats and Waxes," 2, 5th Ed., p. 197.

other process to remove the remaining glycerol radicals. In either case the end result is a mixture of free fatty acids ready for the distillation process.

Two distillations were run on entirely different lots of fatty acids, since several months elapsed before the distillation plant could be spared for the second determination, and it was not practical under the existing conditions to store a portion of the first lot of stock for use in the second determination.

METHOD

The method employed in making the determination was as follows: The distillation plant was operated on fresh fatty acid stock, with no fatty acid or pitch left in the still from previous distillations. Before starting the tests, distillation was carried on until conditions were constant. A flying start and stop of the tests were used in a manner similar to that employed in making evaporative boiler tests. Thus, without interruption or disturbance of the distillation, tests were started and stopped immediately after emptying the condenser drums.

Measurements of all such quantities as were required to write a heat balance of the plant were made. Assistants were stationed at points where manual control was necessary. All readings were taken at 5-min. intervals.

The accompanying diagram gives an outline of the distillation plant employed. It consisted of a coal-fired still, supplied with superheated steam through a reducing valve and a separately fired superheater. This still was fed continuously so that the volume in the still was constant, the level in a gauge glass being

carefully watched. The combined fatty acid vapor and superheated steam passed over from the still to a water-cooled tubular type of condenser. Here the fatty acids were condensed out and collected in a drum in the bottom of the condenser. At intervals the fatty acids were transferred to the separatory drums, whence, by means of compressed air, they were transferred to a scale tank for weighing. This scale tank is shown on the same floor level as the still. Actually, as a matter of convenience, it was placed on an upper floor, so that the fatty acid after weighing could be dropped into a storage tank on a lower level. Here entrained moisture was settled out and weighed. The distilled fatty acids were allowed to stand until laboratory tests showed that all the moisture had settled out.

The steam passed over to a barometric condenser, and the fixed gases were taken care of by a vacuum pump. The barometric condenser discharged into a separatory catch basin, where the small quantity of entrained fatty acid was recovered by skimming.

The cooling water was carefully weighed, allowance being made for surface evaporation from the top of the open condenser and the scale tanks. This allowance was based on actual loss in evaporation of an open vessel immersed in the top of the condenser.

EXPERIMENTAL DATA ON FATTY ACID RECOVERY

	TEST A MARCH	TEST B MAY
Length of test.....	7 hrs.	7 hrs.
Average barometer corrected to 58.4° F.....	29.62 in. Hg	29.10 in. Hg
Vacuum condenser vapor inlet corrected to 58.4° F.....	24.13 in. Hg	24.24 in. Hg
Vacuum condenser vapor outlet corrected to 58.4° F.....	25.17 in. Hg	24.68 in. Hg
Average temperature at vapor inlet	433.8° F.	445.5° F.
Average temperature at vapor outlet	141.9° F.	134.0° F.
Average temperature of stock into separatory drums.....	136.8° F.	131.9° F.
Average temperature of condensing water into condenser.....	33.6° F.	52.6° F.
Average temperature of condensing water out of condenser.....	123.1° F.	178.0° F.
Average temperature of condensing water, bottom of condenser.....	116.0° F.	118.5° F.
Total steam consumed.....	7229.0 lbs.	5901.0 lbs.
Total stock into separatory drums (after deducting settled water and moisture).....	11077.0 lbs.	13091.0 lbs.
Total stock from 1st catch basin (after deducting moisture content).....	3176.0 lbs.	2839.0 lbs.
Total stock from 2nd catch basin (after deducting moisture content).....	286.0 lbs.	278.0 lbs.
Total stock (dry).....	14539.0 lbs.	16208.0 lbs.
Average moisture content of stock.....	0.713 per cent	0.4 per cent
Water carried with stock into separatory drums and settled out.....	538.0 lbs.	272.0 lbs.
Water present in stock into separatory drums as moisture.....	80.0 lbs.	53.0 lbs.
Water present in stock from catch basin.....	25.0 lbs.	12.0 lbs.
Total water carried over and present as moisture.....	643.0 lbs.	337.0 lbs.
Condensing water.....	57740.0 lbs.	36719.0 lbs.
Rate of evaporation of condensing water (lbs. per sq. ft. per hr.).....	0.54 lb.	1.46 lbs.
Area from which evaporation took place.....	31.0 sq. ft.	31.0 sq. ft.
Area from which radiation took place.....	357.9 sq. ft.	357.9 sq. ft.
CALCULATED RESULTS		
Total heat to raise 1 lb. of stock from 32° F. to vapor under the test conditions.....	315.3 B. t. u.	308.2 B. t. u.
Heat to vaporize 1 lb. of stock under test conditions.....	130.5 B. t. u.	118.0 B. t. u.

To measure the vacuum, mercury U-gauges were placed at the inlet to the surface condenser and in the vapor pipe outlet, as close to the water surface as possible.

Temperatures were taken at these two points, also at the following points: the surface of water in sur-

face condenser, water near bottom of condenser shell, cold water supply, and fatty acids at outlet of condenser drum. Wherever possible, thermometer wells were used, filled with appropriate liquids—at the vapor inlet, oil, on account of the high temperature, and elsewhere mercury.

The quantity of steam was determined by means of a specially calibrated Gebhart steam flow meter located between the reducing valve and the superheater. The quality of the steam was not determined. It was calculated that with a drop of pressure in the reducing valve, of some sixty odd pounds, the steam was practically dry and saturated.

With one exception, conditions were kept very close to the average, there being little or no variations. This exception was the temperature of the cooling water at the top of the condenser. This fluctuated somewhat more than was desirable, because of the difficulty in the water control and the large volume present in the condenser shell.

In the table are given the data of the two tests and the calculated results.

CALCULATION OF RESULTS

The method of arriving at the calculated results was as follows: A heat balance was struck. In this balance it was assumed that the steam as measured was dry and saturated. An error of perhaps one-half of one per cent enters here. It was further assumed that there was practically no condensation in the vapor pipe connecting the still with the condenser. As this pipe pitched toward the condenser, any condensation would be collected in the condenser drum. This pipe was 12 in. in diameter and 14 ft. long, and was insulated with very heavy magnesia covering. The heat loss was a negligible percentage of the total passing.

The temperature at the condenser inlet was assumed to be that of saturated fatty acid vapor, *i. e.*, it was assumed the fatty acid vapor carried no entrained liquid fatty acid and was not superheated. The very large diameter and height of the still precluded entrainment, and laboratory tests showed that at 28.25 in. to 29.5 in. of vacuum the boiling point is very close to 440° F. This checks very well with the actual large-scale tests where steam for distillation was used. As might be expected, somewhat lower temperatures were found.

The total heat in the superheated steam was obtained from the Molière chart and data published by Prof. C. C. Thomas.¹

The heat lost by radiation from the condenser shell was calculated from the values in Kent's "Mechanical Engineers' Handbook."² The value used for the mean specific heat of the fatty acids was 0.46.

The heat balance as struck is given at top of next page.

The close agreements of the results is rather astonishing. There is a difference of only about 2 per cent in the values for total heat per pound of stock. However, the values for heat of vaporization differ by about

¹ *Trans. Am. Soc. Mech. Eng.*, 29 (1907).

² 1898 Ed., p. 536.

$$\begin{array}{rcccc}
 \text{Total heat above } 32^{\circ} \text{ F.} & & \text{Heat taken up by con-} & & \text{Heat lost by evaporation} & & \text{Heat lost by radiation} \\
 \text{in the fatty acid dis-} & = & \text{densing water} & + & \text{of condensing water} & + & \text{from condenser shell} \\
 \text{tilled} & & & & & & \\
 & & & & & & \\
 & - & \left[\begin{array}{l} \text{Total heat above } 32^{\circ} \text{ F.} \\ \text{of steam entering con-} \\ \text{denser} \end{array} \right. & - & \left. \begin{array}{l} \text{Total heat above } 32^{\circ} \\ \text{F. in steam leaving} \\ \text{condenser} \end{array} \right. & - & \left. \begin{array}{l} \text{Heat above } 32^{\circ} \text{ in water} \\ \text{carried over with stock} \\ \text{into settling tank} \end{array} \right] \\
 & + & \begin{array}{l} \text{Heat to raise stock in condenser drum from } 32^{\circ} \text{ F.} \\ \text{to drum temperature} \end{array} & + & \begin{array}{l} \text{Heat to raise stock from catch basin from } 32^{\circ} \text{ F.} \\ \text{to condenser temperature} \end{array} & &
 \end{array}$$

Substituting the proper figures in this heat balance, and dividing by the quantity of fatty acid distilled, we find: Total heat above 32° F. per lb. of fatty acid distilled = 315.3 B. t. u. for Test A

Whence, on the assumptions before stated, we have: Heat of vaporization per lb. of fatty acid at 24.51 in. vacuum = 315.3-0.46 (433.8-32) = 130.5 B. t. u.

Similarly we find for Test B the values 308.2 B. t. u. and 118.0 B. t. u., but at 25.14 in. vacuum.

9 per cent. This discrepancy can be accounted for in two ways: Firstly, the stock was not the same in both cases. Undoubtedly, there was some difference in the mixtures being distilled, but unfortunately no chemical analyses were made to determine the difference; secondly, the two tests were made at somewhat different pressures and temperatures.

The heat of vaporization differs with temperature. If it did not the vapor pressure curve would be a straight line, and this we know is not the case. Probably the discrepancy is due to a combination of these two factors.

The writer desires to express his appreciation of the assistance rendered in this work by Messrs. S. B. Murdock, J. W. Bodman, and A. D. Whitby, and to thank the N. K. Fairbanks Company, through whose courtesy these figures are published.

NUMERICAL RELATION BETWEEN CELLS AND TREATMENTS IN EXTRACTION PROCESSES

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In a previous study of extraction processes¹ conducted at the U. S. Forest Products Laboratory, Madison, Wisconsin, it was found that no definite relation had ever been developed between the number of solvent treatments received by each charge of material and the number of cells or extractors required to furnish this number of treatments. It was also evident that the ideas in regard to such a relation were very much confused, and that the usual conception was the entirely unfounded one that for a certain number of treatments an equal number of cells was required or the same number plus one.² After it had been determined how many treatments in series were required for efficient extraction and how much time would be required for boiling, pumping, discharging, charging, etc., there was still no method by which it could be determined how many cells or extractors would be required by the process.

In the development of the numerical relation between the cells and the treatments it will be necessary to consider a general case covering all conditions; and this will require the use of terms and the assumption of conditions that may seem unusual or impractical to operators who have had experience with only one kind of extraction process and have used a peculiar

set of terms for describing the various operations. On this account indulgence is requested from the readers who find unusual processes described in unusual terms, and they are asked to overlook these until the general principles become clear.

There is, of course, a definite relation between the number of cells and the number of treatments. It varies with different methods of manipulation, however, and several conditions of treatment must be known accurately before the relation can be developed. It is necessary to know the amount of time required for the operations performed on each charge other than the typical extraction operations, and this time must be known in terms of the typical extraction operations. These latter operations are:

1—The movement of solvent from one charge to another, from the last charge to storage or from the solvent tank to the first charge, which movements will be called "pumping" and designated by *p*.

2—The period of the actual solution action of the solvent on the charge, whether by standing, boiling, or agitating, which will be called "boiling" and designated by *b*. These typical operations are necessary parts of every discontinuous extraction process and may vary only in the time required for their fulfillment.¹ While there are only these two kinds of extraction operations, each may take place a variable number of times in a complete cycle of extractions. The non-extraction operations may vary widely in number, kind, and time required, and include charging and discharging the cells, or any preliminary or after-treatment performed on the charge while in the cells.

SIMULTANEOUS PUMPING

The simplest way to determine the relation between the number of treatments and the number of cells in terms of the time required for the various operations seems to be to use cut and try methods under several different sets of conditions and then to find the complete mathematical relation by inspection. This can be readily done by representing a series of extractions graphically. The easiest system to start with is one in which the pumping of all cells in action is done simultaneously. A series of extractions in four cells by this system can be represented as in Fig. 1.

In this method of representation the arrows indicate movement of the solvent or pumping; for instance, the first line of the diagram indicates simultaneous move-

¹ THIS JOURNAL, 9 (1917), 866.

² Kressmann, *Met. & Chem. Eng.*, 15 (1916), 78.

¹ As will be seen later, *b* may equal zero in some cases.

ment of solvent from cell 2 to cell 3, cell 3 to cell 4, and cell 4 to storage, while line 3 indicates simultaneous application of fresh solvent to cell 3, and movement of solvent from cell 3 to cell 4, and from cell 4 to cell 1. All cells are assumed to contain solvent except those with a double circle. Those indicated in line 2 as containing solvent but without solvent movement show the extraction or boiling period. By following this series of operations we find that a complete cycle has been finished in 16 lines and that line 17 is the same as line 1. During this cycle each cell receives 5 treatments and each batch of solvent is used 5 times. It will be noted that for a period of 3 boilings and 2 pumpings (indicated by braces) each cell stands idle so far as solvent action is concerned; this period is the time available for charging and discharging and for other preliminary or after-treatment of the charge. The relation between this idle period, n , the number of cells, c , and the number of treatments, t , in terms of the boiling period, b , and the pumping period, p , will now be worked out.¹ In the case just shown $n = 3b + 2p$ when $c = 4$ and $t = 5$.

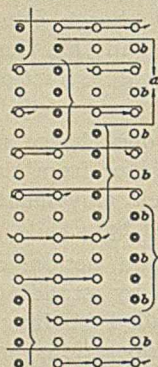


FIG. 1

SIMULTANEOUS PUMPING 4 CELLS 5 TREATMENTS

$$n = 3b + 2p$$

$$a = 2b + 2p$$

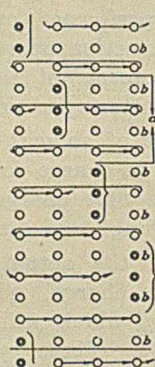


FIG. 2

SIMULTANEOUS PUMPING 4 CELLS 6 TREATMENTS

$$n = 2b + p$$

$$a = 2b + 2p$$

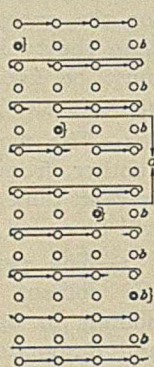


FIG. 3

SIMULTANEOUS PUMPING 4 CELLS 7 TREATMENTS

$$n = b$$

$$a = 2b + 2p$$

In Fig. 2 is shown the same number of cells so used as to give each cell one more treatment. This arrangement shortens the idle period n to $2b + 1p$ and our next relation is that when $c = 4$ and $t = 6$, $n = 2b + p$. In the same way when one more treatment is obtained, the maximum possible in 4 cells, the time available for discharging, charging, etc., is reduced to a single boiling period. (See Fig. 3.)

Similar diagrams for various other numbers of cells and treatments were made and from them was prepared Table I in which the general relation between the factors c , t , n , b , and p becomes apparent.

From inspection of the table it is seen that n is composed of boiling periods equal in number to twice the number of cells minus the number of treatments, and of pumping periods always one less than the number of boiling periods, or

$$n = b(2c - t) + p(2c - t - 1) \text{ or } t = 2c - \frac{n + p}{b + p}$$

It is suggested that more confidence in the table

¹ The letters used in designating the different variables are all shown either in this sentence or in the headings of Tables I and II.

and in the formula derived from it will be produced if the reader will verify a few of the relations shown in the table by preparing diagrams similar to these given, but for different values of c or t . This procedure will also make it possible to follow the further similar developments more readily.

TABLE I—ALL CELLS PUMPED AT ONE TIME

CELLS c	TREATMENTS t	Time Available for Charging, Discharging, etc. n	Time between Discharges a
2	2	$2b + 1p$	$2b + 2p$
2	3	$1b$	$2b + 2p$
3	2	$4b + 3p$	$2b + 2p$
3	3	$3b + 2p$	$2b + 2p$
3	4	$2b + 1p$	$2b + 2p$
3	5	$1b$	$2b + 2p$
4	3	$5b + 4p$	$2b + 2p$
4	4	$4b + 3p$	$2b + 2p$
4	5	$3b + 2p$	$2b + 2p$
4	6	$2b + 1p$	$2b + 2p$
4	7	$1b$	$2b + 2p$
5	3	$7b + 6p$	$2b + 2p$

It should be noted further that in this method of operation, *viz.*, simultaneous pumping of all cells with a boiling period after every pumping period, the time elapsed between any similar operations on successive cells (which is the measure of the output of the system) is always the same, $2b + 2p$, whatever the number of cells or treatments. This period is indicated by a on the diagrams and shows the time elapsed between the beginning discharge of two consecutive cells.

CONTINUOUS EXTRACTION

It should be noticed here that these formulas are applicable also to continuous extraction. Continuous extraction is the same as the system of simultaneous pumping just described in which $b = 0$ and p is the period of time required for moving the liquid contents of one cell to the next or to storage. Under these conditions, $n = p(2c - t - 1)$ or $t = 2c - \frac{n + p}{p}$, and $a = 2p$.

SEPARATE PUMPING

It has been shown¹ that the method of pumping all cells simultaneously gives much less efficient extraction than pumping one cell at a time. It might possibly be thought that the pumping of only one cell at a time would take so much longer that in the case of a large number of cells it would lose more in time than would be gained in efficiency of extraction. Some very interesting comparative figures are obtained by developing the relations between c , t , n , etc., under this method of pumping. The method of representation of the extraction process is the same as in the other case, and the diagram, Fig. 4, shows that in an operation of 6 treatments in 4 cells $n = 2b + 4p$.

In this method there are two other variable time periods which are important and will also be studied; these are the average length of time each charge is boiled with each separate batch of solvent, designated by e , and the time elapsed between successive discharges, designated by a .

In this method of pumping the actual length of time each charge is boiled with each batch of solvent is not always the boiling period b only, but is variable. This is due to the fact that sometimes only one and

¹ THIS JOURNAL, *Loc. cit.*

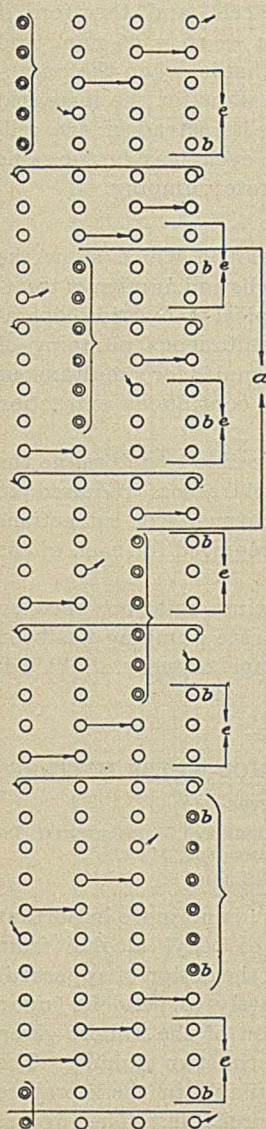


FIG. 4

SEPARATE PUMPING
4 CELLS 6 TREATMENTS

$$n = 2b + 4p$$

$$a = 2b + 7p$$

$$e = b + 2p$$

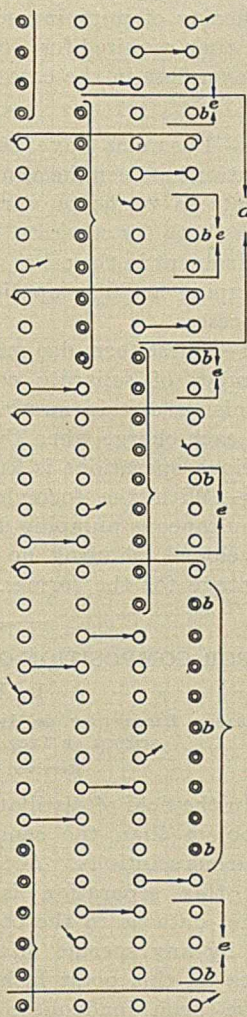


FIG. 5

SEPARATE PUMPING
4 CELLS 5 TREATMENTS

$$n = 3b + 6p$$

$$a = 2b + 6p$$

$$e = b + 3/5p$$

never more than two cells are in the pumping stage at one time, and as soon as one cell is pumped full it can immediately enter the boiling stage while the others are being pumped.

TABLE II—EACH CELL PUMPED SEPARATELY

CELLS	TREATMENTS	Time Available for Charging, Discharging, etc.	Time between Discharges	Average Single Boiling Period
<i>c</i>	<i>t</i>	<i>n</i>	<i>a</i>	<i>e</i>
2	3	1 <i>b</i> + 4 <i>p</i>	2 <i>b</i> + 4 <i>p</i>	<i>b</i> + 2/3 <i>p</i>
2	2	2 <i>b</i> + 2 <i>p</i>	2 <i>b</i> + 3 <i>p</i>	<i>b</i>
3	5	1 <i>b</i>	2 <i>b</i> + 6 <i>p</i>	<i>b</i> + 8/5 <i>p</i>
3	4	2 <i>b</i> + 3 <i>p</i>	2 <i>b</i> + 5 <i>p</i>	<i>b</i> + <i>p</i>
3	3	3 <i>b</i> + 4 <i>p</i>	2 <i>b</i> + 4 <i>p</i>	<i>b</i> + 2/3 <i>p</i>
3	2	4 <i>b</i> + 5 <i>p</i>	2 <i>b</i> + 3 <i>p</i>	<i>b</i> + 0 <i>p</i>
4	7	1 <i>b</i>	2 <i>b</i> + 8 <i>p</i>	<i>b</i> + 18/7 <i>p</i>
4	6	2 <i>b</i> + 4 <i>p</i>	2 <i>b</i> + 7 <i>p</i>	<i>b</i> + 2 <i>p</i>
4	5	3 <i>b</i> + 6 <i>p</i>	2 <i>b</i> + 6 <i>p</i>	<i>b</i> + 8/5 <i>p</i>
4	4	4 <i>b</i> + 8 <i>p</i>	2 <i>b</i> + 5 <i>p</i>	<i>b</i> + <i>p</i>
4	3		2 <i>b</i> + 4 <i>p</i>	
5	9	1 <i>b</i>	2 <i>b</i> + 10 <i>p</i>	<i>b</i> + 32/9 <i>p</i>
5	8	2 <i>b</i> + 5 <i>p</i>	2 <i>b</i> + 9 <i>p</i>	<i>b</i> + 3 <i>p</i>
5	7	3 <i>b</i> + 8 <i>p</i>	2 <i>b</i> + 8 <i>p</i>	<i>b</i> + 18/7 <i>p</i>
5	6	4 <i>b</i> + 11 <i>p</i>	2 <i>b</i> + 7 <i>p</i>	<i>b</i> + 2 <i>p</i>

In the diagram showing 6 treatments in 4 cells, $e = b + 2p$ and $a = 2b + 7p$. In the next diagram, Fig. 5, showing 5 treatments in 4 cells the time n has increased to $3b + 6p$, $e = b + 8/5p$ and $a = 2b + 6p$.

In the same way diagrams have been prepared showing a wide variation in number of cells and number of treatments, and the resulting values for n , a , and e are given in Table II.

From this table the following formulas were developed¹ showing the various relations between c , t , n , a , and e .

EXTRACTIONS IN WHICH EVERY CHARGE RECEIVES TWO TREATMENTS WITH FRESH SOLVENT

Simultaneous Pumping

$$n = (3c - t)b + (3c - t - 1)p$$

$$a = 3b + 3p$$

$$e = b$$

Separate Pumping

$$n = bc(3c - t - 1) - (3c - t) \left(\frac{3c - t - 1}{3} \right) p \text{ when } t \text{ is multiple of } 3$$

$$n = bc(3c - t - 1) - \frac{(3c - t - 1)^2 - (3c - t - 1)}{3(3c - t)} p \text{ when } t \text{ is not multiple of } 3$$

$$a = 3b + (t + 2)p$$

$$e = b + (t/3 - 1)p \text{ when } t \text{ is multiple of } 3$$

$$e = b + \frac{(t - 1)^2 - (t - 1)}{3t^2} p, \text{ when } t \text{ is not multiple of } 3$$

$$n = (2c - t)b + c(2c - t - 5)p + (2t + 4)p \tag{1}$$

$$a = 2b + (t + 1)p \tag{2}$$

$$e = b + \frac{(t - 1)^2}{2t} p \text{ (when } t \text{ is odd)} \tag{3}$$

$$e = b + \frac{t - 2}{2} p \text{ (when } t \text{ is even)}$$

These formulas are of particular interest in showing that:

1—The extra time available for charging, discharging, etc., n , varies directly with the number of cells and inversely with the number of treatments obtained.

2—The length of time between successive discharges of material, a , which is really the reciprocal of the capacity of the system, is *not* influenced by the number of cells but is increased to a slight extent by an increased number of treatments.

3—The average actual time each charge is exposed to the boiling operation is not affected by the number of cells but varies to a slight extent with the number of treatments.

APPLICATION OF FORMULAS

Suppose for example it had been determined that a certain extraction process would require 6 treatments and that the pumping would take 10 min., the boiling 15 min., and the charging, discharging and solvent recovery 1.5 hrs. How many cells would be required for the process?

¹ The development of similar formulas for extractions in which every charge receives two treatments with fresh solvent (THIS JOURNAL, *Loc. cit.*) will not be given in detail but following are the formulas for this method of extraction which have been developed by the same method as the foregoing.

$$\begin{aligned}
 n &= b(2c - t) + p(2c - t - 1) \\
 90 &= 15(2c - 6) + 10(2c - 6 - 1) \\
 &= 30c - 90 + 20c - 70 \\
 90 &= 50c - 160 \\
 250 &= 50c \\
 c &= 5
 \end{aligned}$$

If, however, n had been 60 min. instead of 90 min. the value of c would have come to 4.4; this would mean that either 5 cells would have to be used with a wait of 30 min. after charging before the first batch of solvent is ready to run on or else the time of boiling or of pumping would have to be lengthened so that 4 cells could do the work. If, for instance, 4 cells are to do the work, the boiling period must be lengthened to 25 min.

$$\begin{aligned}
 60 &= b(8 - 6) + 10(8 - 6 - 1) \\
 60 &= 2b + 10 \\
 b &= 25
 \end{aligned}$$

or the pumping period to 30 min.

$$\begin{aligned}
 60 &= 15(8 - 6) + p(8 - 6 - 1) \\
 60 &= 30 + p \\
 p &= 30.
 \end{aligned}$$

These examples show the method of using the formula in determining the number of cells required under different conditions of treatment.

COMPARISON OF SIMULTANEOUS AND SEPARATE PUMPING SYSTEMS

The formulas also make it possible to compare the time consumed between discharges when pumping simultaneously with that consumed when pumping separately, other conditions of extraction being the same. If we take, for instance, a series of 6 extractions in 4 cells, $b = 15$ and $p = 5$. With separate pumping n will be $15(8 - 6) + 4(8 - 6 - 5)5 + (12 + 4)5 = 30 - 60 + 80 = 50$
 a will be $30 + (6 + 1)5 = 65$, and
 e will be $15 + \frac{6 - 2}{2} p = 25$.

For a comparison it would not be fair to use b with the same value in simultaneous pumping as in separate pumping because in the latter case, in the example just given, although b is 15, the *actual* total boiling time for each charge, e , is 25 min. ($b + 2p$); in order to have conditions the same in the two systems, therefore, the value of b in simultaneous pumping (which is always the actual total time of the boiling) must be 25 min. With $c = 4$, $t = 6$, $b = 25$, and $p = 5$ in simultaneous pumping

$$\begin{aligned}
 n &= 25(8 - 6) + 5(8 - 6 - 1) = 50 + 5 = 55 \\
 a &= 2b + 2p = 50 + 10 = 60 \\
 e &= b = 25.
 \end{aligned}$$

That is, a , the time required between successive discharges of material is a little less in the case of simultaneous pumping.¹ But there is a practical consideration which changes this relation slightly; pumping several cells at the same time is likely to take

¹ Although this is only one case it may be shown that the difference between a (sep) and a (sim), with other conditions the same, is always p when t is even and $\frac{t-1}{2}p$ when t is odd.

longer than pumping a single cell, and therefore if p (sim) = 8, then a (sim) = $50 + 16 = 66$ min., and a (sep) = $30 + 35 = 65$ min., that is, when the pumping time is increased a little to allow for the increased difficulty of pumping several cells instead of one cell, the time required for a complete cycle may be the same or even less in the case of separate pumping.

SUMMARY

1—Formulas have been developed which show the relation between the number of cells and number of treatments in terms of various typical extraction operations both for separate and simultaneous pumping of the solvent in the cells. The formulas for simultaneous pumping are also applicable to continuous extraction processes.

2—These formulas make it possible to determine the number of separate cells or extractors required to furnish a certain number of treatments or extractions for each charge when the time required for each of the various operations is known.

3—With the formulas the time relation between simultaneous pumping and separate pumping has been developed, showing no advantage or very slight advantage for the former.

VAPOR COMPOSITION OF ALCOHOL-WATER MIXTURES

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Received December 18, 1919

In the field of distillation there is no more important problem than the separation of ethyl alcohol from aqueous solutions. Accuracy in the design of apparatus for this separation is absolutely dependent upon exact data as to the composition of the vapors given off by any specific mixture of the two liquids. The data on this point hitherto available in the literature have been inadequate and unreliable. The figures usually accepted are those recalculated for Maercker¹ by Dönitz from the original experimental results of Gröning. These data check reasonably the results obtained a half century ago by Duclaux.² The more recent work of Sorel does not check that of Gröning and is apparently less reliable. The work of Evans³ is obviously unreliable in view of the fact that he finds the composition of vapor and liquid identical at 92 per cent by weight, whereas a distillate of higher than 95 per cent alcohol can be obtained in commercial practice.

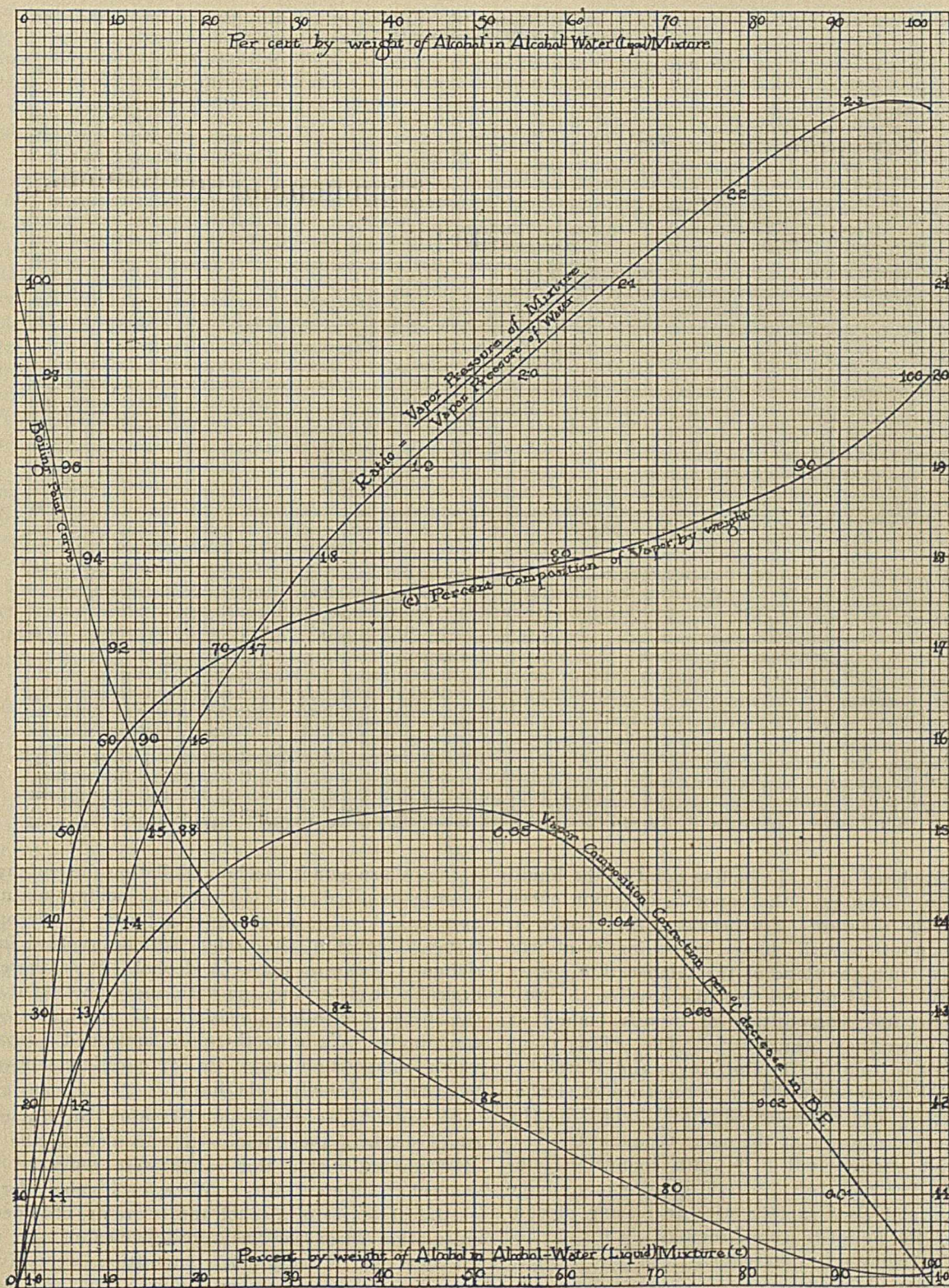
In 1913 Wrewsky⁴ published the results of a series of careful determinations of the vapor composition of alcohol-water mixtures, which bear the earmarks of reliability. On the other hand, Wrewsky's work was done, not at constant pressure, but at constant temperature. Furthermore, he operated at only three temperatures, approximately 40°, 55°, and 75° C. His work is, therefore, not directly available for industrial practice because the industrial distillation

¹ "Handbuch der Spiritus Fabrikation," 7th Ed., Berlin (1898), 590.

² *Ann. chim. phys.*, **14** (1878), 305.

³ *This Journal*, **8** (1916), 261.

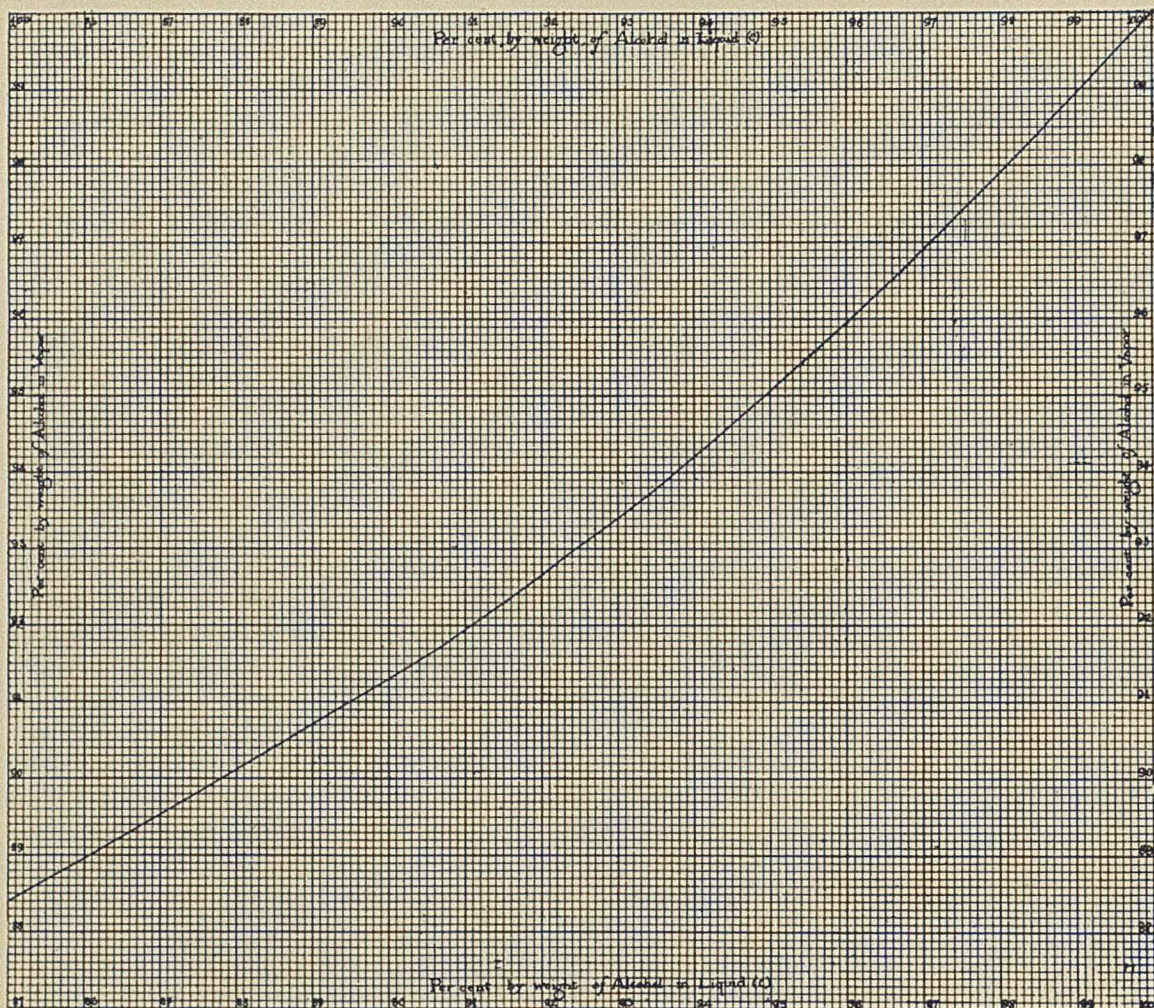
⁴ *Z. phys. Chem.*, **81** (1912), 1.



of alcohol is carried on entirely at temperatures above 78°, *i. e.*, at atmospheric pressure and, in the bottom of rectifying columns, even higher. In order to make these data available for industrial use we have recalculated Wreusky's results, extrapolating them up to the boiling point. The resulting data are given graphically in the accompanying plots and are believed to be by far the most accurate figures avail-

able on the vapor compositions of alcohol-water mixtures.

At each of the temperatures mentioned above Wreusky determined the vapor composition curve. From these isothermal curves the vapor composition was read off at stated intervals of liquid composition and, for each definite liquid composition, plotted against the temperature. These temperature curves



were extrapolated up to the boiling point of the liquid in question. This gave the vapor composition curve shown in the diagram. Furthermore, the change in vapor composition with change in boiling point was determined from the slope of the temperature curves just described, and plotted to make possible the estimation of vapor composition of liquids boiling at pressures other than atmospheric. The boiling point curve given is from the data of Noyes and Warfle.¹

The total pressure exerted by any mixture of alcohol and water, at any given temperature, divided by the pressure of pure water at the same temperature, is a ratio which changes but little with change in temperature. The value of this ratio is, of course, unity for water, and rises rapidly with increasing alcohol content of the liquid to approximately 2.3 for alcohol itself. The ratio was determined from the pressure measurements of Wrewsky at the temperatures employed by him, and graphically extrapolated to the boiling point in exactly the same way as the vapor composition. The value of this ratio at the boiling point is plotted as a separate curve, and will make it possible to estimate temperature-pressure relationships other than atmospheric, as for example in the lower part of a rectifying column.

In any problem in alcohol distillation the enrichment is easily realized until the constant boiling mixture is approached. The most important part of the operation and hence of the design is that involved in enrichment above 90 per cent alcohol. It is, therefore, desirable to determine the data above this point with the greatest possible precision and to plot it correspondingly. We have, therefore, appended a separate vapor composition curve for compositions above 85 per cent of alcohol in the liquid.

In order to secure greater accuracy for the vapor composition of these liquids rich in alcohol, the data of Wrewsky within this range were replotted in a somewhat different way. Wrewsky's values for the partial pressures of alcohol and of water, obtained by calculation from the measured values of the total pressure and of the vapor composition, were divided in each case by the pressure of pure alcohol and of pure water at the temperature of the determination. These ratios change but slightly with the temperature. For each of the three temperatures used by Wrewsky they were plotted against the molal composition of the liquid, and smooth curves drawn among the individual points. The ratios were read off these curves for each mol per cent of alcohol in the liquid, and these ratios plotted against the temperature. These last

¹ *J. Am. Chem. Soc.*, 23 (1901), 154.

curves, either straight lines or very flat, were extrapolated up to the boiling point, and from the values thus obtained the vapor composition calculated. It will be noted that the extrapolation was in no case greater than four degrees, because the boiling points of these rich alcohol mixtures are nearly constant.

In order to show the applicability of these curves, let it be assumed that a 50 per cent alcohol-water mixture is boiling at a temperature of 100° C. at some point in the lower part of a rectifying column. It is required to determine the composition of the vapor and the pressure under which the liquid is boiling.

Were the liquid boiling at atmospheric pressure it would have a vapor composition at 77.7 per cent. Under these conditions it would boil at 82° . Since it is boiling at 100° , its boiling point has been increased 18° by pressure. From the correction curve it is seen that the vapor composition changes 0.524 per cent per degree. This corresponds to a correction of 0.9 per cent. The correction is positive for a decrease in boiling point and is, therefore, negative for the case in question. Hence the vapor composition of the 50 per cent alcohol at 100° is 76.8 per cent.

At 100° water exerts one atmosphere pressure. It is seen from the vapor pressure ratio curve that a 50 per cent solution exerts 1.97 times as much, *i. e.*, the pressure at the given point in the still is 1.97 atmospheres or 1497 mm.

A SIMPLE METHOD FOR THE PREPARATION OF SODIUM AMALGAM IN FLAKES¹

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Received November 28, 1919

In the course of preparation of saligenin by a slight modification of Hutchinson's method made by Mr. Hurd,² it has been necessary for us to use a 2.75 per cent sodium amalgam. This amalgam, when poured on to porcelain plates, forms solid layers, which require a good deal of work and time to reduce to a powder in a mortar. We have been able to prepare a powder of very finely flocculent amalgam by pouring the hot liquid amalgam slowly into a battery jar of xylene or kerosene, which is already being rapidly agitated by an electric stirrer. The amalgam is broken up by the currents as fast as it is poured into the liquid and the fine flocculi settle to the bottom. They are then dried in a current of air on a porcelain plate. When the flocculi are clumped they are readily pulverized by a couple of blows with a pestle.

AN IMPROVED COMPARATOR

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Received January 12, 1920

While studying the change in hydrogen ion concentration³ of water or of a solution of broth due to

¹ From the Department of Pharmacology, University of Minnesota, with the aid of funds granted by the United States Interdepartmental Social Hygiene Board for Research in the prevention and care of venereal diseases.

² A. Hutchinson, *Ber.*, **24** (1891), 173; Charles D. Hurd, personal communication.

³ W. M. Clark and H. A. Lubs, *J. Bact.*, **2** (1917), Nos. 1, 2, and 3.

adding 0.1 cc. of milk to be tested and incubating, the writer found that the time required to match the colors, and to make the necessary changes of tubes to and from the comparator, was so great that a large number of tubes could not be read each hour by the colorimetric method.

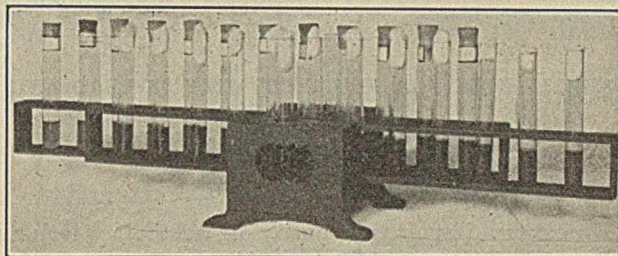


FIG. 1

To make it possible to compare a great number of unknowns with the standard tubes, the comparator of Hurwitz, Meyer and Ostenberg¹ was modified as shown in the accompanying illustrations.

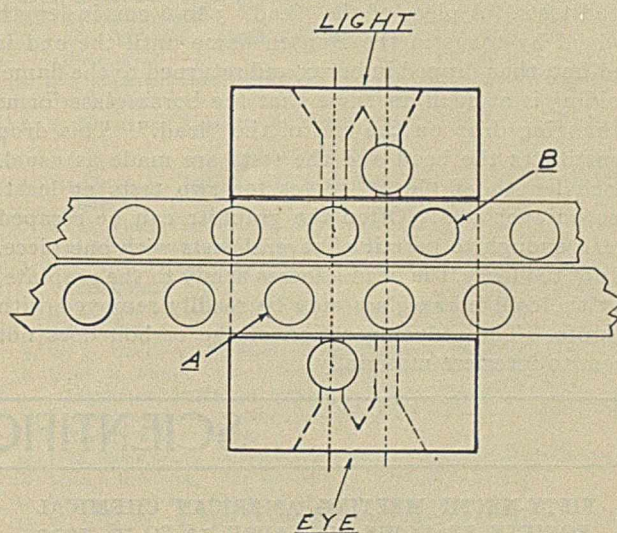


FIG. 2

Two sliding test-tube racks were constructed and so arranged in the comparator that the one carrying the standard tubes, B (Fig. 2), could be slid back and forth until a tube was found that compared in color with the first unknown in the second rack A. The second rack A carrying the unknowns, was then moved along one place and the reading of the second unknown found in the same way. The under surface of each sliding rack was indented opposite each test-tube, the indentation being designed to receive a cog supplied with a spring. By means of this arrangement the tubes stopped with a click at the proper place in the path of light as the racks were moved back and forth. A cog plate and thumb screw to match might be used to facilitate the movement of the sliding racks, but these have not been found necessary. Rack B is so constructed that it can be removed readily and replaced by other racks containing standard tubes for other ranges of P_H .

¹ Johns Hopkins Hospital, *Bulletin* **27** (1916), 16.

The readings may be made by using light directly transmitted or the comparator may be tilted by placing books under the end toward the observer and light may be reflected from a white surface through the tubes.

It is possible by using this improved comparator to examine, in a given time, many times the number of samples which could be examined by the old method.

SUBSTITUTES FOR PLATINUM WIRE IN BEAD AND FLAME TESTS

By C. C. Kiplinger

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Received January 2, 1920

One very important source of waste in platinum arises from its extensive use in the qualitative laboratory. The wire alloys with many of the metals with which it comes in contact in the several tests, becomes brittle, breaks, and soon disappears. Under these conditions, the platinum is a total loss.

To avoid this waste it has been found feasible to use a lead pencil "lead" in place of the wire for making bead tests. A piece of the "lead" 5 to 6 cm. in length is held by tongs in the Bunsen flame until the end is red-hot, then dipped in borax and returned to the flame, holding it at such an angle that the borax glass forms a hanging drop on the end of the "lead." This drop constitutes the bead and the tests are made as usual. Each length of "lead" serves for two tests at least, one on each end. Often the graphite can be scraped clean enough to permit of several tests with one piece. If not too large, the bead adheres firmly to the graphite, yet the cold borax glass may be readily removed with a knife. The reducing action of the carbon does not seem to interfere materially.

In a method described by Ehringhaus,¹ a small strip of filter paper is wet with the solution to be tested and held in the flame, or one end of the paper is placed in the solution and the other end in the flame. The writer has found the latter method very convenient if used in conjunction with the following device: A glass tube 15 cm. long and 6 mm. in internal diameter is bent at an angle of 45° 2 cm. from one end, and the latter is held in the flame until the opening is reduced to a diameter of 3 mm. to 4 mm. A strip of filter paper 4 cm. long is rolled to fit tightly the reduced opening and forced into the tube, leaving about 3 cm. projecting. The longer arm of the tube is filled with the solution to be tested, or with dilute hydrochloric acid, if a powder is to be tested on the paper, and the paper wick is inserted in the flame, holding the tube at such an angle that it feeds properly. A flame of 3 or 4 min. duration can easily be obtained. The burning carbon tends, however, to mask the color of the substances looked for. While this is of no consequence in spectroscopic tests, it is of vital importance in direct vision work. It is possible to eliminate the carbon flame partly by supporting a burner horizontally on a stand, and bringing the upper edge of the paper wick barely within the bottom portion of the flame. The salt solution is vaporized and colors the flame with a minimum consumption of paper.

A clean iron nail not less than 3 mm. in diameter will be found to be of value in these tests. Owing to the relatively large surface offered by the head of the nail, the latter seems peculiarly adapted for this purpose. The nail can be easily cleaned and burned free from sodium, and in general the spectra of the iron salts do not interfere.

¹ *Chem. Abs.*, 13 (1919), 3115.

SCIENTIFIC SOCIETIES

FIFTY-NINTH MEETING AMERICAN CHEMICAL SOCIETY, ST. LOUIS, MO., APRIL 12 TO 16, 1920 PROGRAM OF PAPERS

GENERAL SESSION

- HENRY W. KIEL, Mayor of St. Louis. Address of Welcome.
W. A. NOYES, President, American Chemical Society. Response.
PAUL W. BROWN, Editor, *America at Work*. The Physical Basis for the Economic Development of the Mississippi Valley.
CHAS. H. HERTY, Editor, *Journal of Industrial and Engineering Chemistry*. Victory and Its Responsibilities.
AMOS A. FRIES, Director, Chemical Warfare Service. Chemical Warfare.
J. H. HILDEBRAND. The Prediction of Solubility.
VICTOR LENHER. Selenium Oxochloride, a Neglected Inorganic Solvent.
E. T. WHERRY. Studying Plant Distribution with Hydrogen Ion Indicators.
G. H. A. CLOWES. Adsorption of Alkaloids.
O. H. PIERCE. The Chemical Resources of the St. Louis District.

AGRICULTURAL AND FOOD CHEMISTRY DIVISION

- C. E. COATES, *Chairman* T. J. BRYAN, *Secretary*
1. C. E. COATES. Louisiana Molasses and Sirup.
2. GEORGE DEFREN. The Use of Refined Edible Lactic Acid in Food Products.
3. H. S. GRINDLEY. Preliminary Feeding Experiments with Pigs to Determine the Nutritive Value of the Amino Acids of the Proteins of Feedingstuffs. (Lantern.)
4. C. T. DOWELL. Proteins of Pecans.
5. FRED H. SMITH. Body Fat of Hogs Fed on Peanuts.
6. J. W. REED. An Accurate and Rapid Dry Combustion Method for the Simultaneous Determination of Soil Organic Matter and Organic Carbon.

7. J. W. REED AND R. H. RIDGELL. The Actual Carbon Content of Soil Organic Matter and Its Relation to the Use of Conventional Factor.
8. W. D. RICHARDSON. Limitations of the White Rat as an Experimental Animal.
9. W. D. RICHARDSON. Mammalian vs. Avian Dietary Experiments.
10. HERBERT BAILEY AND W. D. BALDSEFEN. The Ether Insoluble Hexabromides of Pure and Adulterated Linseed Oils.
11. J. S. HUGHES AND E. F. FERRIN. The Relative Nutritive Value of Alfalfa as a Supplement to a Diet of Corn and Tankage and Kaffir and Tankage.
12. JAY BARTON. Data on Bacterial Count of Beverages in Missouri.
13. C. O. SWANSON. Hydrocyanic Acid Present in Sudan Grass.
14. C. O. SWANSON AND W. L. LATSHAW. Effect of Alfalfa on the Sulfur of the Soil in Comparison with Grain Crops.
15. L. H. ALMY AND E. FIELD. The Preservation of Fish Frozen in Chilled Brine. I—The Penetration of Salt.

BIOLOGICAL CHEMISTRY DIVISION

- R. A. GORTNER, *Chairman* A. W. DOX, *Secretary*
1. C. O. JOHNS AND H. C. WATERMAN. Some Proteins from the Georgia Velvet Bean, *Stizolobium Deeringianum*. (Lantern.)
2. C. O. JOHNS AND A. J. FINKS. The Deficiency of Cystine in Proteins of the Genus *Phaseolus*. (Lantern.)
3. P. A. KOBER. Studies on Neocarphenamines.
4. R. A. GORTNER AND GEORGE E. HOLM. On the Colorimetric Estimation of Tyrosin by Folin's Phenol Reagent.
5. GEORGE E. HOLM AND R. A. GORTNER. On the Origin of the Humin Formed by the Acid Hydrolysis of Protein. VI—The Effect of Acid Hydrolysis upon Tryptophane.
6. M. X. SULLIVAN AND R. E. STANTON. The Alkali Reserve in Pellagra.
7. S. L. JODIDI, S. C. MOULTON AND K. S. MARKLEY. The Mosaic Disease of Spinach as Characterized by its Nitrogen Constituents.

8. B. M. DUGGAR. The Effect of Conditions on the Relation of Seed Plants to H-Ion Concentration of Nutrient Solutions.
9. W. H. CHAMBERS. The Relation of Dextrose to H-Ion Concentration with *Bacillus Coli*.
10. RICHARD D. BELL AND EDWARD A. DOISY. The Determination of Small Amounts of Chlorine in Tissues.
11. H. E. PATTEN AND T. O. KELLEMS. Pectin Studies. I—Effect of Pectin on the Hydrogen Ion Concentration of Acid and of Alkaline Solutions.
12. P. A. SCHAFFER. The Oxidation of Acetoacetic Acid by Hydrogen Peroxide in the Presence of Glucose.
13. A. W. DOX AND LESTER YODER. Influence of Fermentation on the Starch Content of Experimental Silage.
14. A. D. EMMETT AND MABEL STOCKHOLM. Water-Soluble β -Vitamines: Are the Antineuritic and the Growth-Promoting Vitamines the Same?
15. A. D. EMMETT AND MARGUERITE STURTEVANT. The Fat-Soluble A. Vitamine and Xerophthalmia.
16. C. R. MOULTON. Biological Changes in the Flesh of Beef Animals during Partial Starvation.
17. J. S. HUGHES AND F. E. FOX. Scurvy in Poultry.
18. J. S. HUGHES AND J. B. FITCH. The Relation of the Vitamine Content of Feed to the Vitamine Content of Milk Produced.
19. VICTOR E. LEVINE AND EBEN J. CAREY. Studies in Embryo-Chemistry. I—The Enzymes of the Embryonic Pancreas. A. Lipase.
20. VICTOR E. LEVINE. A New Test for Sugar in the Urine.
21. EDGAR J. WITZEMANN. Disodium Phosphate as a Specific Catalyst for the Quantitative Oxidation of Glucose to Carbon Dioxide with 3 Per cent H_2O_2 .
22. HARPER F. ZOLLER. The Standardization of the Borax Solubility Test for Commercial Casein and Its Application. (Lantern.)
23. HARPER F. ZOLLER. The Precipitation of Grain Curd Casein from Pasteurized Milk Including Pasteurized Sweet Cream Buttermilk. (Lantern.)
24. MANSFIELD CLARK, HARPER F. ZOLLER, A. O. DAHLBERG AND A. C. WEIMER. Grain Curd Casein. (Lantern.)
25. HARRISON HALE. Chlorine as a "Flu" Preventive.
26. HOWARD B. LEWIS AND LUCIE E. ROOT. The Synthesis of Lysine in the Organism of the White Rat.
27. C. H. BAILEY AND A. M. GURJAR. Respiration in Cereals. II—The Respiration of Sprouted Wheat. (By title.)
28. C. H. BAILEY AND A. M. GURJAR. Respiration in Cereals. III—The Respiration of Rice Paddy and Milled Rice. (By title.)
29. C. H. BAILEY AND A. M. GURJAR. Respiration in Cereals. IV—The Respiration of Frosted Wheat Plants. (By title.)
30. C. H. BAILEY AND A. M. GURJAR. Respiration in Cereals. V—The Respiration of Wheat Plants Infected with Stem Rust. (By title.)
31. S. D. WILKINS AND R. A. DUTCHER. The Etiology of Limberneck in Fowls. (By title.)
32. S. D. WILKINS AND R. A. DUTCHER. The Relation of Vitamines to the Development of Sex Organs in Cockerels. (By title.)
33. V. E. NELSON AND A. R. LAMB. The Effect of Vitamine Deficiency on Various Species of Animals. I—The Production of Xerophthalmia in the Rabbit. (By title.)
34. E. I. FULMER, F. S. SHERWOOD AND V. E. NELSON. The Role of Vitamines in the Growth of Yeast. Are Vitamines Essential? (By title.)
35. W. P. BOVIE. A Correction of Two Previous Papers: (1) Rate of Recovery from the Action of Fluorite Rays. (2) Sensitization to Heat Due to Exposure to Light of Short Wave Lengths.
36. W. P. BOVIE. The Graphical Representation of Hydrogen Ion Concentration.
37. W. P. BOVIE. Some Notes Concerning Formol Titration of Nitrogen.
13. J. A. AMBLER. Naphthalene Sulfonic Acids. Some Difficultly Soluble Salts of Naphthalene Sulfonic Acids. (Lantern.)
14. J. A. AMBLER. A Method for the Qualitative Detection of Some Naphthalene Sulfonic Acids. (Lantern.)
15. C. E. SENSEMAN. The Preparation of Disulfonic Acids from Benzene Monosulfonic Acid. (Lantern.)
16. H. L. HALLER. Synthesis of *sym*-Xylidine. (Lantern.)
17. MAX PHILLIPS AND H. D. GIBBS. Alkali Fusions. II—The Fusion of Sodium Benzene Disulfonate with Sodium Hydroxide for the Production of Resorcinol.
18. MAX PHILLIPS AND H. D. GIBBS. A Synthesis of Thymol from *p*-Cymene. (Lantern.)
19. K. P. MONROE. The Vapor Pressure of Phthalic Anhydride. (Lantern.)
20. K. P. MONROE. A New Source of Furfural and an Investigation of the Preparation and Properties of "Furfural Green." (Lantern.)
21. ROBERT E. HUSSEY. The Present Independence of American Synthetic Dyes and How It Was Accomplished.

INDUSTRIAL AND ENGINEERING CHEMISTRY DIVISION

H. D. BATCHELOR, *Chairman*H. E. HOWE, *Secretary*

I—Symposium on Cellulose Chemistry

1. JESSIE E. MINOR. The Mechanism of the Reactions of Cellulose.
2. S. A. MAHOOD. Some Observations on the Determination of Cellulose in Wood.
3. R. C. WOODBRIDGE, JR. Nitrocellulose from Wood Pulp.
4. J. O. SMALL AND C. A. HIGGINS. Notes on the Manufacture of Nitrocellulose.
5. G. J. ESSELEN. Certain Aspects of the Chemistry of Cellulose Acetate from the Colloidal Viewpoint.
6. H. E. HOWE. Projects of the Preliminary Committee on American Cotton Research.
7. JASPER E. CRANE. Is It Advisable to Form a Section of Cellulose Chemistry?
8. L. F. HAWLEY. Some Observations on the Determination of Cellulose in Wood.
9. H. A. LEVEY. Cellulose Phthalate: Its Preparation and Properties.

II—Papers

1. CLARENCE W. BALKE. Effect of Impurities on the Metallurgy of Tungsten.
2. W. D. TURNER AND K. K. KERSHNER. Separation and Examination of the Isomers of Xylene.
3. H. L. DUNLAP AND K. K. FISCHLOWITZ. Preparation of Furfural from Corn Cobs.
4. H. L. DUNLAP AND K. K. KERSHNER. Carbonization of Missouri Cannel Coals.
5. W. D. COLLINS. Problems in Specifications for Reagent Chemicals.
6. THOS. B. FREAS. The Standardization of Laboratory Apparatus and Instruments in Respect to Quality, Shape, Size, and Packing.
7. I. N. KUGELMASS. Recovery of the Grease from the Soapy Wash Waters in Laundering.
8. I. N. KUGELMASS. A Rapid Soap Dissolving-Distributing System.
9. MERLE RANDALL. The Recovery of Iodine from Kelp.
10. VICTOR LENHNER. Selenium and Tellurium.
11. W. J. HALE. A Plan for Incentive to Research in Pure and Applied Chemistry.
12. A. M. COMFY. The Economic Status of the Chemist. (Lantern.)
13. EDWARD J. MAHIN. Crystal Growth in Bearing Metals. (Lantern.)
14. F. W. BUSHONG. The Specific Heat of Petroleum at Different Temperatures. (Lantern.)
15. D. R. SPERRY. The Application of the Filter Press to Problems in Chemical Industry.
16. D. T. SHAW. Chemical Corrosion.
17. W. D. RICHARDSON. The Solution of Metal in Acids as Related to Corrosion.
18. W. D. RICHARDSON. A Suggested Basis for an Index of Corrosion for Iron and Steel.
19. W. D. RICHARDSON. A New Type of Catalyzer for the Hydrogenation of Oils and Other Purposes.
20. F. C. VILBRANDT, S. L. SHENEFIELD AND JAMES R. WITHROW. Sulfites as Standards for Oxidizing Reagents.
21. D. C. MAPES. The Crystalline Structure of Paraffin Wax.
22. C. K. FRANCIS. Mid-Continent Gasoline.
23. J. CULVER HARTZELL. The Relation of Chemistry (Analytical and Thermal) to the Fabrication of Steel.
24. J. CULVER HARTZELL. The Relation of the Electric Furnace to the Fabrication of Carbon and Alloy Steels, with Special Reference to the Chemical and Physical Changes Produced.
25. O. L. BARNEBEY. Industrial Uses of Activated Charcoal.
26. EDWARD J. MAHIN. Inclusions and Ferrite Crystallization in Steel: II—Solubility of Inclusions.
27. HERBERT BAILEY AND C. B. EDWARDS. The Determination of the Specific Heat of Heavy Mineral Oils.
28. W. C. HOLMES. The Freezing Points of Mixtures of Sulfuric and Nitric Acids. (By title.)

DYE SECTION

CHARLES L. REESE, *Chairman*R. NORRIS SHREVE, *Secretary*

1. S. A. TUCKER. Dye Patents of the Chemical Foundation, Inc.
2. W. F. MEGGERS AND F. J. STIMSON. Dyes for Photographic Sensitizing. (Lantern.)
3. J. F. RAGSDALE. Anthranilic Acid, Purity and Tests of the Commercial Product.
4. L. A. WATT. Ortho-chlor-para-toluol Sulfonate, Purity and Tests of the Commercial Product.
5. LAUNCELOT W. ANDREWS. Uses of Formic and Oxalic Acids in the Dyeing Industry.
6. GEORGE HEYL. Synthetic Dyes as Applied to Chemicotherapy and Microscopy.
7. ROBERT E. ROSE. Dye Research.
8. I. M. BERNSTEIN. The Hydrogen Exponent in the Classification of Indicators and Some of Its Applications.
9. F. L. ENGLISH. Application of $TiCl_3$ in Analysis of Intermediates.
10. C. L. KNOWLES. Some Physical Constants of Pure Aniline. (By abstract.)
11. E. Q. ADAMS. The Absorption Spectra of the Nitric Esters of Glycerol. (Lantern.)
12. L. A. MIKESKA. Tetramethylquinolines. (Lantern.)

The scientific business of the meeting began with an address by the retiring president, Prof. Bancroft, on "Contact Catalysis," which reviewed thoroughly the history and present status of this knotty problem. It will be found intensely interesting by everyone seeking light on these little understood phenomena. This was followed by a "Symposium on Colloid Chemistry," which contained some notable contributions. Hulett and Nelson, in a long and brilliant paper, brought forward experimental proof that so-called graphitic acid is a colloidal oxide of carbon. They assign it the formula C_4O or $C_{11}O_3$, and describe its properties. Selvig and Ratliff, of the Bureau of Mines (Pittsburgh), discussed the analytical determination of graphitic carbon in various materials. W. C. Moore described examples of selective adsorption by the bacteria *B. pyocyaneus*—an essentially colloidal action. W. A. Deane, of the Dorr Concentrator Company, discussed at length "settling" problems, showing how they involved the detailed utilization and application of all the information we possess about the principles of colloid chemistry; in fact, the practical observations of Mr. Deane go far to explain some so-far unexplained colloid phenomena, such as the stratification of settling solutions. Mr. Deane has made a splendid contribution to applied colloidal chemistry. The other papers presented at this session were: Goodwin and Knobel, on the effect which alternating current, superposed on direct current, has on the over-voltage of hydrogen; Holler and Ritchie, on the concentration of hydrogen ions in dry cells, and its effect on the voltage of the cells; MacInnes, Adler and Joubert on the exact chemical reactions taking place in the lead accumulator, in which they proved that no higher oxide than PbO_2 is formed; Wm. R. Mott, on flame colorations and condensed coatings obtained by introducing substances into the arc. The last paper dealt with several new methods of detection of elements, particularly the rarer ones, and is highly commended as rich in new analytical data of wide practical application.

After lunch, taken in the Walker Memorial Building at the new Institute of Technology group in Cambridge, the afternoon session was held in one of the numerous fine lecture rooms, the particular one selected happening to have "Faraday" inscribed over its portal. With an assembly which taxed the capacity of the hall, many students of "Tech" and Harvard being present, the following papers were read and discussed: W. S. Landis, on the new cyanide formed by fusion of calcium cyanamide with salt and some calcium carbide, in a quick-melting electric furnace, forming a mixture of calcium chloride and sodium cyanide (25 to 40 per cent of the latter), which is sold and used for its cyanide content only; C. J. Rottman, on exact and quick determination of carbon in ferro-alloys—a difficult analytical problem, and T. D. Yensen, on a new method of determining carbon in iron, consisting in condensing the carbon dioxide produced in liquid air and afterwards evaporating the solidified carbon dioxide in an evacuated space, the pressure produced allowing determination of the carbon in the specimen to 0.001 per cent; L. T. Richardson, on the influence of enclosed slag on causing corrosion of iron, showing that it has practically no influence; Langdon and Grossman, on the effect of sand-blast cleaning and acid pickling on the properties of steel, showing a decided decrease of its resistance to rapid alternating stresses; C. H. Brace, on the details of producing calcium electrolytically on a commercial scale, in a special form of fused-salt electrolyzing apparatus; G. J. Sargent, on electrolytic chromium plating, showing how deposits up to one centimeter thickness have been obtained; Mathers and Sowder, on bronze plating, describing two baths which work industrially; Hartman, Hougén and Kobler, of the Carborundum Company, on the resistance of twelve different special refractory materials to spalling, as tested by spraying when hot with cold water, and to abrasion, as tested when hot by grinding against a cold carborundum wheel. Several of the above papers led to lively discussion, particularly that on the corrosion of iron.

After this session, many of the visitors were guided through the magnificent new buildings of the Institute, while a considerable number stayed behind discussing with the Electric Furnace Association the training of men for running electric furnaces. This topic was discussed with great fervor until it was time to adjourn for dinner.

In the evening, Arthur D. Little, Inc., was host to the Society at its model industrial research laboratories in Cambridge. The museums, library, laboratories and semi-commercial plant in the basement were admired by a gathering which literally filled the building. Later, there were speeches and singing and a good deal of fun, with unlimited smokes, and Professor Armstrong, of the Institute, entertained most charmingly with a pictorial account of hunting with a camera in the wilds of New Brunswick. He delighted and enthused his audience by his breezy account of a most fascinating summer vacation.

Friday was set for joint sessions with the American Institute of Electrical Engineers. The program was a morning session with a Symposium on Electrically Produced Alloys, an afternoon trip to the General Electric Works at Lynn, an informal subscription dinner, followed by an evening session with a Symposium on Power for Electrochemical Purposes. President Bancroft was chairman at the morning session, and President Townley of the Electrical Engineers, in the evening. The 250 electrochemists, together with about 150 electrical engineers, crowded the meeting place—the Swiss Room of the Copley Plaza Hotel—to its full capacity, discussion was active, and the occasion was indeed a notable one.

At the morning session, in the Symposium on Electrically Produced Alloys, the following papers were read and discussed: H. E. Howe, a review of the fundamental problems in alloys-research, particularly reciting the physical and chemical data which are needed for thoroughly studying alloys and alloying phenomena; R. J. Anderson and C. B. Gibson each discussed the broad outlines of the ferro-alloy industry, its rise and present commercial importance, and the technique of the general problem of producing ferro-alloys; B. D. Saklatwalla described the ferrovanadium industry, giving occasion for some pungent discussion of the limits of supply of vanadium and its commercial exploitation; F. A. Raven described the physical properties and chemical uses of ferrosilicon; E. S. Bardwell, ferromanganese; Alcan Hirsch, ferrocerium; R. M. Major, nickel chromium and similar alloys; R. C. McKenna, on producing high tungsten high-speed steel in the electric furnace, resulting in considerable discussion of the best form of electric furnace for getting a good mixture of these slowly alloying metals; E. Haynes, on cobalt-chromium alloys, known as stellite, used as high-speed cutting tools and for surgical instruments; T. D. Yensen, on the magnetic and electric properties of iron-nickel alloys; Hunter and Bacon on similar properties of iron-titanium alloys. Finally, C. F. Harding related his experiments in fixing the nitrogen of the air by a silent discharge process which is at present of very low efficiency (one per cent) but which contains the promise of better things.

The trip to Lynn was made in eight electric cars, directly from and returning to Copley Square, both societies being invited guests of the General Electric Company. A printed itinerary gave all the details of the visit with great accuracy, and it was carried out according to plan with military precision. While duly impressed by the magnitude of the Lynn plants, some of the more critical visitors were not as favorably impressed by the details of execution of the work in some parts of the plants; the Heroult electric steel furnace, however, appeared to be casting steel dead—melted "to the queen's taste;" this operation was given unstinted commendation.

The informal dinner was attended by about a hundred and fifty, and was graced by the presence of our life-long friend, Professor Elihu Thompson.

The evening session was almost a crush. The Symposium.

on Power for Electrochemical Purposes was calculated to draw out every electrical engineer and every electrochemist within possible reach of Boston. Chairman Townley divided the program into three groups, *viz.*, papers treating on the general question, discussion of specific electric furnace requirements, and presentation of the advantages of specific power sites. In the first class came the following: J. L. Harper, discussing the general requirements of power by electrochemical industries of various kinds; E. A. Wilcox, on the central station man's view of electrochemical customers and their requirements; A. Smith, on abuse of power demands by electric furnace operators, in particular the necessity of controlling excessive surges on the lines. In the second class, H. L. Hess discussed the requirements of electric steel furnace plants; H. A. Winne the use of reactors on electric furnace circuits to reduce the momentary surges or peaks in the power demands; J. A. Seede, on automatic controllers for regulating arc furnace electrodes so as to stabilize the current. In the third class of papers, C. T. Maynard described the power available at Rumford Falls, Maine, only two hundred miles from Boston; F. F. Fowler, the water powers of the Pacific Coast, developed and potential; J. W. Beckman, the power developments of Norway and Sweden, showing how far these countries have out-distanced the United States in developing their power for electrochemical industries.

The Saturday morning session in the new Lecture Hall at Harvard University was lively and interesting. Professor T. W. Richards—"Atomic Weights" Richards—gave the visitors a hearty greeting. The proceedings included the following: J. W. Richards—"Metallurgical Calculations" Richards—described the Söderberg self-baking, continuous electrode, which is already extensively used in Europe and is being installed on some ferrosilicon furnaces at Anniston, Alabama; C. A. Keller's paper on synthetic, electric-furnace pig iron, described the great importance this product attained in France during the war, in utilizing steel turnings and producing cast-iron shells; E. F. Kern described experiments on reducing manganiferous silicate slags in an electric furnace to silico-spiegel; P. B. Short, a step-induction regulator of new form for electric furnaces;

C. J. Weed, the general application of the electric furnace to metallurgy; L. B. Lindemuth described in detail the position of the electric furnace in manufacturing steel, concluding that it is the most flexible and most generally useful of all steel-making apparatus; H. M. St. John, on the evolution of the electric brass furnace. Some statements in the last paper as to advantages and power required for melting brass were questioned by Mr. Winder, giving rise to a very lively interchange of opinion between Messrs. Gillette, Baily, FitzGerald, Hering, Richards, and others, which, in general, substantiated the statements in the paper.

After the session, Professor Richards invited those present to inspect the Gibbs Research Laboratory, which opportunity was eagerly accepted. Lunch followed at the Harvard Union, and then a visit to the Huff Electrostatic Separator plant at Arlington closed the program. The visitors found the last item very instructive and interesting.

Thus Boston and the Electrochemical Society came together for the third time, to their mutual profit and advantage.

LEHIGH UNIVERSITY
SOUTH BETHLEHEM, PA.
April 13, 1920

J. W. RICHARDS

CALENDAR OF MEETINGS

- American Pharmaceutical Association—Annual Convention, Washington, D. C., May 6 to 8, 1920.
- Society of Cotton Product Analysts—11th Annual Convention, Grunewald Hotel, New Orleans, La., May 10 and 11, 1920.
- National Foreign Trade Council—Convention, San Francisco, Cal., May 12 to 15, 1920.
- National Fertilizer Association—27th Annual Convention, White Sulphur Springs, W. Va., week of June 21, 1920.
- American Institute of Chemical Engineers—Semi-Annual Meeting, Montreal, June 21 and 22; Ottawa, June 23; Shawinigan, June 24 and 25; La Tuque, June 26, 1920.
- Sixth National Exposition of Chemical Industries—Grand Central Palace, New York, N. Y., September 20 to 25, 1920.

NOTES AND CORRESPONDENCE

FRENCH GAS WARFARE¹

Little by little the mysteries of the great battle are being cleared up. Soon all the secrets will be known. To-day those of the murderous gas warfare are disclosed by Colonel Vinet, head of the office of chemical research at the Ministry of War.

Our enemies first used this barbarous weapon on April 22, 1915, on the Belgian front between Bixchoote and Langemark. At five o'clock in the evening, a heavy cloud of greenish vapor appeared over the German trenches and, pushed along by a light wind, covered the French lines. The assaulting waves followed. An entire division of the tenth army was overcome. Our men were seized with violent coughing fits and suffocated. Many held firm but paid for their resistance with their lives.

Germany had once more flagrantly violated the laws of war. On July 22, 1899, at the Hague she had indeed signed the international convention prohibiting combatants to "spread asphyxiating or poisonous gases."

This attack most evidently took us by surprise. We had to improvise everything. The task was pressing. We had first of all to seek the means to protect our soldiers against these poisons, then give them the means to retaliate.

M. Kling, director of the Municipal Laboratory of the City of Paris, was hurriedly called to the front, and after a quick survey gave the preliminary instructions with decided farsightedness. The means at hand had to be used—wads of

¹ Translation of an article by Lucien Chassaing in *Le Journal*, Paris, February 24, 1920.

cotton saturated with hyposulfite of soda, ordinary goggles to protect the eyes. An appeal was then sent to the chemists and the work was soon organized.

A commission for chemical research was created, happily realizing the importance of its role, and jostling a little the established bureaucrats who were a little wary of these things which they had not foreseen. Every source was utilized, and the young school of French chemistry showed itself equal to its task. The Pasteur Institute, the School of Pharmacy, the Sorbonne, the College of France, the Conservatory of Arts and Trades, the Faculty of Medicine, all cooperated with ardor in the necessary research work.

Three sections were established: Studies on the front, directed by M. Kling; studies of means of offense, directed by Professor Mouren; and studies of means of protection, directed by Professor Vincent. Intimate relations were also established with general headquarters, which had started a gas warfare service, and had nominated "officers, army chemists." In 1917 this service became interallied, and soon we were ready for both attack and defense.

Germany, in April 1915, had employed simple liquid chlorine escaping from metallic cylinders in which it had been compressed. In June, bromine first put in its appearance, being used in projectiles fired from mine throwers. It was from this time on that gases were used in artillery projectiles. The first serious use was in the Argonne, at the Bois de la Gruerie. Successively, new compounds, of which formaldehyde and chlorine

were generally the bases, were used by the enemy. A salvage service allowed us to make investigations very quickly so as to determine the exact nature of these products, their physiological action, and to deduce the therapeutics and the means of effective protection.

These last arrived at a fine degree of perfection. Our soldiers, wearing their masks, could in the last months of the war remain without inconvenience several hours in an atmosphere containing a high percentage of poisonous gases. Horses, dogs, and carrier pigeons, all had their protective devices.

From September 1915 to September 1918, 12,400,000 masks of different models were manufactured and delivered. From August 1915 to April 1916, 10,000,000 pairs of goggles were also delivered.

The effort to find means to retaliate was no less active. Beginning in September 1915, during the Champagne offensive, we used again the German shells loaded with tetrachlorosulfide of carbon, the only product which we had in sufficient quantities.

Messrs. Urbain and Lebeau then invented a gas with hydrocyanic acid base, calling it "Vincennite," which produced instant mortal action, but which our chiefs through an excess of humanitarian scruples, which were indeed superfluous, decided not to employ. The same decision was arrived at for phosgene, used in the beginning of the Battle of Verdun in February 1916, and which produced a panic which is recorded in the German orders of the day.

The chemists, not discouraged, then turned their efforts towards other products of chlorine or formaldehyde. Finally the choice fell on a product which the Germans had first used in July 1917 at the Battle of Ypres, and which was called for that reason "Yperite," and which is chemically a sulfide of dichloroethyl. This possesses both toxic and blistering qualities.

The manufacture, despite the great difficulties and the dangers which it entailed, was pushed fast. Eleven factories made the necessary chlorine, and up to the armistice had produced 24,000 tons. In the seven months, from April to November 1918, 2,172,334 shells for the "75," 90,810 for the "105," and 142,035 for the "155" had been loaded with yperite.

In all, there had been loaded and fired against the enemy 13,193,000 "75" shells, 3,930,000 heavy shells and trench bombs, and 1,140,000 grenades loaded with gas. We have, besides, ceded 8,000 tons of asphyxiating gases and over a million loaded projectiles to our allies.

These figures are enough to show what a formidable effort was made by our French scientists and manufacturers, an effort of which they may be justly proud, and how well merited was the glorious citation given by Marshal Petain to the chemical services in the war.

CHEMICAL PREPAREDNESS

Editor of the Journal of Industrial and Engineering Chemistry:

Some time ago you gave a talk before some of the chemists of our company assembled in the library. At that time it occurred to me that in spite of receiving encouragement from such men as yourself to keep up our good work, there has been one phase of the general subject of patriotic protective activities not generally considered.

It is doubtless true that as a body of highly trained scientists the American chemists can easily adapt themselves to possible war-time activities, but most of us are really very far from knowing just what we can best do in an emergency. What a chaotic condition is bound to follow when a few thousand men are all suddenly attempting to produce something new and to learn how to do it at the same time!

To parallel the general agitation for universal military training, why should not the AMERICAN CHEMICAL SOCIETY and the large chemical industries get behind the development of a val-

uable system of peace-time education of their chemists for the pursuit of definite, coördinated and specialized activities in times of possible war? How many of us know anything definite about the manufacture of phenol, picric acid, ethylene, mustard gas, formaldehyde, acetone or wood alcohol, nitrogen fixation, or about large-scale work upon such fundamental processes as nitration, sulfonation, etc., to say nothing of many other technical processes, doubtless of great war-time importance?

It has occurred to me that the American chemical industry might in part repay the country for its many recent advancements if in peace-time in connection with the Government it should adopt a policy of lending its chemists two weeks each year for the purpose of pursuing special studies in "chemical war technology." A so-called "Chemical War College" would in fact be more of a university with its various specializing "colleges" made up of the laboratories and plants of actually functioning industrial plants in various parts of the country. If the chemists were to be loaned the Government then the Government might pay traveling expenses and any peculiar living expenses necessarily incurred by the "war technology" student during his course of training. It would not be many years before we would have in this country an army of chemists each of whom would know exactly what his duty should be, where he should go, with what he would have to work, and how to go about his task in any emergency. These men should be classified and their names and records kept on file, each being subject to emergency call for technical or scientific service at proper salary.

Would it not be worth while to give this matter some discussion in the columns of the JOURNAL?

NATIONAL ANILINE & CHEMICAL CO.
BUFFALO, N. Y., April 3, 1920

C. A. LYFORD

DECENNIAL CELEBRATION FOR THE FOREST PRODUCTS LABORATORY

The Madison Association of Commerce and Forest Products Laboratory have made preliminary arrangements for the celebration at Madison of the tenth anniversary of the opening of the Laboratory. Present plans call for a two-day meeting during the latter part of June, with addresses by men of national reputation in science and industry, a banquet, tours of inspection through the laboratory, informal discussions, and various forms of entertainment. Detailed arrangements will be announced later.

The Forest Products Laboratory is a branch of the United States Forest Service, established in 1910 in coöperation with the University of Wisconsin, and is a consolidation of a number of testing laboratories and other units of the Forest Service which had been located at various points throughout the United States. It is engaged principally in industrial research on problems connected with the manufacture and use of forest products, including beside lumber, posts, poles, ties, and similar products, pulp and paper, naval stores, hardwood and softwood distillation products, and other chemicals and pharmaceuticals. At the present time the laboratory employs about 200 people and occupies five buildings in whole or in part.

AMERICAN FELLOWSHIPS IN FRENCH UNIVERSITIES

The Society for American Fellowships in French Universities offers each year for open competition among graduates of American colleges and other suitably qualified candidates, 25 fellowships to encourage advanced study and research in French universities. Fellowships of the annual value of \$1,000 are granted for one year and are renewable for a second year. Application blanks and further information may be obtained from the Secretary, Dr. I. L. Kandel, 576 Fifth Ave., New York City.

FIRE CAUSED BY YELLOW PHOSPHORUS

Editor of the Journal of Industrial and Engineering Chemistry:

Recently two notes appeared in your JOURNAL about fires caused by yellow phosphorus. In this connection I wish to relate a similar occurrence in our building. All stock chemicals of combustible nature are kept in a fireproof vault which is arranged with shelves made of concrete. The vault is heated with a steam radiator regulated to 55° F. with a thermoregulator. On the evening of February 12, 1920, the steam pipe in the vault burst and the escaping steam melted the yellow phosphorus in the cans and bottles. One bottle containing 3 lbs. of phosphorus broke and the molten phosphorus being on the top shelf, dripped

down and ignited. The fire caused only slight damage as it was soon extinguished with sand.

Whenever yellow phosphorus is received in cans it should be immediately transferred to glass-stoppered bottles containing water. Such glass containers should be placed in pails or other receptacles containing water and these in turn imbedded in sand. The container should always be kept on the floor and never on shelves. Periodic inspection of combustible substances of this nature will help to avoid future fires.

ALEXANDER LOWY

SCHOOL OF CHEMISTRY
UNIVERSITY OF PITTSBURGH
PITTSBURGH, PA., March 18, 1920

WASHINGTON LETTER

By J. B. McDONNELL, Union Trust Building, Washington, D. C.

Caught in the cross currents of politics, filibuster and straight out-and-out opposition to its provisions, the dye bill is still before the Senate awaiting action.

The last month has seen but few favorable developments of importance in matters of keen interest to the chemical world which are pending in Washington. The delay in action on the dye bill promises to be lengthy and the chances of failure to get action this session are growing. The army reorganization bill, appropriation bills, peace resolution, and perhaps the soldier bonus measures will all get right of way in the Senate over the dye bill, under present plans.

An effort may be made by Senator Watson to get the measure up within the near future, but whether he will try sufficiently hard to succeed will depend largely upon the result of a conference which he is to have with Senator Penrose, chairman of the Finance Committee, at the latter's home in Philadelphia in the near future. Senator Penrose, who has been absent for several months in Florida because of ill health and only recently returned to Philadelphia, is a high protectionist and, of course, in favor of protecting the dye industry, but he has not given any opinion as to the merits of the measure the committee reported out. It is to get his support of the committee measure and have him exert his very powerful influence on the Senate to remove much of the obstruction tactics that the conference has been decided on.

A peculiar situation has developed among the Republicans. Western senators, through a filibuster which showed itself on the floor when the bill enjoyed its fleeting consideration of a day, just prior to the taking up of the Treaty by the Senate for the second time, have succeeded in forcing the Finance Committee to report out tariff measures it had been determined to "smother" for this session. Since then, they have been at work trying to assure action on the floor on these measures and have used obstructionist tactics against the dye bill as a means of bringing pressure to win their point.

Several amendments to the bill are pending. The United States Tariff Commission in a report made to the Finance Committee on the Moses amendment criticizes it and states that it is unworkable in certain of its provisions. The Moses amendment would eliminate the embargo provisions of the committee bill and substitute therefor a system of special tariffs. It is practically an anti-dumping measure. Senator Keyes, also of New Hampshire, has introduced four amendments to the committee bill, upon the assumption, however, that the bill will be adopted without very material changes. His amendments are designed to afford more certain and greater protection to the dye consumer by changing the phraseology of the bill in places.

Senator Thomas, Democrat, of Colorado, has proposed as an amendment to the dye bill a measure imposing a tax equal to one hundred per cent on all political contributions of more than a thousand dollars. He has attempted to get action on this measure several times without success. He has been accused of indulging himself in sarcastic humor in this move. Needless to say, the dye bill has been the target of many charges hurled by opponents, who charge that possible campaign contributions have had the effect of enlisting supporters. It is also needless to say that charges of "lobby" and the crew of additional charges and innuendoes that usually accompanies the first, have been made in connection with other tariff measures pending, few of them escaping.

Favorable report also has been made by the Finance Committee on Senator Smoot's anti-dumping measure. It has been sug-

gested that this be made an amendment to the dye bill, but plans were changed and it was reported as a separate measure. While it is not proposed as a substitute for any section of the dye bill, it is interesting to note that Senator Smoot several times has expressed the opinion that anti-dumping legislation, supplemental to a high protective tariff, would be adequate to protect the dye industry. Although not an ardent supporter of the committee bill, Senator Smoot is not radically opposed to it, however, and will support it when it comes to a vote, according to those in charge of the bill.

As the situation stands at the present time the outlook for action on the bill in the near future is dark; while chances of action on the laboratory and scientific instruments bill are considerably less at this session. The same is true of other tariff measures. Only strong pressure upon members of the Senate to convince them of the necessity of enacting legislation to protect the industry can bring any action, unless the Penrose-Watson conference results in the strong support of the former. There are many senators who would vote for the bill if brought up for action, but the fact that we still are technically at war, with war-time restrictions in force, has been a potent argument in the hands of those Republicans who are opposed to any tariff legislation until after election.

The Senate at the present time is engaged in consideration of the army reorganization measure, which was passed recently by the House. Establishment of the Chemical Warfare Service as a separate department of the Army was provided for in the bill as it passed the House, and although the Senate has not yet acted finally on the provisions of the bill, it also will accord to this important warfare machinery the recognition it should have by making it a separate department, instead of placing it as a sub-division of another department as was proposed by General March, Chief of Staff.

Following hearings which brought scientific and technical men from all over the country to testify before the committee, the House passed the Nolan bill providing for reorganization of the United States Patent Office. This measure is now pending in the Senate Patents Committee of which Senator Norris is chairman. The bill provides for a general reorganization of the patent office, and increases the pay of the staff so as to prevent complete disintegration through resignations. Hearings were held on the bill on April 8, but no action was taken by the committee at that time, because of lack of a quorum. Senator Norris is now in Nebraska and is expected to return here the latter part of April. Present plans are for the prompt calling of the committee and an immediate favorable report to the Senate, where it is proposed to press it for passage at this session.

St. John Perret, chief of the War Trade Board Section of the Department of State, has left here and is now in Europe, where he is acting as the representative of the United States in the allocation of the German dyestuffs through the allied commissions provided for in the Treaty.

Latest official advices are to the effect that there is a prospect of the dock strike, which has tied up shipping at Amsterdam and Rotterdam for many weeks, being broken. Officials state that practically all of the dyes obtained from Germany through the reparations commission and under the option obtained by Dr. Charles H. Herty, from the German chemical cartel, are now in Rotterdam awaiting shipment. They have been there for several weeks, arriving just about the time the strike was begun. Shipping has been paralyzed however and it has been found impossible to move them. Once the strike is ended the barrier will be removed and the dyes will come forward to this

country. Only a small percentage of the dyes were returned to the German factories because of failure to come up to the standards required, advices state.

In its program of economy, the keynote of which has been cutting of appropriations allowed Government departments, Congress has severely cut the appropriations allowed the Bureau of Standards. The House, in cutting the estimates presented

by the House, pruned so well that the appropriation allowed is less than sufficient to maintain the present work of the Bureau. The Senate refused to materially increase the House appropriations. This will mean that the Bureau cannot carry out some of the plans for extending the work and in some instances must cease work it is now doing.

April 14, 1920

INDUSTRIAL NOTES

The Bureau of Mines reports the results of investigations on electric sparking in mines from lightning. A remarkable explosion of fire-damp was recently caused by a discharge of lightning at the Sitalpur coal mine, India, and numerous accidents have been caused by lightning entering by shot-firing wires. Suggestions are made as to precautions to be taken in wiring, in order to prevent such accidents.

Cibucaco wood, which is imported from the Philippines to China, is largely used in the preparation of dyes for coloring cheap papers such as those used in the manufacture of fire crackers and cheap stationery. The Chinese process of extracting the dye is very crude and wasteful, but the business is closely controlled by a strong syndicate which regulates the value of the manufacture and the price of the product.

The Bureau of Mines report on Petroleum Refineries in the United States indicates that the petroleum refinery capacity of the United States is considerably overbuilt. Refineries now have a total rated capacity over 50 per cent in excess of the refinable oil supplies, and when refineries now being constructed are completed the excess may reach 80 per cent. In 1919 the oil runs were 361,520,153 barrels of crude oil, or 990,466 barrels per day.

The attar of rose production by Bulgaria has shown a heavy drop since the First Balkan War in 1912, the production falling from more than 126,800 oz. in 1912 to 52,000 oz. in 1919, with a probable further decrease in 1920. The acreage planted to roses has decreased about 25 per cent in this time. During the war large stocks remained unsold in Bulgaria until early in 1919, when a considerable part of the stock was sent to the United States in exchange for flour. It is estimated that the present available stock in Bulgaria hardly exceeds 50,000 ounces.

A joint Army-Navy Board has been created by the Secretary of War and the Secretary of the Navy for the purpose of coordinating the various helium gas activities. The Bureau of Mines of the Department of the Interior has been invited to have a representative meet with this board.

Representative Graham of Illinois, chairman of the Committee on War Expenditures, which is investigating the government's nitrate and explosive manufacturing operations, recently told the House that the Nitro explosive plant near Charleston, W. Va., which cost the Government \$70,000,000, has been old to the Charleston Improvement Co. for \$8,550,000, payable in installments in about 10 years. The company, Representative Graham said, is holding the property for the purpose of investment and speculation.

The Far Eastern Division of the Bureau of Foreign and Domestic Commerce reports that the outlook for American dyes in Japan is good. American dyes do not suffer in manufacture on account of the humidity of the climate, which prevents effective crystallization in the dye products of some countries.

Tests made years ago at the Forest Products Laboratory indicated that sodium fluoride might be successfully used as a wood preservative because it had high toxicity, was not injurious to metal, and was convenient to handle. Tests extending over 5 years on sap-pine ties used in a mine in Birmingham, Alabama, and on red oak ties in the tracks of the Baltimore and Ohio Railway Company show that ties treated with sodium fluoride remain in as good condition as those treated with zinc chloride, both showing very little deterioration. Ties treated with creosote were in still better condition, while untreated ties were in various advanced stages of decay.

The Senate has passed the bill authorizing the Federal Trade Commission to administer inventions for public welfare.

The National Research Council has received a gift of \$10,000 from the Southern Pine Association to pay for the incidental expenses of a scientific study by a number of investigators, under the advice of the Research Council's special committee on forestry, of the re-growth of trees or cut-over forest lands, with the aim of determining the best forestry methods for obtaining the highest productivity.

The United States Civil Service Commission announces a competitive examination for analyst in the Bureau of Mines, Pittsburgh, Pa., at \$1,200 to \$1,500 a year, open to both men and women. Ability to make routine analyses of coal, calorimetric experiments, and analyses of ores is required. Applicants must be high school graduates and have had three years' analytical experience in a chemical laboratory, or have had three years' college training which included quantitative analysis. Applications must be filed with the Civil Service Commission, Washington, D. C., on or before May 11, 1920.

The United States Civil Service Commission announces an examination for metallurgist at the Engineering Experiment Station, Naval Academy, Annapolis, Md., at \$7.60 per day. Applicants must be college graduates who have majored in chemistry, engineering or metallurgy, and have had one year's experience in the use of the microscope in the examination of metals. Applications must be filed with the Civil Service Commission, Washington, D. C., on or before May 11, 1920.

The United States Civil Service Commission has announced an examination for surveillance inspector in the Ordnance Department at Large, at \$1,600 to \$2,400 a year. Appointees whose services are satisfactory may be allowed the temporary increase of \$20 a month. The duties of appointees will be general supervision of storage and testing of smokeless powder, high explosives and loaded ammunition, making stability tests on smokeless powders and high explosives, and inspection of explosives in storage. Graduation from a standard high school and two years of experience with smokeless powder and explosives are required. Applications must be filed with the United States Civil Service Commission, Washington, D. C., by May 11, 1920.

The specifications recommended by the Inter-Departmental Committee for the Standardization of Paint Specification in the purchase of paint for government use show two significant features: the recognition by paint technologists of the increasing importance of zinc oxide, and the latitude of the specifications. Less than ten years ago no zinc oxide was used by the Government except in the navy and lighthouse service, while now battleship gray pigment is 60 per cent zinc oxide.

The *Technical Review* describes investigations recently made in Switzerland on the use of acetylene as a motive power for automobiles. Two-stroke, four-stroke and Diesel engines were used, with a mixing valve which allows for the variation of the amount of air to be added to the gas. Comparisons of a motor using gasoline and acetylene are given. With acetylene the motor is less liable to overheat and the cylinders and valves keep unusually clean. Oil consumption is heavy when pure acetylene gas is used, but if a small amount of warm water is drawn into the cylinders in the form of a spray, the consumption is less than with gasoline.

Sixteen new companies were chartered during the month of February 1920 for the production and distribution of chemicals, with a total capitalization of more than twelve and a half millions.

The Bureau of Mines has issued a report on the motor gasoline situation, embodying the results of investigations of the motor fuel supply of the United States. The number of automobiles in service increased about 1700 per cent from 1909 to 1918, while the production of gasoline increased 560 per cent, and the production of crude oil only 95 per cent. Because of the great demand for kerosene, it is unlikely that the quantity of gasoline can be increased by making it less volatile than now. The extraction of gasoline from natural gas is approaching its maximum. The supply of fuel oil from which gasoline may be obtained by cracking is sufficiently large, although the demand for fuel oil has increased rapidly and the price has more than doubled, but the process of cracking is expensive and would be unprofitable without a further increase in the price of gasoline. The use of alcohol as a motor fuel will probably increase, as well as the use of benzene produced from coal tar. The solution of the problem will probably be found in the designing of an automobile engine which will use fuel oil satisfactorily, since this will be the most efficient way of using fuel oil, and the supply of motor fuel will be limited only by the total supply of fuel oil.

The Bureau of Mines, Department of the Interior, has recently issued a complete statement of coal-mine fatalities in the United States during 1919. The report shows a reduction of 10.58 per cent in fatalities as compared with 1918, although there was an increase in accidents due to gas and dust explosions. While the actual number of fatalities is considerably less than in 1918, the ratio on a tonnage basis is slightly higher.

The Bureau of Crop Estimates, Department of Agriculture, reports the area of winter wheat planted in foreign lands as follows: Spain, 9,511,000 acres, or 109 per cent of the 5 year average from 1914 to 1918; France, 11,369,000 acres, or 89.5 per cent of the 5 year average; Roumania 1,321,000 acres, or 28.1 per cent of the 5 year average; British India, 27,429,000 acres, or 85.9 per cent of the 5 year average. The condition of cereals is good.

A National First-Aid and Mine-Rescue Contest will be held under the auspices of the Bureau of Mines, Department of the Interior, at Denver, Colorado, August 20 and 21, with a view toward stimulating the safety movement among the mines and metallurgical plants of the country. Teams from the principal mining companies of the country will participate in the contest for the national championship in first-aid and mine-rescue work, and cups, medals and prizes will be awarded to the winners. Arrangements are being made to hold at the same time and place the annual meetings of the Rocky Mountain Coal Mining Institute, the Colorado Metal Mining Association, the American Mining Congress and other mining associations. Mr. D. J. Parker, of the U. S. Bureau of Mines, Pittsburgh, Pa., is in charge of the arrangements for the contests.

The Bureau of Mines has prepared a report on diatomaceous or infusorial earth, showing its uses, method of preparation, chemical composition of various samples, and distribution of deposits in the United States. Contrary to the general impression, diatomaceous earth is not confined to the western part of the United States, but is well distributed in many eastern states.

Westinghouse, Church, Kerr & Co., Inc., engineers and constructors, have recently been merged with Dwight P. Robinson & Co., Inc., constructing and consulting engineers, of New York City. The new company which will be called Dwight P. Robinson and Co., Inc., will have executive offices at 61 Broadway, New York City.

The American-Scandinavian Foundation has established twenty traveling fellowships, each with a stipend of \$1,000 to \$1,200, to be awarded to men and women of American birth for technological research and humanistic study in the Universities of Sweden, Denmark, and Norway. Ten students will be sent to Sweden, and five each to Denmark and Norway, for a period of study extending over one academic year. Selection of the fellows will be made by a jury of university professors and technical experts appointed by the Foundation. Further information in regard to the fellowships may be obtained from The American-Scandinavian Foundation, 25 West 45th Street, New York City.

The Pittsburgh Experiment Station of the Bureau of Mines is conducting a series of experiments on automobile exhaust gases in connection with the proper ventilation of tunnels and subways through which automobiles must pass. Tests are made on all sizes of cars and trucks under the various conditions, which are found in actual street use, to determine the amount of carbon monoxide produced. Dr. Yandell Henderson, of Yale University, is determining how many parts of carbon monoxide in 10,000 parts of air may be considered safe for a person to breathe for several hours without any ill effects. It is expected that the test will not only solve the problems of tunnel ventilation, but will be of great value to automobile owners and dealers in furnishing unbiased information on the efficiency of any given machine under particular conditions and on obtaining greater mileage through proper adjustment of the carburetor.

A Chilean Section of the Société de Chimie Industrielle has been organized with forty members. It is hoped to include in this new section every industrial chemist who has studied in France.

In a report made to the Natural Gas Conservation Committee the rapidly diminishing supply of natural gas is being felt keenly by the printing industry of the country, including the manufacturers of ink and paper for books, magazines and newspapers. Carbon black obtained from natural gas is the cheapest and best-known color base for printing inks, carbon paper, and type-writer ribbons, and is also used in the manufacture of phonograph records and automobile tires. Because of the short life of natural gas fields, which prevents the manufacture in one place for a long time, manufacturers of carbon black must follow the opening of all new fields.

The War Department has recently sold the picric acid plant at Picon, Arkansas, to H. C. Couch, president of the Arkansas Light & Power Co., who is understood to represent a group of Little Rock business men. Mr. Couch recently purchased the power plant, the water supply, and the gas supply systems. It is planned to make the plant an industrial center.

Experiments on the manufacture of dyes have been in progress for some time near Copenhagen, and although the results have been kept secret they are understood to be successful. Various intermediate products have already been made, and it is hoped to build up a dye industry which will render Denmark independent of foreign dyes.

The Southern Agricultural Chemical Corporation, subsidiary of the Tennessee Copper and Chemical Corporation of New York, is planning a \$600,000 fertilizer factory at Atlanta, which will be one of ten fertilizer plants to be erected by the company in the south.

A new edition of the chart for blending casinghead gasoline and naphtha has been prepared for the Bureau of Mines, Department of the Interior, which provides an easy method of determining the amount of naphtha to be used per gallon of casinghead gasoline to obtain a product of any required gravity. Copies may be obtained on application to the Bureau of Mines, Washington, D. C.

The Vinegar Hill Zinc Co. has purchased from the Government the acid plant near Cuba City, Wisconsin, which was erected during the war, and plans to rush the completion of the plant for the manufacture of sulfuric acid. Production is expected to begin about June 1.

The chemical industry in the Netherlands is reviving in spite of many difficulties which it has to face in the very low rate of exchange, the difficulty in obtaining raw materials, high wages, high ocean freights, and foreign competition.

The recent discovery of a valuable molybdenum steel alloy by a British metallurgist has resulted in an increased demand for molybdenum and an increase in price. Low-grade deposits of the mineral molybdenite are found in Australia, India, South Africa, and Peru. Peru mines are now producing nearly 80 per cent of the world's supply. The mines are being exploited by American capitalists, who export the ores to the United States.

At the recent formal inspection of the new laboratories at University College, London, by Prince Arthur of Connaught, Lord Moulton gave an address on "The Training and Functions of the Chemical Engineer." Another £50,000 is still required for the new chemical laboratories.

At the 1919 meeting of the board of governors of the New Zealand Institute it was decided to establish a fellowship of the Institute, to be limited to forty fellows, based on research or distinction in science. Not more than four are to be elected in any one year until the number is complete, after which vacancies will be filled as they occur.

The growing scarcity of gum camphor and the steadily increasing price has led to an investigation of the production of camphor oils from California sages. The method of recovery by steam distillation is not complicated or expensive. The entire group of oils are recovered together, true gum camphor frozen out, and the remainder separated by fractional distillation. The most prolific source of supply seems to be the desert sage of the western plains.

In a report on the Necessity for Helium Conservation, the Bureau of Mines describes the immense importance of helium for the filling of military lighter-than-air craft, on account of its perfect inertness and extreme buoyancy. At the time of the armistice about 150,000 cu. ft. of helium were ready on the dock for shipment to France. Since the supply of helium is limited to a special type of natural gas which is being consumed for domestic and industrial purposes, the Government is endeavoring to explore the extent of this resource and provide ways for its conservation. The Bureau of Mines Experiment Station at Ft. Worth, Texas, would like to learn of any such gases, particularly "wind-gas," and in appropriate cases will send cylinders for collecting samples for analysis.

O. Friedlander Chemical Co. has been awarded damages of \$2,000 in a suit against William H. Knox & Co., Inc. On April 5, 1918, the plaintiff agreed to deliver 50 tons of soda ash to the defendant at an agreed price of \$3.20 per hundred pounds. The soda ash was tendered on September 13, 1918, but was refused on the ground that the Government had denied an export license to the defendant and that the price charged was in excess of that allowed by the authorities.

Prohibition of the importation of dyestuffs into British India, which went into effect September 6, 1919, has recently been removed.

PERSONAL NOTES

Mr. Leon R. Whitcomb has resigned as chemical engineer with Hill & Ferguson, New York City, and is now associated with the Refinite Co., of Omaha, Neb.

Mr. R. E. Hardee, formerly with the Florida Cotton Oil Co., Jacksonville, Fla., is at present associated with the Florida Industrial Corporation, Gainesville, Fla., as chemist.

Mr. Ivan P. Tashof, specialist in inventions relating to the chemical, electrochemical, and metallurgical industries, recently announced the opening of an office for the practice of patent and trade-mark law, in Washington, D. C.

Dr. Lawrence T. Fairhall, formerly Major in the Sanitary Corps, U. S. Army, is research chemist with the O'Bannon Corporation, West Barrington, Rhode Island.

Mr. W. F. Rudd, of the department of chemistry, at the Medical College of Virginia, has been elected president of the American Conference of Pharmaceutical Faculties.

Mr. Ernest Jenkins Hoffman, who recently resigned as assistant chemist, U. S. Bureau of Mines, Pittsburgh, Pa., has accepted a position in organic research with W. B. Pratt, Inc., Boston, Massachusetts.

Mr. Douglas E. Scott, who for several years has been in charge of the Experimental Paper Mill of Arthur D. Little, Inc., Cambridge, Mass., has resigned and is now with the United Fruit Co., located at Boston, Massachusetts.

Mr. B. P. Caldwell is now with the Polytechnic Institute, Brooklyn, N. Y., as professor of analytical chemistry.

Mr. J. M. Clark, formerly chief chemist for the Sherwin-Williams Co., is now connected with the Deloro Smelting and Refining Co., Deloro, Ontario, Canada, in the capacity of superintendent.

Mr. J. B. Krak has left the West Virginia Geological Survey and has taken a position with the General Chemical Co., at the Laurel Hill Laboratories, Laurel Hill, Long Island.

Mr. Henry W. Esterwood, formerly with the F. S. Royster Guano Co., Norfolk, Va., as chemist, is at present analytical chemist in the Jackson Research Laboratories of E. I. du Pont de Nemours & Co., at Deep Water Point, New Jersey.

Mr. Gaston J. Levy, who served as Second Lieutenant in the Chemical Warfare Service, at Hanlon Field, France, has accepted the position of chemist with the Sperry Flour Co., Spokane, Washington, and is in charge of their laboratory there.

Mr. Clarence E. Breckenridge, formerly chief engineer for Merck & Co., New York, recently accepted a similar position with Rockwood & Co., Brooklyn, N. Y.

Mr. J. O. Halverson, for the past three years associate in the department of nutrition in the Ohio Agricultural Experiment Station, recently took charge of nutrition work in the Agricultural Experiment Station at Raleigh, N. C.

Mr. E. Olney Herman has resigned as industrial physicist at the Lynite Laboratories of the Aluminum Castings Co., Cleveland, O., and is now in charge of general technical and efficiency matters for the Eureka Company, North East, Pennsylvania.

Mr. F. Rohde has resigned as vice president and general manager of the Palo Company, New York City.

Mr. E. Gorgas Bashore, formerly chief chemist of the Babcock & Wilcox Co., has become a partner of Mr. Cyrus Wm. Rice under the firm name of Rice & Bashore, with offices located in Pittsburgh, Pennsylvania.

Dr. F. W. Zerban, formerly research chemist for the Louisiana Sugar Experiment Station, has accepted a position as research chemist with the firm of Penick & Ford, Ltd., New Orleans, Louisiana.

Mr. Arthur L. Davis has resigned as assistant catalytical chemist at the Fixed Nitrogen Research Laboratory, American University, Washington, D. C., and is now employed as research chemist with the International Coal Products Corp., Newark, New Jersey.

Mr. E. K. Kanet, formerly traveling superintendent of the Wm. F. Mosser Co., Richwood, W. Va., has become research chemist with the Goodyear Tire & Rubber Co., Akron, Ohio.

Dr. F. M. Beegle, formerly chemist for the American Chemical & Manufacturing Co., Norfolk, Va., is now chief chemist for the Glidden Company, Cleveland, Ohio.

Mr. Charles W. Studt, formerly a manufacturing chemist with the Mallinckrodt Chemical Works, St. Louis, Mo., is now chief geologist for the Eclipse Oil Co., Fort Worth, Texas.

Mr. William C. Dowd has left the American Smelting & Refining Co., Pueblo, Colo., where he was employed as chemist, to accept the position of chief chemist at the Austin Plant of the American Radiator Co., Buffalo, New York.

Mr. H. H. Hanson, formerly in charge of the feedingsuffs laboratory of the West Virginia State Experiment Station, is at present state chemist of the State Board of Agriculture, Dover, Delaware.

Mr. L. B. Shipley, formerly chemist and technical representative, in matters pertaining to coal-tar heavy oils, for the Barrett Company, is now associated with the Bernuth Lembecke Co., Inc., New York City, in a similar capacity.

Mr. Oregon B. Helfrich has just received his Ph.D. degree from the Johns Hopkins University and has accepted a position with E. I. du Pont de Nemours & Co., doing research work in the Delta laboratory of that company at Arlington, New Jersey.

Dr. Percy W. Punnett, formerly in the organic research laboratory of the Butterworth-Judson Corp., Newark, N. J., is at present with the Rochester Button Co., Rochester, New York, as chemist.

Dr. R. L. Kahn, formerly associated with New York University and Bellevue Hospital Medical College in the department of bacteriology, recently accepted an appointment as immunologist in the laboratory of the Michigan Department of Health, Lansing, Michigan.

Dr. Donald K. Tressler has resigned as research chemist of the varnish department of the Acme White Lead and Color Works, Detroit, Mich., to accept an industrial fellowship with the Mellon Institute of Industrial Research, University of Pittsburgh, Pittsburgh, Pa.

Dr. John Charles Hessler has been appointed assistant director of the Mellon Institute of Industrial Research of the University of Pittsburgh. Dr. Hessler, who is now serving as president of James Milliken University, Decatur, Ill., will enter upon his new duties at the close of the present academic year. As a member of the administrative staff of the Institute, Dr. Hessler will be in supervisory charge of certain of the researches in organic chemistry, a field in which he has specialized during the past twenty years.

Dr. J. J. Elbert, a technologist with the American Cyanamid Co., New York City, and a member of both the American Electrochemical Society and the American Chemical Society, died suddenly at his home in Tuckahoe, N. Y., on February 27. He was a graduate of Massachusetts Institute of Technology in chemical engineering, and of the University of Berlin.

Mr. Fred Olsen has resigned as chief of the research department of the Aetna Explosives Co., and is now attached to the Explosives Section of the Ordnance Department, Washington, D. C., directing various researches in explosives.

Mr. W. G. Koupal, a recent graduate of the University of Illinois, Urbana, Ill., is at present with the Pittsburgh Plate Glass Co. as a chemist in their research laboratories at Creighton, Pennsylvania.

Mr. Morris Schrero, formerly in the technology department of the Carnegie Library of Pittsburgh, Pittsburgh, Pa., has accepted a position as associate chemist in the research division, department of technical control, of the American Writing Paper Co., Holyoke, Massachusetts.

Dr. Lathrop E. Roberts, who received the degree of Ph.D. last December at the University of Chicago, Chicago, Ill., is now assistant director of technical control, American Writing Paper Co., Holyoke, Massachusetts.

Mr. W. B. Burnett, formerly in charge of the science work at the Masonic Home, is serving as assistant on the inorganic staff of the University of Illinois, Urbana, Ill., where he is at the same time doing graduate work.

Mr. J. Wm. Barker, recently a student at Kansas State College, Manhattan, Kans., has accepted a position as control chemist in the Marysville, Kansas, mill of the Larabee Flour Mills Corp., Kansas City, Missouri.

Mr. Frederick R. Ward has left the American Manganese Manufacturing Co., Dunbar, Pa., where he was employed as chief chemist and is now connected with the Tennessee Manganese Co., of Cleveland, Tenn., in the same capacity.

Mr. Arthur L. Day has changed from the Corning Glass Works, Corning, N. Y., to the Geophysical Laboratory, Washington, D. C.

Mr. Ralph E. Canfield, formerly with the American Nitrogen Products Co., at their nitric acid factory in Lake Buntzen, B. C., Canada, is now chemist for the Crown Willamette Paper Co., at their Camas, Washington, plant, where the laboratory work for all the paper mills of the company is now being centralized.

Mr. T. B. Peters, a recent graduate from the Pennsylvania State College, has accepted a position in the operating laboratory of E. I. du Pont de Nemours & Co., at their plant in Arlington, New Jersey.

Dr. David Klein has resigned as associate professor of biochemistry in the Johns Hopkins University School of Public Health and Hygiene, and has taken a position with the Hollister Wilson Laboratories, Chicago, Ill., as director of research and control laboratories.

Mr. G. A. Brewster, formerly chief chemist and metallurgist of the American Steel Foundries, has taken a position as metallurgist for the Arco Steel Department of the American Radiator Co., Buffalo, New York.

Mr. H. K. Baumgardner, until recently research chemist for the Crown Cork and Seal Co., Baltimore, Md., is now conducting researches for the H. I. Roosen Co., Brooklyn and New York, manufacturers of printing and lithographic inks and dry colors.

Mr. J. R. Marston, formerly superintendent of lead refinery with the Cia Minera Fundidora y Afinadora, of Monterrey, Mexico, has recently undertaken some research work for the United Verde Copper Co., Clarkdale, Arizona.

Dr. Henry Arnstein has resigned as technical director with the Cereal Products Refining Co., and has opened consulting offices devoted to the manufacture of various food products, in San Francisco, California.

The Rumford Committee of the American Academy of Art and Sciences at its recent meeting appropriated the sum of \$250 to Prof. Julius Stieglitz in aid of the publication of Marie's "Tables of Constants."

Assistant Professor Gerald L. Wendt, of the department of chemistry, University of Chicago, Chicago, Ill., has been appointed associate editor of the *Journal of the Radiological Society of North America*.

Mr. W. K. Buck, formerly in the metallurgical laboratory of the Naval Engineering Experiment Station, Annapolis, Md., has joined the forces of the Aluminum Co. of America, in their technical direction department, New Kensington, Pa.

Mr. Charles A. Stanwick who was honorably discharged from the U. S. Army in July of last year, is at present in the employ of the Western Electric Co., N. Y. C., as electrical engineer. In addition to this Mr. Stanwick is instructor in electrical engineering at Cooper Union Institute, New York City.

Mr. Oliver C. Ralston has left the employ of the Hooker Electrochemical Co., Niagara Falls, N. Y., where he was chief metallurgist, to become superintendent of the Seattle, Wash., station of the U. S. Bureau of Mines.

Dr. Colin G. Fink, head of the laboratories of the Chile Exploration Co., recently lectured to the graduate students of the School of Chemistry, Yale University, on "Industrial Research," and on "The Value of Physical Chemistry to the Organic Chemist."

Associate Professor C. J. Frankforter is in charge of the department of chemistry of the University of Nebraska, Lincoln, Neb. Some new courses in practical chemistry have been introduced by this department and have been working out very successfully since they were opened last September. The department is housed in a very fine new laboratory and the chairman of the chemistry faculty, Dr. F. W. Upson, is arranging for courses devoted to chemical engineering subjects. The department has also put in a course in oil and gas which is primarily for geologists.

Mr. J. Roy Haag, previously assistant chemist of the R. I. Agricultural Experiment Station, Kingston, R. I., has become assistant chemist, department of soil investigations, Agricultural Experiment Station, College Park, Maryland.

Mr. W. B. Hicks has resigned as chemist in the U. S. Geological Survey to take charge of analytical research for the Solvay Process Co., Syracuse, New York.

Mr. S. G. Keller, Jr., has severed his relations as assistant superintendent with the Standard Asphalt & Refining Co., Independence, Kansas, to become assistant superintendent of the Empire Refineries, Inc., Ponca City, Oklahoma.

Mr. D. Fay Hosman for the past three years research and consulting chemist for the Blue Valley Creamery Co., Chicago, Ill., has left that firm to become manager of the creamery department of the Scottsbluff Creamery Co., Scottsbluff, Nebraska.

Mr. Andrew Bender, formerly with the Air Reduction Co., as research chemist in their Jersey City laboratory, has joined the Barrett Co., and will be in charge of the control laboratory of their Frankford plant, Frankford, Philadelphia, Pa.

Mr. Ivar Bull Simonsen, until recently connected with the Niagara Smelting Corp., Niagara Falls, N. Y., as metallurgist, is at present with the Tennessee Manganese Co., as metallurgist at their plant in Cleveland, Tennessee.

Mr. Frederick L. Begtrup has left the Metal & Thermit Co., Jersey City, N. J., where he was employed as analytical chemist, and has taken a position with the Niagara Sprayer Co., Middletown, N. Y.

Mr. P. B. Caster, formerly with the Western Sugar Refinery and Spreckles Sugar Co., San Francisco, Cal., has joined the forces of the California and Hawaiian Sugar Refining Co., Crockett, Cal., as assistant to the plant manager.

Mr. James F. Walsh, chemical engineer, and lately Lieutenant in the Chemical Warfare Service, in charge of the charcoal development at the Astoria Plant of the Gas Defense Division, is at present with Wheeler & Woodruff, chemical engineers, of New York City.

GOVERNMENT PUBLICATIONS

By NELLIE A. PARKINSON, Bureau of Chemistry, Washington, D. C.

NOTICE—Publications for which price is indicated can be purchased from the Superintendent of Documents, Government Printing Office, Washington, D. C. Other publications can usually be supplied from the Bureau or Department from which they originate. Commerce Reports are received by all large libraries and may be consulted there, or single numbers can be secured by application to the Bureau of Foreign and Domestic Commerce, Department of Commerce, Washington. The regular subscription rate for these Commerce Reports mailed daily is \$2.50 per year, payable in advance, to the Superintendent of Documents.

CONGRESSIONAL COMMITTEES

Coal. House Report 600. 21 pp. February 6, 1920. Mining of coal, phosphate, oil, gas, and sodium on public domain. Conference Report to accompany Senate Document 2775, submitted by Mr. Sinnott.

Dyes and Dyeing. Senate Report 425. 5 pp. February 14, 1920. Importation of coal-tar products. Report to accompany House Resolution 8078, submitted by Mr. Watson.

LIBRARY OF CONGRESS

List of References on Dyestuffs. 186 pp. Paper, 15 cents. Compiled under the direction of H. H. B. MEYER. Contains the chemistry, manufacture, and trade in dyestuffs.

PUBLIC HEALTH SERVICE

The Efficiency of Certain Devices Used for the Protection of Sand Blasters against the Dust Hazard. C.-E. A. WINSLOW, LEONARD GREENBURG AND E. H. REEVES. Public Health Reports, 35, 518-34. The article describes the dust problem in sand blasting, previous studies of the efficiency of helmets and respirators designed for the protection of the worker in dusty trades, installation and methods used in the present study, results of the present study, and gives a summary and practical conclusions in regard to the protection of sand-blast operators.

GEOLOGICAL SURVEY

Deposits of Manganese Ore in Arizona. E. L. JONES, JR., AND F. L. RANSOME. Bulletin 710-D. Contributions to Economic Geology, 1919, Part I. 92 pp. Issued January 29, 1920. Fifty scattered deposits were examined. Some of

the deposits are within a few miles of a railroad, but most of them are more than 15 miles distant. No roads have been built to some of the deposits, and from a few of them the ore is packed on animals to the nearest roads. The cost of transporting the ore to the railroad by wagon or automobile truck ranges from 35 to 50 cents per ton-mile. The analysis of the manganese ore from various claims is given.

Deposits of Manganese Ore in Southeastern California. E. L. JONES, JR. Bulletin 710-E. Contributions to Economic Geology, 1919, Part I. 24 pp. Issued December 30, 1919. The possibility of commercial production of manganese ore in this region is determined by the accessibility of the deposits and the cost of transportation and mining. Water and fuel must be hauled to the camps. All the mining is done by hand and the mining costs are high. The ore is sorted by hand or screened in order to bring it to the required grade. Although it appears to be well adapted to mechanical concentration by wet gravity processes, the lack of water near the deposits precludes the installation of such machinery on the ground, and the expense of hauling the crude ore for considerable distances to water would probably not be justified.

The manganese ore consists of the oxides, of which psilomelane, pyrolusite, and manganite have been determined. Psilomelane is the dominant oxide in all these deposits. Calcite is universally present in the ores, though it varies largely in quantity. Iron oxides are scarce in most of these deposits. Quartz was nowhere observed as a primary constituent of the manganese deposits. No manganese mineral other than oxides was recognized in any of these deposits, but none of the workings had passed through the oxidized zone.

Oils in the Warm Springs and Hamilton Domes, near Thermopolis, Wyoming. A. J. COLLIER. Bulletin 711-D. Contributions to Economic Geology, 1919, Part II. 12 pp. Issued January 16, 1920.

Preliminary Report on the Chromite of Kenai Peninsula, Alaska. A. G. GILL. Bulletin 712-D. Mineral Resources of Alaska, 1918-D. 31 pp. The amount of ore above sea level at Port Chatham, on the assumption that material containing 10 per cent of chromic oxide can be profitably concentrated, has been estimated at 32,300 tons. Below sea level there may be a much greater amount, as the lower deposits are as a rule richer and heavier than those at higher altitudes. The exposures examined lead to an estimate of 12,600 tons below tide. The amount of chromite in sight at Red Mountain is placed at 195,600 tons. So long as chrome ore containing 40 or 45 per cent of chromic oxide is marketable, it will probably pay to continue the present process of hand sorting. It is estimated that one-half to three-quarters of the available tonnage of chromite in this region would require concentration, and if it is found that ore carrying less than 10 per cent chromic oxide can be profitably milled, the tonnage of available chromite will be much greater than these estimates.

Deposits of Manganese Ore in Nevada. J. T. PARDEE AND E. L. JONES, JR. Bulletin 710-F. Contributions to Economic Geology, 1919, Part I. 40 pp. Issued March 15, 1920. Manganese is rather widely distributed in Nevada. In addition to the small amounts that occur in rocks generally it is more or less abundant in many of the metalliferous quartz lodes and in deposits of other types. The classification and general features of the manganese deposits include deposits formed by replacement of country rock by carbonate or silicate minerals that have become partly or completely oxidized; deposits formed by replacement of country rock by oxide minerals; and silicate and carbonate lenses in metamorphic rocks.

Metals and Nonmetals, Except Fuels. F. L. RANSOME AND E. F. BURCHARD. Bulletin 710. Contributions to Economic Geology (Short Papers and Preliminary Reports) 1919, Part I. 8 pp. 1920.

Gold, Silver, Copper, Lead, and Zinc in Utah in 1918. V. C. HEIKES. Separate from Mineral Resources of the United States, 1918, Part I. 35 pp. Issued March 4, 1920. There was a decrease in 1918, as compared with 1917, in the output of ore and in the production and value of all the metals except silver, as shown by the following table:

MINE PRODUCTION OF GOLD, SILVER, COPPER, LEAD, AND ZINC IN UTAH 1917-1918

	1917	1918	Decrease in 1918	1864-1918
No. of producers...	334	251
Ore, short tons.....	15,358,481	14,705,718	652,763
Gold ¹ , fine ounces.....	\$3,355,156	2,949,170	405,986	96,068,503
Silver, fine ounces.....	13,479,133	13,455,597	23,536	379,476,124
Copper, lbs.....	246,674,153	227,169,630	19,504,523	2,189,332,043
Lead, lbs.....	178,521,958	167,008,224	11,513,734	4,142,608,381
Recoverable zinc, lbs.....	21,286,871	18,399,417	2,887,454	210,357,026
Total Value.....	\$99,328,155	86,047,597	13,280,558	1,002,202,934 ²

¹ Includes small placer production.

² The total by years to the end of 1913 appears in Mineral Resources for 1913, pt. 1, p. 366, 1914.

Mineral Waters in 1918. A. J. ELLIS. Separate from Mineral Resources of the United States, 1918, Part II. 37 pp. Issued March 19, 1920. The statistics given refer only to domestic mineral waters that have been sold, imports being excepted. Three uses of mineral waters are recognized—table, medicinal, and in the manufacture of soft drinks. The sources of mineral waters are described, and the mineral water trade in 1918 is reviewed as well as the mineral water trade by states.

Architectural Concrete Stone and Concrete Blocks in 1917 and 1918. G. F. LOUGHLIN. Separate from Mineral Resources of the United States, 1918, Part II. 8 pp. Issued March 16, 1920. The publication was prepared in response to numerous requests for statistical information on the production of artificial stone.

Asbestos in 1918. J. S. DILLER. Separate from Mineral Resources of the United States, 1918, Part II. 12 pp. Issued March 20, 1920. Includes an account of the domestic output, varieties and types of asbestos, asbestos in the United States, prices, imports, exports, foreign deposits and production, and the world's production.

The Data of Geochemistry. F. W. CLARKE. Bulletin 695, 4th edition. 832 pp. 1920.

Bauxite and Aluminum in 1918. J. M. HILL. Separate from Mineral Resources of the United States, 1918, Part I. 14 pp. Issued March 16, 1920. The quantity of bauxite marketed in the United States in 1918 showed an increase over the production of 1917 of about 7 per cent in quantity and about 11 per cent in value. The production from the Georgia, Alabama, and Tennessee fields in 1918 showed a decrease of 31 per cent, but the Arkansas production showed an increase of approximately 1 per cent. Of the quantity of domestic bauxite marketed in 1918, apparently the producers of aluminum took about 419,043 long tons, the makers of chemicals about 63,896 tons, makers of abrasives about 112,908 tons, and the makers of refractories about 9,874 tons.

The value of primary aluminum produced in the United States in 1918 showed a decrease of about 10 per cent from the value of the output in 1917. The decrease was due very largely to the decline in price of aluminum during 1918 and does not represent a corresponding decline in quantity of output.

Copper in 1917. B. S. BUTLER. Separate from Mineral Resources of the United States, 1917, Part I. 74 pp. Issued March 13, 1920. The report includes: A summary of statistics of the copper industry in the United States in 1912, 1913, 1914, 1915, 1916, and 1917; an account of the general condition of the industry, including the world's production and consumption and domestic conditions; statistics showing the production, imports, exports, consumption, uses, prices and costs of copper; and a review of foreign production.

BUREAU OF MINES

A Glossary of the Mining and Mineral Industry. A. H. FAY. Bulletin 95. 754 pp. Reprint. Cloth, 75 cents. 1920.

Absorption as Applied to Recovery of Gasoline Left in Residual Gas from Compression Plants. W. P. DYKEMA AND R. O. NEAL. Technical Paper 232. Petroleum Technology 53. 43 pp. Paper, 15 cents. 1920.

Metal-Mine Accounting. C. B. HOLMES. Technical Paper 250. 63 pp. Paper, 10 cents. February 1920.

Metal-Mine Accidents in the United States during the Calendar Year 1918. Compiled by A. H. FAY. Technical Paper 252. 113 pp. Paper, 10 cents. 1920. Includes supplemental labor and accident tables for the years 1911 to 1918, inclusive.

Accidents at Metallurgical Works in the United States during Calendar Year 1918. Compiled by A. H. FAY. Technical Paper 256. 23 pp. Paper, 5 cents. Issued January 1920.

BUREAU OF STANDARDS

Melting Points of Chemical Elements, and Other Standard Temperatures. Circular 35. 4th ed. 2 pp. Paper, 5 cents. Issued December 1, 1919.

Specifications for the Manufacture and Installation of Railroad Track Scales. Circular 83. 35 pp. Paper, 5 cents. Issued January 31, 1920.

Recommended Specification for Basic Carbonate White Lead, Dry and Paste. Circular 84. 10 pp. Paper, 5 cents. Issued December 27, 1919. Prepared and recommended by the United States Interdepartmental Committee on Paint Specification Standardization, August 11, 1919. Basic carbonate white lead may be ordered in the form of dry pigment or paste ground in linseed oil. Material shall be purchased by net weight.

(a) DRY PIGMENT—The pigment shall be the product made from metallic lead and shall have a composition corresponding approximately to the formula $2PbCO_3 \cdot Pb(OH)_2$. It shall be thoroughly washed after corroding, shall be free from impurities and adulterants, and shall meet the following requirements:

Color—Color Strength—When specified, shall be equal to that of a sample mutually agreed upon by buyer and seller.

	Minimum Per cent	Maximum Per cent
Coarse particles:		
Retained on Standard No. 200 ¹ screen..	..	None
Retained on Standard No. 325 ¹ screen..	..	2.0
Lead carbonate.....	65.0	75.0
Total impurities, including moisture.....	..	2.0

¹The No. 200 and No. 325 screens are the same as screens formerly known as 200 mesh and 350 mesh.

(b) PASTE—The paste shall be made by thoroughly grinding the above-described pigment with pure raw or refined linseed oil.

The paste as received shall not be caked in the container and shall break up readily in oil to form a smooth paint of brushing consistency. The paste shall consist of:

	Minimum Per cent	Maximum Per cent
Pigment.....	90	92
Linseed oil.....	8	10
Moisture and other volatile matter.....	..	0.7
Coarse particles and "skins" (total residue retained on No. 325 screen based on pigment)	2.0

Recommended Specification for Basic Sulfate White Lead, Dry and Paste. Circular 85. 11 pp. Paper, 5 cents. Issued January 26, 1920. Prepared and recommended by the United States Interdepartmental Committee on Paint Specification Standardization, September 22, 1919.

Basic sulfate white lead may be ordered in the form of dry pigment or paste ground in linseed oil. Material shall be purchased by net weight.

(a) DRY PIGMENT—The pigment shall be the sublimed product prepared from lead sulfide ores, free from impurities and adulterants, and shall meet the following requirements:

Color—Color Strength—When specified shall be equal to that of a sample mutually agreed upon by buyer and seller.

	Minimum Per cent	Maximum Per cent
Coarse particles:		
Retained on standard No. 200 ¹ screen...	None
Retained on standard No. 325 ¹ screen...	0.5
Composition:		
Lead oxide.....	11.0	18.0
Zinc oxide.....	..	9.0
Total impurities, including moisture.....	..	1.0
The remainder shall be lead sulfate		

¹The No. 200 and No. 325 screens are the same as screens formerly known as 200 and 350 mesh.

(b) PASTES—The paste shall be made by thoroughly grinding the dry pigment with pure raw or refined linseed oil.

The paste as received shall not be caked in the container and shall break up readily in oil to form a smooth paint of brushing consistency. It shall mix readily in all proportions without curdling with linseed oil, turpentine, or volatile mineral spirits, or any combination of these substances.

The paste shall consist of:

	Minimum Per cent	Maximum Per cent
Pigment.....	89	91.0
Linseed oil.....	9	11.0
Moisture and other volatile matter.....	..	0.7
Coarse particles and "skins" (total residue retained on No. 325 screen, based on pigment).....	..	0.5

Metals: Standard Samples Issued or in Preparation. Circular 25, supplement. 5 pp. Issued February 28, 1920. Supersedes all previous supplements.

Recommended Specifications for White Paint and Tinted Paints Made on a White Base—Semi-paste and Ready Mixed. Circular 89. Prepared and recommended by the United States Interdepartmental Committee on Paint Specification Standardization, January 26, 1920.

Prescribes specifications for pigment, liquid, semipaste, and ready-mixed paint, methods of sampling, laboratory examinations of semi-paste and mixed paint, analysis of pigment and reagents employed. It is believed that this specification admits practically all high-grade prepared paints generally available in the United States, and which are therefore obtainable without requiring manufacturers to make up special lots.

Concerning the Annealing and Characteristics of Glass. A. Q. TOOL AND J. VALASEK. Scientific Paper 358. 45 pp. Paper 10 cents. Issued January 31, 1920. Certain methods for determining the annealing range were tested, and the "annealing temperatures" obtained are given for a number of optical glasses. The critical temperature range for these glasses was determined. Some of the methods available for determining the relaxation time were tested. A cooling procedure which proved very satisfactory in the laboratory is discussed.

Estimation of Nitrates and Nitrites in Battery Acid. L. B. SEFTON. Technologic Paper 149. 10 pp. Paper, 5 cents. Issued February 24, 1920.

Determination of Cellulose in Rubber Goods. S. W. EPSTEIN AND R. L. MOORE. Technologic Paper 154. 16 pp. Paper, 5 cents. Issued February 20, 1920. A method is presented which is readily applicable to the determination of fabric in rubber sheeting, raincoat materials, waterproofed fabrics, spread goods, frictioned and calend red fabrics in general.

Cements for Spark-Plug Electrodes. H. F. STALEY. Technologic Paper 155. 10 pp. Paper, 5 cents. Issued February 21, 1920.

Metallographic Features Revealed by the Deep Etching of Steel. H. S. RAWDON AND SAMUEL EPSTEIN. Technologic Paper 156. 24 pp. Paper, 10 cents. Issued March 19, 1920. The method of deep etching of steel by means of concentrated acids was examined in detail. The metallographic features of steel revealed by deep etching are of three general types: Chemical inhomogeneity, mechanical nonuniformity, and physical discontinuities.

DEPARTMENT OF AGRICULTURE

A Pink Yeast Causing Spoilage in Oysters. A. C. HUNTER. Department Bulletin 819. 24 pp. Issued March 10, 1920. Paper, 5 cents.

The Microscopical Examination of Flour. G. L. KEENAN AND M. A. LYONS. Department Bulletin 839. 32 pp.

COMMERCE REPORTS—MARCH 1920

Camphor allotments for January, February, and March, 1920, are as follows: United States, 379,635 lbs.; France, 56,876 lbs.; England, 134,915 lbs. The price per hundred weight has been increased greatly. (P. 1220)

After extensive investigations in Sumatra and the Federated Malay States, the conclusion has been reached that it would not pay the rubber plantations to collect seed especially for the purpose of extracting oil therefrom unless other oil-bearing seeds could be produced in the same locality. (P. 1221)

Nothing is being done in New Zealand to utilize the wool scouring waste. (P. 1221)

Prices of tin plate in France continue to rise and available supplies tend to decline. Tin plates of 1 meter and 25, 29, and 33 centimeters in thickness are completely lacking on the market and are greatly needed. (P. 1231)

The difficulties of supply and price of gasoline in the British Isles can only be overcome by the discovery of new fields, increased production in and better distribution from existing fields, and the substitution of other liquid fuels for gasoline. Benzene, power alcohol, and coal gas are suggested as substitutes. (Pp. 1236-7)

The production of cooking utensils from stainless or rustless steel is reported. (P. 1244)

Conditions in the petroleum refineries of Budapest are critical from want of raw material. (P. 1250)

The large German nitrogen works producing calcium cyanamide would face immediate ruin were it not for the protection of a syndicate organized under the patronage of the state to maintain uniform prices for nitrogen products. The low exchange also acts as a high tariff on imports into Germany. (Pp. 1251-2)

It is expected that the manufacture of textile substitutes will be highly profitable in Germany and strenuous efforts are being made to develop this industry. The production depends upon the availability of large supplies of both alcohol and nitric acid. (P. 1252)

The German aluminum industry was overdeveloped during the war when aluminum was used as a substitute for copper in most electrotechnical products. The low exchange value of the mark affords protection to the domestic industry, but under normal conditions the industry would be at a disadvantage as against the aluminum industry of other countries as the German works use brown coal, while other countries use water power. The difficulty of securing raw materials is another source of trouble. (P. 1252)

Another great German war industry is the manufacture of synthetic rubber. Whether this industry will continue to be profitable will depend upon the protection afforded by the low exchange value of the mark. (Pp. 1253-4)

The soap making industry has been revolutionized by the production of a colloidal clay soap. The clay increases the lathering and detergent properties of the soap and by its use the present proportion of fatty acids can be halved. (Pp. 1262-3)

Statistics showing the production of beet sugar in Spain during the seasons July 1 to November 1, 1918 and 1919 are given. (P. 1263)

A market for brass and copper is reported in Argentina. (P. 1267)

The manufacture of paints in South Africa promises to become eventually an industry of considerable importance. Almost inexhaustible supplies of earth pigments, ochres, and oxides exist there. Imports of ochres and linseed oil are diminishing. (Pp. 1293-4)

A large stock of salt on hand is reported at Cadiz, Spain, due to the scarcity of shipping facilities and extremely high ocean freights. (Pp. 1294-5)

All restrictions on the purchase or sale of hides, calfskins or kips in Great Britain and Ireland have been withdrawn, but existing restriction on the export of these commodities will be maintained. (P. 1297)

Italy's rubber industry is reported to be in a favorable condition. (Pp. 1299-1300)

The Venezuelan government has imposed certain regulations in regard to the tapping of trees producing gums, such as rubber, balata, etc. (P. 1302)

Statistics giving the final detailed estimate of India's 1919-20 sesame crop are given. The condition of the crop is reported to be generally good. (P. 1309)

A French decree of March 2, 1920, prohibits from March 5 the exportation and reexportation of coal tar and products obtained direct by distillation of coal tar, except under license from the Ministry of Finance. (P. 1329)

The test of concrete tanks used for water storage, undertaken by the Bureau of Standards, has been discontinued because the daily loss has become constant. These tests seem to demonstrate that the 1:2:4 concrete is sufficiently waterproof for this purpose. (P. 1331)

The French resources of iron ore are described. (Pp. 1356-61)

The British government has placed a fund of £1,000,000 at the disposal of its Research Department to encourage industries to undertake research. The Advisory Council for Scientific and Industrial Research has therefore recommended that the fund be expended on a coöperative basis in the form of liberal contributions toward the income raised by voluntary associations of manufacturers established for the purpose of research. The privileges accorded members, the establishment of information bureaus and research companies are described and a list is given of the associations organized and contemplated. The results of any research belong to the association, which will hold them in trust for the benefit of its members. (Pp. 1362-4)

A technical committee has been appointed to study and report upon arrangements on a large scale for the distillation of the crude petroleum obtained from the Argentina oil wells with the object of extracting from it light oils and essences such as naphtha, gas oil, and benzene for industrial use. The oil is said to be of a very high quality. (Pp. 1368-9)

The production of antimony in the Province of Hunan, China, has fallen off considerably since the close of the war, many smelters having closed down. (P. 1370)

The Bureau of Standards is conducting an investigation with a view to the elimination of "fish scaling" in enamels for sheet steel. (P. 1374)

A market for rubber goods in the Canary Islands is reported (P. 1385)

Belgium is still in need of the following products to carry on her reconstruction program: hematite pig iron, puddled pig iron, semi-steel products, gas coal, mineral substances for the manufacture of chemical and pharmaceutical products and drugs, potassium chlorate, amorphous phosphorus, sesquisulfide of phosphorus, glue, paraffin, and aniline colors. The following products are now ready for export: silver sand, glue stock, precipitated phosphate of lime, white lead, litharge, red lead, blueing, window glass, and sole leather. (Pp. 1399-1405)

The shortage of Belgian plate glass continues and there are no prospects of immediate increase. (P. 1425)

The tanning industry in Sao Paulo, Brazil, has been established on a secure footing, due to conditions brought about by the war. (Pp. 1429-32)

The Indian Department of Statistics has issued its first forecast of the 1919-20 crop of winter rape, mustard, and linseed oils. (P. 1437)

A German trust company has been organized for financing the purchase of raw materials. (P. 1473)

The crude potash situation in France is reviewed. (P. 1474)

Probably none of the wartime industries of Great Britain has shown greater development or more progress than that of glass making. (P. 1486)

The Balkan Peninsula is said to be a good region for the manufacture of nitrates from the air because of the abundant water power. (P. 1490)

A chemical company has been founded in Rumania to manufacture chemicals from raw materials. (P. 1490)

The total production of whale oil in Norway during 1919 was 23,654 metric tons, which is considerably less than the normal output. (P. 1505)

Foreign markets for inks are reviewed. (Pp. 1528-9)

The condition of the French mines and the iron and steel industries is reviewed. (Pp. 1588-91)

The tin supply of South China will soon go out of the markets of the world unless there is a rapid and complete change in the situation. Chinese tin is produced on the basis of a silver dollar whose exchange value is substantially that of \$1 gold, while Straits Settlements tin, the chief competitor of Chinese tin in the markets of the world, is produced on the basis of a Straits dollar worth about 56 cents gold. (Pp. 1596-7)

The great scarcity of coal in France has caused some railway companies to transform their motive power from coal to fuel oil consumption. (P. 1597)

The gutta-percha industry is reviewed and statistics are given showing the quantities, values, and countries of destination of gutta-percha and gutta inferior exported from Singapore during 1918. (Pp. 1608-9)

For some years the production of kauri gum in New Zealand has been decreasing. (P. 1611)

It is anticipated that the production and exportation of palm oil on the east coast of Sumatra will continue to increase. Exporters have been handicapped by a lack of suitable containers. Some shipments made in locally constructed wooden barrels resulted in a loss through leakage of 30 per cent. (P. 1614)

Statistics showing the production of copper in Sweden are given. (P. 1615)

The British dyestuffs industry has made considerable progress during the past year, but there is still a scarcity of certain colors. It is hoped with the greater attention now being paid to research departments the results will be more satisfactory, and that England will secure a powerful place in the world's markets. (P. 1636)

An account is given of foreign markets for soaps and soap-making materials, including Algeria, Argentina, China, Jamaica, and Switzerland. (Pp. 1638-41)

A description is given of the vegetable oil-bearing materials and products in Spain. (Pp. 1646-54)

The international metric system of weights and measures is the only system authorized in Belgium. Although on account of the disturbed state of affairs in Belgium there has been laxity in the enforcement of the use of the metric system, the disposition is to have it obeyed. (P. 1654)

The American purchase of certain Bolivian petroleum deposits is announced and a railway line from Santa Cruz to Formosa is contemplated. This will serve as a very important gateway to a hitherto untouched section of Bolivia, the riches of which are known to be considerable. (P. 1656)

Statistics are given showing the production of metals in Mexico since 1916. (P. 1661)

Aluminum is being considered by the Japanese hydroelectric plants to replace copper in their heavy transmission cables. (P. 1662)

A reward of £10,000 is offered for the discovery in Australia of petroleum in commercial quantities. (P. 1670)

There is a demand for heavy chemicals in the Far East which should increase with the industrial development of that region. (P. 1705)

Considerable quantities of sole leather have been produced in China and beginning with 1920 the production of chrome leather of practically all ordinary grades was commenced. The quality of leather produced is of the cheaper grades and it is believed that there will be little clashing of interest between the American and European and the Chinese leathers. (P. 1708)

The prohibition on the importation of dyestuffs into British India has been removed. (P. 1729)

Artificial silk is being manufactured by the British Cellulose and Chemical Manufacturing Co. in Great Britain. The silk thread is of such texture and quality that when converted into articles of clothing it will have the appearance and finish of natural silk. When fully developed one factory alone will produce 9 tons of artificial silk a day. The company also manufactures noninflammable films, synthetic perfumes, and aspirin. (P. 1739)

An Australian manufacturer has invented and patented a new composition called "Keltona," made partly from waste products, which can be used for imitation tiling, chair bottoms, and for most purposes for which thin woods and stones can be used. (P. 1756)

The exportation of nitrate of soda from Chile during January 1920 was the largest in any one month in the history of the nitrate trade. (P. 1763)

There is said to be a steady demand in Sweden for drugs from the United States. In the case of salvarsan there is a most serious opposition to be met on account of the distinct predilection among Swedish medical men to the use of the original German product. Among other drugs which could be advantageously introduced are senega root, golden seal, sabadilla seed, and barbital. (P. 1779)

A decrease in the opium crop in Greek Macedonia is reported in 1919. (P. 1788)

The drug and chemical trade of Japan is briefly reviewed. (P. 1799)

SPECIAL SUPPLEMENTS ISSUED

SCOTLAND—22a	GUATEMALA—32g
BRITISH WEST INDIES—25a	SALVADOR—39g
COSTA RICA—27a	BRITISH INDIA—53c
CUBA—28a	EGYPT—71g
	MADAGASCAR—72b

STATISTICS OF EXPORTS TO THE UNITED STATES

HONGKONG, CHINA— (pp. 1254-5)	Paper and manufac- tures	Potash
Antimony	Tin	Rubber goods
Camphor	Wood, unmanufac- tured and manufac- tured	Saffron
Chemicals	PERSIA—(p. 1787)	BELGIUM—(pp. 1403- 4)
Explosives	Gum tragacanth	Horsehides
Fibers	Opium	Rabbit skins
Hides	Skins, raw	Glass
Leathers and manu- factures	FRANCE—(pp. 1305-9)	Calfskins
Peanut oil	Aluminum	Anilin colors
Wood oils	Chemicals	Red powdered arsenic
Aniseed oil	Hides and skins	Silver sand
Cassia oil	India rubber scrap	Marble and flint stone
Bismuth	Paper	Crude rubber
Manganese	Perfumery and soap	Medicinal plants
Wolfram		Fusel oil
		Muriate of potash

BOOK REVIEWS

Technical Handbook of Oils, Fats and Waxes. Vol. II—Practical and Analytical. By PERCIVAL J. FRYER, F.I.C., F.C.S., Chief Chemist and Director, Yalding Mfg. Co., Ltd., Lecturer in Oils, Fats and Waxes at the Polytechnic, Regent St., W. London Institute, and FRANK E. WESTON, B.Sc. (1st Hons.), F.I.C., Head of the Chemistry Dept., the Polytechnic, Regent St., W. xvi + 314 pp. 69 figures. Cambridge University Press, London, England, 1918. Price, \$4.50.

The present volume considers the practical and analytical sides of oil and fat chemistry in the condensed and systematic manner characteristic of Volume I. The work is useful to the technical and industrial analyst, and perhaps even more so to the advanced college student of the subject. Practically all of the well-known methods for the testing and analysis of oils, fats, waxes, fatty acids, higher alcohols, glycerol, hydrocarbons and rosin and turpentine are given. It will be remembered that analytical methods were merely catalogued in the first volume. Excellent discrimination and judgment are shown in the selection of methods and all of the more important and some not so important are included. The illustrations are mainly from photographs, including apparatus of the fixed metallic and optical sort, and also assembled glass apparatus used in various tests. Diagrams and tables are frequent. The greatest drawback to the work is the lack of references to original literature for use in connection with various methods of analysis and tests, which tends to restrict the use of the book to beginners. Although a few such references are given, they are not general and numerous tests and methods are without references. The book is well printed on good paper. It will be found useful in all oil and fat libraries, particularly for quick reference.

W. D. RICHARDSON

Chemical Calculation Tables. By HORACE L. WELLS. 2nd Ed., Revised. John Wiley & Sons, Inc., New York. Price, \$1.35.

In preparing the second edition of this volume, first issued in 1903, the author has taken the opportunity to revise the volume by recalculating the constants in accordance with what the author designates as the latest international table of atomic weights, leaving the reader to ascertain what particular table has been used. The recalculation of the constants seems to have been made with great care, the author realizing the importance of accuracy in tables of this kind.

The volume includes a rather brief table of Gravimetric Factors with a table of Formula-Weights, Factors for Indirect Analyses and a table for the Reduction of Gas Volumes, and also a table for Calculation of Nitrogen from the Gas Volumes. These tables do not cover at all completely the factors required by the analytical chemist.

The Gravimetric Factors in addition to the substance weighed include only metals and their oxides and occasionally carbonates or sulfates so that the analytical chemist would have to add a great many factors for his every-day use. The selection of the factors to be included is not always the best possible. For instance, under lead, the factors for converting PbS to Pb and PbO are given, while the factors for conversion from PbO₂ are not given. It is doubtful if lead is ever weighed as PbS, while in electrolytic work small quantities are frequently obtained as PbO₂ which would have been a much better factor to have included.

The table of Multiples for Organic Compounds will no doubt be found useful as well as the table for Molecular-Weight Determinations. The table for Weights and Measures is very limited.

The five-place table of Logarithms is undoubtedly the most valuable part of the volume as this table is very well printed and is extremely convenient for use, being very well thumb-indexed and very legible.

J. C. OLSEN

The Petroleum Handbook. By STEPHEN O. ANDROS, A.B., B.Sc., E.M. 206 pp. 48 illustrations, 17 tables. Shaw Publishing Co., 910 South Michigan Ave., Chicago, Ill. Price, \$2.00.

This book, as stated by the author, is a compilation of general information on the origin, production, refining and marketing of petroleum and its products. It contains a chapter on Gasoline Specifications and State Laws covering gasoline and kerosene.

There is no new information contained in the book. The compiler has evidently made no attempt to discover and point out the errors in the original publications. The description of the methods of refining is very general and is incorrect in a great many cases. He states that the vacuum distillation process is in use in some refineries, whereas I know of no place where it is in use at the present time.

The compiler states that the method of refining asphalt base petroleum is substantially the same as in paraffin base, whereas, in fact, it is handled very differently.

On page 87 he states that the final boiling point of kerosene distillate is 450° F., whereas it is nearer 600° F.

The operation of the Trumble process (p. 95) is described in such a manner that one perfectly familiar with the process would not even recognize it.

The tabulation of the State Laws and Gasoline and Kerosene Specifications is also incorrect in a great many cases.

This book has the same objectionable features that I have noticed in others of this kind which have been recently published, in that although it is a useful compilation of information for one who had has experience in oil, it is a very poor guide for a beginner.

G. W. GRAY

Creative Chemistry. By EDWIN E. SLOSSON. 311 pp. Century Company, New York City, 1919. Price, \$2.50 net.

The prominence given to science by war activities has so emphasized it in the minds of the public that there is to-day a greater demand for scientific discussions presented in a popular way than has ever been the case heretofore. There is real need for a popular presentation of the interesting problems in the field of chemistry, and "Creative Chemistry" does much toward fulfilling this need. It is well written and illustrated and the public is sure to find it very readable.

From the chemist's standpoint one might question the wisdom of giving prices (as on page 141) in view of the constantly changing conditions in the chemical industry. At times there is a crossing of subjects which in no way detracts from the readability of the volume but suggests the possibility of a somewhat better arrangement in another edition of the work. There are a few inaccuracies of small consequence that might be eliminated the next time. The author seems to have had the same difficulty in adhering to his text as do some of our best-known lecturers who find a discussion of things chemical leading them into by-paths suggested as the subject is developed. It would seem worth while to present such valuable diagrams as those presented on page 147 in more legible form and the addition of discussions at

some points or the inclusion of a glossary would seem helpful.

The reading references are particularly valuable, especially to those who desire supplementary reading on various chemical subjects. The numerous bits of humor which Mr. Slosson has sprinkled through the text will be appreciated, and the book should have a wide appeal, doing much toward creating the scientific public of which the chemist is in need.

H. E. HOWE

White Lead—Its Use in Paint. By A. H. SABIN. 128 pp. John Wiley & Sons, Inc., New York, 1920. Price, \$1.25.

The purpose of this book is to give simple information regarding white pigments rather than to present the matter from the strictly chemical or scientific standpoint. It outlines briefly the history of the white lead industry, touching upon the methods used in the production of white lead by the Old Dutch, Carter, Matheson, mild, chamber, and various precipitation methods, and the process of sublimation. It describes briefly the methods used for drying, pulp mixing, and washing white lead, commenting upon the fineness, opacity, adhesion, water resistance, durability, methods of mixing, application, defects, etc. In the discussion of water resistance, affinity between lead and oil, etc., the author's statements are rather vague and not in agreement with present conceptions of physical chemistry.

Information is given regarding methods of mixing white lead, how the pigment affects light tints made with blue or green, and how the white lead may occasionally be discolored through effect of sulfur gases in certain industrial communities. The discussion on lead-poisoning is well presented, but the statements made on page 59 are contradictory.

Information regarding leaded zinc, lithopone, zinc oxide, and their use in modern flat and gloss paints is presented in an interesting but not impartial manner.

An outline of the proper method of reducing, thinning, and tinting white lead paste for application to various kinds of lumber follows.

The volume should prove of value to the painter and to the student interested in paints.

H. A. GARDNER

Mining and Manufacture of Fertilizing Materials and Their Relation to Soils. By STRAUSS L. LLOYD, E.M. vi + 153 pp.; illustrated. D. Van Nostrand Company, New York City, 1919. Price, \$2.00.

The author expresses the hope that the book "will appeal not only to manure manufacturers, but to farmers, as well as to agricultural students and all those who take an intelligent interest in the subject of agricultural chemistry." The book is divided into fourteen chapters. It treats of the functions and sources of nitrogen, phosphoric acid and potash, the action of lime and gypsum, nitrification and denitrification, the origin and composition of soils, and the relation between soils and fertilizing materials. The author presents in much detail the subjects of pebble phosphate ore and hard rock phosphate ore dressing and milling. Chapters are devoted to the more common fertilizer ingredients containing nitrogen, potash and phosphoric acid, and to the manufacture of superphosphate, precipitated phosphate, ammoniated phosphates, and complete fertilizers. Considerable space is devoted to the occurrence, mining and purification of nitrate of soda, and to the fixation of nitrogen as calcium cyanamide and calcium nitrate. Much attention is given to the mining and purification of the German potash salts, and to the liberation of potash from feldspars by artificial means. The book closes with two chapters on the analysis of fertilizers and soils.

It will be noted that the book covers a rather unusual combination of subjects and is more suited to the general reader than to the student or analyst seeking special information. Most students will have neither the time nor desire to go into so much detail in regard to phosphate mining and fertilizer manufacture and the analyst will prefer a book which is not so restricted in its scope.

Greater care should have been taken in the preparation of the text. For example, the author after speaking of the formation of starch, says that "in some unknown way it becomes soluble," so that it can be transferred from one part of the plant to another. He states that available phosphate "appears to hasten the maturity of plant." It would seem that he could have said "it does" so.

In discussing the action of lime he asserts that its chief function "is to improve the mechanical condition of the soil," whereas most authorities hold that its chief function is to counteract soil acidity and to break up or transform certain toxic substances which occur in such soils.

The author says that chemical analysis "does not attempt to ascertain and state in which form or forms the nitrogen is present in a fertilizer." He is evidently not aware that the forms of nitrogen are determined and published in some of the northern states. He adds that "compounds of ammonia are quite uncommon in commercial fertilizers," which is not borne out by the large amount of sulfate of ammonia used in mixed fertilizers.

The sweeping statement that "the mineral forms of nitrogen, such as nitrate of soda and sulfate of ammonia, both dissolve easily in water, hence they would soon wash into the subsoil and out of reach of the plants," certainly needs great qualification.

The writer makes the assertion that "before nitrogen in ammonium sulfate or organic substances can be taken up by plant life, it must be converted into nitrates." It is, nevertheless, well established that many plants may use nitrogen in one or both of those forms without change to nitrates. It is also asserted that "the nitrogen in organic substances is chiefly in an albuminoid form," no reference being made to other proteins and simpler organic compounds.

In speaking of common salt no mention is made of the fact that sodium may replace some potassium in connection with the growth of at least certain cultivated plants, only the indirect action of the salt being mentioned.

Silica in the form of grains of sand is referred to as "forming the bulk of most soils,"—a statement which may perhaps be excused in the case of a resident of Florida. Reference is made to the formation of carbonate of lime by "organic activity" in the shells of the "crustaceans," whereas a reference to the mollusca would have seemed more appropriate if a single reference were to be made. In one place gypsum is referred to as "an important fertilizing material," and elsewhere it is stated that it does not furnish "needed plant food."

One is rather amazed at the broad, unqualified statement that "there is no way to tell, without experiment, what food constituents a soil lacks."

It is asserted that sulfate of ammonia "should be used only when the soil has been lately limed," regardless of the fact that large areas of soil require no lime.

A strong odor in a fertilizer is said to be "an indication that organic matter is decomposing and nitrogen is being lost," which is a rather surprising statement from one supposed to be familiar with the strong odor of sludge acid.

Other parts of the book are relatively much better, showing greater care in preparation or greater familiarity with the subject. Many an agricultural student or general reader will find in the book much that will be new to him, and consequently interesting.

H. J. WHEELER

NEW PUBLICATIONS

By CLARA M. GUPPY, Librarian, Mellon Institute of Industrial Research, Pittsburgh

- Acids, Alkalies and Salts.** G. H. J. ADLAM. 8vo. 121 pp. Price, 2s. 6d. Sir Isaac Pitman & Sons, New York.
- Asbestos and the Asbestos Industry; the World's Most Wonderful Mineral, and Other Fireproof Materials.** A. L. SUMMERS. 12mo. 107 pp. Price, \$1.00. Sir Isaac Pitman & Sons, New York.
- Chemical Molecule: La Molecule Chimique.** R. LESPIEAU. 287 pp. Price, 3 fr. 50. F. Alcan, Paris.
- Chemistry: A Class Book of Organic Chemistry.** Vol. II. J. B. COHEN. 156 pp. Price, 4s. 6d. Macmillan & Co., Ltd., London.
- Chemistry and Technology of the Diazo-Compounds.** J. C. CAIN. 2nd Ed. 199 pp. Price, 12s. 6d. E. Arnold, London.
- Chemistry for Textile Students.** BARKER NORTH AND NORMAN BLAND. 379 pp. Price, 30s. University Press, Cambridge.
- Chemistry: Service Chemistry; Being a Short Manual of Chemistry and Metallurgy and Their Application in the Naval and Military Services.** V. B. LEWES AND J. S. S. BRAME. 5th Ed. 8vo. 576 pp. 21s. Edward Arnold, London.
- Chemists' Manual of Nonferrous Alloys.** J. R. DOWNIE. 168 pp. Price, 10s. E. & F. N. Spon, London; Spon & Chamberlain, Ltd., New York.
- Coal: Chemistry of Coal.** J. B. ROBINSON. 96 pp. Price, 3s. 6d. Gurney and Jackson, London.
- Cordage and Cordage Hemp and Fibers.** THOMAS WOODHOUSE AND P. KILGOUR. 12mo. 113 pp. Price, \$1.00. Sir Isaac Pitman & Sons, New York.
- Dyes: Manufacture of Intermediate Products for Dyes.** J. C. CAIN. 2nd Ed. 273 pp. Price, 10s. Macmillan & Co., Ltd., London.
- Galvanizing and Tinning.** W. T. FLANDERS. 8vo. 354 pp. Price, \$4.00. U. P. C. Book Co., Inc., New York.
- Hendricks' Commercial Register of the United States for Buyers and Sellers, 1919-1920.** 28th Annual Ed. 2703 pp. Price, \$12.50. S. E. Hendricks Co., Inc., New York.
- Ions, Electrons, and Ionizing Radiations.** J. A. CROWTHER. 276 pp. Price, 12s. 6d. E. Arnold, London.
- Iron: An Outline of the Metallurgy of Iron and Steel.** A. H. SEXTON AND J. S. PRIMROSE. 2nd Ed. 8vo. 572 pp. Price, 15s. Scientific Publishing Co., Salford, England.
- Limes and Cements: Their Nature, Manufacture and Use. An Elementary Treatise.** E. A. DANCASTER. 2nd Ed. revised and enlarged. 220 pp. 7s. 6d. C. Lockwood & Son, London.
- Lubricants: Memorandum on Solid Lubricants.** T. C. THOMSEN AND L. ARCHBUTT. 28 pp. H. M. Stationery Office, London.
- Metallurgy: Practical Metallurgy.** THOMAS TURNER. 2nd Ed. 108 pp. Price, 3s. 6d. C. Griffin & Co., London.
- Oil Analysis: A Short Handbook of Oil Analysis.** A. H. GILL. 9th Ed. revised. 215 pp. Price, \$2.50. J. B. Lippincott Co., Philadelphia.
- Organic Chemistry: Laboratory Manual of Organic Chemistry.** H. L. FISHER. 8vo. 331 pp. Price, \$2.25. John Wiley & Sons, Inc., New York.
- Rarer Metals: Mineralogy of the Rarer Metals.** E. CAHEN AND W. O. WOOTTON. 2nd Ed. revised. 246 pp. Price, 10s. 6d. C. Griffin & Co., London.
- Reagent Chemicals: Standards and Tests for Reagent Chemicals.** B. L. MURRAY. 8vo. 400 pp. Price, \$2.00. D. Van Nostrand Co., New York.
- Silk Industry and Trade: A Study of the Economic Organization of the Export Trade of Kashmir and Indian Silks with Special Reference to the Utilization in the British and French Markets.** R. C. HAWLEY. 172 pp. Price, 10s. 6d. P. S. King & Son, Ltd., London.
- Textiles: Piece Goods Manual.** A. E. BLANCO. Price, \$1.25. Statistical Dept. of the Inspectorate General of Chinese Maritime Customs, Shanghai. (Sold by G. D. Stechert & Co., New York.)
- Zinc and Its Alloys.** T. E. LONES. 8vo. 136 pp. Price, \$1.00. Sir Isaac Pitman & Sons, New York.
- Aluminum Bronze: The Still Process for Casting Aluminum Bronze. A Complete Description of a New Method for Preventing the Formation of Alumina in Aluminum Bronze.** P. H. G. DURVILLE. *The Metal Industry*, Vol. 18 (1920), No. 3, pp. 118-120.
- Catalysis from an Industrial Standpoint.** E. B. MAXTED. *The Chemical Age* (London), Vol. 2 (1920), No. 37, pp. 227.
- Catalyzers in Paint and Varnish Manufacture.** F. P. INGALLS. *Chemical and Metallurgical Engineering*, Vol. 22 (1920), No. 13, pp. 590-592.
- Cellulose: Titration of Cellulose.** WALTER QVIST. *Pulp and Paper Magazine*, Vol. 18 (1920), No. 11, pp. 261-262.
- Chemical Industry: Need of Coöperative Educational Work in the Chemical Industry.** J. G. COHEN. *Chemical and Metallurgical Engineering*, Vol. 22 (1920), No. 11, pp. 509-512.
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- Colloidal Clay as a Catalyst in Oxidation and Hydrogenation.** J. G. HAY. *The Chemical Age* (London), Vol. 2 (1920), No. 36, p. 194.
- Coloring Paper: The Theory and Art of Coloring Paper.** OTTO KRESS. *Pulp and Paper Magazine*, Vol. 18 (1920), No. 11, pp. 255-259.
- Copper: The Requirements of Refined Copper.** LAWRENCE ADDICKS. *Chemical and Metallurgical Engineering*, Vol. 22 (1920), No. 10, pp. 449-453.
- Cotton Oil Industry: Pictorial Description of the Cotton Oil Industry.** DAVID WESSON. *Chemical and Metallurgical Engineering*, Vol. 22 (1920), No. 10, pp. 465-472.
- Dye Exports Essential to Upkeep of American Foreign Trade.** L. W. ALWYN-SCHMIDT. *Color Trade Journal*, Vol. 6 (1920), No. 3, pp. 69-72.
- Fused Silica: Commercial Development of Fused Silica; an Account of the Development of the Electrothermal Processes of Fusing Glass Sand.** JOHN SCHARL AND WALLACE SAVAGE. *Chemical and Metallurgical Engineering*, Vol. 22 (1920), No. 13, pp. 593-595.
- Glass: Scientific Research and the Glass Industry in the United States.** M. W. TRAVERS. *Nature*, Vol. 105 (1920), No. 2627, pp. 9-10.
- Indigo Carmine.** I. J. M. MATTHEWS. *Color Trade Journal*, Vol. 6 (1920), No. 3, p. 96.
- Lead: Action of Water on Lead.** J. F. LIVERSEEGE AND A. W. KNAPP. *Journal of the Society of Chemical Industry*, Vol. 39 (1920), No. 3, pp. 271-331.
- Malt: On the Influence of the Temperature, Concentration, Duration of the Mash, and Slackness of Malt on the Starch Products of the Extract of Malt.** JAMES O'SULLIVAN. *Journal of the Society of Chemical Industry*, Vol. 39 (1920), No. 3, pp. 221-271.
- Mineral Oils: Some Laboratory Tests on Mineral Oils.** ARNOLD PHILIP. *Journal of the Institute of Petroleum Technologists*, Vol. 6 (1920), No. 21, pp. 5-61.
- Mustard Gas: History of Mustard Gas.** C. J. WEST. *Chemical and Metallurgical Engineering*, Vol. 22 (1920), No. 12, pp. 541-542.
- Nitrogen: Political and Commercial Control of the Nitrogen Resources of the World.** I. C. G. GILBERT. *Chemical and Metallurgical Engineering*, Vol. 22 (1920), No. 10, pp. 443-448.
- Oil: The Conservation of Oil.** PHILIP DUMAS. *Journal of the Institute of Petroleum Technologists*, Vol. 6 (1920), No. 21, pp. 82-97.
- Oxygen, Nitrogen and Rare Gases from the Air.** C. R. HOUSEMAN. *The Chemical Age* (London), Vol. 2 (1920), No. 36, pp. 198-199.
- Paper from Bagasse; How Sugarcane Refuse Is Treated to Make Good Paper Pulp.** C. J. WEST. *Paper*, Vol. 26 (1920), No. 2, pp. 26, 28.
- Saccharin versus Sugar.** J. F. QUEENY. *Chemical Age*, Vol. 2 (1920), No. 2, pp. 367-369.
- Shale-Oil: Distillation of Shale-Oil.** J. A. BISHOP. *Mining and Scientific Press*, Vol. 120 (1920), No. 11, pp. 371-375.
- Solidification Points of Mixtures of Metallic Chlorides. Part I. Mixtures of Sodium, Potassium, and Calcium Chlorides.** F. C. A. H. LANTSBERRY AND R. A. PAGE. *Journal of the Society of Chemical Industry*, Vol. 39 (1920), No. 4, pp. 371-411.
- Sugar: Extraction and Production of Sugar from Different Vegetable Sources.** A. R. LING. *Chemical News*, Vol. 120 (1919), No. 3124, pp. 98-101.
- Talc: A Talk on Talc.** FREELAND JEWETT. *Chemical Age*, Vol. 2 (1920), No. 2, pp. 389-390.
- Tungsten: The Metallurgy of Tungsten.** C. W. BALKE. *Chemical Age*, Vol. 2 (1920), No. 2, p. 366.
- Tungsten Ore Deposits in Burma, Notes on.** J. C. BROWN. *Journal of the Society of Chemical Industry*, Vol. 39 (1920), No. 4, pp. 441-481.
- Water: Filtration and Softening of Water.** G. G. HEPBURN. *The Chemical Age* (London), Vol. 2 (1920), No. 37, pp. 226-227.

NEW JOURNAL

- Discovery: A Monthly Popular Journal of Knowledge.** Ed. by A. S. RUSSELL, 8 Severn Road, Sheffield. Price, 7s. 6d. Published by Mr. John Murray, 50a Albemarle St., London, W. 1, England. (Sold by W. Wesley & Son, 28 Essex St. Strand, London, W. C., England.)

RECENT JOURNAL ARTICLES

- Abrasives: Manufacture of Artificial Abrasives in the Electric Furnace.** C. J. BROCKBANK. *Journal of the Society of Chemical Industry*, Vol. 39 (1920), No. 4, pp. 411-441.
- Alcohol: Industrial Alcohol under the New Law and Regulations.** B. R. TUNISON. *Chemical and Metallurgical Engineering*, Vol. 22 (1920), No. 11, pp. 513-518.

MARKET REPORT—APRIL, 1920

FIRST-HAND PRICES FOR GOODS IN ORIGINAL PACKAGES PREVAILING IN THE NEW YORK MARKET

INORGANIC CHEMICALS

	Apr. 1	Apr. 15
Acid, Boric, cryst., bbls.....lb.	.16 ¹ / ₂	.16 ¹ / ₂
Hydrochloric, com'l, 22°.....lb.	.02	.02
Hydriodic.....oz.	.19	.19
Nitric, 42°.....lb.	.08 ¹ / ₂	.08 ¹ / ₂
Phosphoric, 50% tech.....lb.	.25	.25
Sulfuric, C. P.....lb.	.08	.08
Chamber, 66°.....ton	22.00	22.00
Oleum.....ton	27.00	27.00
Alum, ammonia, lump.....lb.	.04	.04
Aluminum Sulfate (iron-free).....lb.	.03	.03
Ammonium Carbonate, pwd.....lb.	.13 ¹ / ₂	.14 ¹ / ₂
Ammonium Chloride, gran.....lb.	.18	.18
Ammonia Water, carboys, 26°.....lb.	.10	.10
Arsenic, white.....lb.	.14	.14
Barium Chloride.....ton	175.00	175.00
Nitrate.....lb.	.11	.11
Barytes, white.....ton	25.00	25.00
Bleaching Powd., 35%, Works, 100 lbs.	4.00	4.50
Borax, cryst., bbls.....lb.	.09 ¹ / ₂	.09 ¹ / ₂
Bromine, tech., bulk.....lb.	.85	.85
Calcium Chloride, fused.....ton	22.00	22.00
Chalk, precipitated.....lb.	.05	.05
China Clay, imported.....ton	18.00	18.00
Copper Sulfate.....100 lbs.	8.25	8.25
Feldspar.....ton	8.00	8.00
Fuller's Earth.....100 lbs.	1.50	1.50
Iodine, resublimed.....lb.	4.10	4.35
Lead Acetate, white crystals.....lb.	.14	.14
Nitrate.....lb.	.15	.15
Red American.....100 lbs.	.10 ¹ / ₂	.10 ¹ / ₂
White American.....100 lbs.	.09 ¹ / ₂	.09 ¹ / ₂
Lime Acetate.....100 lbs.	3.00	3.25
Lithium Carbonate.....lb.	1.50	1.50
Magnesium Carbonate, Tech.....lb.	.12	.12
Magnesite.....ton	65.00	65.00
Mercury flask.....75 lbs.	98.00	104.00
Phosphorus, yellow.....lb.	.35	.35
Plaster of Paris.....100 lbs.	1.50	1.50
Potassium Bichromate.....lb.	.40	.40
Bromide.....lb.	.90	.90
Carbonate, calc., 80-85%.....lb.	.22	.22
Chlorate, cryst.....lb.	.15	.15
Cyanide, bulk, 98-99%.....lb.	.30	.30
Hydroxide, 88-92%.....lb.	.30	.30
Iodide, bulk.....lb.	3.10	3.35
Nitrate.....lb.	.14	.14
Permanganate, U. S. P.....lb.	.75	.75
Salt Cake.....ton	18.00	25.00
Silver Nitrate.....oz.	.80	.80
Soapstone, in bags.....ton	12.00	12.00
Soda Ash, 58%, bags.....100 lbs.	3.25	4.00
Caustic, 76%.....100 lbs.	6.25	6.00
Sodium Acetate.....lb.	.08 ¹ / ₂	.08 ¹ / ₂
Bicarbonate.....100 lbs.	2.25	2.25
Bichromate.....lb.	.30	.30
Chlorate.....lb.	.10	.10
Cyanide.....lb.	.27	.27
Fluoride, technical.....lb.	.16	.18
Hyposulfite.....100 lbs.	3.60	3.60
Nitrate, 95%.....100 lbs.	3.85	3.80
Silicate, 40°.....lb.	.02	.02
Sulfide.....lb.	.06 ¹ / ₂	.06 ¹ / ₂
Bisulfite, powdered.....lb.	.06 ¹ / ₂	.06 ¹ / ₂
Strontium Nitrate.....lb.	.25	.27
Sulfur, flowers.....100 lbs.	3.75	3.75
Crude.....long ton	30.00	30.00
Talc, American, white.....ton	20.00	20.00
Tin Bichloride.....lb.	.21 ¹ / ₂	.21 ¹ / ₂
Oxide.....lb.	.60	.60
Zinc Chloride, U. S. P.....lb.	.50	.50
Oxide, bbls.....lb.	.22	.22

ORGANIC CHEMICALS

Acetanilid.....lb.	.65	.75
Acid, Acetic, 28 p. c.....100 lbs.	3.00	3.25
Glacial.....lb.	.14	.16 ¹ / ₂
Acetylsalicylic.....lb.	1.00	.95
Benzoic, U. S. P., ex-toluol.....lb.	.85	.85
Carbolic, cryst., U. S. P., drs.....lb.	.15	.15
50- to 110-lb. tins.....lb.	.21	.21

Acid (Concluded)

	Apr. 1	Apr. 15
Citric, crystals, bbls.....lb.	1.20	1.18
Oxalic, cryst., bbls.....lb.	.45	.50
Pyrogallic, resublimed.....lb.	2.50	2.50
Salicylic, bulk, U. S. P.....lb.	.55	.55
Tartaric, crystals, U. S. P.....lb.	.78	.83
Trichloracetic, U. S. P.....lb.	4.40	4.40
Acetone, drums.....lb.	.16	.20
Alcohol, denatured, 190 proof....gal.	1.01	1.01
Ethyl, 190 proof.....gal.	7.50	8.00
Wood, Pure.....gal.	3.00	3.50
Amyl Acetate.....gal.	4.00	4.00
Camphor, Jap. refined.....lb.	2.50	2.25
Carbon Bisulfide.....lb.	.06	.06
Tetrachloride.....lb.	.11	.11
Chloroform, U. S. P.....lb.	.30	.35
Cresote, U. S. P.....lb.	.75	.75
Cresol, U. S. P.....lb.	.18	.18
Dextrine, corn.....lb.	.06 ¹ / ₄	.06 ¹ / ₄
Imported Potato.....lb.	.14	.14
Ether, U. S. P., conc., 100 lbs.....lb.	.19	.19
Formaldehyde.....lb.	.57	.53
Glycerin, dynamite, drums.....lb.	.22	.21 ¹ / ₂
Starch corn.....100 lbs.	5.25	5.25
Potato, Jap.....lb.	.06	.06
Rice.....lb.	.25	.25
Sago.....lb.	.05	.05

OILS, WAXES, ETC.

Beeswax, pure, white.....lb.	.65	.65
Black Mineral Oil, 29 gravity....gal.	.22	.22
Castor Oil, No. 3.....lb.	.18	.18
Ceresin, yellow.....lb.	.14	.14
Corn Oil, crude.....lb.	.18	.17 ¹ / ₂
Cottonseed Oil, crude, f. o. b. mill..lb.	.17 ¹ / ₄	.15 ¹ / ₂
Menhaden Oil, crude (southern). gal.	.95	.95
Neat's-foot Oil, 20°.....gal.	2.25	2.25
Paraffin, 128-130 m. p., ref.....lb.	.11 ¹ / ₂	.11 ¹ / ₂
Paraffin Oil, high viscosity.....gal.	.45	.45
Rosin, "F" Grade, 280 lbs.....bbl.	18.55	19.75
Rosin Oil, first run.....gal.	.90	.90
Shellac, T. N.....lb.	1.35	1.35
Spermaceti, cake.....lb.	.30	.30
Sperm Oil, bleached winter, 38°....gal.	1.95	1.95
Stearic Acid, double-pressed.....lb.	.27 ¹ / ₂	.27 ¹ / ₂
Tallow Oil, acidless.....gal.	1.70	1.60
Tar Oil, distilled.....gal.	.60	.60
Turpentine, spirits of.....gal.	2.35	nominal

METALS

Aluminum, No. 1, ingots.....lb.	.31 ¹ / ₂	.31 ¹ / ₂
Antimony, ordinary.....100 lbs.	11.75	11.75
Bismuth.....lb.	2.52	2.52
Copper, electrolytic.....lb.	.18 ¹ / ₂	.19
Lake.....lb.	.18 ³ / ₄	.19
Lead, N. Y.....lb.	.09 ¹ / ₄	.09 ¹ / ₄
Nickel, electrolytic.....lb.	.45	.45
Platinum, refined, soft.....oz.	155.00	155.00
Quicksilver, flask.....75 lbs ea.	98.00	104.00
Silver.....oz.	1.35	1.27
Tin.....lb.	.61 ¹ / ₄	.62 ¹ / ₄
Tungsten Wolframite.....per unit	7.00	7.00
Zinc, N. Y.....100 lbs.	9.00	9.00

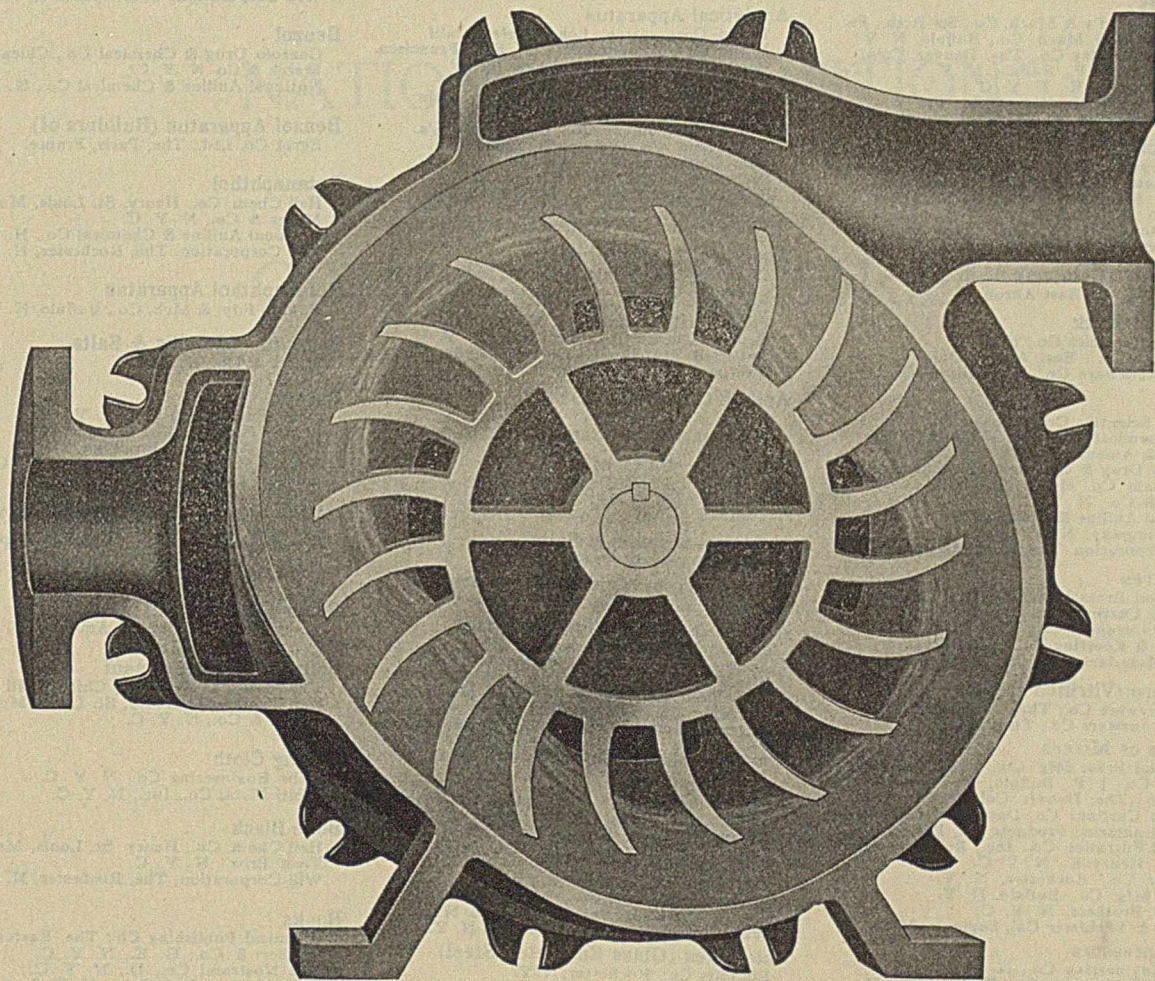
FERTILIZER MATERIALS

Ammonium Sulfate.....100 lbs.	7.10	7.10
Blood, dried, f. o. b. N. Y.....unit	8.00	8.00
Bone, 3 and 50, ground, raw.....ton	48.00	48.00
Calcium Cyanamide, unit of Ammonia.....	4.00	4.00
Fish Scrap, domestic, dried, f. o. b. works.....unit	7.25	7.25
Phosphate Rock, f. o. b. mine:		
Florida pebble, 68%.....ton	6.85	6.85
Tennessee, 78-80%.....ton	11.00	11.00
Potassium muriate, 80%.....unit	2.60	2.60
Pyrites, furnace size, imported....unit	.18	.18
Tankage, high-grade, f. o. b. Chicago.....unit	7.75	7.75

COAL-TAR CHEMICALS		Apr. 1	Apr. 15	Acid Colors (Continued)		Apr. 1	Apr. 15
Crudes							
Benzol, C. P.	gal.	.33	.33	Fuchsin	lb.	2.50	2.50
Cresol, U. S. P.	lb.	.18	.18	Orange III	lb.	1.00	1.00
Naphthalene, flake	lb.	.10	.12 1/2	Red	lb.	1.10	1.10
Phenol, drums	lb.	.15	.15	Violet 10B	lb.	6.50	6.50
Toluol, 90%	gal.	.34	.34	Alkali Blue, domestic	lb.	4.75	4.75
Xylol, water white	gal.	.40	.40	Imported	lb.	8.00	8.00
Intermediates							
Acids:							
Anthranilic	lb.	2.75	2.75	Azo Carmine	lb.	4.00	4.00
Benzoic	lb.	.85	.85	Azo Yellow	lb.	2.00	2.00
Broenner's	lb.	1.75	1.75	Erythrosine	lb.	12.00	12.00
Cleves	lb.	2.00	2.00	Indigotine, conc.	lb.	3.00	3.00
Cresylic, 97-99%	gal.	1.00	1.10	Paste	lb.	1.50	1.50
F	lb.	3.00	3.00	Naphthol Green	lb.	1.50	1.50
Gamma	lb.	2.50	2.50	Ponceau	lb.	1.00	1.00
H	lb.	1.85	1.85	Scarlet 2R	lb.	1.00	1.00
Metanilic	lb.	1.70	1.70	Direct Colors			
Monosulfonic F	lb.	3.00	3.00	Black	lb.	1.00	1.00
Naphthionic crude	lb.	.70	.70	Blue 2B	lb.	.70	.70
Neville & Winther's	lb.	1.90	1.90	Brown R	lb.	1.80	1.80
Phthalic	lb.	.60	.60	Fast Red	lb.	3.50	3.50
Picric	lb.	.25	.25	Yellow	lb.	2.00	2.00
Sulfanilic	lb.	.35	.35	Violet, con't.	lb.	2.20	2.20
Tobias	lb.	2.25	2.25	Chrysophenine, domestic	lb.	2.25	2.25
Amido Azo Benzol	lb.	1.15	1.15	Imported	lb.	3.80	3.80
Aniline Oil	lb.	.35	.36	Congo Red, 4B Type	lb.	.90	.90
For Red	lb.	.65	.65	Primuline, domestic	lb.	3.00	3.00
Aniline Salt	lb.	.47	.47	Oil Colors			
Anthracene, 80-85%	lb.	.75	.75	Black	lb.	.70	.70
Anthraquinone	lb.	4.50	4.50	Blue	lb.	1.65	1.65
Benzaldehyde, tech.	lb.	.75	.75	Orange	lb.	1.40	1.40
U. S. P.	lb.	1.25	1.10	Red III	lb.	1.65	1.65
Benzidine Base	lb.	1.40	1.40	Scarlet	lb.	1.75	1.75
Benzidine Sulfate	lb.	1.15	1.15	Yellow	lb.	1.70	1.70
Diamidophenol	lb.	6.00	6.00	Nigrosine Oil, solution	lb.	.90	.90
Dianisidine	lb.	9.00	9.00	Sulfur Colors			
p-Dichlorobenzol	lb.	.10	.10	Black	lb.	.20	.20
Diethylaniline	lb.	1.40	1.40	Blue, domestic	lb.	.70	.70
Dimethylaniline	lb.	2.00	1.45	Brown	lb.	.35	.35
Dinitrobenzol	lb.	.36	.36	Green	lb.	1.00	1.00
Dinitrotoluol	lb.	.43	.43	Yellow	lb.	.90	.90
Diphenylamine	lb.	.85	.80	Chrome Colors			
G Salt	lb.	.75	.90	Alizarin Blue, bright	lb.	7.75	7.75
Hydroquinone	lb.	2.00	2.00	Alizarin Red, 20% Paste	lb.	1.10	1.10
Metol	lb.	11.50	11.50	Alizarin Yellow G	lb.	1.00	1.00
Monochlorobenzol	lb.	.12	.12	Chrome Black, domestic	lb.	1.25	1.25
Monoethylaniline	lb.	2.15	2.15	Imported	lb.	2.20	2.20
a-Naphthylamine	lb.	.45	.45	Chrome Blue	lb.	2.50	2.50
b-Naphthylamine	lb.	1.15	1.15	Chrome Green, domestic	lb.	1.50	1.50
b-Naphthol, crude	lb.	.65	.70	Chrome Red	lb.	2.00	2.00
m-Nitraniline	lb.	1.00	1.00	Gallocyanine	lb.	3.25	3.25
p-Nitraniline	lb.	1.50	1.55	Basic Colors			
Nitrobenzol, crude	lb.	.16	.16	Auramine, O, domestic	lb.	2.50	2.50
Rectified (Oil Myrbane)	lb.	.18	.18	Auramine, OO	lb.	4.15	4.15
p-Nitrophenol	lb.	.80	.80	Bismarck Brown Y	lb.	.90	.90
p-Nitrosodimethylaniline	lb.	1.90	1.90	Bismarck Brown R	lb.	1.20	1.20
o-Nitrotoluol	lb.	.22	.22	Chrysoidine R	lb.	.75	.75
p-Nitrotoluol	lb.	1.15	1.15	Chrysoidine Y	lb.	.75	.75
m-Phenylenediamine	lb.	1.35	1.35	Green Crystals, Brilliant	lb.	6.00	6.00
p-Phenylenediamine	lb.	2.50	2.50	Indigo, 20 p. c. paste	lb.	.85	.85
Phthalic Anhydride	lb.	.45	.45	Fuchsin Crystals, domestic	lb.	5.00	5.50
Primuline Base	lb.	3.00	3.00	Imported	lb.	12.00	12.00
R Salt	lb.	.75	.75	Magenta Acid, domestic	lb.	4.25	4.25
Resorcin, tech.	lb.	4.00	4.00	Malachite Green, crystals	lb.	3.75	3.75
U. S. P.	lb.	6.00	6.00	Methylene Blue, tech.	lb.	2.50	2.75
Schaeffer Salt	lb.	.75	.75	Methyl Violet 3 B	lb.	2.60	2.60
Sodium Naphthionate	lb.	1.10	1.10	Nigrosine, spts. sol.	lb.	.85	.85
Thiocarbanilid	lb.	.60	.60	Water sol., blue	lb.	.65	.65
Tolidine, Base	lb.	1.75	1.65	Jet	lb.	.90	.90
Toluidine, mixed	lb.	.44	.44	Phosphine G., domestic	lb.	7.00	7.00
o-Toluidine	lb.	.30	.30	Rhodamine B, ex-con't.	lb.	35.00	35.00
m-Toluylenediamine	lb.	1.35	1.35	Victoria Blue, base, domestic	lb.	6.00	6.00
p-Toluidine	lb.	1.75	1.75	Victoria Green	lb.	6.00	6.00
Xylidene, crude	lb.	.40	.40	Victoria Red	lb.	7.00	7.00
COAL-TAR COLORS							
Acid Colors							
Black	lb.	1.15	1.15	Victoria Yellow	lb.	7.00	7.00
Blus	lb.	3.00	3.00				

NASH HYTOR—

THE SIMPLEST VACUUM PUMP OR COMPRESSOR



THE pump for air, industrial gas, or acid gas. No valves. No gears. No pistons. No piston packing. No sliding vanes. No interior lubrication necessary. Absolutely clean air, delivered without pulsation. Rotor only moving part, cast in one piece, heavily shrouded, and mounted on ball bearings outside of casing. Long life. Constant efficiency. Positive service.

NASH ENGINEERING CO.

South Norwalk, Conn., U. S.

CLASSIFIED LIST OF CHEMICAL EQUIPMENT

Absorption Towers

Duriron Castings Co., Dayton, O. (Acid Proof)
 General Ceramics Co., N. Y. C.
 Knight, Maurice A., East Akron, Ohio.
 Lummus Co., The Walter E. Boston, Mass.
 Thermal Syndicate, Ltd., The N. Y. C.
 U. S. Stoneware Co., The, Akron, O.

Acid and Alkali Resistant Apparatus

Buffalo Fdy & Mch. Co., Buffalo, N. Y.
 Cleveland Brass Mfg. Co., The, Cleveland, O.
 Duriron Castings Co., Dayton, O. (Acid Proof)
 Elyria Enameled Prod. Co., Elyria, O.
 Jacoby, Henry E., N. Y. C.
 Pfaudler Co., Rochester, N. Y.

Acid Distillation Apparatus

Thermal Syndicate, Ltd., The, N. Y. C.

Acid Eggs

Bethlehem Fdy & Mch. Co., So Beth, Pa.
 Buffalo Fdy & Mch. Co., Buffalo, N. Y.
 Denver Fire Clay Co., The, Denver, Colo.
 Devine Co., J. P., Buffalo, N. Y.
 Jacoby, Henry E., N. Y. C.
 Schutte & Koerting Co., Phila., Pa.
 U. S. Stoneware Co., The, Akron, O.

Acid Pitchers

Guernseyware Co., The, Cambridge, O.
 Heil Chem. Co., Henry, St. Louis, Mo.
 Knight, Maurice A., East Akron, O.
 Palo Co., N. Y. C.
 U. S. Stoneware Co., The, Akron, O.
 Will Corporation, The, Rochester, N. Y.

Acid Plants (Complete or in Part)

Knight, M. A., East Akron, O.

Acid Proof Brick

General Ceramics Co., N. Y. C.
 Knight, M. A., East Akron, Ohio.
 U. S. Stoneware Co., The, Akron, O.

Acids

Central Scientific Co., Chicago, Ill.
 Dow Chemical Co., The, Midland, Mich.
 Eimer & Amend, N. Y. C.
 Gazzolo Drug & Chemical Co., Chicago, Ill.
 Heil Chem. Co., Henry, St. Louis, Mo.
 Merck & Co., N. Y. C.
 National Aniline & Chemical Co., N. Y. C.
 Palo Company, N. Y. C.
 Will Corporation, The, Rochester, N. Y.

Acid Valves

Cleveland Brass Mfg. Co., The, Cleveland, O.
 Duriron Castings Co., Dayton, O.
 General Ceramics Co., N. Y. C.
 Schutte & Koerting Co., Phila., Pa.
 York Manufacturing Co., York, Pa.

Acid Ware (Vitrified Clay)

Guernseyware Co., The, Cambridge, O.
 U. S. Stoneware Co., The, Akron, O.

Agitators or Mixers

Cleveland Brass Mfg. Co., The, Cleveland, O.
 Devine Co., J. P., Buffalo, N. Y.
 Dorr Co., The, Denver, Col. & N. Y. C.
 Duriron Castings Co., Dayton, O. (Acid Proof)
 Elyria Enameled Products Co., Elyria, O.
 General Filtration Co., Inc., Rochester, N. Y.
 Jacoby, Henry E., N. Y. C.
 Pfaudler Co., Rochester, N. Y.
 Sowers Mfg. Co., Buffalo, N. Y.
 Stevens Brothers, N. Y. C.
 Werner & Pfeleiderer Co., Saginaw, Mich.

Air Compressors

Abbé Engineering Co., N. Y. C.
 Crowell Engineering Co., Brooklyn, N. Y.
 General Electric Co., Schenectady, N. Y.
 Hubbard's Sons Norman, Brooklyn, N. Y.
 Nash Engineering Co., So. Norwalk, Conn.
 Schutte & Koerting Co., Phila., Pa.

Air-Diffusing Plates

General Filtration Co., Inc., Rochester, N. Y.

Air Washers

Carrier Engineering Corp., N. Y. C.

Alcohol Apparatus (Builders of)

Egrot Co., Ltd., The, Paris, France.

Alkalis

Arnold Hoffman & Co., Inc., N. Y. C.
 Lithflux Mineral & Chem. Wks., Chicago, Ill.
 National Aniline & Chemical Co., N. Y. C.

Alundum Ware

Denver Fire Clay Co., The, Denver, Colo.
 Heil Chem. Co., Henry, St. Louis, Mo.
 Norton Co., Worcester, Mass.
 Scientific Materials Co., Pittsburgh, Pa.
 Will Corporation, The, Rochester, N. Y.

Ammeters

Brown Instrument Co., Philadelphia, Pa.
 General Electric Co., Schenectady, N. Y.

Ammonia

Heil Chem. Co., Henry, St. Louis, Mo.

Ammonia Fittings and Supplies

York Manufacturing Co., York, Pa.

Ammonia—Salts of

Merck & Co., N. Y. C.

Analysis—Gas

Sarco Co., Inc., N. Y. C.

Analysis—Gas Apparatus

Braun Corporation, Los Angeles, Calif.
 Braun-Knecht-Heimann Co., San Francisco.
 Central Scientific Co., Chicago, Ill.
 Daigger & Co., A., Chicago, Ill.
 Denver Fire Clay Co., The, Denver, Colo.
 Eimer & Amend, N. Y. C.
 Emil Greiner Co., The, N. Y. C.
 Griebel Instrument Co., Carbondale, Pa.
 Heil Chem. Co., Henry, St. Louis, Mo.
 Palo Company, N. Y. C.
 Sarco Co., Inc., N. Y. C.
 Sargent & Co., E. H., Chicago, Ill.
 Scientific Materials Co., Pittsburgh, Pa.
 Thomas Co., Arthur H., Philadelphia, Pa.
 Will Corporation, The, Rochester, N. Y.

Analytical Apparatus

Braun Corporation, Los Angeles, Calif.
 Braun-Knecht-Heimann Co., San Francisco.
 Central Scientific Co., Chicago, Ill.
 Daigger & Co., A., Chicago, Ill.
 Denver Fire Clay Co., The, Denver, Colo.
 Eimer & Amend, N. Y. C.
 Griebel Instrument Co., Carbondale, Pa.
 Heil Chem. Co., Henry, St. Louis, Mo.
 Palo Company, N. Y. C.
 Sargent & Co., E. H., Chicago, Ill.
 Scientific Materials Co., Pittsburgh, Pa.
 Thomas Co., Arthur H., Philadelphia, Pa.
 Will Corporation, The, Rochester, N. Y.

Analytical Laboratories

Chemical Laboratories of N. Y., Inc., N. Y. C.
 Columbus Laboratories, Chicago, Ill.
 Dearborn Chemical Co., Chicago, Ill.
 Fort Worth Laboratories, Fort Worth, Texas.
 General Chemical Laboratories, N. Y. C.
 Sadtler & Son, Samuel P., Philadelphia, Pa.
 Westport Mill, Westport, Conn.

Aniline Oil—Salts of

Merck & Co., N. Y. C.
 National Aniline & Chemical Co., N. Y. C.

Antimony—Salts of

Merck & Co., N. Y. C.

Arresters (Dust)**Arsenic—Metallic & Salts**

Merck & Co., N. Y. C.

Arsenic Plants (Acid Proof Stoneware)

Knight, M. A., East Akron, O.

Asphalt & Tar Testing Apparatus

Central Scientific Co., Chicago, Ill.
 Denver Fire Clay Co., The, Denver, Colo.
 Eimer & Amend, N. Y. C.
 Heil Chem. Co., Henry, St. Louis, Mo.
 Scientific Materials Co., Pittsburgh, Pa.
 Thermo-Electric Instrument Co., Newark, N. J.
 Thomas Co., Arthur H., Philadelphia, Pa.
 Will Corporation, The, Rochester, N. Y.

Autoclaves

Central Scientific Co., Chicago, Ill.
 Denver Fire Clay Co., The, Denver, Colo.
 Devine Co., J. P., Buffalo, N. Y.
 Duriron Castings Co., Dayton, O. (Acid-Proof)
 Eimer & Amend, N. Y. C.
 Heil Chem. Co., Henry, St. Louis, Mo.
 Scientific Materials Co., Pittsburgh, Pa.
 Sowers Manufacturing Co., Buffalo, N. Y.
 Will Corporation, The, Rochester, N. Y.

Autoclaves (Glass Enameled Steel)

Pfaudler Co., Rochester, N. Y.

Autoclaves (High and Low Pressure)

Buffalo Fdy & Mch. Co., Buffalo, N. Y.
 Devine Co., J. P., Buffalo, N. Y.

Automatic Pressure Regulators

Brown Instrument Co., The, Phila., Pa.
 Taylor Instrument Cos., Rochester, N. Y.

Automatic Temperature Regulators

Brown Instrument Co., The, Phila., Pa.
 Engelhard, Charles, N. Y. C.
 Taylor Instrument Cos., Rochester, N. Y.

Bakelite

General Bakelite Co., N. Y. C.

Balances and Weights

Becker Inc., Christian, N. Y. C.
 Braun Corporation, Los Angeles, Calif.
 Braun-Knecht-Heimann Co., San Francisco.
 Central Scientific Co., Chicago, Ill.
 Daigger & Co., A., Chicago, Ill.
 Denver Fire Clay Co., The, Denver, Colo.
 Eimer & Amend, N. Y. C.
 Griebel Instrument Co., Carbondale, Pa.
 Heil Chem. Co., Henry, St. Louis, Mo.
 Palo Company, N. Y. C.
 Sargent & Co., E. H., Chicago, Ill.
 Scientific Materials Co., Pittsburgh, Pa.
 Thomas Co., Arthur H., Philadelphia, Pa.
 Will Corporation, The, Rochester, N. Y.

Barium—Salts of

Dow Chemical Co., The, Midland, Mich.
 Heil Chem. Co., Henry, St. Louis, Mo.
 Merck & Co., N. Y. C.
 Will Corporation, The, Rochester, N. Y.

Barometers

Brown Instrument Co., The, Phila., Pa.
 Heil Chem. Co., Henry, St. Louis, Mo.
 Taylor Instrument Cos., Rochester, N. Y.
 Will Corporation, The, Rochester, N. Y.

Beakers

Braun-Knecht-Heimann Corp., San Francisco
 Central Scientific Co., Chicago, Ill.
 Daigger & Co., A., Chicago, Ill.
 Denver Fire Clay Co., The, Denver, Colo.
 Eimer & Amend, N. Y. C.
 Guernseyware Co., The, Cambridge, O.
 Heil Chem. Co., Henry, St. Louis, Mo.
 Herold China & Pottery Co., Golden, Col.
 Palo Company, N. Y. C.
 Sargent & Co., E. H., Chicago, Ill.
 Scientific Materials Co., Pittsburgh, Pa.
 Will Corporation, The, Rochester, N. Y.

Benzol

Gazzolo Drug & Chemical Co., Chicago, Ill.
 Merck & Co., N. Y. C.
 National Aniline & Chemical Co., N. Y. C.

Benzol Apparatus (Builders of)

Egrot Co. Ltd., The, Paris, France.

Betanaphthol

Heil Chem. Co., Henry, St. Louis, Mo.
 Merck & Co., N. Y. C.
 National Aniline & Chemical Co., N. Y. C.
 Will Corporation, The, Rochester, N. Y.

Betanaphthol Apparatus

Buffalo Fdy & Mch. Co., Buffalo, N. Y.

Bismuth—Metallic & Salts

Merck & Co., N. Y. C.

Blowers

Abbé Engineering Co., N. Y. C.
 Central Scientific Co., Chicago, Ill.
 Crowell Mfg. Co., Brooklyn, N. Y.
 Denver Fire Clay Co., The, Denver, Colo.
 Duriron Castings Co., Dayton, O.
 Eimer & Amend, N. Y. C.
 Heil Chem. Co., Henry, St. Louis, Mo.
 Palo Company, N. Y. C.
 Scientific Materials Co., Pittsburgh, Pa.
 Schutte & Koerting Co., Phila., Pa.
 Will Corporation, The, Rochester, N. Y.

Boiler Coverings

Magnesia Association of America, N. Y. C.

Boiler Water Treatment

Dearborn Chemical Co., Chicago, Ill.
 Heil Chem. Co., Henry, St. Louis, Mo.
 Merck & Co., N. Y. C.

Bolting Cloth

Abbé Engineering Co., N. Y. C.
 Multi-Metal Co., Inc., N. Y. C.

Bone Black

Heil Chem. Co., Henry, St. Louis, Mo.
 Toeb Bros., N. Y. C.
 Will Corporation, The, Rochester, N. Y.

Books

Chemical Publishing Co., The, Easton, Pa.
 Stechert & Co., G. E., N. Y. C.
 Van Nostrand Co., D., N. Y. C.
 Wiley & Sons, Inc., John, N. Y. C.

Bottles (Glass)

Central Scientific Co., Chicago, Ill.
 Denver Fire Clay Co., The, Denver, Colo.
 Eimer & Amend, N. Y. C.
 Heil Chem. Co., Henry, St. Louis, Mo.
 Scientific Materials Co., Pittsburgh, Pa.
 Wheaton Co. T. C., Millville, N. J.
 Will Corporation, The, Rochester, N. Y.

Brinell Machines

Central Scientific Co., Chicago, Ill.
 Denver Fire Clay Co., The, Denver, Colo.
 Eimer & Amend, N. Y. C.
 Heil Chem. Co., Henry, St. Louis, Mo.
 Palo Company, N. Y. C.
 Scientific Materials Co., Pittsburgh, Pa.
 Will Corporation, The, Rochester, N. Y.

Burner Guards

Heil Chem. Co., Henry, St. Louis, Mo.
 Knight M. A., East Akron, O.
 Will Corporation, The, Rochester, N. Y.

Burners—Gas and Oil

Braun Corporation, Los Angeles, Calif.
 Braun-Knecht-Heimann Co., San Francisco.
 Central Scientific Co., Chicago, Ill.
 Daigger & Co., A., Chicago, Ill.
 Denver Fire Clay Co., The, Denver, Colo.
 Detroit Heating & Lighting Co., Detroit, Mich.
 Eimer & Amend, N. Y. C.
 Griebel Instrument Co., Carbondale, Pa.
 Heil Chemical Co., Henry, St. Louis, Mo.



BULLETIN

SIXTH NATIONAL EXPOSITION OF CHEMICAL INDUSTRIES

WILL BE AT

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NEW YORK

SEPT. 20th-25th

1920



This will be the largest Industrial Exposition ever held, occupying four floors of the World's biggest Exposition Building.

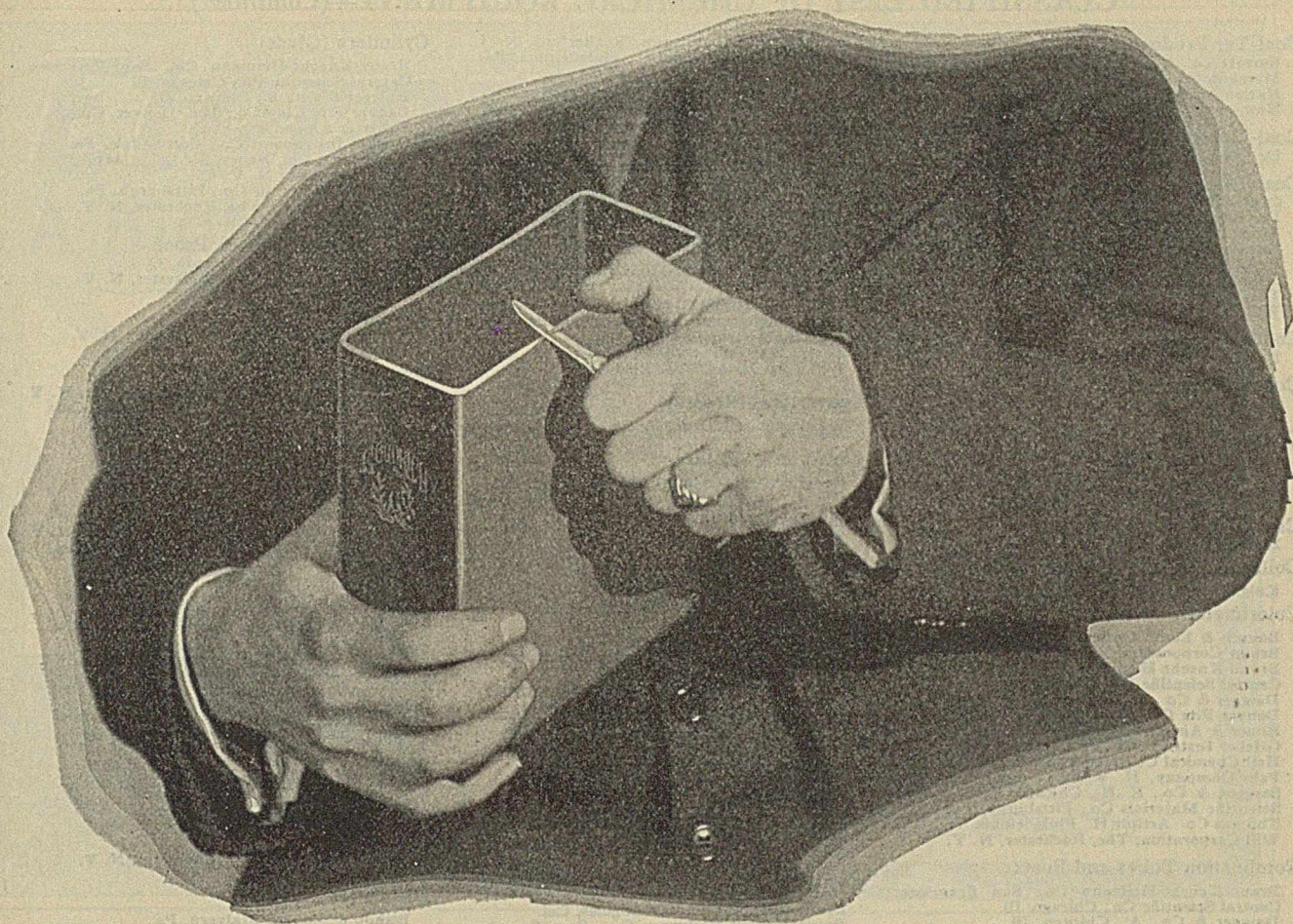
For particulars, address:

National Exposition of Chemical Industries

Grand Central Palace, New York City

CLASSIFIED LIST OF CHEMICAL EQUIPMENT—(Continued)

- Palo Company, N. Y. C.**
Sargent & Co., E. H., Chicago, Ill.
Schutte & Koerting Co., Phila., Pa.
Scientific Materials Co., Pittsburgh, Pa.
Thomas Co. Arthur H., Philadelphia, Pa.
Will Corporation, The, Rochester, N. Y.
- Burners—Sulphur**
Schutte & Koerting Co., Phila., Pa.
- Burnishing Pots and Baskets (Acid Proof)**
Knight, M. A., East Akron, Ohio.
- By-Product Machinery**
Devine Co., J. P., Buffalo, N. Y.
Swenson Evaporator Co., Chicago, Ill.
- Cabinets (Laboratory, Chemical, Sample and Specimen)**
Schwartz Sectional System, Indianapolis, Ind.
- Cable & Wire**
General Electric Co., Schenectady, N. Y.
- Cadmium—Salts of**
Dow Chemical Co., The, Midland, Mich.
Merck & Co., N. Y. C.
- Calcium—Salts of**
Dow Chemical Co., The, Midland, Mich.
Merck & Co., N. Y. C.
- Calorimeters—Fuel and Gas**
Braun Corporation, Los Angeles, Calif.
Braun-Knecht-Heimann Co., San Francisco.
Central Scientific Co., Chicago, Ill.
Daigger & Co. A., Chicago, Ill.
Denver Fire Clay Co., The, Denver, Colo.
Eimer & Amend, N. Y. C.
Griebel Instrument Co., Carbondale, Pa.
Heil Chem. Co., Henry, St. Louis, Mo.
Palo Company, N. Y. C.
Sarco Co., Inc., N. Y. C.
Sargent & Co., E. H., Chicago, Ill.
Scientific Materials Co., Pittsburgh, Pa.
Thomas Co. Arthur H., Philadelphia, Pa.
Will Corporation, The, Rochester, N. Y.
- Capsules**
Braun-Knecht-Heimann Co., San Francisco.
Central Scientific Co., Chicago, Ill.
Denver Fire Clay Co., The, Denver, Colo.
Eimer & Amend, N. Y. C.
Engelhard, Charles, N. Y. C. ("Impervite")
Heil Chem. Co., Henry, St. Louis, Mo.
Norton Co., Worcester, Mass.
Thermal Syndicate Ltd., The, N. Y. C.
Will Corporation, The, Rochester, N. Y.
- Carbon Bisulfide**
Merck & Co., N. Y. C.
National Aniline & Chemical Co., N. Y. C.
- Carbon Tetrachloride**
Dow Chemical Co., The, Midland, Mich.
Gazzolo Drug & Chemical Co., Chicago, Ill.
Merck & Co., N. Y. C.
- Carboys**
Whitall Tatum Co., Philadelphia, Pa.
Will Corporation, The, Rochester, N. Y.
- Carboy Inclinator**
Central Scientific Co., Chicago, Ill.
Denver Fire Clay Co., The, Denver, Colo.
Eimer & Amend, N. Y. C.
Heil Chem. Co., Henry, St. Louis, Mo.
Will Corporation, The, Rochester, N. Y.
- Carboy Stoppers (Acid Proof)**
Knight M. A., East Akron, O.
Whitall Tatum Co., Philadelphia, Pa.
- Casseroles**
Braun-Knecht-Heimann Co., San Francisco.
Central Scientific Co., Chicago, Ill.
Daigger & Co. A., Chicago, Ill.
Denver Fire Clay Co., The, Denver, Colo. (Acid-Proof)
Duriron Castings Co., Dayton, O.
Eimer & Amend, N. Y. C.
Guernseyware Co., The, Cambridge, O.
Heil Chem. Co., Henry, St. Louis, Mo.
Herold China & Pottery Co., Golden, Colo.
Palo Company, N. Y. C.
Sargent & Co., E. H., Chicago, Ill.
Scientific Materials Co., Pittsburgh, Pa.
Will Corporation, The, Rochester, N. Y.
- Castings (Acid and Heat Resisting)**
Bethlehem Fdy & Mach. Co., So. Beth., Pa.
Buffalo Fdy & Mch Co., Buffalo, N. Y.
Cleveland Brass Mfg Co., The, Cleveland, O.
Devine Co., J. P., Buffalo, N. Y.
Duriron Castings Co., Dayton, O. (Acid Proof)
Jacoby, Henry E., N. Y. C.
- Castings (Chemical, Acid and Caustic)**
Bethlehem Fdy & Mach Co., So. Beth., Pa.
Buffalo Fdy & Mach Co., Buffalo, N. Y.
Cast Iron Pipe Publicity Bureau, N. Y. C.
Cleveland Brass Mfg Co., The, Cleveland, O.
Devine Co., J. P., Buffalo, N. Y.
Duriron Castings Co., Dayton, O. (Acid Proof)
Ferguson & Lange Foundry Co., Chicago, Ill.
Garrigue & Co., William, Chicago, Ill.
- Castings (Chilled)**
Jacoby, Henry E., N. Y. C.
Sowers Mfg Co., Buffalo, N. Y.
- Castings—Evaporator & Vacuum Pan**
Buffalo Fdy & Mch Co., Buffalo, N. Y.
Devine Co., J. P., Buffalo, N. Y.
Garrigue & Co., William, Chicago, Ill.
Sowers Manufacturing Co., Buffalo, N. Y.
- Castings (Machinery)**
Ferguson & Lange Foundry Co., Chicago, Ill.
- Castings (Semi-steel)**
Ferguson & Lange Foundry Co., Chicago, Ill.
- Caustic Potash**
Gazzolo Drug & Chemical Co., Chicago, Ill.
Merck & Co., N. Y. C.
- Caustic Pots**
Bethlehem Fdy & Mach. Co., So. Beth., Pa.
Buffalo Fdy & Mch Co., Buffalo, N. Y.
Devine Co., J. P., Buffalo, N. Y.
- Caustic Soda**
Arnold, Hoffman & Co., Inc., N. Y. C.
General Chem. Co., N. Y. C.
Merck & Co., N. Y. C.
National Aniline & Chemical Co., N. Y. C.
- Caustic Soda Dissolvers—Automatic**
Lummus Co., The, Walter E., Boston, Mass.
- Cement Filler**
Toch Bros., N. Y. C.
- Cement Refractory**
Heil Chem. Co., Henry, St. Louis, Mo.
Norton Co., Worcester, Mass.
- Cement Testing Apparatus**
Bausch & Lomb Optical Co., Rochester, N. Y.
Braun-Knecht-Heimann Co., San Francisco.
Central Scientific Co., Chicago, Ill.
Daigger & Co. A., Chicago, Ill.
Denver Fire Clay Co., The, Denver, Colo.
Eimer & Amend, N. Y. C.
Griebel Instrument Co., Carbondale, Pa.
Heil Chemical Co., Henry, St. Louis, Mo.
Palo Company, N. Y. C.
Sargent & Co., E. H., Chicago, Ill.
Scientific Materials Co., Pittsburgh, Pa.
Thomas Co. Arthur H., Philadelphia, Pa.
Will Corporation, The, Rochester, N. Y.
- Centrifugals**
Bausch & Lomb Optical Co., Rochester, N. Y.
Braun Corporation, Los Angeles, Cal.
Braun-Knecht-Heimann Co., San Francisco.
Central Scientific Co., Chicago, Ill.
Daigger & Co. A., Chicago, Ill.
DeLaval Separator Co., N. Y. C.
Denver Fire Clay Co., The, Denver, Colo.
Eimer & Amend, N. Y. C.
Fletcher Works, Philadelphia, Pa.
Heil Chemical Co., Henry, St. Louis, Mo.
International Equipment Co., Boston, Mass.
Palo Company, N. Y. C.
Sargent & Co., E. H., Chicago, Ill.
Scientific Materials Co., Pittsburgh, Pa.
Sharples Specialty Co., The, West Chester, Pa.
Tolhurst Machine Works, Troy, N. Y.
Will Corporation, The, Rochester, N. Y.
- Centrifugal Dryers**
Fletcher Works, Philadelphia, Pa.
- Centrifugal Engineers**
Fletcher Works, Philadelphia, Pa.
Sharples Specialty Co., The, West Chester, Pa.
Tolhurst Machine Works, Troy, N. Y.
- Cerium Oxalate**
Merck & Co., N. Y. C.
- Charcoal**
Heil Chem. Co., Henry, St. Louis, Mo.
- Chemical Apparatus**
Buffalo Fdy & Mch Co., Buffalo, N. Y.
Central Scientific Co., Chicago, Ill.
Denver Fire Clay Co., The, Denver, Colo.
Detroit Heating & Lighting Co., Detroit, Mich.
Devine Co., J. P., Buffalo, N. Y.
Eimer & Amend, N. Y. C.
Fletcher Works, Philadelphia, Pa.
Heil Chem. Co., Henry, St. Louis, Mo.
Laboratory Materials Co., Chicago, Ill.
Schaar & Co., Chicago, Ill.
Scientific Materials Co., Pittsburgh, Pa.
Sowers Manufacturing Co., Buffalo, N. Y.
Werner & Pfeiderer Co., Inc., Saginaw, Mich.
Will Corporation, The, Rochester, N. Y.
- Chemical Glass Tubing**
Central Scientific Co., Chicago, Ill.
Denver Fire Clay Co., The, Denver, Colo.
Eimer & Amend, N. Y. C.
Heil Chem. Co., Henry, St. Louis, Mo.
Will Corporation, The, Rochester, N. Y.
- Chemical Glassware**
Braun Knecht Heimann Co., San Francisco.
Central Scientific Co., Chicago, Ill.
Daigger & Co. A., Chicago, Ill.
Denver Fire Clay Co., The, Denver, Colo.
Eimer & Amend, N. Y. C.
Griebel Instrument Co., Carbondale, Pa.
Heil Chemical Co., Henry, St. Louis, Mo.
Palo Company, N. Y. C.
Sargent & Co., E. H., Chicago, Ill.
Scientific Materials Co., Pittsburgh, Pa.
Thomas Co. Arthur H., Philadelphia, Pa.
Wheaton Co. T. C., Millville, N. J.
Will Corporation, The, Rochester, N. Y.
- Chemical Plants (Complete Installation)**
Buffalo Fdy & Mch Co., Buffalo, N. Y.
Devine Co., J. P., Buffalo, N. Y.
Garrigue & Co. William, Chicago, Ill.
Hercules Engineering Corp., N. Y. C.
Knight, M. A., East Akron, O.
Lummus Co., The, Walter E., Boston, Mass.
Mantius Engineering Co., N. Y. C.
Swenson Evaporator Co., Chicago, Ill.
Technical Products Co., Inc., N. Y. C. (used equipment)
- Chemical Plants (Erected and Supervised)**
Buffalo Fdy & Mch Co., Buffalo, N. Y.
Egrot Co. Ltd., The, Paris, France.
McNamara, M., Everett, Mass.
Swenson Evaporator Co., Chicago, Ill.
- Chemical Reagents**
Baker Chem. Co., J. T., Phillipsburg, N. J.
Central Scientific Co., Chicago, Ill.
Daigger & Co. A., Chicago, Ill.
Denver Fire Clay Co., The, Denver, Colo.
Eimer & Amend, N. Y. C.
General Chemical Co., Baker & Adamsons Works, Easton, Pa.
Heil Chemical Co., Henry, St. Louis, Mo.
Merck & Co., N. Y. C.
National Aniline & Chemical Co., N. Y. C.
Palo Company, N. Y. C.
Scientific Materials Co., Pittsburgh, Pa.
Will Corporation, The, Rochester, N. Y.
- Chemicals**
Baker Chemical Co., J. T., Phillipsburg, N. J.
Braun Corporation, Los Angeles, Calif.
Braun-Knecht-Heimann Co., San Francisco.
Central Scientific Co., Chicago, Ill.
Daigger & Co. A., Chicago, Ill.
Dearborn Chemical Co., Chicago, Ill.
Denver Fire Clay Co., The, Denver, Colo.
Dow Chemical Co., The, Midland, Mich.
Eastman Kodak Co., Rochester, N. Y.
Eimer & Amend, N. Y. C.
Gazzolo Drug & Chemical Co., Chicago, Ill.
General Chemical Co., N. Y. C.
General Chem. Co., B. & A. Wks., Easton, Pa.
Griebel Instrument Co., Carbondale, Pa.
Heil Chemical Co., Henry, St. Louis, Mo.
Hercules Powder Co., N. Y. C.
Laboratory Materials Co., Chicago, Ill.
Merck & Co., N. Y. C.
Monsanto Chemical Works, St. Louis, Mo.
National Aniline & Chemical Co., N. Y. C.
Newport Chemical Works, Inc., N. Y. C.
Palo Company, N. Y. C.
Pfeizer & Co., Charles, N. Y. C.
Roessler & Hasslacher Chemical Co., N. Y. C.
Sargent & Co., E. H., Chicago, Ill.
Schaar & Co., Chicago, Ill.
Scientific Materials Co., Pittsburgh, Pa.
Thomas Co., Arthur H., Philadelphia, Pa.
Toch Bros., N. Y. C.
Will Corporation, The, Rochester, N. Y.
- Chlorine Generators—Acid Proof**
Knight M. A., East Akron, O.
- Chlorine—Liquid**
Arnold, Hoffman & Co., Inc., N. Y. C.
Electro Bleaching Gas Co., N. Y. C.
- Chromel Metal**
Hoskins Mfg Co., Detroit, Mich.
- Chromium (Metal & Salts)**
Merck & Co., N. Y. C.
- Circuit Breakers and Switches**
General Electric Co., Schenectady, N. Y.
- Clarifiers**
Sharples Specialty Co., The, West Chester, Pa.
- Classifiers**
Dorr Co., The, Denver, Colo., & N. Y. C.
- Classifiers or Washers**
Dorr Co., The, Denver, Colo., & N. Y. C.



One Way to Tell a Good Battery Jar

TAKE your knife and cut into the edge of the jar—as indicated in the illustration. Cut into it about a half inch. You have made a protruding sliver, slightly curled. Flick the end of the sliver lightly with your finger—as you would the string of a banjo or mandolin. Do this several times.

If it bends, will move back and forth under the play of your finger—it's a good jar, a jar made of tough, high quality rubber.

If it snaps off the moment you touch it, or keeps breaking off while cutting—the rubber is too brittle, and may crack in service.

The knife test is the surest quick way to convince yourself of the merit of Goodrich Jars.

THE B. F. GOODRICH RUBBER CO.

Akron, Ohio

Goodrich Battery Jars



CLASSIFIED LIST OF CHEMICAL EQUIPMENT—(Continued)

Coal-Tar Products

Barrett Co., The, N. Y. C.
 Merck & Co., N. Y. C.
 National Aniline & Chemical Co., N. Y. C.
 Newport Chemical Works, Inc., N. Y. C.

Coal-Tar Products (Plants for)

Devine Co., J. P., Buffalo, N. Y.

Coal Testing Apparatus

Central Scientific Co., Chicago, Ill.
 Denver Fire Clay Co., The, Denver, Colo.
 Eimer & Amend, N. Y. C.
 Engelhard, Charles, N. Y. C.
 Heil Chem. Co., Henry, St. Louis, Mo.
 Scientific Materials Co., Pittsburgh, Pa.
 Thermo-Electric Instrument Co., Newark, N. J.
 Will Corporation, The, Rochester, N. Y.

Coating Sprays

Eureka Machine Co., Cleveland, O.

Cobalt—Metal & Salts

Merck & Co., N. Y. C.

Cocks—Plug (Acid Proof)

Cleveland Brass Mfg. Co., The, Cleveland, O.
 Duriron Castings Co., Dayton, O.
 Knight, M. A., East Akron, O.
 U S Stoneware Co., The, Akron, O.

Cocks—Laboratory (Brass)

Mueller Mfg. Co., H., Decatur, Ill.

Coils or Worms (Acid Proof Stoneware)

Knight, M. A., East Akron, Ohio.

Colorimeters

Bausch & Lomb Optical Co., Rochester, N. Y.
 Braun Corporation, Los Angeles, Calif.
 Braun-Knecht-Heimann Co., San Francisco.
 Central Scientific Co., Chicago, Ill.
 Daigger & Co., A., Chicago, Ill.
 Denver Fire Clay Co., The, Denver, Colo.
 Eimer & Amend, N. Y. C.
 Griebel Instrument Co., Carbondale, Pa.
 Heil Chemical Co., Henry, St. Louis, Mo.
 Palo Company, N. Y. C.
 Sargent & Co., E. H., Chicago, Ill.
 Scientific Materials Co., Pittsburgh, Pa.
 Thomas Co. Arthur H., Philadelphia, Pa.
 Will Corporation, The, Rochester, N. Y.

Combustion Tubes and Boats

Braun-Knecht-Heimann Co., San Francisco.
 Central Scientific Co., Chicago, Ill.
 Daigger & Co., A., Chicago, Ill.
 Denver Fire Clay Co., The, Denver, Colo.
 Eimer & Amend, N. Y. C.
 Engelhard, Charles, N. Y. C. ("Impervite")
 General Ceramics Co., N. Y. C.
 Guernseyware Co., The, Cambridge, O.
 Heil Chemical Co., Henry, St. Louis, Mo.
 Norton Co., Worcester, Mass.
 Palo Company, N. Y. C.
 Sargent & Co., E. H., Chicago, Ill.
 Scientific Materials Co., Pittsburgh, Pa.
 Thermal Syndicate, Ltd., The, N. Y. C.
 Will Corporation, The, Rochester, N. Y.

Combustion Tubes and Boats (Platinum)

Baker & Co., Inc., Newark, N. J.
 Heil Chem. Co., Henry, St. Louis, Mo.
 Will Corporation, The, Rochester, N. Y.

Compressors, Rotary

Crowell Mfg. Co., Brooklyn, N. Y.
 Devine Co., J. P., Buffalo, N. Y.
 Heil Chem. Co., Henry, St. Louis, Mo.

Concentrators—Sulphuric Acid

Thermal Syndicate, Ltd., The, N. Y. C.

Condensers (Barometric and Surface)

Buffalo Fdy. & Mch. Co., Buffalo, N. Y.
 Devine Co., J. P., Buffalo, N. Y.
 Duriron Castings Co., Dayton, O. (Acid Proof)
 Jacoby, Henry E., N. Y. C.
 Lummus Co., The, Walter E., Boston, Mass.
 Schutte & Koerting Co., Phila., Pa.
 Stevens Brothers, N. Y. C.

Condensers—Nitric Acid

Thermal Syndicate, Ltd., The, N. Y. C.

Conductivity Measuring Apparatus

Central Scientific Co., Chicago, Ill.
 Leeds & Northrup Co., The, Philadelphia, Pa.

Consulting Chemists & Chemical Engineers

Abbott, W. G., Jr., Wilton, N. H.
 Andrews, A. B., Lewiston, Me.
 Brickenstein, John H., Washington, D. C.
 Buffalo Fdy. & Mch. Co., Buffalo, N. Y.
 Carlitz & Co., J. S., Philadelphia, Pa.
 Columbus Laboratories, Chicago, Ill.
 Ellis-Foster Co., Montclair, N. J.
 Fort Worth Laboratories, The, Ft. Worth, Tex.
 Foster, A. B., Washington, D. C.
 Gorzo, Julius, Pittsburgh, Pa.
 Maas, Arthur R., Los Angeles, Cal.
 McNamara M. Everett, Mass.
 Mantius Engineering Co., N. Y. C.
 Marvin Davis Laboratories, Inc., N. Y. C.
 Mount, W. D., Roanoke, Va.
 Parker, C. L., Washington, D. C.
 Pease Laboratories, N. Y. C.

Rutherford Laboratories, Rutherford, N. J.
 Sadtler & Son Samuel P., Philadelphia, Pa.
 Schwarz Laboratories, N. Y. C.
 Sieck & Drucker, Chicago, Ill.
 Singer-Perlstein Co., Milwaukee, Wis.
 Stillwell Laboratories, N. Y. C.
 Sutton, Frank, N. Y. C.
 Swenson Evaporator Co., Chicago, Ill.
 Townsend, Clinton P., Washington, D. C.
 Westport Mill, The (Dorr Co.), Westport, Conn.
 Wheeler & Woodruff, N. Y. C.
 Wiley & Co., Inc., Baltimore, Md.

Contracting Engineers

Swenson Evaporator Co., Chicago, Ill.

Controlling Instruments

Brown Instrument Co., The, Phila., Pa.
 Engelhard, Charles, N. Y. C.
 Taylor Instrument Cos., Rochester, N. Y.
 Will Corporation, The, Rochester, N. Y.

Converters—Booster

Westinghouse Elec. & Mfg. Co., E. Pittsburgh, Pa.

Converters—Rotary

General Electric Co., Schenectady, N. Y.

Copper—Salts of

Merck & Co., N. Y. C.

Cores

Norton Co., Worcester, Mass.

Counters—Revolution

Brown Instrument Co., Philadelphia, Pa.

Critical Point Determination Apparatus

Brown Instrument Co., Philadelphia, Pa.
 Denver Fire Clay Co., The, Denver, Colo.
 Eimer & Amend, N. Y. C.
 Engelhard, Charles, N. Y. C.
 Heil Chem. Co., Henry, St. Louis, Mo.
 Hoskins Mfg. Co., Detroit, Mich.
 Palo Company, N. Y. C.
 Scientific Materials Co., Pittsburgh, Pa.
 Thwing Instrument Co., Philadelphia, Pa.
 Will Corporation, The, Rochester, N. Y.

Crucibles

Braun Corporation, Los Angeles, Cal.
 Braun-Knecht-Heimann Co., San Francisco.
 Central Scientific Co., Chicago, Ill.
 Daigger & Co., A., Chicago, Ill.
 Denver Fire Clay Co., The, Denver, Colo.
 Duriron Castings Co., Dayton, O. (Acid proof).
 Eimer & Amend, N. Y. C.
 Engelhard, Charles, N. Y. C. ("Impervite")
 Guernseyware Co., The, Cambridge, O.
 Heil Chemical Co., Henry, St. Louis, Mo.
 Herold China & Pottery Co., Golden, Col.
 Norton Co., Worcester, Mass. (Alundum).
 Palo Company, N. Y. C.
 Sargent & Co., E. H., Chicago, Ill.
 Scientific Materials Co., Pittsburgh, Pa.
 Thermal Syndicate, Ltd., The, N. Y. C. (Vitroasil).
 Will Corporation, The, Rochester, N. Y.

Crucibles, Platinum

American Platinum Works, Newark, N. J.
 Baker & Co., Inc., Newark, N. J.
 Bishop & Co., J. Malvern, Pa.
 Central Scientific Co., Chicago, Ill.
 Denver Fire Clay Co., The, Denver, Colo.
 Eimer & Amend, N. Y. C.
 Heil Chem. Co., Henry, St. Louis, Mo.
 Scientific Materials Co., Pittsburgh, Pa.
 Will Corporation, The, Rochester, N. Y.

Crushers & Grinders (Ind. & Lab'y)

Abbé Engineering Co., N. Y. C.
 Braun Corporation, Los Angeles, Calif.
 Braun-Knecht-Heimann Co., San Francisco.
 Central Scientific Co., Chicago, Ill.
 Daigger & Co., A., Chicago, Ill.
 Denver Fire Clay Co., The, Denver, Colo.
 Eimer & Amend, N. Y. C.
 Heil Chemical Co., Henry, St. Louis, Mo.
 Palo Company, N. Y. C.
 Raymond Bros Impact Pulv. Co., Chicago, Ill.
 Sargent & Co., E. H., Chicago, Ill.
 Scientific Materials Co., Pittsburgh, Pa.
 Thomas Co., Arthur H., Philadelphia, Pa.
 Will Corporation, The, Rochester, N. Y.
 Williams Patent Crusher & Pulv. Co., Chicago.

Crystallizers—Ammonia Nitrate

Buffalo Fdy. & Mch. Co., Buffalo, N. Y.
 Devine Co., J. P., Buffalo, N. Y.

Crystallizing Pans (Acid Proof)

Knight, M. A., East Akron, Ohio.

Cupels

Braun Corporation, Los Angeles, Cal.
 Braun-Knecht-Heimann Co., San Francisco.
 Central Scientific Co., Chicago, Ill.
 Daigger & Co., A., Chicago, Ill.
 Denver Fire Clay Co., The, Denver, Colo.
 Eimer & Amend, N. Y. C.
 Heil Chemical Company, Henry, St. Louis, Mo.
 Hoskins Mfg. Co., Detroit, Michigan.
 Norton Company, Worcester, Mass.

Cylinders (Glass)

Braun-Knecht-Heimann Co., San Francisco.
 Central Scientific Co., Chicago, Ill.
 Daigger & Co., A., Chicago, Ill.
 Denver Fire Clay Co., The, Denver, Colo.
 Eimer & Amend, N. Y. C.
 Griebel Instrument Co., Carbondale, Pa.
 Heil Chem. Co., Henry, St. Louis, Mo.
 Palo Company, N. Y. C.
 Scientific Materials Co., Pittsburgh, Pa.
 Will Corporation, The, Rochester, N. Y.

Decanting Pots—Acid Proof

Knight, M. A., East Akron O.
 Will Corporation, The, Rochester, N. Y.

Denitrating Apparatus

Buffalo Fdy. & Mch. Co., Buffalo, N. Y.

Diaphragms—Acid Proof

General Filtration Co., Inc., Rochester, N. Y.
 Herold China & Pottery Co., Golden, Colo.

Dictionary—Chemical

Chemical Catalog Co., The, N. Y. C.

Diffusion Apparatus

Devine Co., J. P., Buffalo, N. Y.
 General Filtration Co., Inc., Rochester, N. Y.
 Lummus Co., The, Walter E., Boston, Mass.
 Swenson Evaporator Co., Chicago, Ill.

Dipping Baskets & Jars—(Acid Proof)

Knight, M. A., East Akron, Ohio.

Dishes (Crystallizing)

Will Corporation, The, Rochester, N. Y.

Dishes (Glass)

Will Corporation, The, Rochester, N. Y.

Dishes ("Imperite")

Engelhard, Charles, N. Y. C.

Dishes (Petri)

Will Corporation, The, Rochester, N. Y.

Dishes (Platinum)

Bishop & Co., J., Malvern, Pa.
 Denver Fire Clay Co., The, Denver, Colo.
 Eimer & Amend, N. Y. C.
 Heil Chem. Co., Henry, St. Louis, Mo.
 Will Corporation, The, Rochester, N. Y.

Dissolving Tanks—Automatic

Lummus Co., The, Walter E., Boston, Mass.
 Werner & Pfleiderer Co., Saginaw, Mich.

Distilling Machinery and Apparatus

Braun Corporation, Los Angeles, Calif.
 Braun-Knecht-Heimann Co., San Francisco.
 Buffalo Fdy. & Mch. Co., Buffalo, N. Y.
 Central Scientific Co., Chicago, Ill.
 Cleveland Brass Mfg. Co., The, Cleveland, O.
 Daigger & Co., A., Chicago, Ill.
 Denver Fire Clay Co., The, Denver, Colo.
 Detroit Heating & Lighting Co., Detroit, Mich.
 Devine Company, J. P., Buffalo, N. Y.
 DuPont Chemical Co., Inc., Wilmington, Del.
 Duriron Castings Co., Dayton, O.
 Egot Co., The, Paris, France.
 Eimer & Amend, N. Y. C.
 Electric Heating Apparatus Co., N. Y. C.
 Elyria Enameled Prod. Co., Elyria, O.
 Garrigue Co., Wm., Chicago, Ill. (Fatty Acids)
 General Ceramics Co., N. Y. C.
 Griebel Instrument Co., Carbondale, Pa.
 Heil Chem. Co., Henry, St. Louis, Mo.
 Jacoby, Henry E., N. Y. C.
 Lummus Co., The, Walter E., Boston, Mass.
 Palo Company, N. Y. C.
 Pfaunder Co., Rochester, N. Y.
 Sargent & Co., E. H., Chicago, Ill.
 Scientific Materials Co., Pittsburgh, Pa.
 Sowers Mfg. Co., Buffalo, N. Y.
 Stevens Bros., N. Y. C.
 Stokes Machine Company, F. J., Phila., Pa.
 Swenson Evaporator Co., Chicago, Ill.
 Thomas Co., Arthur H., Philadelphia, Pa.
 Wheeler Condenser & Eng. Co., Carteret, N. J.
 Will Corporation, The, Rochester, N. Y.
 Zarembo Company, Buffalo, N. Y.

Draft Gauges

Braun-Knecht-Heimann Co., San Francisco.
 Brown Instrument Co., Philadelphia, Pa.
 Central Scientific Co., Chicago, Ill.
 Denver Fire Clay Co., The, Denver, Colo.
 Heil Chem. Co., Henry, St. Louis, Mo.
 Palo Company, N. Y. C.
 Scientific Materials Co., Pittsburgh, Pa.

Drum Dryers—Vacuum and Atmospheric

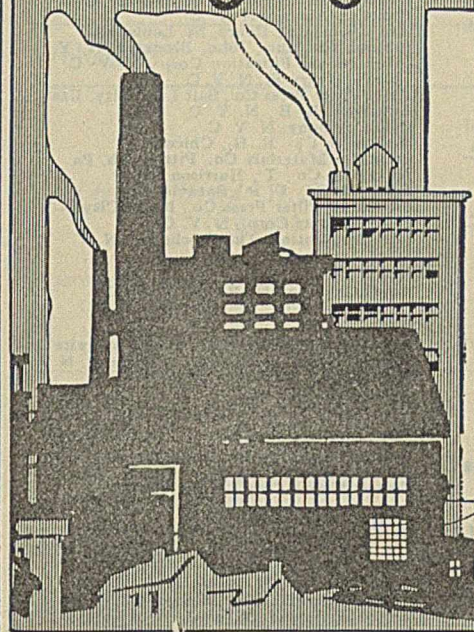
Buffalo Fdy. & Mch. Co., Buffalo, N. Y.
 Devine Co., J. P., Buffalo, N. Y.

Dry Blast Plants

Carrier Engineering Corp., N. Y. C.

Eternium Paint

A high grade Black coal-tar Paint



Keeps New Metal New

Even the newest of metal is liable to small defects and these may soon grow into large patches of corrosion.

Eternium is a high-grade black, coal-tar product that flows easily into these places. It covers freely, with an impenetrable protective coating that is proof against acid, vapor, alkali and steam.

Use Eternium freely. It is put up in handy sized cans, drums and barrels. It costs little and goes far.

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Dryers—Centrifugals

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Heil Chem Co., Henry, St. Louis, Mo.
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Braun-Knecht-Heimann Co., San Francisco, Cal.
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Scientific Materials Co., Pittsburgh, Pa.
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Central Scientific Co., Chicago, Ill.
Daigger & Co., A., Chicago, Ill.
Denver Fire Clay Co., The, Denver, Colo.
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Engelhard, Chas., N. Y. C.
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Electric Heating Apparatus Co., N. Y. C.
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Sowers Mfg. Co., Buffalo, N. Y.
Sperry & Co., D. R., Batavia, Ill.
Stokes Mach. Co., F. J., Philadelphia, Pa.
Swenson Evaporator Co., Chicago, Ill.
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Wheeler Condenser & Eng. Co., Carteret, N. J.
Zaremba Company, Buffalo, N. Y.

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Central Scientific Co., Chicago, Ill.
Daigger & Co., A., Chicago, Ill.
Denver Fire Clay Co., The, Denver, Colo.
Devine Co., J. P., Buffalo, N. Y.
Duriron Castings Co., Dayton, O. (Acid Proof).
Eimer & Amend, N. Y. C.
Elyria Enameled Prod. Co., Elyria, O. & N. Y. C.
Engelhard, Charles, N. Y. C. ("Impervite")
General Ceramics Co., N. Y. C.
Guernseyware Co., The, Cambridge, O.
Heil Chemical Co., Henry, St. Louis, Mo.
Herald China & Pottery Co., Golden, Colo.
Knight, Maurice A., East Akron, Ohio
Palo Company, N. Y. C.
Pfauder Co., Rochester, N. Y.
Sargent & Co., E. H., Chicago, Ill.
Scientific Materials Co., Pittsburgh, Pa.
Sowers Manufacturing Co., Buffalo, N. Y.
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Will Corporation, The, Rochester, N. Y.

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Chemical Equipment Co., Chicago, Ill.
Devine Co., J. P., Buffalo, N. Y.
Wheeler Condenser & Eng. Co., Carteret, N. J.

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General Electric Co., Schenectady, N. Y.

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Duriron Castings Co., Dayton, O.

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Central Scientific Co., Chicago, Ill.
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Tolhurst Machine Works, Troy, N. Y.
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Denver Fire Clay Co., The, Denver, Colo.
Eimer & Amend, N. Y. C.
Engelhard, Charles, N. Y. C. ("Impervite")
Heil Chem. Co., Henry, St. Louis, Mo.
Norton Co., Worcester, Mass.
Will Corporation, The, Rochester, N. Y.

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Scientific Materials Co., Pittsburgh, Pa.
Thomas Co., Arthur H., Philadelphia, Pa.
Will Corporation, The, Rochester, N. Y.

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General Electric Co., Schenectady, N. Y.

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Filter Cloth

United Filters Corp., N. Y. C.

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Multi-Metal Co., Inc., N. Y. C. (Monel Metal)
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General Filtration Co., Inc., Rochester, N. Y.
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Braun-Knecht-Heimann Co., San Francisco, Cal.
Central Scientific Co., Chicago, Ill.
Daigger & Co., A., Chicago, Ill.
Denver Fire Clay Co., The, Denver, Colo.
Eimer & Amend, N. Y. C.
Gazzolo Drug & Chem. Co., Chicago, Ill.
Heil Chemical Co., Henry, St. Louis, Mo.

Palo Company, N. Y. C.
Sargent & Co., E. H., Chicago, Ill.
Scientific Materials Co., Pittsburgh, Pa.
Thomas Co., Arthur H., Philadelphia, Pa.
Will Corporation, The, Rochester, N. Y.

Filter Paper Clippings

Angel Co., Inc., H. Reeve, N. Y. C.

Filter Presses (Laboratory & Industrial)

Abbé Engineering Co., N. Y. C.
Braun Corporation, Los Angeles, Calif.
Braun-Knecht-Heimann Co., San Francisco, Cal.
Central Scientific Co., Chicago, Ill.
Denver Fire Clay Co., The, Denver, Colo.
Eimer & Amend, N. Y. C.
Heil Chem. Co., Henry, St. Louis, Mo.
Johnson Co., Inc., John, Brooklyn, N. Y.
International Filtration Corp., N. Y. C.
Jacoby, Henry E., N. Y. C.
Kelly Filter Press Co., Salt Lake City, Utah
Lungwitz, E. E., N. Y. C.
Palo Company, N. Y. C.
Sargent & Co., E. H., Chicago, Ill.
Scientific Materials Co., Pittsburgh, Pa.
Shriver & Co., T., Harrison, N. J.
Sperry & Co., D. R., Batavia, Ill.
Sweetland Filter Press Co., N. Y. City.
United Filters Corp., N. Y. C.
Will Corporation, The, Rochester, N. Y.

Filter Pulp

Angel Co., Inc., H. Reeve, N. Y. C.

Filters

Engelhard, Charles, N. Y. C. ("Impervite")
General Filtration Co., Inc., Rochester, N. Y.
Norton Co., Worcester, Mass.
United Filters Corp., N. Y. C.
Will Corporation, The, Rochester, N. Y.

Filters—(Acid Proof)

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Knight, Maurice A., East Akron, Ohio
United Filters Corp., N. Y. C.

Filters (Bone Black)

Colwell, Lewis, Chicago, Ill.

Filters, Folded

Angel Co., Inc., H. Reeve, N. Y. C.
Central Scientific Co., Chicago, Ill.
Eimer & Amend, N. Y. C.
Heil Chem. Co., Henry, St. Louis, Mo.
Sargent & Co., E. H., Chicago, Ill.
Scientific Materials Co., Pittsburgh, Pa.
Thomas Co., Arthur H., Philadelphia, Pa.
Will Corporation, The, Rochester, N. Y.

Filters—Vacuum

American Continuous Suction Filter
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Devine Co., J. P., Buffalo, N. Y.
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United Filters Corp., N. Y. C.

Filters—Water

Braun Corporation, Los Angeles, Calif.
Braun-Knecht-Heimann Co., San Francisco, Cal.
Booth Apparatus Co., Syracuse, N. Y.
Central Scientific Co., Chicago, Ill.
Daigger & Co., A., Chicago, Ill.
Denver Fire Clay Co., The, Denver, Colo.
Eimer & Amend, N. Y. C.
General Ceramics Co., N. Y. C.
Griebel Instrument Co., Carbondale, Pa.
Heil Chemical Co., Henry, St. Louis, Mo.
Palo Company, N. Y. C.
Sargent & Co., E. H., Chicago, Ill.
Schutte & Koerting Co., Phila., Pa.
Thomas Co., Arthur H., Philadelphia, Pa.
Will Corporation, The, Rochester, N. Y.

Filtros

General Filtration Co., Inc., Rochester, N. Y.

Filtros—Vacuum

Devine Company, J. P., Buffalo, N. Y.

Fire Brick and Clay

Denver Fire Clay Co., The, Denver, Colo.

Flasks

Braun-Knecht-Heimann Co., San Francisco, Cal.
Central Scientific Co., Chicago, Ill.
Daigger & Co., A., Chicago, Ill.
Denver Fire Clay Co., The, Denver, Colo.
Eimer & Amend, N. Y. C.
Heil Chem. Co., Henry, St. Louis, Mo.
Palo Company, N. Y. C.
Scientific Materials Co., Pittsburgh, Pa.
Will Corporation, The, Rochester, N. Y.

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Foamite Firefoam Co., N. Y. C.

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Detroit Heating & Lighting Co., Detroit, Mich.
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Heil Chem. Co., Henry, St. Louis, Mo.

Funnels

Denver Fire Clay Co., The, Denver, Colo.
Heil Chem. Co., Henry, St. Louis, Mo.
Knight, M. A., East Akron, Ohio
Will Corporation, The, Rochester, N. Y.