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OFFICERS FOR 1918

The following officers have been elected by the American Chemical Society for the year 1918:

President: William H. Nichols, General Chemical Company, New York City.

Directors: H. E. Barnard, State Laboratory of Hygiene, Indianapolis, Ind.; and G. D. Rosengarten, Powers-Weightman-Rosengarten Co., Philadelphia, Pa.

Councilors-at-Large: H. E. Howe, A. D. Little, Inc., Cambridge, Mass.; G. A. Hulett, Princeton University, Princeton, N. J.; W. A. Noyes, University of Illinois, Urbana, Ill.; and Allen Rogers, Pratt Institute, Brooklyn, N. Y.

DR. NICHOLS—LEADER IN CHEMICAL INDUSTRY

By C. F. Chandler

Dr. William H. Nichols was one of the small group of New York chemists who, in 1876, originated this by far the largest chemical society in the world. It now has 51 local sections and approximately 11,000 members, and publishes three distinct chemical journals.

Dr. Nichols was born January 9, 1852, in Brooklyn, N. Y. He graduated from the Brooklyn Polytechnic Institute in 1868 and then entered New York University, where he had the good fortune to study chemistry under Dr. John W. Draper, the first President of the American Chemical Society. He received his B.S. in 1870. In 1873 he received his M.S. from the same institution; in 1904, LL.D. from Lafayette and Sc.D. from Columbia. In 1912 he was decorated by the King of Italy with the Order of Commendatore of the Crown of Italy. He was president of the English Society of Chemical Industry 1904–1905 and of the Eighth International Congress of Applied Chemistry held in Washington and New York in 1912.

William H. Nichols, President American Chemical Society

In 1870, when only eighteen years old, he founded his own chemical business under the title G. H. Nichols and Company, using his father's name because he was not yet of age. Later the business was incorporated as the Nichols Chemical Company.

The instincts of the pioneer in Dr. Nichols led to the origin in his plant of many ideas and appliances used in chemical industry to-day, for example, the well-known practice of storing and transporting sulfuric acid in steel.

The manufacture of sulfuric acid from pyrites was first carried out profitably in the Nichols Chemical Works at Laurel Hill. The pyrites used contained some copper and the search for the proper metallurgical treatment of it led to the invention of methods still employed for smelting such ores and also to the devising of a method for analyzing copper by electrolysis, which was the foundation of the industry of the electrolytic refining of copper. These processes for smelting and refining copper ores were so successful that the business grew rapidly to such dimensions that in 1898 it was transferred to a special company, the Nichols Copper Company, of which Dr. Nichols is president. The works, located on Newtown Creek, Brooklyn, constitute one of the most extensive copper plants in the world. In 1899, the chemical branch of the business went into the General Chemical Company.

The superior executive ability of Dr. Nichols shows in the success he has had in such enterprises as the rehabilitating of the Granby Consolidated Mining, Smelting and Power Company, Ltd., converting it into one of the best-managed copper companies in the world; the recent organizing of the National Aniline and Chemical Company, Inc., looking to the permanent relief of American textile manufacturers and others; the bringing of a new lease of life to his Alma Mater, the Polytechnic Institute of Brooklyn, which seemed to be on the decline but is today a school of engineering of high mark, due largely to the good work of Dr. Nichols.

With all these business activities Dr. Nichols has acted as chairman of the Committee on Chemicals for the government and just recently has been appointed by Secretary Lane chairman of the Committee of Chemists advisory to the Bureau of Mines.

New York City

EDITORIALS

ON WITH THE INVESTIGATION

The massing of German reinforcements from the East on the western front led Winston Churchill, British Minister of Munitions, in an address at the American Luncheon Club recently, to exclaim:

"America! Come and aid us with all your might and speed, for this is a matter for action on the largest scale ever planned. * * We are sure to win the whole of President Wilson's program if we will utilize all our resources fully." Such an appeal, coming from a member of a race strong in self-reliance and bull-dog tenacity, must sink deep into the heart of every American.

That we have lacked speed in many of our preparations has been shown clearly by the investigations of the Senate Committee on Military Affairs. The reports show that the Committee felt fully capable of probing deep into the supply of cannon, machine guns and rifles, but when the subject of ammunition for such arms, and particularly of the basic chemicals needed in its manufacture, was approached, the investigation seemed at once to veer from so technical a subject. This is not difficult to understand, and yet we feel that the Committee can perform further public service if it will extend its investigations to cover thoroughly this field, particularly as to acetic acid for aeroplane dope and toluol for high explosives.

Enormous quantities of acetic acid are needed immediately, and until this is supplied the aviation program will be held up. The present total output of this product is already engaged for the Navy and our Allies. New factories must be built for further output. With the liberty motor completed, with all arrangements made for the supply of spruce wood in abundance, the startling fact remains that, unless action has been taken within the twenty-four hours previous to this writing, not even the method of manufacture of the necessary acetic acid has been decided upon, much less has the erection of any plant begun. In view of the tremendous difficulties of plant construction in these times, it is appalling to think of the delays ahead in this work which even in peace times and under normal conditions would prove an extremely formidable undertaking. It looks as if someone has blundered seriously, especially when we reflect upon the unprecedented speed with which Congress at the outset appropriated $650,000,000 for the aviation service.

The fundamental importance of toluol, the great need for it in the production of high explosives, and the method of its manufacture by stripping gas are well understood at the present time. Appropriations have been available since the adjournment of the previous session of Congress. Nearly six months have elapsed since the conference was held in Washington between representatives of the War Department, the gas producers, and the public service commissions. Yet to-day there are many gas plants with which no final arrangements have been made by the War Department for the erection of scrubbers to strip the gas. That this condition is not due to lack of coöperation by the companies is indicated in a letter to us from Brig.-Gen. William H. Crozier. Under date of October 17, 1917, he states: "We have received a ready response to coöperate with us from every company that we have written to so far." We have been informed by the Ordnance Department that for the present at least negotiations for the installation of apparatus for the recovery of toluol will not be conducted with gas plants whose capacity would be less than 40,000 gallons per year. It would be interesting to learn through a public investigation how far these negotiations have resulted in actual contracts and inauguration of construction work, and what dates such contracts bear. Unfortunately we are not in position to give much detailed information on this point, but we know of one contract which has been shifting forward and backward for months, and is not yet signed. Whether the delay in settling the petty features of the contract is due to the attitude of the manufacturer or to the methods of the War Department it is not for us to judge, but we do know that the construction firm in question would not be at all adverse to an investigation of the reasons for this delay. When all is said, it is not a question of this or that manufacturer; if any such firm delays the prompt execution of government plans, turn aside from him and get a contractor who will start the work promptly. It is toluol that is needed, and not the saving of a few cents per gallon in its production. It would seem that officials of the War Department are still following the leisurely ways of contract making characteristic of peace times, while material which may be of the utmost importance at a critical moment is now being burned, and can never be recovered.

Of course the decision to use mixtures of toluol and ammonium nitrate for high explosives relieves the situation somewhat, nevertheless the ammonia plants are not yet completed. We have upon us the responsibility of supplying not only the needs of our own Army, but of aiding in every way possible those of our Allies. This applies particularly to Italy, fighting so resolutely to-day, its very existence immediately threatened.

If it be held that toluol recovery must not exceed nitration capacity because of lack of storage tanks, will not the War Department contemplate the many cases of seemingly autocratic procedure adopted by government officials during the last few weeks, acts which have been accepted cheerfully by the country because they were war measures? In the light of such procedure the storage question can readily be solved by commandeering some of the many storage tanks scattered throughout the country and now filled with petroleum products. Can anyone doubt the relative value to ourselves and our Allies of a half dozen such storage tanks filled on the one hand with kerosene, or on the other hand with toluol? Then too, is the

War Department certain that the nitration capacity of the country is not in excess of toluol recovery, or that it will not be so by the time the recovery plants are installed? Already the coal shortage has seriously diminished the production of toluol from the by-product coke ovens, until now the chief source of supply.

This country can possess no more valuable reserve than ample quantities of stored toluol. Another Halifax disaster, the bombing of a few munitions stations, the sinking of a few supply ships stored with this material might at any moment make a serious shortage, a shortage which would be criminal with all the lives at stake, if the possibility of such can be avoided. On with the investigation! Senator Chamberlain can perform a distinct service if through his Committee he can speed up matters in the supply of such materials. The country will hereafter crucify with its scorn any manufacturer who now seeks to profiteer at its expense in this its hour of trial. So, too, will the country hold accountable those of its public servants who dilly-dally over minor details in fundamental matters.

SOMEBODY, PLEASE CUT THE TAPE

If the National Retail Merchants' Association should arbitrarily rule that all would-be purchasers of hats must be supplied with the hat most convenient to the reach of the clerk in attendance, without regard to the shape, color or size of the hat, what manner of Easter parade would result from males and females thus adorned! Or suppose the Amalgamated Employment Bureau should decree that seekers of help could secure only "the next on the list," regardless of qualifications. What would eventually result to the regular processes of commercial life if, seeking a stenographer, one should draw a cook! These suggestions are not intended to reflect on the good qualities of the number six hat on the seven and a half head or on the abilities of the domestic in her proper sphere. Wait a minute—these cogitations are not trivial.

Burns was all right when he wrote "A Man's a Man for a' That," but there are all kinds of men and there are all kinds of chemists: analytical and research chemists, organics and inorganics, chemists fresh from the universities and chemists who have been able to add to their university training valuable plant experience. Some have specialized in explosives, others in metal alloys. Some are accurate in analytical work, others excel in planning research. If, however, a government department, bureau or division wishes to increase its chemical force by securing the transfer of a specially qualified chemist from a cantonment to a government laboratory, such coördinate branch of the government service must send out to the camps and simply ask for a chemist. Chemists must not be sought by name. To request a specially qualified man, designating the man you want, is no longer permitted. Such is the ruling of the General Staff of the Army, to which ruling the War Department has strictly adhered for some weeks past. Shades of common-sense America, what an absurd situation! Is this the final outworking of the spirit of the selective draft which President Wilson assured us was to fashion this nation into the most efficient fighting machine, which law, the record of these columns will testify, we have striven steadfastly to uphold? Is Secretary Baker aware of this ruling, a ruling which was not brought into being three thousand miles away, but right in the city of Washington in his own Department?

The results of such procedure are not only disaster to government chemical work but serious demoralization of the staffs of the chemical industries, which are supplying the very sinews of war. This can be illustrated best by two specific cases. A colonel in the Ordnance Department wrote recently to a prominent chemical manufacturing company stating that the Department was desirous of securing the services of a number of chemists and factory foremen for use as inspectors at munitions plants. He specified that they should have had such experience as would enable them to carry out intelligent inspection of explosives manufactured for the government in this emergency. The manufacturer was asked to go over his organization and advise as to any men who might be available and whom he could recommend. That is all right from one point of view. Of course the government must have competent inspectors, and chemical manufacturers are just as patriotic as other men and will gladly sacrifice their staffs if need be. That is the real question, "if need be." There are more than three hundred chemists in cantonments to-day, practically inaccessible for government chemical work because of this remarkable ruling of the General Staff. One of these is a graduate of two leading American universities, in each of which he specialized in chemistry. Furthermore he has had three and a half years of experience in research and in the manufacture of explosives, dyestuffs and pharmaceuticals, and is familiar with the installation and operation of chemical machinery. Yet his daily duties consist of scrubbing floors, shoveling coal or cinders, chopping wood, digging ditches (not trenches) and general work around the stable or kitchen.

We do not seek to arouse sympathy for this young soldier chemist. He is having a good experience and loyally doing his duty as any other young American would; his clear eye and soldierly bearing show, too, that he has made good as a soldier. He makes not the slightest complaint. But we do feel that the government is not getting from him the most efficient service he could perform; and it is a shame for a similarly qualified man to be taken at this time from the industries for government work while such a man could be made available in a few hours were it not for the weird ruling of the General Staff.

Sixteen thousand chemists at the outbreak of the war filed with the Bureau of Mines complete data concerning their training and specialization in order that their services might be promptly and intelligently availed of as need arose. Alas, these cards of voluntary information are now bound tightly together by the red tape of this rule-beyond-understanding promulgated by the General Staff.

Somebody, please cut the tape!

PLATINUM OSCILLATIONS

At the recent meeting of Section C (Chemistry) of the American Association for the Advancement of Science at Pittsburgh the platinum resolution (page 159, this issue), introduced by Mr. George F. Kunz, of Tiffany & Co., New York City, was unanimously passed. To present this matter to the proper authorities a committee was appointed consisting of Dr. W. A. Noyes, Chairman, and Dr. W. F. Hillebrand.

The admirable suggestions contained in the resolution will appeal immediately to every chemist in university or technical laboratories. Unfortunately for the ray of hope held out by this movement, Dr. Hillebrand, of the Bureau of Standards, in declining to serve upon the committee, felt compelled to take this step because of his knowledge that all of this platinum supply is needed immediately by the government, and has indeed already been turned over to the Nitrate Committee for catalyzer purposes in the oxidation of ammonia. There seems, at least for the present, no hope that the use of residues for research on the platinum group of minerals can be undertaken, as the question of government ownership of this material is undetermined.

The accounts of the energy, resourcefulness, and peregrinative ability of Mr. Draper, who safely transported from Russia 21,000 ounces of platinum and platinum ore and delivered it to the Department of Commerce, furnish very interesting reading and command unquestioned appreciation of his achievement. The necessity for such a journey, however, suggests further thought as to the accuracy of judgment of the Secretary of Commerce, who last Spring, evidently while this material was being collected, gave ample assurances to the jewelers of the country that the government had an abundance of platinum, either on hand or available from stocks known to be existing abroad, which statement was heralded very widely by the Jewelers' Committee. We tremble to think of the fix the government might have been in if Mr. Draper had stubbed his toe in going aboard ship and spilled the precious metal into the sea, or had lost his trunk in the mazes of the Union Station baggage room in Washington, or had met with the same delay in express shipments which the average citizen encounters nowadays. Seriously, we have been running very close to the danger line in government supplies of this material for munitions manufacture, while the advertising campaign for platinum jewelry has gone merrily on. Meanwhile, the university and industrial chemist cannot hope for relief from the present high prices of platinum ware from the source contemplated by the resolution of Mr. Kunz.

The communication from Dr. Jas. Lewis Howe, printed in this issue, views the platinum situation solely from the standpoint of "business as usual," an antiquated slogan whose pernicious effect upon war programs has already made itself plainly evident.

Two new methods of attacking the problem of platinum conservation have developed. Individuals and local Sections can use their influence with the local press to persuade them to refuse advertisements of platinum jewelry. The *New York Times* and the *New York World* have adopted such a policy, and it it is a pleasure to state that this has been done on the urgent appeal of Mr. Kunz, who is endeavoring to bring all of the New York newspapers into line.

The second interesting development is the recent organization of the Women's National League for the Conservation of Platinum. May the good work of this new organization prosper in every way. It is for women that this platinum jewelry is designed; it is through women that its use can be most effectually discountenanced. Perhaps through this League the real punch will be put into the platinum conservation movement.

AN APPRECIATION AND A GREETING

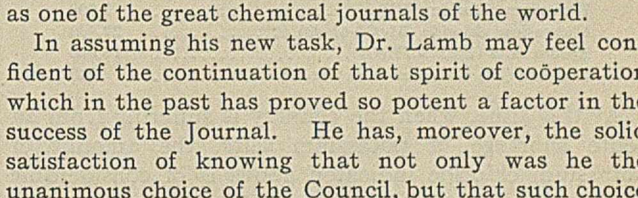
The appearance of the January number of the *Journal of the American Chemical Society* marks the retirement of Dr. W. A. Noyes from its editorship and the entrance upon his duties of the newly elected editor, Dr. A. B. Lamb.

After fifteen years of splendid service, Dr. Noyes carries with him on retirement universal grateful appreciation of the devotion he has shown to the upbuilding of that Journal. To its list of contributors he has called all of the research workers in pure chemistry in this country. As a result of his accurate and conscientious editorial work the publication stands to-day as one of the great chemical journals of the world.

In assuming his new task, Dr. Lamb may feel confident of the continuation of that spirit of coöperation which in the past has proved so potent a factor in the success of the Journal. He has, moreover, the solid satisfaction of knowing that not only was he the unanimous choice of the Council, but that such choice was based upon the unanimous report of a committee of our ablest men who canvassed the field with closest scrutiny. The fact that Dr. Lamb begins his editorship while devoting much of his time to government service constitutes an additional reason for loyal support.

CHEMISTRY INSIGNIA

For the first time in the history of the United States an Army group will wear a design typifying chemistry as a recognized branch of war service. Through the courtesy of the manufacturers we are the proud possessors of the first insignia and collar design struck from the dies.



Officer Enlisted Man

Insignia for Chemical Service

From the record of attainments of the first men selected to wear such insignia it can be predicted with certainty that they will be worthily worn.

Good luck to the Chemical Service Section of the National Army!

ORIGINAL PAPERS

THE EXTRACTION OF POTASH AND OTHER CONSTITUENTS FROM SEA WATER BITTERN[1]

By Joel H. Hildebrand

Received December 5, 1917

COMPOSITION OF SEA WATER

The main constituents of sea water, besides sodium chloride, are magnesium sulfate, magnesium chloride and potassium chloride, together with a small quantity of magnesium bromide and calcium salts. During the evaporation of the sea water to secure sodium chloride, the calcium present is almost completely deposited as calcium sulfate, so that calcium salts are practically absent from the mother liquor. By considering the various analyses of sea water we may calculate the relative amounts of the solid salts that might be obtained by evaporation. The salt works around San Francisco Bay, with which we are primarily concerned, produce something over 100,000 tons of sodium chloride per annum; the amounts of the other salts associated with this amount of sodium chloride would be as follows:

	Tons
Sodium chloride (NaCl)	100,000
Potassium chloride (KCl)	2,800
Magnesium chloride ($MgCl_2.6H_2O$)	27,300
Epsom salts ($MgSO_4.7H_2O$)	16,000
Bromine (Br) extracted from the bromides	240

At this time, when the country is suffering from an acute shortage of potassium salts, the amount of potassium chloride indicated above is of considerable importance. During the first half of 1917 the total potash production of the country, calculated on the basis of K_2O, was 14,000 tons, which amount was but 10 per cent of the normal amount used before the war. It is evident that the amount of potash that could be extracted from the bitterns of the salt works on San Francisco Bay alone would add about 10 per cent to the country's present annual production of potash. The amount of salt actually produced in this region is nearly 140,000 tons per annum, so that a liberal allowance for losses in working up the bittern should leave still 3000 tons of potassium chloride. By utilizing the bitterns from other regions on the Pacific Coast, notably San Diego, this amount would be very greatly increased.

The other materials mentioned in the above table also represent very considerable values, although they have less relation to the present national emergency.

After the removal of most of the common salt in the salt ponds, the other salts would be contained in approximately 100,000 tons of bittern, having a volume of approximately 100,000 cubic yards.

The values represented by these materials, and their importance both as a natural resource of California and in supplying the country with potash in the present acute emergency, made the study of this problem seem a proper one to undertake at this time.

SCIENTIFIC BASIS OF METHODS FOR RECOVERY OF THE CONSTITUENTS OF BITTERN

We are very fortunate to possess a vast fund of information upon the solubility relationships of the various salts obtainable from sea water through the classic work of van't Hoff and his co-workers. This work is described in great detail in "Über die Bildungsverhältnisse der ozeanischen Salzablagerungen" (Leipzig Verlagsgesellschaft, 1912). During the progress of the work two smaller volumes were published in 1905 and 1909 by van't Hoff, entitled "Zur Bildung der ozeanischen Salzablagerungen" (Braunschweig, Vieweg).

Inasmuch as very little of this work has been translated into English, and in view of the difficulty of interpreting it in its formidable complexity, it seems desirable to give a general outline of its nature.

The solubility of a single salt in its relation to changes in temperature may be represented by simple diagrams of the type familiar to all trained chemists.

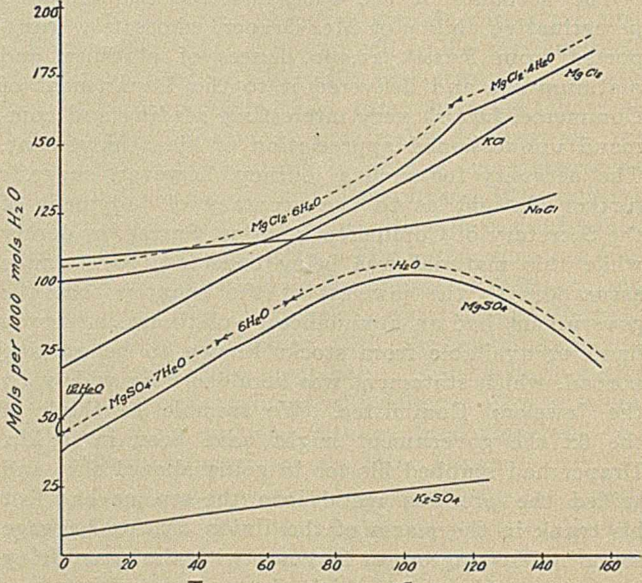


Fig. I

In Fig. I are represented the solubility curves for the main salts with which we have to deal, *viz.*, sodium chloride, potassium chloride, potassium sulfate, magnesium sulfate and magnesium chloride. In this figure solubility is expressed as the number of mols of anhydrous salt per 1000 mols of water. Of course, other units may be used, such as mols or grams of salt in a certain number of grams of water or of solution, or in a certain number of cubic centimeters in solution. If we know the solubility expressed in any of these terms it is possible to calculate it in any other terms, the density of the solution being required where the conversion is between a weight and volume basis.

The laws of dilute solutions may frequently be extended to give an approximate idea of the behavior of concentrated solutions. The solubility of a given

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[1] This work has been supported by the Council of Defense of the State of California.

salt is varied by the introduction into the solution of another salt. The effect of the second salt can be predicted qualitatively by remembering that where the salts possess a common ion the solubility of each is usually decreased by the presence of the other. If, however, there is a strong tendency to form a complex salt the solubility of one may be increased by the presence of the other. Again, where there is no common ion the solubility of one is increased by the presence of the other owing to the interaction of the two salts.

There are various ways of representing graphically the solubility relationships of salt pairs. The method adopted by van't Hoff is to represent the amount of each salt in the solution in terms of mols of anhydrous salt per 1000 mols of water, measured along two axes at right angles to each other, as illustrated in Fig. II. Each curve here represents the composition of a solution saturated with one component. The intersections of the curves represent the composition of a solution saturated with both components. A point between these curves and the origin denotes the composition of an unsaturated solution. A point outside of the curves would represent a mixture of a saturated solution with one or both solid salts, depending upon its position. On evaporation of an unsaturated solution the relative amounts of the two salts would remain the same until the solution becomes saturated, so that, for example, a solution having the composition represented by the point *a* in Fig. II would, on evaporation, change in composition as represented by the motion along the line *ab*. As soon as the curve *AB* is reached, representing in this case the composition of a solution saturated with potassium chloride, solid potassium chloride will separate and the solution must become relatively richer in sodium chloride, so that as the evaporation proceeds from *b* the solution will change in composition along the solubility curve towards *B*. Similarly, an unsaturated solution having the composition represented by *c* would, on evaporation, change in composition as represented by movement along the line *cd*. At *d* sodium chloride would begin to crystallize, whereupon the solution would become richer in potassium chloride, its composition changing along the line *dB*. It is evident that the final result in the evaporation of any solution of these two salts would be a saturated solution having the composition represented by *B*, changing into a mixture of the two solid salts.

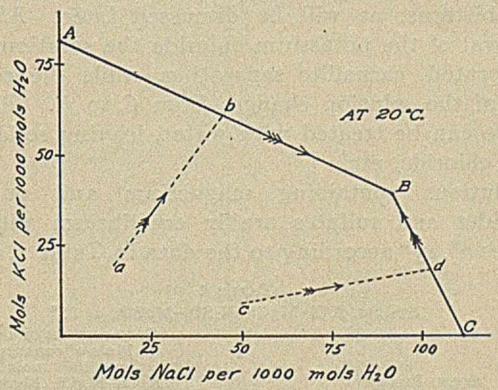

FIG. II

The effect of temperature may be indicated on a third axis at right angles to the others, giving a solid figure, as represented in perspective in Fig. III.

Where a double salt may be formed, the solubility relationships at a given temperature would be represented by a diagram such as that in Fig. IV. This diagram represents the solubility at 30° of mixtures of sodium sulfate decahydrate, and magnesium sulfate heptahydrate, which form the double salt, astrakanite, $Na_2Mg(SO_4)_2.4H_2O$. The middle portion of the curve seen in this figure represents the composition of solutions saturated with astrakanite. Solid astrakanite, which contains equivalent quantities of the two salts, has a composition lying upon a line bisecting the angle between the two axes. The composition of the solid salt is represented by a point on this line at *E*, expressing the number of mols per 1000 mols of water in the solid salt. The composition of solid sodium sulfate, $Na_2SO_4.10H_2O$, which lies along the line *OA*, is at a distance from the origin corresponding to its water content at *F*. Similarly, solid magnesium sulfate has the composition represented by the point *G*. When an unsaturated solution containing these salts is evaporated, its composition will, as in the previous case, move along a line away from the origin until one of the curves representing the composition of the saturated solution is reached, when the solution will change in composition along this line in the direction away from the line representing the composition of the solid which is separating. Thus a solution having the composition represented by *a* would, on evaporation, change in composition along the line *ab*, when, on further evaporation, sodium sulfate would separate, and finally, at *B*, both sodium sulfate and the double salt would separate, the solution remaining constant in composition until it had all disappeared. Similarly an unsaturated solution of composition represented by *c* would change in composition in the direction *cdB*, the solids separating being first pure astrakanite and then a mixture of astrakanite and sodium sulfate. The point *B* represents, therefore, the end-point of crystallization for solutions which contain more sodium sulfate than magnesium sulfate.

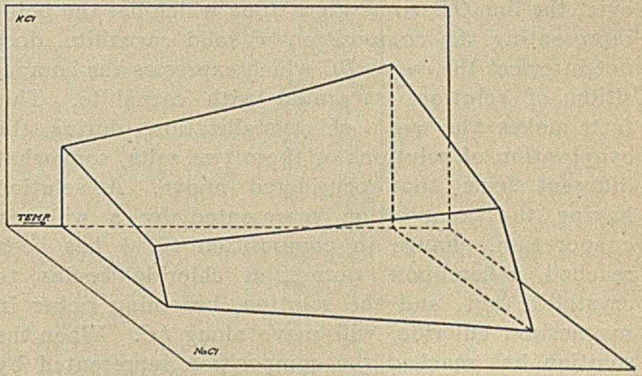

FIG. III

Fig. V represents the solubility of mixtures of magnesium chloride and potassium chloride, from which

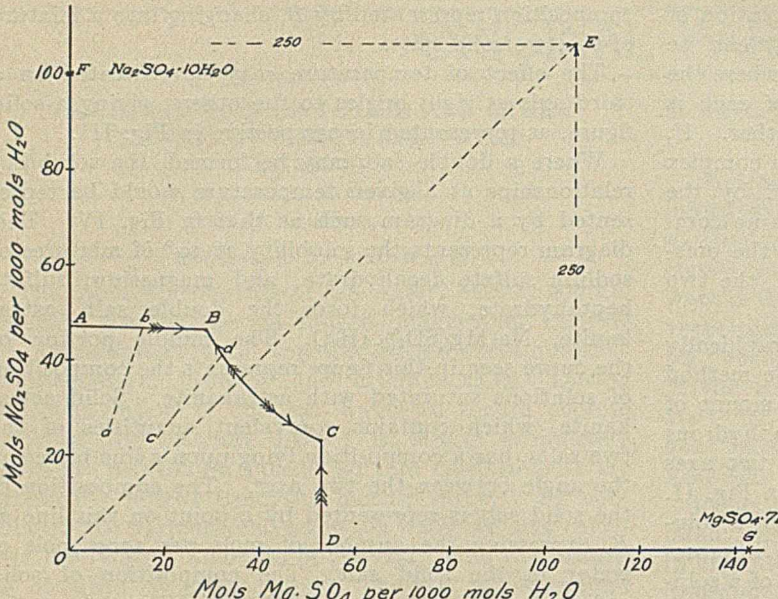

Fig. IV

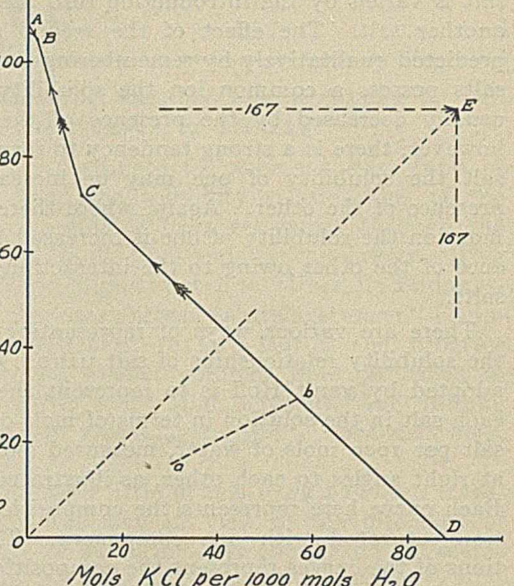

Fig. V

it is possible to crystallize the double salt carnallite, $KMgCl_3.6H_2O$. Unlike the previous instance, however, the line *OE*, somewhere upon which lies the point representing the composition of solid carnallite, does not intersect the curve *BC* which expresses the composition of solutions saturated with carnallite. This fact makes the path of crystallization, during the evaporation of solutions of these two salts, somewhat different from that considered above. A solution having the composition represented by *a* will, on evaporation, change in composition till *b* has been reached, whereupon potassium chloride begins to crystallize out, and the solution, becoming richer in magnesium chloride, will move along *bC*. When the solution has reached the composition represented by *C*, carnallite will begin to separate, but since carnallite contains more potassium chloride than does the saturated solution at *C*, it is evident that while carnallite crystallizes, the solution will tend to move along the line *CB* instead of remaining at *C*. The phase rule, however, requires that while both potassium chloride and carnallite are present, the solution must remain constant in composition at *C*. Therefore, instead of the liquid phase disappearing at this point, as was the case in the former salt pair, it is one of the solid phases, potassium chloride, which will now disappear, being changed over into carnallite. It is not until all of the potassium chloride has been so changed that the solution can move from *C* to *B*. *B* will thus represent an end-point of crystallization, while *C* will not. It is evident, therefore, that in order to prepare crystals of carnallite it is necessary to use a solution containing more than the equivalent amount of magnesium chloride, the relative amounts of the two salts being such that, on evaporation, the line *BC* will be intersected slightly above *C*. Similar considerations show us that on treating solid carnallite with water, instead of dissolving as such, it would tend to change into solid potassium chloride and a solution whose composition is that represented by *C*. It is obvious, therefore, that it is not difficult to obtain potassium chloride from carnallite, a point of importance in the treatment of salt bitterns, as will be discussed later. After the removal of the potassium chloride the solution can be evaporated, carnallite separating, while the composition of the solution changes from *C* to *B*. This carnallite can be treated with water, leaving solid potassium chloride, etc.

Solutions containing magnesium and potassium chlorides and sulfates are in equilibrium with solid phases at 25° according to the data in Table 1, and are represented by van't Hoff along four axes, as in Fig. VI, each pair of axes representing solutions containing a common ion. The boundary lines correspond to solutions saturated with the two constituents represented by the enclosing axes. Where salt pairs containing no common ion are present it is impossible to represent the composition by a point in the plane of this figure. A mixture of equivalent quantities of potassium sulfate and magnesium chloride would evidently lie at the origin *O* and would be indistinguishable from pure water by its position in the plane. In order to make this distinction it is necessary to intro-

Table 1

System, $KCl-MgCl_2-K_2SO_4-MgSO_4$, at 25°

		Composition of Solution Mols of constituents per 1000 mols H_2O			
	Solid Phases	K_2Cl_2	$MgCl_2$	$MgSO_4$	K_2SO_4
A	KCl	44	...	..	..
B	$MgCl_2.6H_2O$	..	108	..	..
C	$MgSO_4.7H_2O$	..	...	55	..
D	K_2SO_4	..	...	..	12
E	KCl and $KMgCl_3.6H_2O$	5.5	72.5	..	..
F	$MgCl_2.6H_2O$ and $KMgCl_3.6H_2O$	1	105	..	..
G	$MgCl_2.6H_2O$ and $MgSO_4.6H_2O$	..	104	14	..
H	$MgSO_4.7H_2O$ and $MgSO_4.6H_2O$	..	73	15	..
I(*a*)	$MgSO_4.7H_2O$ and $K_2Mg(SO_4)_2.6H_2O$	..	...	58.5	5.5
K	K_2SO_4 and $K_2Mg(SO_4)_2.6H_2O$	..	...	22	16
L	K_2SO_4 and KCl	42	...	..	1.5
M	KCl; K_2SO_4 and $K_2Mg(SO_4)_2.6H_2O$	25	21	11	..
N	KCl; $MgSO_4.7H_2O$ and $K_2Mg(SO_4)_2.6H_2O$	9	55	16	..
P	KCl; $MgSO_4.7H_2O$ and $MgSO_4.6H_2O$	8	62	15	..
Q	KCl; $MgSO_4.6H_2O$ and $KMgCl_3.6H_2O$	4.5	70	13.5	..
R	$MgCl_2.6H_2O$; $KMgCl_3.6H_2O$ and $MgSO_4.6H_2O$	2	99	12	..

(*a*) The composition of the solution at this point is given by different figures in van't Hoff's earlier and later books. The latter are doubtless incorrect, as the former agree with those of H. S. van Klooster, *J. Phys. Chem.*, **21** (1917), 513.

duce a vertical axis representing the sum of the constituents of the solution. Again, since equivalent quantities of potassium sulfate and magnesium chloride in solution may be represented equally well as equivalent amounts of magnesium sulfate and potassium chloride, by plotting along the potassium chloride axis not mols of potassium chloride, but double mols, namely, K_2Cl_2, it is possible to construct a solid model expressing the composition of solutions containing any amounts of these ions. Such a model, a perspective drawing of which is seen in Fig. VII, may be constructed by inserting needles at the intersections of the lines in the plane figure, the heights of the needles being equal to the total number of mols of dissolved salt in the solution at this point, always remembering to consider the mol of potassium chloride to be K_2Cl_2. The tops of these needles may be connected by threads which mark off surface within which a saturated solution is in equilibrium with a single salt. Along the threads separating two fields the solution is saturated with two salts, while at the intersections three salts are present. The composition of unsaturated solutions is represented by points within the model. For example, a solution containing 2 mols of K_2Cl_2, 8 mols of $MgSO_4$ and 10 mols of $MgCl_2$ will be represented by a point found by counting 2 divisions along the potassium chloride axis, 8 divisions to the left in the direction of magnesium sulfate, which would then be 6 divisions to the left of the magnesium chloride axis, 10 divisions along the magnesium chloride axis, and then upwards 20 divisions, representing the total number of mols. This solution could also be represented as containing 6 mols of magnesium sulfate, 12 mols of magnesium chloride, and 2 mols of potassium sulfate, which would give the same locus to the point. On the removal of water from this solution all of the solid constituents will increase in the same proportion, so that the solution would move along the line in space away from the origin O until one of the fields is reached representing the separation of a solid salt. Further evaporation would then result in a change in composition of the solution equivalent to the removal of a saturated solution of the solid which is separated, since both this solid and water are being removed simultaneously. The composition of the solution would thus move along one of the faces of the model until one of the boundary lines has been reached, when a second salt would begin to separate along with the first. The solution would then change in composition as represented by motion along this line. By drawing lines on these surfaces it is possible to represent in the projection of the solid model such crystallization paths, so that the projection may be used in many cases instead of the solid model.

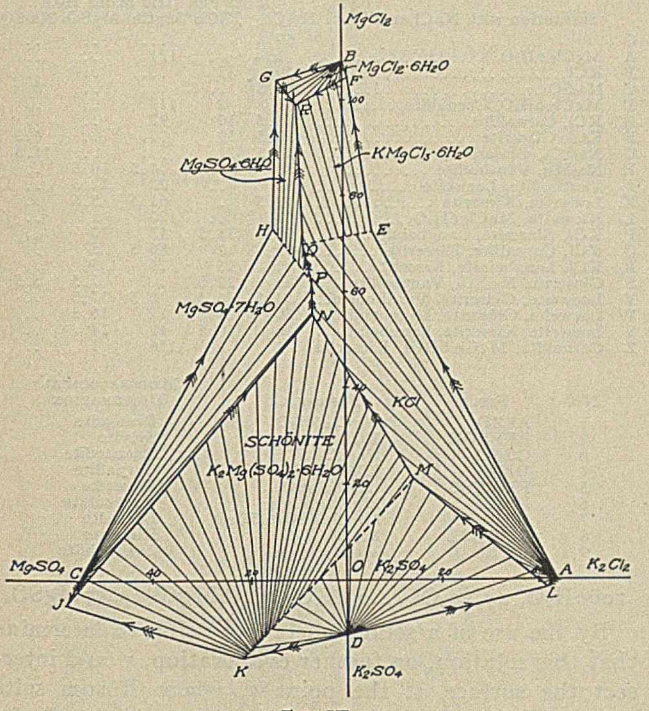

FIG. VI

It is possible to calculate in this way the amounts of the various solid salts and the amount of water which have separated from any solution when a certain point on a crystallization path has been reached. Thus, in the case of the solution considered above, the diagonal which joins the origin with the point representing the composition of this solution will be found to intersect the schönite face, showing that this would be the first salt to separate on evaporation. As evaporation proceeds the crystallization path would meet the boundary line of this face with the magnesium sulfate face, after which these two salts would separate together. Suppose, for example, we wish to calculate the actual amounts of these two salts separating and the amount of water that must be removed when the point N has just been reached, at which potassium chloride just begins to separate. The solution at N has the following composition: $1000H_2O + 9K_2Cl_2 + 16MgSO_4 + 55MgCl_2$. The amount of this solution we may represent as an unknown quantity by p, the amount of schönite separating we may represent by q, the magnesium sulfate by r, and the amount

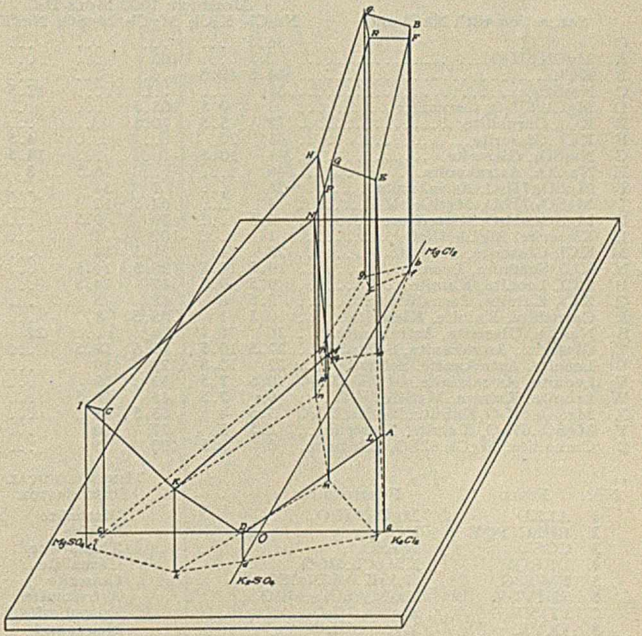

FIG. VII

of water removed by s. We may then represent what has become of the original solution during evaporation by means of the following equation:

$$1000H_2O + 8MgSO_4 + 10MgCl_2 + 2K_2Cl_2 = p(1000H_2O + 16MgSO_4 + 55MgCl_2 + 9K_2Cl_2) + q(K_2Mg(SO_4)_2.6H_2O) + r(MgSO_4.7H_2O) + sH_2O$$

By equating coefficients of the various substances present, it is possible to set up the following equations:

$$\begin{aligned} &\text{Coefficients of } H_2O\text{:} & 1000 &= 1000p + 6q + 7r + s \\ &\text{Coefficients of Mg:} & 18 &= 71p + q + r \\ &\text{Coefficients of } K_2\text{:} & 2 &= 9p + q \\ &\text{Coefficients of } Cl_2\text{:} & 12 &= 64p \end{aligned}$$

The solution of these equations gives the following values:

$$p = 0.188;\ q = 0.31;\ r = 4.38;\ s = 779$$

These values of q, r and s represent the amounts of the respective substances which have separated by evaporating the original solution and p represents the amount of solution left. If, instead of taking the amount of the original solution represented by 1000 mols of water, a different amount is taken, proportionate amounts of the solids and water are obtained from the solution on evaporation to the same point. When we come to consider the evaporation of sea water, we have in addition to the above components large amounts of sodium salts. Since during evaporation sodium chloride is always present, it is possible to represent saturated solutions such as are obtained on evaporating sea water by solid models similar to the one considered above. By introducing sodium chloride as another component no new degrees of freedom are introduced, provided it is stipulated that solid sodium chloride shall always be present. Van't Hoff and his co-workers have determined the solubility relationships at 25° and 83°. Fig. VIII represents the results for 25° contained in Table 2; results for 83° are found in Table 3 and Fig. IX. The amount of sodium chloride present is not considered in the projection, but is counted in the total number of dissolved mols which would be represented in a solid model. Sodium sulfate may be expressed in terms of the other salts present, since $Na_2SO_4 = Na_2Cl_2 + MgSO_4 - MgCl_2$, or, $= Na_2Cl_2 + K_2SO_4 - K_2Cl_2$. Thus point C, Table 2, is represented in Fig. VIII by counting $12^1/_2$ divisions to the left of the origin and $12^1/_2$ divisions along the K_2SO_4 axis. Its position in a space model would be $63^1/_2$ divisions vertically above the point so obtained. Such a model may be constructed in a way similar to that previously described.

TABLE 2

System, NaCl–KCl–MgCl₂–MgSO₄–Na₂SO₄, at 25°

	Saturation with NaCl and	Mols per 1000 Mols H_2O				
		Na_2Cl_2	K_2Cl_2	$MgCl_2$	$MgSO_4$	Na_2SO_4
O		55.5	...	...	...	...
A	$MgCl_2.6H_2O$	2.5	...	103	...	...
B	KCl	44.5	19.5	...	...	...
C	Na_2SO_4	51	...	...	...	12.5
D	$MgCl_2.6H_2O$, Carnallite	1	0.5	103.5	...	...
E	KCl, Carnallite	2	5.5	70.5	...	...
F	KCl, Glaserite	44	20	...	...	4.5
G	Na_2SO_4, Glaserite	44	10.5	...	...	14.5
H	Na_2SO_4, Astrakanite	46	...	...	16.5	3
I	$MgSO_4.7H_2O$, Astrakanite	26	...	7	34	...
J	$MgSO_4.7H_2O$, $MgSO_4.6H_2O$	4	....	67.5	12	...
K	$MgSO_4.6H_2O$, Kieserite	2.5	...	79	9.5	...
L	Kieserite, $MgCl_2.6H_2O$	1	...	102	5	...
M	KCl, Glaserite, Schönite	23	14	21.5	14	...
N	KCl, Schönite, Leonite	19.5	14.5	25.5	14.5	...
P	KCl, Leonite, Kainite	9.5	9.5	47	14.5	...
Q	KCl, Kainite, Carnallite	2.5	6	68	5	...
R	Carnallite, Kainite, Kieserite	0.5	1	85.5	8	...
S	Na_2SO_4, Glaserite, Astrakanite	26	8	16	...	22
T	Glaserite, Astrakanite, Schönite	27.5	10.5	16.5	18.5	...
U	Leonite, Astrakanite, Schönite	22	10.5	23	19	...
V	Leonite, Astrakanite, $MgSO_4.7H_2O$	10.5	7.5	42	19	...
W	Leonite, Kainite, $MgSO_4.7H_2O$	9	7.5	45	19.5	...
X	$MgSO_4.6H_2O$, Kainite, $MgSO_4.7H_2O$	3.5	4	65.5	13	...
Y	$MgSO_4.6H_2O$, Kainite, Kieserite	1.5	2	77	10	...
Z	Carnallite, $MgCl_2.6H_2O$, Kieserite	0	0.5	100	5	...

No.	Field	Formula	Mineralogical Designation
1	ALZD	$MgCl_2.6H_2O$	Bischofite
2	BFMNPQE	KCl	Sylvite
3	CGSH	Na_2SO_4	Thenardite
4	DZRQE	$KMgCl_3.6H_2O$	Carnallite
5	FMTSG	$(1.33K\ 0.67Na)SO_4$	Glaserite
6	SHIVUT	$Na_2Mg(SO_4)_2.4H_2O$	Astrakanite
7	VIJXW	$MgSO_4.7H_2O$	Epsom salts
8	JXYK	$MgSO_4.6H_2O$	Not found
9	KYRZL	$MgSO_4.H_2O$	Kieserite
10	TUNM	$K_2Mg(SO_4)_2.6H_2O$	Schönite
11	NUVWP	$Mg(1.52K\ 0.48Na)(SO_4)_2.4H_2O$	Leonite
12	PWXYRQ	$MgSO_4.KCl.3H_2O$	Kainite

The composition of sea water which has been evaporated until it is saturated with sodium chloride is as follows, expressed in mols of each constituent:

TABLE 3

System, NaCl–KCl–MgCl₂–MgSO₄–Na₂SO₄, at 83°

	Saturation with NaCl and	Mols per 1000 Mols H_2O				
		Na_2Cl_2	K_2Cl_2	$MgCl_2$	$MgSO_4$	Na_2SO_4
O		59	..	...	..	..
A	$MgCl_2.6H_2O$	1	..	121	..	..
B	KCl	39	37	...	..	..
C	Na_2SO_4	56.5	..	...	..	8
D	$MgCl_2.6H_2O$, Carnallite	1	2	117	..	..
E	KCl, Carnallite	1.5	10	92	..	..
F	KCl, Glaserite	39.5	39	...	..	4.5
G	Na_2SO_4, Glaserite	43.5	21	...	..	11.5
H	Na_2SO_4, Vanthoffite	51	..	4.5	10.5	..
I	Vanthoffite, Loeweite	35	..	22	12.5	..
K	Loeweite, Kieserite	12.5	..	61.5	5.5	..
L	Kieserite, $MgCl_2.6H_2O$	1	..	120	1	..
P	KCl, Glaserite, Langbeinite	29.5	33.5	13	10	..
Q	KCl, Carnallite, Kieserite	2	12	86.5	5	..
R	KCl, Langbeinite, Kieserite	11	15	76	5	..
S	Glaserite, Na_2SO_4, Vanthoffite	43	22.5	...	7.5	5.5
V	Loeweite, Glaserite, Vanthoffite	34.5	26.5	8.5	17.5	..
W	Loeweite, Glaserite, Langbeinite	30	24.5	12	16.5	..
Y	Loeweite, Kieserite, Langbeinite	16	10.5	42	14	..
Z	Carnallite, $MgCl_2.6H_2O$, Kieserite	1	2	116	1	..

No.	Field	Formula	Mineralogical Designation
1	ALZD	$MgCl_2.6H_2O$	Bishcofite
2	BFPRQE	KCl	Sylvite
3	CGSH	Na_2SO_4	Thenardite
4	DZQE	$KMgCl_3.6H_2O$	Carnallite
5	FPWVSG	$(K, Na)_2SO_4$	Glaserite
6	HSVI	$MgNa_6(SO_4)_4$	Vanthoffite
7	IVWYK	$Mg_2Na_4(SO_4)_4.5H_2O$	Loeweite
8	KYRQZL	$MgSO_4.H_2O$	Kieserite
9	WPRY	$Mg_2K_2(SO_4)_3$	Langbeinite

$$1000H_2O,\ 47Na_2Cl_2,\ 1.03K_2Cl_2,\ 7.36MgCl_2,\ 3.57MgSO_4$$

By the use of a solid model it is possible to determine that this solution, on further evaporation, would intersect the surface at the point a, where Epsom salts would begin to separate. The composition of the solution at this point is approximately $1000H_2O$, $11Na_2Cl_2$, $6K_2Cl_2$, $20MgSO_4$, $41MgCl_2$. Further evaporation would lead to the boundary between this field and the kainite field WX, after which these two salts would separate together. It is possible to calculate as before the amounts of each substance removed from the solution when the latter has the composition indicated, say, by X. Suppose that 10,000 g. of the original solution are used. The number of grams corresponding to the number of mols of each substance in the original solution is 24,790, so that 10,000 g. of solution would contain, instead of the previous number of mols of each constituent, only 0.404 of these quantities, namely, $404H_2O$, $19Na_2Cl_2$, $0.42K_2Cl_2$, $3MgCl_2$ and $1.44MgSO_4$. On evaporation this solution would yield p mols of the solution saturated at X, containing its constituents in the proportions indicated in Table 2, together with $qMgSO_4.7H_2O + rNa_2Cl_2 + sKCl.$-

$MgSO_4.3H_2O + tH_2O$. We can, therefore, write the following equation:

$$404H_2O + 19Na_2Cl_2 + 0.42K_2Cl_2 + 3MgCl_2 + 1.44MgSO_4 = p(1000H_2O + 3.5Na_2Cl_2 + 4K_2Cl_2 + 65.5MgCl_2 + 13MgSO_4) + qMgSO_4.7H_2O + rNa_2Cl_2 + sKCl.MgSO_4.3H_2O + tH_2O$$

By equating corresponding coefficients and solving the resulting equations, we obtain the following values:

$$p = 0.0458;\ q = 0.37;\ r = 18.9;\ s = 0.47;\ t = 354$$

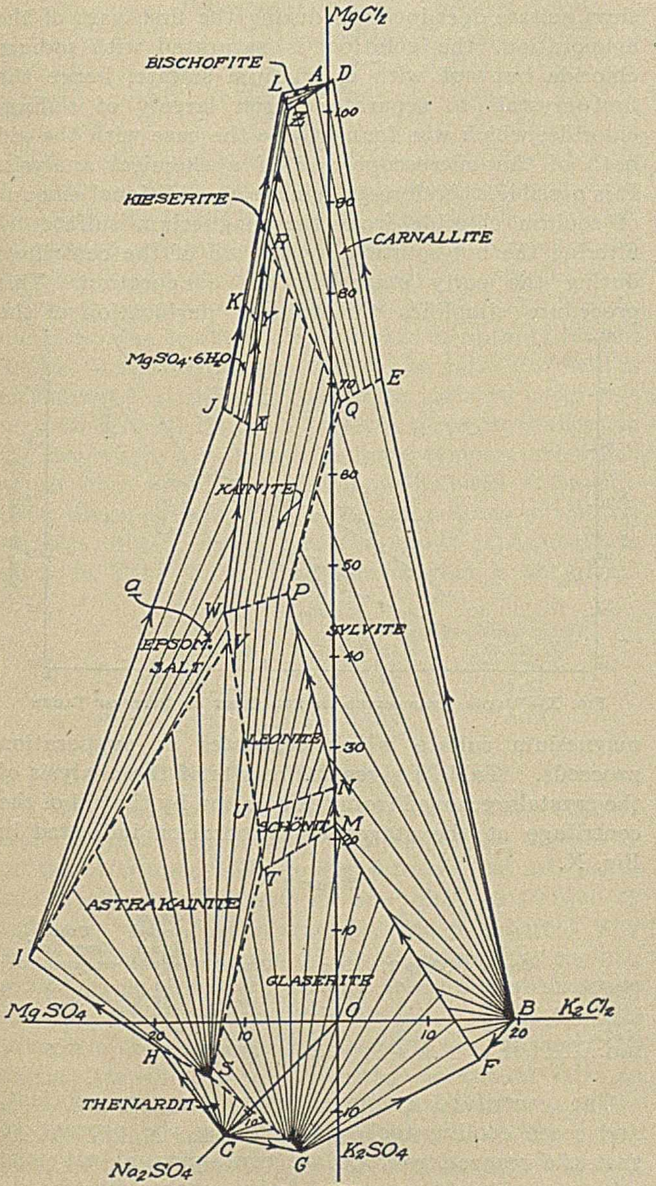

Fig. VIII

Hence we conclude that 354 mols of water have been evaporated, and 0.37 mol of Epsom salts, 18.9 mols of Na_2Cl_2 and 0.47 mol of kainite are in the solid portion. Similar calculations may be made to determine what will happen during all sorts of changes. For example, instead of removing water, a certain salt may be added to a solution saturated with other salts, and by the aid of geometric and algebraic considerations it will be possible to determine what will take place.

It is evident from the position of point *a* in the diagram for 25° that only a small amount of Epsom salts will have been crystallized by evaporation of the mother liquor from sea water before kainite will begin to separate. It is true that kainite shows a great tendency to supersaturation, and unless suitable nuclei are present this field might not be present, which will allow the evaporation and separation of Epsom salts to continue somewhat further until the potassium chloride field is reached. The conditions obtaining in solar evaporation are, however, very favorable to the crystallization of such a substance because of the presence of many impurities. In order to get a good separation of the potassium salts from the magnesium

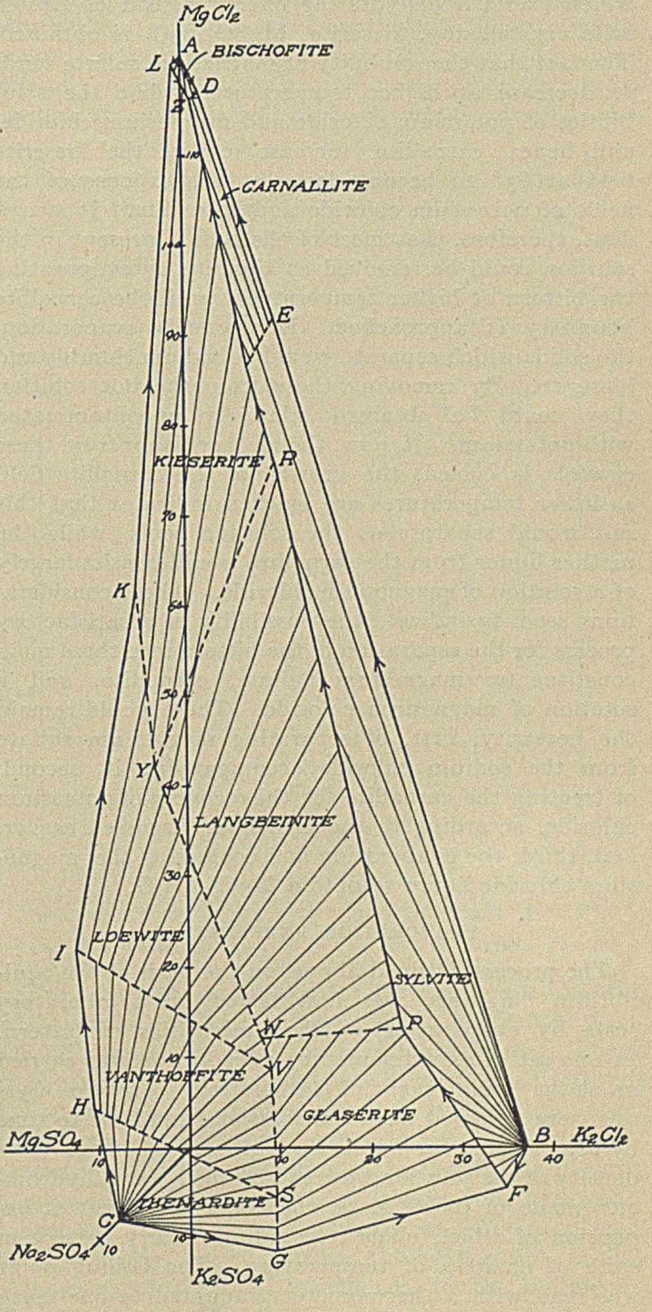

Fig. IX

salts in the bittern of sea water it is not desirable to carry on the evaporation so as to separate more than a small amount of Epsom salts at 25°. On comparing Figs. VIII and IX it will be seen that at the higher temperatures it is possible to continue the evaporation much farther before any salt containing potassium will crystallize from the hot solution. At this temperature and in the presence of the magnesium chloride which exerts a dehydrating effect, instead of Epsom salts crystallizing, kieserite, $MgSO_4.H_2O$, is obtained. The fact that the solubility of magnesium sulfate tends to decrease at higher temperatures, while the solubilities of potassium chloride and magnesium chloride, and hence, carnallite, increase, causes the kieserite field at 83° to become large at the expense of the fields of potassium chloride and carnallite. It is evident, therefore, that most of the sulfate present in the solution could be removed as kieserite by evaporating the bittern at higher temperatures until the carnallite boundary is approached. During this evaporation, the solids which separate would be sodium chloride and kieserite. By removing these from the hot solution they could be obtained relatively uncontaminated with potassium. If, now, the mother liquor from these crystals is cooled, the growth of the carnallite field as lower temperatures are reached indicates that this salt would separate as the solution cools, while the mother liquor from the carnallite would consist largely of a solution of magnesium chloride. These considerations seem to indicate the possibility of a satisfactory process for the separation of the bittern into three main constituents: magnesium sulfate, carnallite, and a solution of magnesium chloride. There would remain the necessity, first, of separating magnesium sulfate from the sodium chloride accompanying it, second, of treating the carnallite for the recovery of potassium chloride, according to the principles discussed earlier, and, third, the evaporation and cooling of the magnesium chloride liquor to obtain $MgCl_2.6H_2O$.

EVAPORATION EXPERIMENTS

The process outlined above, on the basis of the equilibrium diagrams, was first tested on a laboratory scale by evaporating weighed quantities of bittern. In one set of experiments the evaporation was carried on at the boiling point of the solution. Crops of crystals were removed from the solution from time to time by centrifuging the liquid through a muslin bag. The density of the solution was read by the aid of a hydrometer made of pyrex glass, the small coefficient of expansion of which made its readings nearly correct in spite of changes of temperature. The boiling point was read with a thermometer graduated to one-tenth of a degree. The amount of water, when each reading of density and boiling point was made, was determined by weighing the vessel containing the hot solution. It will be seen from the results, plotted in Fig. X, that the density and boiling point rise gradually until water has been removed amounting to about 36 per cent of the weight of the original bittern. The density and boiling point from here on increase more rapidly with the further removal of water. This more rapid increase is caused by encountering the boundary of the carnallite field shown in Fig. IX, and the subsequent separation of carnallite. The crystals which are deposited from the solution after this point is reached contain a considerable amount of potassium in the form of carnallite. A calculation of the amount of water which should be removed in order to reach the carnallite boundary at 83° gave a figure corresponding very closely with that indicated by the above curve.

The original mother liquor at 25° is saturated both with sodium chloride and magnesium sulfate, but since the solubility of sodium chloride does not materially change with the temperature, whereas that of magnesium sulfate does increase during the first part of the evaporation, the solution is saturated with sodium chloride but not with magnesium sulfate, hence the first crystals to separate consist largely of sodium chloride, which was found to be the case with the aid both of the microscope and of a chemical analysis. It is possible, therefore, to remove an additional amount of sodium chloride from the magnesium sulfate by filtering the hot solution by the aid of the centrifuge during the early stages of the evaporation. This procedure simplifies the further purification of the magnesium sulfate which separates as evaporation proceeds. Table 4 gives the results of the analysis of the crystals removed from the solution by the aid of the centrifuge at the stages of evaporation indicated in Fig. X.

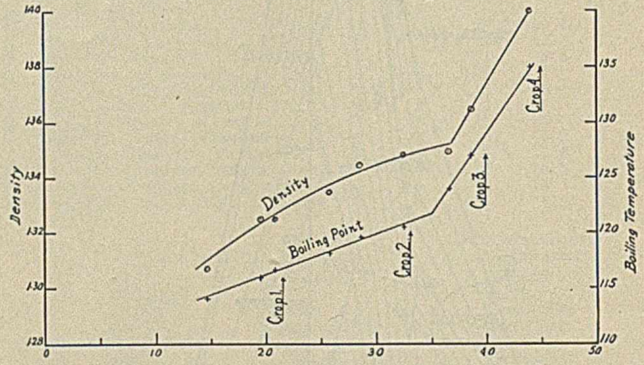

FIG. X—WATER EVAPORATED IN PER CENT OF BITTERN TAKEN

TABLE 4

	Crop 1	Crop 2	Crop 3	Crop 4	Crop from cooled mother liquor from 4	Crop from final evaporation
$MgSO_4$.....	15.1	24.6	20.1	24.1	1.3	3.5
$MgCl_2$.....	12.0	12.3	21.8	16.9	39.2	38.2
KCl.......	4.8	6.3	4.4	17.1	3.6	1.1
NaCl......	20.3	20.3	13.3	17.0	1.3	..
H_2O.......	52.8	36.4	40.5	24.9	54.7	57.5

The centrifuge employed was not very efficient, and some cooling took place during the process, so that the respective crops are contaminated with the salts that should remain in the mother liquor. It is evident from the results of this analysis, together with the course of the density and boiling-point curves, that the process contemplated furnishes the desired separation. In a second experiment, based upon the results of the first, the solution was evaporated until the density had reached a value of 1.35 at the boiling point of the solution, 121°. The crystals separating up to this point were removed and the mother liquor allowed to cool. The crystals separating on cooling should be carnallite, and it will be seen from the analysis of these crystals in Table 5 that their composition approximates closely to that of carnallite. The mother liquor from the carnallite consists principally of a solution of magnesium chloride, as is confirmed by its analysis. The potassium content of the first two fractions may be attributed to the cooling in the centrifuge inevitable in working on such a small scale. As is to be expected, the proportion of $MgSO_4.H_2O$ to NaCl is greater in the second crop of crystals than in the first.

TABLE 5—COMPOSITION OF MATERIAL OBTAINED AT VARIOUS STAGES IN PER CENT

	B. p. 116°	Removed at b. p. 121°, d. 1.35	Removed from cooled liquor	Theoretical for carnallite	Final mother liquor
K..........	3.1	5.7	10.0	14.1	0.4
Cl..........	23.5	19.1	36.6	38.4	23.4
SO_4..........	16.9	32.2	trace	0.0	2.7
Mg..........	3.9	9.2	7.6	8.7	7.9

The curve given in Fig. XI was obtained by an evaporation in which no crystals were removed, thus avoiding the inevitable losses occurring through attempts to remove crystals from the hot solution. The break in the boiling-point curve in Fig. XI is at a higher temperature than that in Fig. X. This is doubtless due to the use of different samples of bittern in the two experiments, so that the carnallite field is encountered at different points in the two cases. It may be noticed that the break is more pronounced in the case where it occurs at the lower temperature which is just what would be expected on the basis of the solubility diagram in Fig. IX. The composition of the two samples of bittern used in the above experiments is given in Table 6. The sodium content is not given.

TABLE 6

	Bittern used in getting curves in Fig. X	in Fig. XI
K....................	1.48	1.76
Cl....................	15.82	18.22
SO_4....................	5.81	3.88
Mg....................	5.38	6.32

OUTLINE OF PROPOSED PROCESS

1. EVAPORATION OF THE BITTERN—The bitterns from various sources will vary somewhat depending on the temperature of the liquid in the last salt pond, and whether or not any Epsom salts are allowed to separate. There is, in fact, no reason apparent why a crop of Epsom salts should not be removed by cooling, either artificially or by storage till winter, before the subsequent process of separation is applied. The process of solar evaporation should not, however, be carried far enough to cause any potassium salts to crystallize, as it is probably not desirable to separate the potassium content into two portions. The variations in the composition of the bittern caused by any of the above factors would not cause any serious difficulty, as during the later evaporation the separation of NaCl and $MgSO_4.H_2O$, kieserite, would take place in such proportion as to make the resulting liquid converge towards a fairly uniform composition.

It is more important, under present conditions, to recover all of the potassium salts, and hence to prevent their contaminating the NaCl and kieserite fraction, than it is to recover all of the Epsom salts, or to obtain pure magnesium chloride from the final liquor. Such contamination would result, if the evaporation were continued as far as the carnallite boundary, for some cooling during the separation of the kieserite from the mother liquor is inevitable, and if the solution is saturated with carnallite before this separation begins, some of it will crystallize along with the kieserite. On the other hand, if the evaporation is not continued so far, a little of the sulfate will remain in the solution, and will probably pass through the succeeding operations and come down with the magnesium chloride at the final stage of the process. Since very pure magnesium chloride will probably not be desired, the presence of this sulfate can do no harm.

Instead, therefore, of continuing the evaporation as far as the break in the boiling-point curves, as in Figs. X and XI, it will doubtless be better to evaporate till the boiling point is about 120° C. This will result in the recovery of practically all of the carnallite and still allow leeway for variations in the bitterns used.

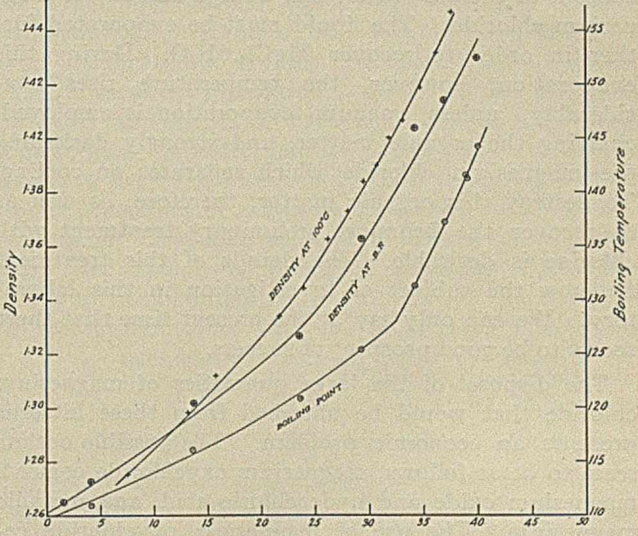

FIG. XI—WATER EVAPORATED IN PER CENT OF BITTERN TAKEN

The best type of evaporator for this operation will doubtless be of the film type, where a given part of the liquid is not boiling for a very long time. There is a tendency for magnesium chloride to hydrolyze, giving magnesium hydroxide and hydrochloric acid, which escapes with the steam. If the liquid is evaporated in a kettle it is boiling for such a length of time that a considerable amount of magnesium hydroxide is formed. If, on the other hand, the liquid is allowed to flow over a heated surface, the evaporation taking place very quickly, there is little time for this hydrolysis to take place. This liquid may then be kept in a settling tank without further loss of hydrochloric acid, even near the boiling temperature, provided actual boiling does not take place.

In principle, then, the process indicated is as follows: Evaporate until the boiling point of the liquid is raised to about 120° C. and the density is approximately 1.35. The liquid running off from the evaporator should be caught in a steam-jacketed tank where it is allowed to settle. The clear liquor is allowed to run off to a cooling tank, in which the carnallite will separate. The sludge of NaCl and kieserite is run into centrifuges, previously heated, where it is separated from the adhering mother liquor, which is run into the cooling tank mentioned above. The separation of the NaCl from the material remaining in the centrifuges and the recovery of Epsom salts will be discussed later.

2. THE RECOVERY OF THE CARNALLITE—The liquor containing the carnallite may be cooled by the fresh bittern going to the evaporator, in order to utilize the heat content of the latter. After it has been thoroughly cooled, the carnallite which has separated is removed and freed from its mother liquor by centrifuging. The recovery of the potassium chloride from this carnallite will be discussed later.

3. THE RECOVERY OF BROMINE AND MAGNESIUM CHLORIDE—The mother liquor from the carnallite contains a very little potassium, a little sulfate, a considerable amount of colloidal organic matter, the bromine content of the sea water, and a large amount of magnesium chloride. The liquid must be evaporated further in order to recover $MgCl_2.6H_2O$. During this evaporation, however, the temperature rises considerably, unless vacuum evaporation is employed, charring the organic matter, and strongly darkening the magnesium chloride which separates on cooling. To destroy this organic matter, therefore, as well as to recover the bromine, preliminary treatment with chlorine is desirable. The details of this treatment are now the subject of investigation in this laboratory. We can only say at the present time that there seems to be good prospect of success.

The disposal of the large quantities of magnesium chloride that would be obtained from these bitterns presents an economic problem. The possible outlets seem to be as follows: magnesium oxychloride cement, magnesium oxide and hydrochloric acid, and metallic magnesium. The use of magnesium oxychloride cements might be greatly increased by skilful advertising, hydrochloric acid might be substituted for sulfuric acid, for certain purposes, and there seems to be good reason to anticipate a large production of magnesium in the future.

4. THE SEPARATION OF SODIUM CHLORIDE AND MAGNESIUM SULFATE—The separation of the sodium chloride and the kieserite obtained in the first part of the process is complicated by the possibility of forming astrakanite, $Na_2Mg(SO_4)_2.4H_2O$, at ordinary temperatures and of loeweite, $Na_4Mg_2(SO_4)_4.5H_2O$, or vanthoffite, $Na_6Mg(SO_4)_4$, at higher temperatures. In order to put the separation of the magnesium from the sodium salts on an exact basis it is desirable to have a knowledge of the solubility relationships of the chlorides and sulfates of these two metals. It has been found possible, by using data given by van't Hoff, Seidell[1] and Roozeboom,[2] to construct the equilibrium diagram for all but two points which are unimportant for the present purpose. The data used are given in Table 7, and are represented graphically in Fig. XII, giving a diagram similar to that in Fig. VI, where potassium chloride is considered instead of sodium chloride. The two undetermined points have been added more or less at random, for the sake of completing the fields, and are denoted by interrogation marks on the figure.

Now the material obtained from the first stage of our process contains $MgSO_4$ and NaCl in nearly equivalent amounts, and hence, if dissolved in water, would be represented by a point lying nearly vertically above the origin, at a distance increasing as the solution is evaporated. It might, therefore, cut the surface of the solid model in the astrakanite face, which would prevent the separation of the sodium from the magnesium. A little magnesium chloride, however, would, if added, raise the solution away from the astrakanite field, so that we would have only NaCl and $MgSO_4.7H_2O$ to deal with.

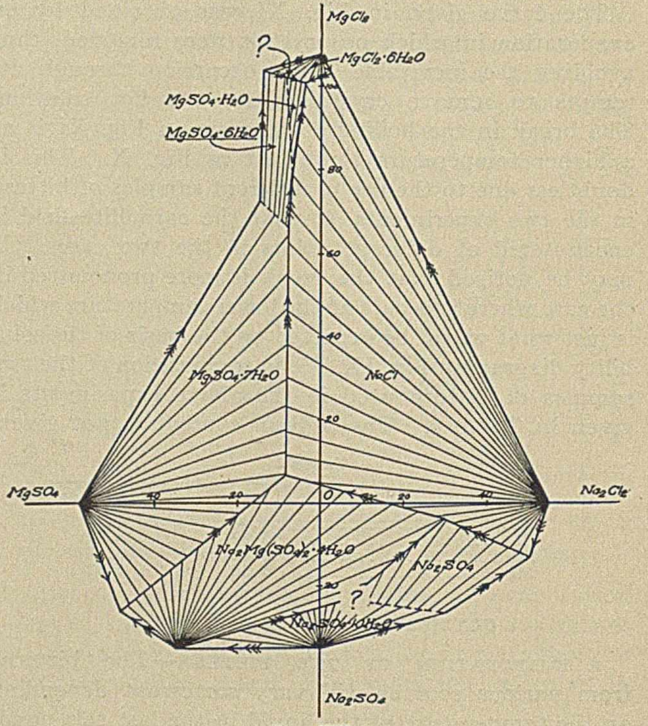

FIG XII

The solubilities of these two salts are affected so differently by the temperature that we may anticipate their separation by first cooling, removing Epsom salts, then evaporating partly at higher temperatures, removing sodium chloride, then cooling again, etc. The portion of the equilibrium diagram that can be constructed for 83° from van't Hoff's data shows that at that temperature loeweite and vanthoffite intrude themselves between the magnesium sulfate and sodium chloride fields, even when a considerable amount of magnesium chloride is added, so that it may not be advisable to evaporate the solution for the removal of sodium chloride at too high a temperature. The great tendency of these double salts towards supersaturation might allow the evaporation to proceed without their formation.

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[1] *Am. Chem. J.*, **27** (1902), 52; see also Schreinemakers and Baat, *Z. physik. Chem.*, **67** (1909), 533.

[2] *Z. physik. Chem.*, **2** (1888), 518.

TABLE 7

System, $NaCl-MgCl_2-MgSO_4-Na_2SO_4$, at 25°

Solid phases	Na_2Cl_2	$MgCl_2$	$MgSO_4$	Na_2SO_4
Na_2Cl_2	55.5	...	..	..
$Na_2SO_4.10H_2O$	..	...	..	35.5
$MgSO_4.7H_2O$	..	...	58	..
$MgCl_2.6H_2O$	..	108	..	..
$MgCl_2.6H_2O$; Na_2Cl_2	2.5	103	..	..
Na_2Cl_2; Na_2SO_4	51	...	..	12.5
Na_2SO_4; $Na_2SO_4.10H_2O$(*)	30	...	..	27
$Na_2SO_4.10H_2O$; $Na_2Mg(SO_4)_2.4H_2O$	..	...	35	35
$MgSO_4.7H_2O$; $Na_2Mg(SO_4)_2.4H_2O$	..	...	48	26
$MgSO_4.7H_2O$; $MgSO_4.6H_2O$	..	73	15	..
$MgSO_4.6H_2O$; $MgSO_4.H_2O$		Not determined		
$MgSO_4.6H_2O$; $MgCl_2.6H_2O$	..	104	14	..
Na_2Cl_2; Na_2SO_4; $Na_2Mg(SO_4)_2.4H_2O$	46	...	16.5	3
Na_2SO_4; $Na_2SO_4.10H_2O$; $Na_2Mg(SO_4)_2.4H_2O$		Not determined		
Na_2Cl_2; $MgSO_4.7H_2O$; $Na_2Mg(SO_4)_2.4H_2O$	26	7	34	..
Na_2Cl_2; $MgSO_4.7H_2O$; $MgSO_4.6H_2O$	4	67.5	12	..
Na_2Cl_2; $MgSO_4.6H_2O$; $MgSO_4.H_2O$	2.5	71	9.5	..
Na_2Cl_2; $MgSO_4.H_2O$; $MgCl_2.6H_2O$	1	102	5	..

(*) From the experimental work of Professor W. C. Blasdale, which is still in progress in this laboratory, this point may be considerably in error.

In order to have the desired data it is very important that the equilibria here involved should be determined for temperatures both lower and higher than 25°. Work on the solubilities at 0° is now in progress in this laboratory and will be published as soon as possible. It is planned, also, to include potassium salts in this work, so that a diagram for 0° similar to that in Fig. VIII can be constructed, and which might suggest a modification of the first treatment of the bittern.

5. THE RECOVERY OF POTASSIUM CHLORIDE FROM CARNALLITE—The recovery of potassium chloride from the carnallite was discussed earlier in connection with Fig. V. We may ask whether hot or cold water should be used for this purpose. A reference to the tables shows that the proportion of potassium chloride to magnesium chloride in the solution at equilibrium with potassium chloride and carnallite is much less at 25° than at 83°. This makes it obvious that a much smaller proportion of potassium chloride goes into solution at the lower temperature. The composition of the solution in equilibrium with potassium chloride and carnallite at 25° is as follows: $1000H_2O + 5.5K_2Cl_2 + 72.5MgCl_2$. From this it is possible to calculate the amount of water to be used in extracting the magnesium chloride from the carnallite at this temperature. If 1 mol of carnallite is used we can write the following equation:

$$KMgCl_3.6H_2O + xH_2O = yK_2Cl_2 + z(1000H_2O + 5.5K_2Cl_2 + 72.5MgCl_2)$$

From this we find $x = 7.8$; $y = 0.425$; $z = 0.0138$. That is, 1 mol, or 277.5 g. of carnallite, requires 7.8 mols, or 140.4 g. of water, or, the weight of water required is approximately half the weight of the carnallite. At the lower temperatures that would naturally be used somewhat more water would be required, but relatively less KCl would be dissolved.

The liquor used in extracting the carnallite may then be partly evaporated and cooled, whereupon another crop of carnallite crystals will be obtained. To obtain the maximum amount of carnallite but no magnesium chloride the solution should be evaporated to such an extent that on cooling with separation of carnallite its composition will correspond to point *B* in Fig. V, which is $1000H_2O + 105MgCl_2 + K_2Cl_2$. The amount of evaporation necessary is calculated from the following equation:

$$1000H_2O + 72.5MgCl_2 + 5.5K_2Cl_2 = xH_2O + yKMgCl_3.6H_2O + z(1000H_2O + 105MgCl_2 + K_2Cl_2)$$

This gives $x = 340$; $y = 9.8$; $z = 0.6$; hence 25720 g. of solution should lose 6120 g. of water, giving 2720 g. of carnallite on cooling; or, 1 ton of solution should lose 0.238 ton of water, and deposit 0.107 ton of carnallite.

It will probably not pay, here or in stage 2 of the process, to recover the small amount of potassium chloride remaining in the mother liquor from the carnallite.

Condensed summary of the above process:

Evaporate bittern till boiling point becomes about 120°, and density (hot) 1.35. Separate solid and liquid while hot (settling tank and centrifuge).

A. Solid. NaCl and $MgSO_4.H_2O$. Dissolve out NaCl with cold water (containing some $MgCl_2$?); dissolve residue in hot water and cool with ice machine, getting $MgSO_4.7H_2O$.

B. Liquid. Cool.

I. Solid carnallite. Extract with minimum amount cold water, leaving
  1. Solid KCl.
  2. Solution. Evaporate partly, cool.
    a. Solid carnallite, add to I.
    b. Solution of $MgCl_2$, add to II.

II. Solution, mainly $MgCl_2$. Bleach with Cl_2 and remove Br_2. Evaporate, cool, recover solid $MgCl_2.6H_2O$.

The above process is being tested in this laboratory on a semi-commercial scale under the direction of Professor Merle Randall, and will be described in a later publication. It may be mentioned, however, that an excellent separation of actual bittern has been obtained into one lot of material consisting of kieserite and sodium chloride, another consisting of carnallite of a high degree of purity and whiteness, and a mother liquor consisting of magnesium chloride solution containing but very small amounts of sulfate and of potassium. For example, using 150 lbs. of bittern, and evaporating till the boiling point was 118°, the three fractions of material obtained had the following composition:

	NaCl, Kieserite Fraction	Carnallite Fraction		Mother Liquor
		Found	Theoretical	
K	1.6	11.0	14.1	Trace
Cl	21.1	37.4	38.4	23.4
SO_4	22.2	0.4	0.0	1.25
Mg	10.0	8.1	8.7	...

The writer wishes, in conclusion, to express great appreciation for the cordial coöperation of the Oliver Salt Company, which has given information and has furnished samples of material and bittern.

Generous credit should be given to Messrs. A. H. Foster, W. D. Coughlan, Carl Iddings and W. D. Ramage for much of the experimental work herein described, and to Mr. Iddings for drawing the illustrations. Professor W. C. Bray has given considerable time to the final criticism of the manuscript and the checking of the figures necessitated by the absence of the author from Berkeley due to his acceptance of a commission in the army.

Since concluding the above work there has appeared in *Chemical Abstracts*, Vol. 11 (1917), 2719, a brief outline of a process by T. Nishimura, *J. Chem. Ind. Tokyo*, Vol. 20 (1917), 587, for extracting potassium from bittern which, apart from certain serious errors in translation, seems to be fundamentally similar to that herein described, and which we may welcome, therefore, as additional evidence of the feasibility of working up these bitterns instead of allowing them to be largely wasted, as at present.

BERKELEY, CAL.

THE DIRECT HEAT TREATMENT OF CEMENT MILL DUST TO INCREASE ITS WATER-SOLUBLE POTASH CONTENT

By Albert R. Merz

Received November 9, 1917

When a potash-containing silicate mixed with lime is ignited at temperatures above 1000°, the lime displaces more or less potash which is volatilized. This fact suggests a possible procedure for the immediate commercial production of potash from potash silicates. Since, however, commercial grades of any insoluble potash silicate in quantity contain comparatively small percentages of potash no process for recovering potash from such material can offer much promise of profitable application unless there is also produced some other product of value in addition to the potash. Fortunately, and by strange coincidence, the two manufacturing industries of this country which have the largest outputs are industries wherein raw materials which contain potash silicates are heated with limestone to temperatures so high that the potash is more or less displaced and volatilized. These two industries, *viz.*, the blast-furnace and Portland cement industries, are already firmly established and for them the problem becomes merely one of the successful recovery of the volatilized potash as a by-product. It may be stated that the chief potential source of potash in this country is the raw material which is or may be used in these industries.

Some potash is contained in all cement materials. In a recent publication by this Bureau[1] it was shown that in the raw mix as fed into the kiln the potash varies from 0.20 to 1.16 per cent in the various cement mills of the United States and Canada, and that the percentage of this potash driven from the kilns in the different plants varies from 24.5 to 95.9. From the results of the analysis of samples of raw mix and of cement from 102 plants it has been calculated that the potash escaping from the kilns of these plants ranges from 0.35 to 5.14 lbs. per barrel of cement produced with an average for the plants of this country of 1.93 lbs. On the basis of an average production of 90,000,000 bbls., the total potash escaping from the cement plants of this country, as at present operated, amounts to about 87,000 tons annually. The profitable recovery of this potash is not dependent upon successful collection alone, but it must be obtained in a form so concentrated as to be merchantable and at a cost sufficiently low to permit of a profit under normal market conditions. Three factors determine such profitable recovery and must be considered in any attempt advantageously to obtain potash as a by-product in the cement industry: liberation of the potash, recombination and collection.

A full discussion of the various conditions which affect the liberation of potash from the raw mix in the kiln and of the methods which have been devised to increase the percentage of potash volatilized is to be found in the publication already mentioned. Similarly the methods of collection of the potash that escapes from the kilns have received ample treatment in this bulletin. The work presented here deals with a method for making water-soluble the "recombined" potash of cement dust.

The recovery of the potash which escapes from the kilns of cement mills was first made by the Riverside Portland Cement Co., at Riverside, Cal., using the Cottrell process of electrical precipitation. The cement dust recovered at this plant was found to contain upward of 90 per cent of its potash in the water-soluble form. A sample of precipitator dust secured from this mill was analyzed by the author and found to give a total potash content of 10.7 per cent, and a water-soluble potash content of 9.8 per cent. The water-soluble potash of this particular dust was 92 per cent of the total potash present in the dust. It was natural to expect that the dust which would be recovered by a similar method at other plants would have a water-soluble potash content approximating the same percentage of the total potash present. It was found, however, when the Cottrell process was installed at the mill of the Security Cement and Lime Company, at Hagerstown, Md., that the dust collected contained a considerable portion of its potash content in a form which was not readily soluble in water. A sample of dust obtained from this plant was found to contain 11.4 per cent total potash, whereas the water-soluble potash content was but 6.8 per cent. Instead of the anticipated 90 per cent or over, this particular sample of dust contained but 60 per cent of its potash in water-soluble form. It has been found at another plant, the Alpha Portland Cement Company at Cementon, N. Y., where installation of the Cottrell process for the recovery of dust has been made, that the water-soluble potash in the dust recovered constitutes a considerably lower proportion of the total potash present than at the Security plant. A sample of the dust recovered at this mill was found by the author to contain 7.0 per cent total potash and only 2.9 per cent water-soluble potash This sample of dust, therefore, had but 41 per cent of its potash present in the water-soluble form.

The term "water-soluble potash" as used above refers to that potash which is obtained in solution by the procedure given in the "Methods of the Association of Official Agricultural Chemists," in accordance with which 10 g. of the sample are boiled with 300 cc. of water for 30 min. and the volume subsequently brought to 500 cc. As it is customary in the fertilizer trade to make payment for none other than this potash it becomes a matter of vital importance to the cement manufacturer who recovers his dust for sale to the fertilizer trade on the basis of its water-soluble potash content to ascertain the cause of the failure to secure the maximum amount of his potash in this form, to devise methods, if possible, to obtain most of the potash in his dust directly as water-soluble potash, to adopt some procedure which will profitably increase the water-soluble potash of his dust as at present obtained or, finally, to endeavor to secure from the fertilizer trade credit for that available potash of his dust which is but slowly water-soluble and for which at present he receives no compensation.

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[1] W. H. Ross, A. R. Merz and C. R. Wagner, U. S. Department of Agriculture, *Bull.* 572.

As stated in a previous publication,[1] the potassium compounds occurring in cement dust may be divided into three groups: (1) those which are readily water-soluble; (2) those which are slowly soluble; and (3) those which are insoluble.

The insoluble potash represents the combinations occurring in the original silicates of the raw mix carried over mechanically in the dust before being subjected to a temperature sufficiently high to bring about decomposition. The form of combination which is slowly soluble in water has been attributed to the recombination of the volatilized potash with the finely divided incandescent particles of siliceous coal ash carried over in the dust[2] and it is claimed[3] to be proportional to the amount of coal ash present in the gases from the kiln. Where oil or gas is used for fuel this combination of the potash occurs in comparatively small amount but, where coal is used for burning, the extent to which the potash occurs in this "recombined" form may be considerable. Of the three cement plants mentioned above, the Riverside Portland Cement Company uses oil for fuel while the Security Cement and Lime Company and the Alpha Portland Cement Company use coal in their operation. The insoluble potash is assumed to be that portion of the total potash which remains undissolved after the dust is boiled in a 5 per cent solution of hydrochloric acid. The difference between the total potash and the sum of the water-soluble and insoluble portions is taken as the slowly soluble or recombined potash. It has been shown by Nestell and Anderson[4] that continued extraction of cement dust with boiling water for 10 hrs. is sufficient to dissolve practically all this slowly soluble potash.

Various methods have been tried to prevent the recombination of potash and it is reported by J. J. Porter,[4] of the Security Cement and Lime Company, that the use of salt has been found beneficial in this connection.

In a publication by W. H. Ross[5] it was shown that when feldspar and lime are digested with water under a steam pressure of 10 to 5 atmospheres about 90 per cent of the potash in the feldspar passes into solution. In cement dust as it escapes from the kilns, the slowly soluble and insoluble potash are already associated with a considerable percentage of free lime and consequently he concluded that the greater part of the constituents might be recovered in water-soluble form by digesting the dust with steam under pressure. Experimental work has shown this to be the case and the results obtained by this procedure will be published separately.

Since ignition of a potash-containing silicate in the presence of lime liberates potash and since the cement dust, as has already been stated, contains a considerable proportion of free lime, it occurred to the author that the recombined potash might be rendered water-soluble by simple ignition of the dust. A preliminary experiment was carried out in which a sample of the above-mentioned treater dust from the Security Cement and Lime Company was ignited with an equal weight of calcium carbonate in a J. Lawrence Smith crucible at about 1000° for a period of 40 minutes. The water-soluble potash was found to have been increased by this treatment from 6.8 to 10.4 per cent on the basis of the original sample. In other words, the water-soluble potash had been increased from 60 per cent to 91 per cent of the total potash content. The addition of calcium carbonate served, however, to considerably lower the percentage of water-soluble potash in the resultant product so that the latter contained 7.1 per cent of water-soluble potash. It is possible that a procedure somewhat similar to this on a large scale should prove profitable to the cement manufacturer who at present receives nothing for his recombined potash, for although he has not appreciably increased the percentage of water-soluble potash in the material he sells, he has increased the quantity of his product over 40 per cent.

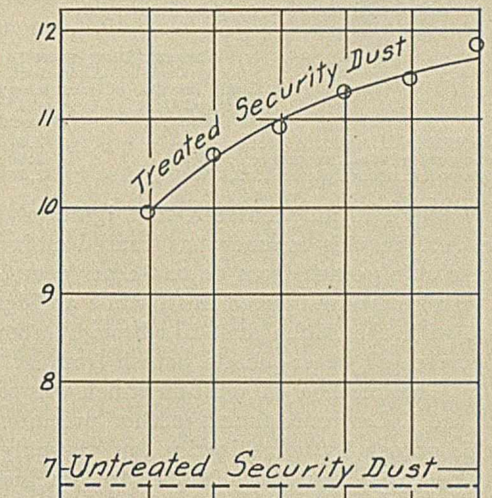

Fig. I—Percentages of Water-Soluble Potash in Untreated and Treated Dusts

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[1] W. H. Ross and A. R. Merz, This Journal, 9 (1917), 1035.

[2] R. J. Nestell and E. Anderson, *Ibid.*, 9 (1917), 646.

[3] J. J. Porter, paper presented at the meeting of the Portland Cement Association, Chicago, September, 1917.

[4] *Loc. cit.*

[5] This Journal, 9 (1917), 467.

Similar experiments were carried out without the addition of calcium carbonate, using the same dust and also the dust from the Alpha Portland Cement Company already referred to. The results obtained were unexpected—with the former dust there was a decided increase of the water-soluble potash of the original sample, whereas with the latter there was an actual decrease. Table I gives the percentages of total potash in the dusts before treatment and the percentages of water-soluble potash before and after treatment at about 1000° in a closed crucible, all expressed on the basis of the original sample.

TABLE I

	CEMENT DUST FROM	
	Security Cement and Lime Co. Original Dust Per cent	Alpha Portland Cement Company Original Dust Per cent
Total potash, K_2O	11.4	7.0
Water-soluble potash before treatment	6.8	2.9
Water-soluble potash after treatment	9.4	2.1

From Table I it may be seen that ignition of the Security dust in this manner has caused an increase of water-soluble potash from 60 to 82 per cent of the total potash in the un-ignited dust, while an identical procedure with the Alpha dust has brought about an actual decrease of water-soluble potash from 41 to 30 per cent. The method of ignition which was carried out as in the J. Lawrence Smith method precluded the loss of this potash by volatilization and the only conclusion to be drawn was that a further "recombination" of water-soluble potash takes place in the dust.

TABLE II—EFFECTS OF IGNITION FOR 40 MINUTES IN AN OPEN DISH ON PRECIPITATOR DUST EXPRESSED IN PERCENTAGES

	SECURITY DUST							ALPHA DUST						
	Untreated	Temperature of Ignition						Untreated	Temperature of Ignition					
ON BASIS OF ORIGINAL DUST	Dust	600°	700°	800°	900°	1000°	1100°	Dust	600°	700°	800°	900°	1000°	1100°
Loss in weight	...	5.69	6.05	6.40	6.64	7.32	8.70	..	12.91	13.21	13.48	14.55	15.52	17.84
Total potash, K_2O	11.40	11.37	11.42	11.42	11.27	11.19	10.99	7.02	6.98	6.97	7.00	6.48	5.97	5.38
Water-soluble potash	6.79	9.36	9.95	10.18	10.55	10.62	10.82	2.93	4.16	4.69	5.06	4.96	5.15	5.01
ON BASIS OF PRODUCT														
Total potash	11.40	12.06	12.15	12.20	12.07	12.07	12.04	7.02	8.02	8.03	8.09	7.58	7.07	6.55
Water-soluble potash	6.79	9.93	10.59	10.88	11.30	11.46	11.85	2.93	4.78	5.40	5.85	5.81	6.10	6.10

TABLE III—WATER-SOLUBLE POTASH IN IGNITED CEMENT DUSTS EXPRESSED IN PERCENTAGES OF TOTAL POTASH CONTENT

	SECURITY DUST							ALPHA DUST						
	Untreated	Temperature of Ignition						Untreated	Temperature of Ignition					
	Dust	600°	700°	800°	900°	1000°	1100°	Dust	600°	700°	800°	900°	1000°	1100°
On basis of total potash in original dust	60	82	87	89	93	93	95	42	59	67	72	71	73	71
On basis of total potash in ignited dust	..	82	87	89	94	95	98	..	60	67	72	71	86	93

A glance at the dusts in their original condition before ignition would be sufficient to enable anyone to distinguish between them. The Security dust is ash-gray in color while the Alpha dust is black, evidently containing unburnt carbonaceous matter. A sample of the latter dust was boiled with concentrated hydrochloric acid for 30 minutes, filtered on an alundum plate and a carbon determination was made on the residue. The carbon was found to form 9.26 per cent of the dust. Assuming the dust lost from the kiln to be 4 per cent of the raw mix, and 600 lbs. of raw mix as necessary to yield a barrel of cement, then the loss of carbon per barrel of cement produced amounts to 2.2 lbs. The view that this carbonaceous matter may have an influence on the behavior of the cement dust suggested the ignition of a sample of Alpha dust in an open dish placed in a muffle so that the carbonaceous matter might be burned up, combustion of this material not occurring to any great extent in the closed J. Lawrence Smith crucible. The temperature at which such ignition occurred could be approximately controlled by use of a thermo-electric pyrometer and a rheostat in the circuit of the electrically heated muffle, and it was decided to make ignitions of the two cement dusts through a range of temperatures to ascertain the influence of differences of the temperature of ignition on the conversion of "recombined" potash into water-soluble potash or on the further recombination of water-soluble potash. As interest was centered on the behavior of the Alpha dust an initial experiment was run with this dust subjected for 40 min. to a temperature of 600°. This experiment showed that instead of a diminution of water-soluble potash as obtained previously there was an increase thereof when the carbonaceous matter was removed by combustion. It is questionable that the recombination of potash can be attributed solely to the siliceous ash of the coal in the light of the above results. The writer hesitates to ascribe this effect to the carbon, however, because the Alpha dust also contains sulfides in some quantity, a fact forcibly presented when the dust was treated with hydrochloric acid. These sulfides no doubt also undergo change when the dust is ignited in an oxidizing atmosphere and it may be that they have an influence on the recombination of potash in the absence of air.

Table II contains the results of this series of experiments with the two dusts.

The readings of the pyrometer are probably not exact indications of the actual temperatures obtaining in the dishes. In the first place, it was evident that all parts of the muffle were not at a uniformly high temperature and, again, the temperatures locally produced by the combustion of the carbon in the Alpha cement dust may have been considerably in excess of the recorded temperatures. In consequence of this carbon content the manner of distribution of the sample in the dish may also have had an influence on the local temperatures of the samples. These same conditions, however, would obtain in large scale operations.

Table III shows that the ignition at temperatures of 600° or over in an oxidizing atmosphere of treater dust from cement mills results in a liberation of the recombined potash of the dust and that this released potash (at 900°) amounts to 33 per cent of the total potash of the original dust in the one case and to 29 per cent in the other. In view of this fact it is considered possible that a procedure similar to this may find successful application on a manufacturing scale. The use of a kiln using oil or gas as a fuel is to be considered necessary for such operations, or if coal is used some arrangement whereby heat is supplied without contact of the fuel with the dust to be treated.

In case the dust is to be sold directly to the fertilizer trade such a process would have the advantage of yielding a product with its water-soluble potash more concentrated than in the original dust, for not only is the recombined potash liberated by this procedure but the total weight of the material is diminished. This diminution of weight may be attributed to loss of water, of carbon dioxide, and of any carbonaceous matter in the dust. If, however, the preparation of potash salts is aimed at, it is possible that for dusts behaving like the Alpha dust digestion under steam pressure, as mentioned above, would be the preferable method of treatment since in the latter method there is no volatilization of potash and the yield of water-soluble potash on the basis of the total original potash is consequently greater. This volatilization apparently commences at about 900°. As stated before, the temperatures of the Alpha dust very likely were considerably above those indicated by the pyrometer and relatively greater volatilization of potash in this dust at a given temperature may be easily explained on this assumption.

To ascertain the effect of time of heating on the two dusts, samples were ignited at 1000° for 20, 40 and 60 minutes. The results obtained are shown in Table IV.

Table IV—The Effect on Water-Soluble and Total Potash Content of Variations in Duration of Ignition. Temperature 1000°

	Security Dust			Alpha Dust		
	20 min.	40 min.	60 min.	20 min.	40 min.	60 min.
On Basis of Original Dust						
Loss in weight	7.06	7.32	7.37	15.15	15.52	15.80
Total potash	11.33	11.19	11.06	6.12	5.97	5.89
Water-soluble potash	10.60	10.62	10.58	5.02	5.15	4.89
On Basis of Product						
Total potash	12.19	12.07	11.94	7.21	7.07	7.00
Water-soluble potash	11.41	11.46	11.42	5.92	6.10	5.81

It is apparent from inspection of this table that for the periods considered ignition of the dust for a longer period than 20 minutes has no decided effect on the ratio of water-soluble to total potash in the material.

SUMMARY

I—Dusts from cement mills using coal as fuel have considerable proportions of their potash content in a form not readily water-soluble.

II—Ignition of such dusts in an oxidizing atmosphere at temperatures of 600–1100° converts the "recombined" potash into a readily water-soluble form.

III—For the periods considered (20 to 60 minutes) time of ignition apparently has little effect on the water-soluble potash content of the resultant product.

Department of Agriculture
Bureau of Soils, Washington, D. C.

EFFECT OF COAL ASH ON THE LIBERATION AND NATURE OF CEMENT MILL POTASH

By N. S. Potter, Jr., and R. D. Cheesman

Received December 6, 1917

In all articles thus far published relating to potash as a by-product in the manufacture of cement, no consideration has been given to the potash content of the coal used in burning and its attendant effects. It is the purpose of this paper to point out the effect the coal ash has upon the liberation of potash in the kilns and upon the nature of the so-called "treater dust" collected.

The potash collected from the kiln stack gases where coal is used for burning appears in practically two forms, water-soluble potash and the insoluble or slowly soluble potash. The insoluble potash has been attributed to two causes: the potash in the unburned or partly calcined raw material carried over mechanically in the gases and to a recombination of the volatilized potash with the finely divided ash particles of the coal.

R. J. Nestell and E. Anderson in their paper, "The Nature of Cement Mill Potash,"[1] state that "the most important differences in the potash material from coal-fired and oil-fired kilns, as shown by the analyses given, lie in the relative amounts of soluble and insoluble K_2O, and in the wide variation in potash concentration in the lighter fractions of dust obtained from kilns using these two different forms of fuel. In the dust from the oil-fired and coal-fired kilns previously referred to, where in the first case the per cent of insoluble K_2O was 0.56 and in the second case 4.55 per cent, it seems safe to assume that the amount of mechanically carried-over raw material was practically the same, consequently the difference noted in the amount of siliceous material shown cannot be due to a greater amount of calcined raw mix in one case. The only other source of siliceous material is the ash from the coal used as fuel. Since this ash, coming as it does from finely powdered coal, must be in a state of extremely fine subdivisions, approaching that of a true fume, it is reasonable to suppose that part of this ash would be collected among the lighter portions of dust. Since approximately 9 lbs. of coal ash are introduced into the kiln per barrel of clinker burned, if only one-half this amount is carried out with the gases, it would still be sufficient to effect appreciably the composition of the collected dust, as the amount of dust caught per barrel of clinker produced is only about 20 lbs. Consequently it is probable that the considerable amount of insoluble potash shown to be present in the dust from the coal-fired kilns is in reality due to a combination of the volatilized potash with the finely divided incandescent particles of siliceous coal ash."

Wm. H. Ross, in his paper on "The Extraction of Potash from Silicate Rocks—II,"[2] also states that "the slowly soluble combination is explained on the ground that during the burning of the cement part of the volatilized potash undergoes a recombination with the silicates in the dust."

The following is taken from an article on "The Recovery of Water-Soluble Potash as a By-product in the Cement Industry:"[3] "The insoluble potash represents the combinations occurring in the original silicates of the raw mix which is carried over mechanically in the dust before being subjected to a sufficiently high temperature to bring about decomposition. The form of combination which is slowly soluble in water is supposed to be due to a recombination of the volatilized potash with the silicates in the dust. In

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[1] This Journal, 9 (1917), 646.

[2] *Ibid.*, 9 (1917), 467.

[3] *Ibid.*, 9 (1917), 1035.

some plants where coal is used for burning, the extent to which the potash occurs in the "recombined" form may be considerable, while in certain other plants where oil is used for fuel this combination of the potash is present in comparatively small amount." Thus it is evident that the potash content of the coal ash has been quite neglected.

Analyses of the ash from the coal burned in the kiln, Table I, bring out the fact that the potash content of the ash is considerable and to such an extent that it must be taken seriously into consideration in figuring liberation and potash balances. The samples taken for analyses represent a week's composite of the daily averages of coal going into the kiln. Being compelled to buy coal upon the open market due to the coal situation, it was impossible to differentiate the sources relative to the mines furnishing the coal. Work along the line of determining the potash content of the ash of coal from different mines throughout the country is now in progress and the results obtained will be published in due time.

TABLE I—K_2O CONTENT OF KILN COAL ASH

SAMPLE	Per cent K_2O
Week ending Oct. 29, 1917	5.22
Week ending Nov. 5, 1917	4.54
Week ending Nov. 12, 1917	4.85
Week ending Nov. 17, 1917	4.75

The average potash content figures close to 5 per cent. As the coal consumption at the different cement plants throughout the country varies greatly, ranging from 80 lbs. coal per barrel to 250 lbs., it is apparent that the potash entering with the coal is considerable. Assuming the ash content to average 10 per cent and using the above figures, it is evident that the potash introduced by the coal lies between 0.4 lb. and 1.25 lbs. per barrel of clinker. Due to the very finely divided state of these ash particles and the velocity of the gases in the kiln, but a very small percentage of the ash is deposited in the kilns, nearly all passing out with the gases. The per cent being deposited in the kiln is undoubtedly higher in mills using the wet process where the ash particles have a tendency to cling to the wet slurry. In order to observe this effect of the ash upon the nature of the materials in the kiln, samples of the material were taken at intervals of 10 ft. throughout the length of the kiln. The effect upon the K_2O content of the materials is shown graphically in Fig. I.

Curve I takes into consideration the actual K_2O as determined by analyses assuming the original material (slurry) to be previously calcined.

Curve II shows the actual K_2O in these different samples.

Curve III attempts to show the K_2O content of the samples as it would be on the basis of K_2O content of slurry entering the kiln and taking into consideration the actual loss attained at the different intervals as shown by chemical analyses, disregarding any additions of K_2O from other sources.

Curve IV shows the K_2O content at the different intervals calculated on the assumption that the material had been previously calcined and no volatilization of K_2O in the kiln or addition of K_2O from other sources.

Comparing Curves II and III it is clearly shown that the K_2O content of the slurry is effected appreciably from some other source. This source is either the K_2O in the ash from the coal or the K_2O as fume in the gases. Due to the very physical nature of the K_2O from these two causes it is safe to assume that the K_2O in the ash is the principal contaminator.

The appreciable rise in the K_2O content of the slurry at 115 ft. as shown in Curve II is probably only a local condition due to the peculiar construction in our kilns at this point. I-beams about 15 ft. in length are attached to the interior wall of the kiln. These are spaced at intervals of 3 ft. and lie parallel to the axis of the kiln. The kiln in rotating lifts the slurry at this point to approximately the top of the kiln where it drops back through the gases to the floor of the kiln. This very readily acts as a filter causing considerable of the fine dust particles to deposit with the wet slurry.

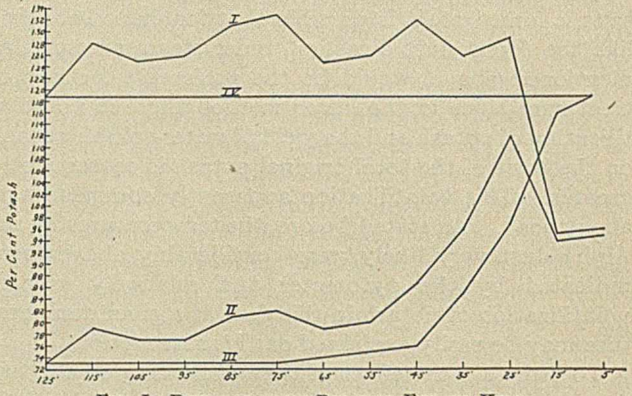

FIG. I—DISTANCE FROM BURNING END OF KILN

I—Actual K_2O, supposing no loss on ignition.
II—Actual K_2O in samples.
III—Theoretical K_2O, supposing no contamination from ash on volatilization of K_2O.
IV—Theoretical K_2O figured to no loss on ignition basis, non-volatilization of K_2O and no adulteration from ash.

TABLE II—CHEMICAL ANALYSES

Distance from Front of Kiln Ft.	SiO_2	Fe_2O_3	Al_2O_3	CaO	MgO	SO_3	S	Loss on Ignition
5	22.68	3.89	6.29	61.75	3.51	1.16	0.05	2.25
15	22.41	3.67	5.95	60.54	3.54	1.14	0.06	4.90
25	20.34	3.29	6.23	56.09	3.23	1.41	0.07	12.05
35	19.74	3.59	6.99	53.97	3.21	1.04	0.17	19.11
45	16.22	3.22	3.96	42.70	2.65	1.05	0.04	32.10
55	14.82	2.62	4.14	40.17	2.66	0.76	0.32	37.15
65	14.12	2.39	3.27	39.47	2.75	0.54	0.52	37.40
75	14.00	2.39	4.15	38.66	2.63	0.19	0.58	38.36
85	14.34	2.39	3.91	38.87	2.40	0.16	0.10	38.77
95	13.92	2.32	4.06	38.76	2.50	0.29	0.22	38.80
105	14.02	2.32	3.36	39.27	2.70	0.21	0.30	39.05
115	14.38	2.02	4.12	38.56	2.55	0.31	0.43	39.01
125	13.82	2.24	4.26	38.97	2.66	0.21	0.39	38.96
Ash	41.63	8.77	33.09	15.23	1.67	....	....	...

The ash deposit with the slurry is further demonstrated by chemical analyses.

Table II shows the chemical analyses of the different samples as taken throughout the kiln and also the ash analyses of the coal used at that time.

As the velocity of the kiln gases is quite high these ash particles will be exposed to the intense heat of the firing zone of the kiln but a fraction of a minute, possibly not more than a few seconds. Further, the very nature of combustion being exothermic precludes the possibility of a very high temperature being attained by the ash particles. Such being the case there should be practically no volatilization of the potash in the ashes from the coal in the kilns.

TABLE III—SOLUBILITY TESTS ON COAL ASH FOR K_2O
Sample Heated to Glow over Meker Burner for 1 Hour
Total K_2O in Ash, 4.47 Per cent

HOW TREATED	Sol. K_2O Per cent H_2O in Sample
Washed on filter paper	0.10
1/2 hour boil	0.16
1 hour boil	0.23
2 hours boil	0.23
4 hours boil	0.23
7 hours boil	0.25
10 hours boil	0.28
15 hours boil	0.28
24 hours boil	0.25

Samples of kiln coal ash containing 4.47 per cent K_2O were heated to a glow for approximately one hour over the Meker burner. This showed no volatilization. Two gram samples of this ash were then boiled vigorously for different periods up to 24 hrs. to determine whether this could be made water-soluble upon boiling. Table III shows that after 1 hour's boiling there is no increase in water-soluble K_2O. This water-soluble is so small as to be negligible. While this does not reproduce the kiln condition exactly it is quite evident that at the temperature attained by the ash in passing through the kiln and for the period to which it is subjected to this temperature, no potash of the ash will be volatilized.

Assuming that 90 per cent of this ash in a dry process plant and 75 per cent in a wet plant passes up the flue with the gases the effect of the insoluble K_2O in the ashes will affect the nature of the treater dust very appreciably.

Due to observations made while assisting in the research work at the Security Cement & Lime Company and to data showing potash balances at different plants at hand, but which we are not at liberty to publish at this time, it is quite evident that the insoluble potash content of the dust collected from the kiln gases is of two sources, that which passes over with the raw material, or partially calcined dust, and that which passes over with the ash from the coal used in burning.

CONCLUSIONS

I—K_2O content of coal ash is considerable.

II—K_2O content of coal ash must not be disregarded in calculating the liberation in kilns.

III—K_2O content of coal ash appears in "treater dust" as insoluble K_2O.

IV—Taking into consideration the K_2O content of ash and the K_2O in raw mix carried over mechanically there is apparently no "recombination" of the volatilized K_2O with the siliceous ash particles.

MICHIGAN PORTLAND CEMENT COMPANY
CHELSEA, MICHIGAN

TOLUOL RECOVERY AND STANDARDS FOR GAS QUALITY[1]

By R. S. McBRIDE

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The removal of benzol and toluol from gas necessarily reduces the heating value and candle-power of the gas; the amount of reduction depends upon the quantity of these constituents originally in the gas, the thoroughness of washing, and the general character of the gas with respect to other heating and lighting constituents. In this paper it is intended, *first*, to present a summary of the more important conditions of operation which determine the probable effect of toluol recovery upon the quality of the gas supplied; *second*, to illustrate the method of estimating the probable effect in any particular case; and *third*, to summarize certain general recommendations as to changes in standards that must be made in order that recovery of toluol can be carried out effectively in a large number of localities. In this memorandum, changes of standards are considered only from the standpoint of toluol recovery. No consideration is given to any other factors which might properly in many cases make desirable a change of standards. Such matters would depend upon a number of factors, not within the scope of the present discussions.

SUMMARY OF PRESENT STANDARDS OF GAS QUALITY AND GAS COMPANY OPERATING CONDITIONS

Both the heating value and the candle-power of gas are used in this country as a measure of the quality of the product supplied. Usually only one of these two characteristics is prescribed by ordinance or administrative ruling, but in some cases both are fixed. In cases where such standards have not been adopted and the quality of the gas supplied is determined by the local gas company, it is of interest to know what quality of gas is being supplied. This information is presented below; for convenience of consideration the companies are classified according to the standard in force. Data are included for all American gas companies making 500,000,000 cu. ft. or more of coal, water, or oil gas per year and for such other companies as have been recommended to the Ordnance Department for consideration by the Sub-Committee on Coal-Tar By-Products.

I—Gas companies in the following cities are expected to supply gas in compliance with the candle-power requirement as follows:

(*a*) *Requirements of 20 candles or higher:*

New York City (including the New York Consolidated System, the Brooklyn Union Gas Company, and the King's County Lighting Company), 22 c.-p. (Permission has recently been given to change to a heating value of 650 B. t. u. at the same price or to any lower heating-value standard if a proportionate reduction in price of gas is made.)

Philadelphia, Pa., 22 c.-p., fixed by a franchise contract with the city.

Des Moines, Iowa, 22 c.-p.

Sioux City, Iowa, 21 c.-p.

Omaha, Neb., 23 c.-p. measured at the gas works, or 21.2 c.-p. measured at the city testing station, and 600 B. t. u. heating value.

Charleston, S. C., 20 c.-p. and 600 B. t. u.

East St. Louis, Ill., 20 c.-p. (an old city ordinance requirement) and 565 B. t. u.

Northern Illinois cities supplied by Public Service Company of Northern Illinois, 22 c.-p. (ordinance) and 565 B. t. u. (state standard).

(*b*) *Requirements of 18 candles:*

Detroit, Mich., 18 c.-p. and 600 B. t. u.

Lansing, Mich., 18 c.-p. and 600 B. t. u. *net* heating value.

Los Angeles, Cal., 18 c.-p. and 600 B. t. u. (most of the gas

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[1] Published with permission of the Director, Bureau of Standards.

supply of this city is natural gas which is not limited by these requirements).

(*c*) *Requirements of 16 candles:*

All cities of Massachusetts, 16 c.-p. (fixed by State statute but subject to some waiver for purposes of investigation by the State Board of Gas and Electric Light Commissioners. This Board has very recently recommended to the State legislature a standard of 528 B. t. u.). The following Massachusetts cities are of interest in this connection: Boston, Brockton, Cambridge, Fall River, Haverhill, Lawrence, Lowell, Lynn, Malden, New Bedford, Pittsfield, Springfield, Worcester.

Nashville, Tenn., 16 c.-p. and 600 B. t. u.
Jackson, Mich., 16 c.-p. (20 c.-p. for water gas) and 600 B. t. u.
Grand Rapids, Mich., 16 c.-p. and 600 B. t. u.
Peoria, Ill., 16 c.-p. and 565 B. t. u.

(*d*) *Requirements of candle-power less than 16:*

Minneapolis, Minn., 15 c.-p. and 600 B. t. u.
Birmingham, Ala., 15 c.-p. and 575 B. t. u.
St. Paul, Minn., 14 c.-p. and 600 B. t. u.

II—Gas companies in the following localities supply gas in compliance with heating-value requirements as follows:

(*a*) *Total heating value, 600 B. t. u.:*

St. Louis, Mo. (This is a municipal requirement; the State requirement is 570 B. t. u.)

Baltimore, Md.
Indianapolis, Ind.
Hammond, Ind.
Peru, Ind.
South Bend, Ind.
Cedar Rapids, Iowa.
Washington, D. C.
Wilmington, Del.
Seattle, Wash.
Tacoma, Wash.
Milwaukee, Wis.
Madison, Wis.
Atlantic City, N. J.
Elizabeth, N. J.
Jersey City, N. J.
Newark, N. J.
Paterson, N. J.
Trenton, N. J.

(*b*) *Total heating values, 585 and below:*

Denver, Colo., 575
Bridgeport, Conn., 575
Hartford, Conn., 575
New Haven, Conn., 575
Waterbury, Conn., 575
Portland, Ore., 570
Ardmore, Pa., 570
San Francisco, Cal., 550
San Diego, 550
Allentown, Pa., 570
Chester, Pa., 570
Reading, Pa., 570
Wilkes-Barre, Pa., 570
Manchester, N. H., 565
Chicago, Ill., 565
Oakland, Cal., 550

Cities of New York State, 585. (Of these cities the following are of interest in this connection: Albany, Binghamton, Buffalo, Poughkeepsie, Rochester, Schenectady, Syracuse, Troy, Utica.)

III—The gas companies in the following cities have no requirements limiting the candle-power or heating value of the gas which they supply but are reported to be supplying gas of candle-power and heating value as given below:

New Orleans, La., 22 c.-p. and 600 B. t. u.
Jacksonville, Fla., 20 c.-p. and 580 B. t. u.
Atlanta, Ga., 19 c.-p. and 600 B. t. u.
Richmond, Va., 18 c.-p. and 590 B. t. u.
Pawtucket, R. I., 17 c.-p. and 600 B. t. u.
Providence, R. I., 17 c.-p. and 600 B. t. u.
Salt Lake City, Utah, 17 c.-p. and 600 B. t. u.
Houston, Tex., 17 c.-p. and 585 B. t. u.
Mobile, Ala., 15 c.-p. and 600 B. t. u.
Portland, Me., 15 c.-p. and 570 B. t. u.
Savannah, Ga., 575 B. t. u.
Battle Creek, Mich., quality not reported.
San Antonio, Tex., quality not reported.

METHOD OF ESTIMATING INFLUENCE OF TOLUOL RECOVERY UPON GAS QUALITY

As previously stated the quantity of toluol or benzol in the gas initially is a large factor in determining the quality of the gas both before and after removal of the toluol, since the conditions which make for the presence of large quantities of these aromatic hydrocarbons are the conditions prevailing during the production of high-candle-power and high-heating-value gases. In general the quantity of toluol and other light oils present in water gas depends upon the amount of gas oil used in the production of this gas. Approximately 10 per cent of the volume of gas oil used can be recovered as crude light oil and of this amount from $^{1}/_{5}$ to $^{1}/_{6}$ can be recovered as pure toluol. Coal gas made by any of the usual horizontal retort processes, which are the only processes of coal-gas manufacture requiring particular consideration in this report, usually contains about one-fourth to one-third of a gallon of light oil per 1000 cu. ft., depending upon the character and treatment of the coal and the quality of the gas. From one-eighth to one-tenth of this light oil is recoverable as pure toluol.

For each one-tenth gallon of light oil removed per 1000 cu. ft. of gas the total heating value is reduced by approximately 10 to 14 B. t. u. per cu. ft. and the candle-power by $2^{1}/_{2}$ to 3 candles. However, restoring part of the light oil removed, for example, enriching with the benzol fraction, may in some measure compensate for the loss in heating value and candle-power brought about by the initial washing. In fact, if a sufficient amount of additional benzol is available the candle-power and heating value can be restored substantially to the original values. (The increase in quality is about the same per unit of volume of benzol returned as was the loss on removal of the light oil.) However, this practice would not generally be practicable since it demands the purchase of benzol or other enriching constituents to take the place of those constituents which are permanently removed from the gas. In estimating the loss of candle-power and heating value, the figures here presented are probably slightly higher than would correspond to the change in quality of gas at the customers since in distributing unwashed gas there is usually considerable loss due to condensation.

From these two generalizations and a knowledge of the initial candle-power and heating value of the gas it is readily possible to estimate approximately the influence upon the quality of the gas of recovering different amounts of toluol or of toluol and benzol. Such estimates are, of course, not exact, but they furnish an excellent guide for readjustment of standards in any case where this is necessary or for approximating the quantity of materials which can be obtained by washing the gas. The following examples will make clear the application of the data:

(1) Assume water gas made from 4 gallons of oil per 1000 cu. ft., having an open-flame candle-power of 20 and a heating value of 625 B. t. u. About 0.4 gallon of light oils per 1000 cu. ft. could be recovered from such gas with practically complete wash-

ing. If none of the benzol was returned to the gas the result would be a gas of about 575 B. t. u. and 10 candle-power. If the light oil were distilled and the benzol fraction were returned to the gas the loss in heating value and candle-power would be, perhaps, one-half as great, and the result a gas of about 600 B. t. u. and 15 candles. In order to restore the heating value and candle-power to substantially their original figures it would be necessary to add benzol to the extent of approximately 0.2 gallon per 1000 cu. ft. of gas manufactured. From the 0.4 gallon of light oil originally obtained, some 0.07 to 0.08 gallon of toluol would probably be obtainable on refining.

(2) Assume carburetted water gas with 3 gallons of gas oil per 1000 cu. ft. and assume a very high oil-efficiency so that the candle-power was 18 and the heating value 570 B. t. u. From this gas about 0.3 gallon of light oil, equivalent to, perhaps, 0.05 to 0.06 gallon toluol, would be obtained per 1000 cu. ft. with commercially complete washing. The result of this washing would be a gas approximately 530 B. t. u. and 9 to 10 candles, which would be restored to 550 B. t. u. and about 12 candles if re-enriched with the benzol portion of the light oil.

(3) A mixture of coal gas and water gas in about equal proportion may be assumed made from water gas for which $3^1/_2$ gallons of gas oil were used to give 18 candles and 600 B. t. u. and coal gas of 580 B. t. u. and 15 candles. Such mixed gas would yield, perhaps, 0.3 gallon light oil per 1000 cu. ft. and the average candle-power would be reduced by washing from $16^1/_2$ to 8 or 9 candles and the heating value from 590 B. t. u. to about 550 B. t. u. Restoration of the benzol fraction would give a product of about 12 candles and 570 B. t. u.

(4) A coal gas made from ordinary grade of gas coal to yield 10,000 cu. ft. of gas per ton is assumed to produce gas of about 14 candles in the open flame and of 585 B. t. u. From this approximately 0.3 gallon of light oil per 1000 cu. ft. of gas could be recovered; and from it 0.025 to 0.03 gallon of toluol per 1000 cu. ft. would be available. The gas after washing would have approximately 8 candles and 550 B. t. u. which would be increased to, perhaps, 12 candles and 570 B. t. u. if the benzol fraction were restored.

In any of the above cases the net loss in heating value and candle-power might readily be reduced if some of the other constituents of the light oils, such as the solvent naphtha fraction, were also restored to the gas; or the loss in heating value and candle-power could be made less by operating the washing equipment in such a way as to accomplish only a partial removal of the light oils. In the latter case if removal of only 75 per cent of the quantity of light oil readily obtainable were considered satisfactory this would make the losses in heating value and candle-power of only about $^3/_4$ as great as above indicated, but, of course it would also somewhat reduce the yield of toluol.

It is probable that complete washing of the gas with restoration of the benzol in most cases will be considered advisable since the need for toluol is considerable and very little sacrifice of toluol yield can be allowed. But the demand for benzol is not so great and the restoration of the benzol to the gas might give results at first more satisfactory to the gas users than would the sale of this benzol with the slight reduction in total costs for the gas which might possibly be accomplished thereby. Especially might the restoration of benzol be necessary where a high candle-power standard has been in force, since otherwise the loss in candle-power would be rather greater than would be desirable at one time. In any computation, therefore, it is probably best to assume, unless other basis is known to be correct, that commercially complete washing of the gas would be necessary and that the benzol fraction of the light oil amounting to approximately one-half the total volume of light oil removed, will be restored to the gas.

RECOMMENDATIONS REGARDING STANDARDS FOR GAS QUALITY

From the estimates in the preceding section it is evident that much greater difficulty is met in complying with a candle-power requirement after removal of toluol or light oil than is encountered if a heating-value standard is to be complied with. Because of this fact it seems desirable that in any case where toluol is to be removed the candle-power standard be altogether eliminated or be made sufficiently low so that it will not interfere seriously with the proposed operations. Many other factors independent of toluol recovery make evident the desirability of eliminating candle-power requirements and substituting heating-value requirements as the primary basis of gas measurement. Therefore, the war is only an added influence tending to hasten an end otherwise desirable.

In all cases where the candle-power has previously been below 18 it would seem that the elimination of the candle-power requirement altogether would be reasonable; although in any event it is expected that the company would supply a gas of at least 8 or 10 candles, which would be sufficient to care for the need of those customers who must use some portion of the gas for open-flame lighting. In cases where 18 or 20 candles or higher have been maintained regularly in the past, it might be undesirable to have the candle-power go below 12 to 14, unless open-flame lights were generally eliminated and a readjustment of the appliances of all customers were made wherever the change in quality might make this necessary. For all companies which have been complying with requirements of 18 candle-power or higher, an understanding might be reached as to the maintenance of at least 12 to 14 candles for such a period as might be necessary to accomplish a general adjustment to the new conditions.

When coal gas is supplied either alone or mixed with very small percentages of water gas it is impracticable to make a very rich gas since the character of the coals available in most instances would preclude economic operation if a higher standard, either of heating value or of candle-power, must be maintained. For cities where only coal gas is supplied the standard could scarcely be higher than about 570 B. t. u. if practically complete toluol recovery is expected. Higher heating-value standards than this would probably have to be modified for such gas supplied.

If water gas is manufactured either alone or as a major constituent of the supply it is entirely practicable to make a gas of reasonably high heating value and candle-power initially and have after removal of the toluol a heating value of 585 to 600 B. t. u. In each case it would be a question as to which procedure was the more economical; that is, whether it would be better to make the same quality of gas as had previously been supplied and supply the customer with a somewhat lower product than formerly after the toluol had been removed from the gas, or to make the gas initially somewhat richer than before by the use of slightly more gas oil per 1000 cu. ft. so that the product after washing would have substantially the same heating value as had previously been supplied. If the quality previously supplied was rather high, approaching the maximum of the range of quality permissible for efficient operation, then any increase in the initial quality would obviously be undesirable; but otherwise an initial increase in quality with subsequent washing down to the original might be the best practice. Since the quantity of toluol available is largely dependent upon the initial richness of the gas which has been washed there is considerable advantage from the standpoint of the Government in having the richest practicable gas made initially; but, of course, in any case the limits of economical operation must be clearly recognized, and conservation of oil might also be an important factor.

As a summary of these points the following suggestions are offered as desirable adjustments to facilitate the recovery of toluol:

I—Eliminate all candle-power requirements now in force except for the cities where 18 candles or higher has been supplied, in which localities reach an understanding that at least 12 candles will be maintained for a period, say a year, during which time readjustments of appliances and substitution of mantle lamps would be accomplished to such an extent as to justify complete elimination of candle-power regulations.

II—For plants making coal gas (or practically only coal gas) let the heating-value standard be from 550 to 570 B. t. u.

III—For plants making water gas, either alone or as a major constituent, let the heating-value standard be 570 to 600 B. t. u. monthly average total heating value, the adjustment being made between these limits according to the economic conditions of operation.

In order to show the number of companies that would be affected by these several recommendations the following tabulation of the companies above listed is prepared. This tabulation does not take account of any unusual local conditions which might affect some of the cases materially. The kind of gas manufactured is also indicated: W, water gas; C, coal gas; O, oil gas; M, mixed coal and water gas; B, by-product coke oven gas; and N, natural gas.

1—Localities in which no change of standard will probably be needed and no serious change in the quality of gas supplied will probably result:

Chester, Pa.	W+B	Schenectady, N. Y.	M
Reading, Pa.	W	Troy, N. Y.	W
Wilkes-Barre, Pa.	W	Utica, N. Y.	W
Ardmore, Pa.	W	Poughkeepsie, N. Y.	W
Allentown, Pa.	W	Syracuse, N. Y.	M
Portland, Ore.	O	Binghamton, N. Y.	W
Manchester, N. H.	M	Rochester, N. Y.	M
Hartford, Conn.	M	Chicago, Ill.	W+B+N
Bridgeport, Conn.	W	San Diego, Cal.	W+O
New Haven, Conn.	M	San Francisco, Cal.	O
Waterbury, Conn.	W	Oakland, Cal.	O
Denver, Colo.	M	Houston, Texas	W
San Antonio, Texas	W	Pawtucket, R. I.	M
Savannah, Ga.	W	Providence, R. I.	M
Jacksonville, Fla.	M	Battle Creek, Mich.	M
Richmond, Va.	M	Portland, Me.	M
Atlanta, Ga.	M	Salt Lake City, Utah	M
New Orleans, La.	W	Mobile, Ala.	M
Albany, N. Y.	W		

2—Localities in which a candle-power standard may have to be abandoned, but with no serious change in the heating value of the gas supplied:

Lynn, Mass.	M	Fall River, Mass.	M
Boston, Mass.	M	Haverhill, Mass.	W
Brockton, Mass.	M	Springfield, Mass.	M
Lawrence, Mass.	M	Malden, Mass.	M
Lowell, Mass.	M	Birmingham, Ala.	M
New Bedford, Mass.	M	Waterloo, Iowa.	W
Pittsfield, Mass.	M	Peoria, Ill.	M
Worcester, Mass.	M	Nashville, Tenn.	M
East St. Louis, Ill.	W+N	Cambridge, Mass.	M

3—Localities in which slight change in heating-value regulations may, perhaps, be required, but in no case probably more than equivalent to 5 per cent of the present value. (The six cities marked (*) have candle-power standards which should be eliminated also.)

Indianapolis, Ind.	B+W	Buffalo, N. Y.	M
Tacoma, Wash.	C+O	Milwaukee, Wis.	M
Seattle, Wash.	B+M	Madison, Wis.	W
Trenton, N. J.	B+M	Cedar Rapids, Iowa.	M
Paterson, N. J.	W	St. Louis, Mo.	M+B
Newark, N. J.	M	Baltimore, Md.	W+B
Jersey City, N. J.	W	Hammond, Ind.	M
Elizabeth, N. J.	W	South Bend, Ind.	M
Atlantic City, N. J.	W	Peru, Ind.	M
Washington, D. C.	M	Wilmington, Del.	W
*St. Paul, Minn.	W	*Grand Rapids, Mich.	M
*Minneapolis, Minn.	M	*Jackson, Mich.	M
*Detroit, Mich.	M+B	*Los Angeles, Cal.	O+N

4—Localities in which high candle-power regulations should be changed or eliminated in order to permit operation on a heating-value basis; the reduction in heating value of the gas delivered would probably be a considerable percentage of the present value. (In the case of Lansing and Omaha a lower heating value than now in force would also be necessary.)

New York City	M	Charleston, S. C.	W
Omaha, Neb.	W	Des Moines, Iowa	W
Lansing, Mich.	M	Sioux City, Iowa	W
Philadelphia, Pa.	M		

Northern Illinois cities supplied by Public Service Company of Northern Illinois, M.

Bureau of Standards
Washington, D. C.

CATALYSTS IN VULCANIZATION[1]

By D. Spence

When the Chairman of this Section wrote me asking if I would read a paper on "Vulcanizing Catalysts," to me one of the most fascinating subjects of rubber chemistry, it was with regret I advised him that I would be unable to do so on account of lack of time to prepare a paper. In the meantime, however, I have found an article prepared by me in 1911, but never published, which, together with a few notes prepared on my trip to Boston yesterday, with your permission I will read, as they should clear up some of the points raised in this discussion, and at the same time serve to show the extent to which the investigation of this subject had been carried by some of us prior to 1911, and our knowledge of this subject in America at that date.

This subject is one which is of particular interest to me, as it has been, I may say, my main field of investigation for the past eight to ten years. During that period many interesting discoveries have been made, more than one hundred widely different vulcanizing catalysts have been prepared, investigated and put on record, and results of scientific as well as of technical importance have been obtained.

By some of our good friends and allies across the water, jealous of their claims to discoveries along this line, I have been recently criticized, for not placing on public record by patent or otherwise, the results of these past years of investigation, but I am confident that by the time they have carried their studies a little further, they will appreciate the difficulties involved in the problem, and my reasons for comparative silence. These remarks will, I hope, serve to answer some of their criticism and, at the same time, outline the history of this subject as far as I am concerned.

In the first place, I would point out that, as a matter of fact, discreet mention will be found in some of my earliest published work on vulcanization, of this basic principle, the importance of which our European friends, as well as enemies, have to-day begun to realize. Thus in an article dealing with the theory of vulcanization[2] and in particular with Axelrod's theory of a varying velocity of reaction with the degree of polymerization of the rubber, I took occasion to point out that "*what Weber, Axelrod, Ostwald and all investigators had overlooked was the fact that the vulcanization of rubber with sulfur, as we know it, is essentially a catalytic reaction.*" Even earlier, however, and in the controversy between Ostwald and myself in 1910–11, over the chemical *vs.* adsorption theories of vulcanization, which some of you probably remember, it was the knowledge of the true nature of the reaction, overlooked by Ostwald, which led me to take the stand I did. In the *Kolloid Zeitschrift*, Vol. 13, pp. 270, 271, will be found further reference to this subject, together with figures and curves showing the influence of varying amounts of a powerful catalyst on the rate of vulcanization, as determined by the amount of combined sulfur. That this basic discovery was not made the subject of a patent as Peachey has suggested,[1] may appear remarkable at first thought, but its very breadth rendered patent protection well-nigh impossible and disadvantageous to seek. The number of substances which can be used in this connection with more or less success is legion, so that patents such as those which have been granted recently, can be readily overcome, and are of no intrinsic value in any case. By the methods of organic chemistry, all manner of substances can be, and have been prepared, which will be found to accelerate vulcanization, and to bring about the physical result to a greater or lesser extent; simple organic substances widely different in constitution, such as aniline oil, piperidine, diazobenzene, etc., metallo-organic compounds such as alkyl derivatives of lead, and the salts of the fatty acids or of aniline oil, with the alkali metals; then there is the great group of substances which by decomposition, natural or induced during vulcanization, produce some very active and efficient catalysts to which class the thioureas belong. Each of these various classes contains several dozen different representatives, which I have already investigated, and there are doubtless hundreds still to be tested, so that I am sure you will agree with me that the task of adequately protecting an invention of the scope of this one is hard, to say the least, and of doubtful value if attempted. It has certainly not been accomplished so far. In 1911, in an effort to cover by patent the results of past years of research in this line, I set down a description of some of the discoveries made by that time, together with four claims, from which, with your permission, I will read the following extracts. The article is entitled "Improvements in and Relating to the Vulcanization of India Rubber" and is dated August, 1911. I was surprised, on referring to it again the other day, to find how fully it deals with the subject.

Since the publication of the classical researches of C. O. Weber into the nature of the vulcanization process, this process has been generally regarded as a simple addition of sulfur or sulfur chloride to the rubber hydrocarbon and the practical vulcanization of the rubber of commerce by sulfur has been handled from the standpoint of a *simple chemical reaction*, as generally understood, between the rubber and the sulfur at the vulcanization temperature. In view of certain difficulties experienced from the earliest of times even to the present day in accepting the simple chemical theory of vulcanization[2] it is therefore the more surprising that what we believe to be the key to successful vulcanization has not been discovered or made known as far as published work shows. The conception of a simple chemical reaction between the rubber and the sulfur at the vulcanization temperature has long been felt to fail to account for certain practical results obtained in vulcanization and only as recently as last year the physico-chemical or adsorption theory of vulcanization was put forward by an eminent authority on the chemistry of rubber on the ground alone that it helped to explain the practical facts of vulcanization better than the simple chemical theory of Weber.

I have cited these facts in order to show that from the earliest days of the industry to the present time what we believe to be the keynote to the vulcanization process as determined by our investigation has never once been sounded * * * * *.

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[1] Read before the Rubber Section, at the 55th Meeting of the American Chemical Society, Boston, September 12, 1917.

[2] *Kolloid-Z.*, **11** (1912), 275.

[1] *J. Soc. Chem. Ind.*, **36** (1917), 321.

[2] Ostwald, *Kolloid-Z.*, **6** (1910), 136.

For the better understanding of the facts leading up to and bearing on the present invention, it is necessary to refer to the following discoveries made by us:

That the vulcanization of pure rubber (by which is meant the rubber of commerce divested of those impurities usually associated with it in greater or lesser amount) by sulfur is an exceedingly slow reaction. The amount of sulfur which enters into combination with the rubber in a given time is relatively small, and even the best of the products obtained have very poor physical properties, usually deteriorate rapidly and are of little or no industrial value. It has been universally reported that this inferiority of so purified rubber is due to the quality of the rubber *per se* or to changes in the rubber *per se* brought about by the purification process. That this is not correct we have established by experiments in which, by returning to the purified rubber, certain of the so-called impurities removed in the first case, and investigated by us, or by adding to the rubber certain substances having an analogous action in vulcanization, the velocity of vulcanization of the original rubber and the superior physical properties of the vulcanized product were restored.

For example, it is known that by dissolving Para rubber in solvents and precipitating the rubber from its solution by acetone or by simple extraction of the rubber by acetone, the vulcanizing capacity of the rubber is greatly reduced, and products with very poor physical properties are obtained. We have discovered, however, that by returning to the rubber certain of the so-called impurities removed in purification, the original vulcanizing characteristics of this rubber are restored.

We have discovered in fact that:

I—The superior qualities of certain raw rubbers with respect to vulcanization are not necessarily due to any superiority in the quality of the rubber itself, but are determined far more by the existence in these rubbers of commerce of certain substances, hitherto regarded as impurities, which are not present in like degree or at all in the other rubbers. These so-called impurities are soluble in acetone and can be extracted in this way. When they are added to rubbers with inferior vulcanizing properties, products which vulcanize rapidly and with superior physical properties are obtained.

II—The acetone extract from Para rubber contains, in addition to the resinous impurities generally held to constitute the acetone extract, *organic substances*, nitrogenous and feebly basic in character, having all the reactions and characteristics of an organic alkaloid.[1] We have found, furthermore, that it is these organic nitrogenous substances present in one form or another in Para rubber that impart to this rubber its characteristic vulcanizing properties and are capable of bringing about substantial improvements in vulcanization when added to other so-called low-grade rubbers. These substances which occur in Para and in some other rubbers to a less extent we shall call henceforth the "active principle" of raw India rubber. To what extent this active principle occurs in Para we have not yet been able to determine, but from calculations made there is probably less than 1/10 of 1 per cent.

III—The function of this active principle in effecting more rapid and better vulcanization of rubber by sulfur is complex but essentially that of a catalyst (as generally understood) accelerating as it does enormously the rate of reaction between the rubber and the sulfur. This has been established by us beyond doubt by comparative curves of the combined sulfur of, purified rubber, vulcanized with sulfur only, (*a*) in absence of, (*b*) in presence of the active principle of India rubber, (*c*) in presence of substances having analogous properties.

IV—Organic substances having analogous reactions and properties can be used as catalysts to replace the "active principle" of Para rubber in the vulcanization of India rubber by sulfur. Thus by adding 1 per cent of the well-known alkaloid quinine to a mixture consisting of 100 parts purified rubber with 8 parts of sulfur, the reaction between the rubber and sulfur is so hastened that the time required using 40 lbs. steam pressure to effect proper vulcanization is reduced from 4 to 5 hrs. when no quinine is present to 25 min. when quinine is present. The physical properties of the product obtained are likewise improved enormously by the use of this substance as catalyst.

V—The number of organic substances which can be used to replace the "active principle" existing in higher-grade rubbers is unlimited; derivatives of these and metallo-organic bodies may be likewise employed and although the substances which may be employed may differ widely in chemical composition, they all have the common property of behaving as catalysts in vulcanization, hastening the velocity of reaction by carrying over the sulfur to the rubber at the temperature of vulcanization. We have found, furthermore, that the power which these substances possess as catalysts in vulcanization varies with their constitution and with the nature and arrangement of their reactive groups. We have discovered that it is possible, by suitably modifying the structure of the substance or its reactive groups by the well-known methods of organic chemistry or by suitably modifying the amount of the substance or substances used in vulcanization, to obtain any desired effect. Thus whereas we have found para-phenetidine to be a very powerful catalyst in vulcanization, ortho-phenetidine is but feebly so and the somewhat weak catalytic action of para-amido-phenol is wonderfully increased by converting it into the corresponding amido-phenetol. Similarly diphenylthiourea is but feebly active compared with the corresponding tetramethyldiaminodiphenylthiourea.

As examples of organic substances having the power to act directly as catalysts in the vulcanization of India rubber by sulfur, we may mention aniline, para-phenetidine, piperidine, and quinine.

As examples of derivatives of organic substances with inorganic radicles and metallo-organic substances acting catalytically in the vulcanization of India rubber by sulfur, we may mention the alkyl derivatives of lead and mercury and the salts of oleic acid with sodium or lead.

As the third class of catalyst in vulcanization we have discovered, furthermore, certain substances which are inactive in themselves, but which at the temperature of vulcanization, it may be alone or it may be in the presence of other substances, break down into a substance or substances having the properties of powerful catalysts. As an example of a substance of this kind, we may mention sulfocarbanilide or diphenylthiourea.

What we claim to have discovered is the basic principle of vulcanization of India rubber by sulfur; that the vulcanization of India rubber by sulfur is not a simple chemical reaction as is to-day assumed, but owes its whole success industrially to its catalytic nature.

We are aware of the well-known action of litharge in the vulcanization of India rubber by sulfur. This inorganic substance has been used from the earliest times in large quantities in rubber compounds (up to 40 per cent) and is known to hasten vulcanization. Further, we are aware of the use of magnesium oxide as an accelerator in vulcanization and of the work of Weber and of Henriques in this connection. Henriques, as the result of investigations into the *inorganic constituents* (the ash) of different rubbers, came to the conclusion that it is the presence of compounds of the alkaline earths (*inorganic elements*, in other words) in quick-curing rubbers, that accounts for the ease with which these rubbers vulcanize. This statement as far as the literature shows is still generally accepted as correct, and gave rise,

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[1] At the time this was written these nitrogenous bodies had not been definitely identified; only their behavior towards the well-known tests for alkaloids had been noted.

> we believe, to the use of magnesium oxide as an accelerator in the vulcanization of rubber by sulfur. We have come to an entirely different conclusion, however, and, although we do not deny the specific results to be obtained by the use of both litharge and magnesium oxide in the vulcanization of India rubber by sulfur, we lay no claim here to the use of any of these inorganic ingredients in vulcanization. We claim, furthermore, that the results obtained by the use of any of these inorganic accelerators in the vulcanization of rubber by sulfur are substantially different from those obtained by the methods of our invention; both these inorganic substances have to be used in large amount to produce any marked result and neither of these inorganic accelerators gives the physical properties to the vulcanized rubber possessed by the products of our invention, which is best illustrated by comparative results and by the fact that the addition of the products of our invention to compounds already containing these inorganic substances causes a still very pronounced improvement in the results of vulcanization.

I will omit the claims at this present time. The article also gives six examples which I have omitted, and defines in full the conditions under which the process may be carried out, and the pros and cons of various classes of reagents. Thus it points out, among other things, that some agents are solids, difficult to handle; others yield colored products on vulcanization (nitrosodimethylaniline, for example); others on account of their poisonous character or odor would not be suitable for practical purposes; nor will the methods of application constitute a novelty in connection with discoveries along this line as the description sets forth in detail.

If our friends across the Atlantic should still doubt our claims to priority of discovery of this fundamental principle of vulcanization let them test the validity of their patents. They will find that the records which I have just cited are a mere indication of the volumes of evidence and facts which can be established to dispose of any claim on their part to novelty of invention as far as America is concerned. As far as the specific claim to the use of *p*-nitrosodimethylaniline is concerned, let me assure Mr. Peachey that when he is ready, I am prepared to demonstrate through several different sources that this material was tried out both scientifically and industrially in this country so long ago as 1910, and was long since abandoned on account of the serious disadvantages to its extensive use. I recall indeed having used it in one instance in the preparation of rubber stoppers for laboratory use, but with unfortunate results. It is a substance which I should not think of employing industrially now, as there are so many objections to its use.

In conclusion, a good reagent must intensify not merely the chemical process of vulcanization, but also the physical; it should toughen the rubber, whether raw or vulcanized; and should render it immune to deterioration. All this has been achieved in America, giving a rubber superior to that from any natural source. A "noble" rubber, similar to the "noble" alloys, is an accomplished fact.

Norwalk Tire and Rubber Co., Inc.
Norwalk, Connecticut

VULCANIZATION OF RUBBER BY SELENIUM

By Charles R. Boggs

Received October 31, 1917

Vulcanized rubber has been manufactured for some years, but there has really been no essential change from the general methods of vulcanization as originally specified by the inventors. The two original methods, which are still in use, are the vulcanization with sulfur by heat and that by sulfur chloride in the cold. Variations in the processes have been introduced and innumerable mixtures made with other materials, but no rubber article of practical importance has been put on the market which has essentially deviated from the two original processes.

There have been made, however, many slight variations in compounding rubber mixtures which have produced vulcanized rubber products that are characterized by properties which fit them to special work better than any previously known compounds. It was with this idea that we thought rubber vulcanized with selenium might give a product of especial adaptability to some of the many uses of rubber. At the time that we first tried to vulcanize rubber with selenium, 1913, we thought that we were the first, although it is evident that anyone with a knowledge of chemistry would expect selenium to act similarly to sulfur. We have since noticed that Pearson in his book, "Crude Rubber and Compounding Ingredients," mentions two methods, one by heating rubber with equal parts of selenium and the other by dropping liquid selenium into a CS_2 solution of rubber at 300° F. under pressure. It is evident that these methods had nothing to recommend them and I believe could never have been developed because of the unsatisfactory product.

Selenium is a metal in the same group of the periodic table as oxygen, sulfur and tellurium, is much more metallic than sulfur and has a higher melting point (217° C.), sufficiently high to discourage one from attempting to use it as a vulcanizer for rubber which is not capable of withstanding such a temperature. Its atomic weight is 79.2. It occurs in two crystalline and one amorphous form and forms a complex molecule when cold, Se_8 being very similar to sulfur. A short table of its properties follows:

	CRYSTAL	Specific Gravity	Solubility	Melting Point
Black or gray crystalline..........	Octahedral Hexagonal	4.80	Insoluble in CS_2	217°
Red crystalline......	Monoclinic	4.46–4.51	Soluble in CS_2	175°
Red amorphous.....	......	4.26–4.28	1 soluble (and 1 insoluble) in CS_2	Softens at 102°

All modifications go over to the black crystalline form when heated at 100 to 150° C. The black crystalline powder can be obtained on the market in small quantities, but it should be procurable in fair amounts if there were a commercial demand for it. Black selenium has the further peculiar property of being an electric conductor under the influence of light although the other forms are insulators. It might, therefore, cause rubber which has been vulcanized with it to show some slightly unusual electrical characteristics.

Our first attempts were with selenium in the form of black powder used in a standard 30 per cent Para compound in the equivalent proportion that sulfur would be used. By heating at about 150° C. for a couple of hours a partial vulcanization resulted. The physical tests showed a normal elongation, but a tensile strength of only about 50 per cent of the similar sulfur compound. The point to be noted is that partial vulcanization was obtained, although the temperature was well below the melting point of Se. Increase in the time did not improve the product. All of these first samples have aged well and after nearly four years give the same elongation and tensile-strength values of about 70 per cent of the original.

Doubling the amount of selenium and using an organic accelerator, which we were then using with sulfur, did not improve the product but did make the samples go to pieces with age, a normal characteristic of overvulcanized and under-vulcanized rubber. A peculiar point about this compound was that when it was removed from the press hot it expanded 25 per cent of its volume. Its volume became normal when cold. This high coefficient of expansion indicated lack of vulcanization.

Using amorphous selenium and an organic accelerator we were able to increase the tensile strength some without sacrificing the elongation. Some other accelerators were tried without much success. The amorphous selenium should go over the metallic form at the temperature used.

The most promising compound we then had was put on wire and has been tested regularly for the last three years and the remarkable point is that it has not deteriorated appreciably in that time. This compound was below normal in its tensile strength. The difficulty seemed to be that the long heating at the relatively high temperature used to effect vulcanization caused too great a depolymerization of the rubber.

By trial we have now found accelerators which enable us to satisfactorily vulcanize rubber with selenium when heated at the ordinary vulcanizing temperature of 275° F. (135° C.) for only about twice the time required with sulfur. The product gives the normal tensile strength (1100 to 1200 lbs.) and elongation (2 to 10 in. or 12 in.) of the same compound with sulfur. It is somewhat soft. This compound shows no deterioration under the short life test of 4 days' heating in air at 70° C.

Electrical tests have been carried out on wire insulated with this compound and the insulation resistance and dielectric strength are somewhat low. Special determinations as dielectric loss, etc., have not yet been determined.

Chemical analysis as applied to ordinary sulfur compounds does not apply to these compounds vulcanized with selenium, as the black selenium is practically insoluble in acetone and consequently the uncombined Se is not separated by extraction with acetone. Also it is only very slightly soluble in $CHCl_3$ and CS_2. The acetone extract contains only the resins from the rubber (provided oils, waxes, etc., are not added). The $CHCl_3$ extract contains some Se and a small amount of unvulcanized rubber as with soft vulcanized rubber when cured with sulfur. A determination of rubber by the tetrabromide method gave 31.7 per cent rubber plus resins, etc., in a compound to which 31.7 per cent rubber had been added. This would indicate that no correction should be made for combined Se, *i. e.*, that the selenium was either not chemically combined or more likely that it was so weakly combined that it was displaced by Br. It is possible that a complete chemical study of the vulcanization of rubber with selenium may throw some additional light on the theory of vulcanization. Also it may help in the study of the nature of the catalytic effect of accelerators as the vulcanization occurs so far below the melting point of the Se.

The product as we now have it has not yet shown any unusual electrical properties, but the indications are that its deterioration with age is much less than with sulfur compounds. It can be brominized and oxidized but the natural oxidation seems to have been slowed up. As the deterioration of rubber goods is the one disadvantage of rubber, especially in those lines of work where permanency is desired, it may be that the use of selenium may partially remove this disadvantage.

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THE PIGMENTS OF THE TOMB OF PERNEB[1]

By MAXIMILIAN TOCH

In 1913 Mr. Edward S. Harkness presented to the Metropolitan Museum of Art of New York City, the Tomb of Perneb, which originally stood in the cemetery of the ancient Memphis. Mr. Harkness acquired this tomb from the Egyptian Government and Dr. Albert M. Lythgoe removed the tomb from Sakkara and re-erected it in the main hall of the Metropolitan Museum of Art.

The tomb was built approximately 2650 B. C. and in it was buried an Egyptian Dignitary named Perneb, who held high office under the king at Memphis. The tomb contains many figures in relief, particularly the side wall in the main chamber, on which the carvings are very profuse. The figures are all colored with various pigments.[2]

The pigments used on the Tomb of Perneb are red, yellow, blue, green, gray and black. There is a popular belief that the red used by the Egyptians was red ochre. This is an error, as ochre is yellow naturally, and only turns red when it is burned or calcined. Ochres all normally contain between 10 and 20 per cent of oxide of iron, whereas the reds of the Egyptians contain more than 50 per cent of oxide of iron, and from their very color it is certain that the red of the ancients was hematite. This is never a very bright

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[1] Paper presented at the meeting of the New York Section of the Society of Chemical Industry, October 19, 1917.

[2] It is a great pleasure for me to acknowledge the assistance that I have received from Dr. Albert M. Lythgoe, through whose courtesy I was given the pieces of limestone and the adherent pigments which were taken from the Tomb of Perneb.

red, but is always one which we associate with brick color.

YELLOW—All the yellows used were the native ochre, which is clay stained with iron rust.

BLUE—The Egyptian blues are very beautiful and range from a light sky-blue to a dark ultramarine. An examination with a microscope of the dark Egyptian blue shows it to be powdered glass or porcelain. This material has been known as "frith" and has of itself no hiding or obscuring power, nor does it seem to have been put on with a binder. This powdered glass has been rubbed into the surface and allowed to set with the Nile clay or the Nile mud, which, on account of its slightly alkaline nature, is cementitious of itself and has both setting and binding power. The appearance of this blue glass, which in modern times is called "smalt," appears blue just the same as snowflakes appear white, because the light is broken up on its crystalline structure, yet a single snowflake is as transparent as pure glass.

GREENISH BLUE is azurite, a hydrated carbonate of copper.

GREEN is malachite, azurite and clay.

BLACK is carbon black composed of charred wood or burnt wood or charred bones.

GRAY is limestone mixed with charcoal or carbon.

Mr. Lythgoe found two paint pots which had evidently been thrown out by the workmen and an examination of these shows the pigment to be hematite mixed with limestone and clay.

It is remarkable that in all investigations of historic materials, many of the tools and implements used have been either forgotten or in many cases not found. I have, in times gone by, paid a great deal of attention to the pigments used by the old masters, beginning with the primitive painters of Italy and going through the history of the Flemish materials down to the English masters, and while I have had abundant matter I have had hardly any historic data concerning the implements used, such as brushes and palette knives. There are practically no brushes left to show us how the wonderful technique of the older painters has been carried out. The same may be said of the musical instruments. The bows used by the early violinists of Italy are not well known and their history, method of manufacture and composition are very largely shrouded in mystery.

There is a brush in the Metropolitan Museum of Art, from one of the excavations of the Palace of Amenhotep III at Thebes, that dates from the XVIIIth dynasty. This brush is similar in size and shape to a medium-sized sash tool used by the house painters of the present day. The bristles are not hair, but evidently of a vegetable stalk similar to bamboo, which has been beaten until the longitudinal fibers have separated. It is bound around with a twine of fiber a good deal like hemp, just the same as a modern round paint brush is bound either with wire or cord.

There has been little or no work done on Egyptian pigments, with the exception of the chapter by Dr. Russell, called "Egyptian Colors," in the book "Medum" by Flinders Petrie, compiled in 1892. Dr. Russell was in error, however, when he stated that the splendid rich blue was a silicate of copper, for the samples that were submitted to me proved to be cobalt, and upon investigation I found that A. W. Hoffman demonstrated that the blue frits of the time of Rameses III were painted with cobalt.

Nearly everyone has made the popular error of assuming that the Egyptians used the white of egg as a binder for their pigments. I cannot find any trace of any albuminous binder in the pigments submitted to me, but they do show some evidence of the use of glue or gelatine. It is well known that the Egyptians manufactured very excellent grades of glue either by boiling parchment or bones and hides of animals. They were excellent cabinet makers and used glue very largely in joining pieces of wood. In the great museum at Cairo there are to-day many samples of furniture glued together with Egyptian glue, which are still in excellent condition. It is, however, more than likely that little or no binder was used when the pigments were applied on the various tombs, even to those built about 1500 years later, like the Temple of Karnak. We all know that the climate of Egypt is exceedingly dry and therefore no rain can wash off or disintegrate a cold water paint made by means of pigment and glue. The Nile clay and Nile mud largely used in building are slightly alkaline and in many respects similar to the adobe mud in New Mexico and Arizona. This mud contains a small percentage of free lime, and any earthy substance which contains free lime will in time act like a weak cement and become firmly bound. It is therefore my opinion that many of the decorations made by the Egyptians were made without any binder other than the lime naturally found in the soil, and in a few cases glue was used. I also judge, from the nature of the implements used, that the pigments were rubbed into the surface and they in time became part of the surface.

I do not refer to the splendid decorative work in the wooden sarcophagi when I say little or no binder was used, for in these coffins and on the outside of the linen wrappings there are some really wonderful decorative paintings in which binders were used. The portraits outside of the mummy wrappings in the second century were done with wax and resins and are excellent works of art, although these paintings have nothing to do with the pigments of the Tomb of Perneb.

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THE PREPARATION OF *N*/100 PERMANGANATE SOLUTIONS

By J. O. HALVERSON AND OLAF BERGEIM

Received August 17, 1917

The preparation, standardization and conditions of use in volumetric analysis of *N* and *N*/10 solutions of potassium permanganate have been studied with some care.[1] Unfortunately, however, certain of the precautions which have thus been shown to be neces-

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[1] See Gooch's "Methods in Chemical Analysis," 1st Ed., New York.

sary for accurate work have been commonly neglected in the literature, even in handbooks of general and applied analytical chemistry. As a result of this and of the widening application of permanganate titration methods, especially in biochemistry, certain methods based on this principle have been recently suggested which possess unnecessary inaccuracies. Common errors are the use of too much sulfuric acid in titrations and a lack of appreciation of the great sensitivity of permanganate solutions to traces of organic matter. In the determination of very small amounts of substance, as, for example, in the estimation of calcium as oxalate in small portions of biological fluids[1] where permanganate solutions approximately $N/100$ must be used, the elimination of these sources of error is a necessity.

Where a standard permanganate weaker than $N/10$ was desired it has been customary to make this shortly before using by dilution of a stronger permanganate solution,[2] in spite of the fact that ordinary distilled water contains appreciable amounts of organic matter which can be removed only with great difficulty. For instance, we have found that water redistilled from both acid and alkaline permanganate may still cause an appreciable deterioration (as much as 2 or 3 per cent) when used in diluting permanganate from $N/10$ to $N/100$. This was true even where the weak solution was used at once. If allowed to stand for any length of time, or if less carefully distilled water was used, the permanganate was almost entirely decomposed, this process being hastened by the catalytic action of the oxides of manganese which were formed.

To avoid the inconvenience as well as inaccuracy of dilution we have endeavored to prepare permanent $N/100$ solutions. The principle used is not new, but the technique as we have adapted it and some data pertaining to the keeping qualities of dilute permanganates and of oxalic acid solutions used as standards may be of interest.

PREPARATION OF $N/100$ POTASSIUM PERMANGANATE

Dissolve 0.40 g. pure potassium permanganate crystals in one liter of redistilled water in a thoroughly clean Florence flask which has been rinsed with the same water. Digest at or near the boiling point for 36 hrs. A funnel covered with a watch-glass may be used as a reflux condenser. Cool and allow to stand over night. Without disturbing the sediment of manganese oxides, filter with gentle suction through a 3-in. Büchner funnel lined with ignited asbestos. Both funnel and filter flask should be rinsed with redistilled water. Transfer the permanganate solution to a glass-stoppered bottle free from traces of organic matter. The solution should be kept in the dark when not in use. If the asbestos becomes clogged with oxides these may be dissolved out with hot concentrated hydrochloric acid, followed by washing with redistilled water without disturbance of the pad.

After standing two or three days this permanganate solution may be conveniently standardized against $N/50$ oxalic acid (0.1261 g. pure crystals to 100 cc.) or sodium oxalate of similar strength. To 10 cc. of the oxalic acid solution add 10 cc. of 10 per cent sulfuric acid which has been treated with just sufficient permanganate solution to give it a faint pink color. Place in a water bath at 65° C. for a few minutes. Then titrate at once to a definite pink color which persists for at least a minute. Correct for the blank obtained by titrating 10 cc. of the sulfuric acid and the same volume of water to the same end-point.

If kept in a dark place, the oxalic acid solution used in standardization does not lose appreciably in strength in from ten days to two weeks. Ordinarily the permanganate solutions after they have stood several days will not vary over 0.1 per cent per week (see Table I). On account of the sensitivity of the reagent it is, nevertheless, desirable to check it up rather frequently. This also serves as a control on technique.

TABLE I—PERMANENCY OF PERMANGANATE SOLUTIONS, ETC.

No.	SOLUTION	Age of Solution	Cc. Permanganate Required for Definite Volume Oxalic Acid
1.	$N/100$ Potassium Permanganate....	10 days	20.14
		25 days	20.20
		148 days	20.62
		185 days	20.65
		386 days	21.05
2.	$N/75$ Potassium Permanganate.....	2 days	11.88
		5 days	12.04
		15 days	12.15
2.	Separate Bottle..................	2 days	11.81
		223 days	11.99
3.	$N/50$ Potassium Permanganate.....	1 day	8.14
		6 days	8.17
		19 days	8.25
		47 days	8.31
		68 days	8.45
		125 days	8.50
		180 days	8.55
4.	$N/80$ Potassium Permanganate (by direct dilution of $N/10$)..........	Theoretical	13.70
		15 minutes	14.20
5.	$N/100$ Potassium Permanganate (by direct dilution of $N/10$)..........	Theoretical	17.50
		15 minutes	17.83
6.	Same as Sol. 5..................	Theoretical	17.50
		15 minutes	17.81
7.	Same as Sol. 5..................	Theoretical	17.43
		15 minutes	17.69
		1 day	17.77
		2 days	18.18
8.	Oxalic acid 0.1101 g./100 cc. (0.0175 normal)........................	1 hour	20.62
		18 days	20.58
		22 days	20.47
		72 days	18.06
9.	Oxalic acid, 0.1101 g./100 cc.......	1 hour	20.54
		11 days	20.62
		14 days	20.62
10.	Oxalic acid, 0.1101 g./100 cc.......	1 hour	8.34
		6 days	8.32
		10 days	8.30
		14 days	8.23
		19 days	8.27

The solutions on which the above data were obtained were kept away from the light except while in use. Solutions 1 and 3 were used almost every day. They were kept at room temperature throughout one summer and in the case of Solution 1 two summers. In the dilution tests redistilled water was used.

CONCLUSIONS

The preparation of weak permanganate solutions by direct dilution is inaccurate and inconvenient. By means of the procedure outlined in this paper $N/100$ potassium permanganate solutions may be prepared which will retain their strength and usefulness for an indefinite period.

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[1] Halverson and Bergeim, *J. Biol. Chem.*, **24** (1916), 22; **29** (1917), 337; Halverson, Mohler and Bergeim, *J. Am. Med. Assn.*, **68** (1917), 1309.

[2] See Michaelis, *Biochem. Z.*, **59** (1914), 166, and Ellinger, *Z. physiol. Chem.*, **38** (1903), 192, for example.

THE USE OF MICROÖRGANISMS TO DETERMINE THE PRESERVATIVE VALUE OF DIFFERENT BRANDS OF SPICES[1]

By Freda M. Bachmann

Received July 25, 1917

In a former paper[2] the sensitiveness to spices of several microörganisms was discussed. In my work at that time I used only one brand of spice. Further studies with spices from different sources gave results widely divergent and indicate that the sensitiveness of one or more microörganisms to a spice may be a criterion of its preservative value.

ORGANISMS

The organisms used for this study were various species of bacteria, yeasts and molds. The bacteria were *Bacillus subtilis*, *Bacillus coli*, *Bacillus prodigiosus*, and *Sarcina lutea*. Five yeasts were used, one of which was isolated from the commercial yeast foam, another from Fleischmann's compressed yeast. The other three were old laboratory cultures of *Saccharomyces cerevisiae*, *Saccharomyces ellipsoideus*, and *Saccharomyces anomalus*. Professor E. M. Gilbert has very kindly examined cultures of the molds and finds them to be *Aspergillus niger* van Tieghem, *Penicillium glaucum* Link, *Rhizopus nigricans* Ehrenb., and an *Alternaria* which is probably *Alternaria tenuis* Nees.

METHOD

The methods used for determining the preservative value of various brands of spices were the same as those used in my former studies.[3] The medium was sterile, nutrient agar containing a definite amount of spice. The bacteria and yeasts were grown in test-tubes, the bacteria on beef broth agar and the yeasts on wort agar. For the molds a shallow watch-glass of 1$^1/_2$ in. diameter was placed inside a Petri dish and then both were sterilized in the oven. Thaxter's potato hard agar, consisting of potato broth with 3 per cent agar and 2 per cent glucose, when sterile was poured into the Petri dish and the same kind of medium containing a definite amount of spice was poured into the watch-glass. When the agar in each was thoroughly hardened it was inoculated with a suspension of mold spores in water by means of a platinum loop. This double plate shows the growth on the spiced agar and also the effect of the volatilized substances on growth of the organism on non-spiced agar. Unless the amount of spice used is so great that the growth of the organism on both the spiced and non-spiced agar is completely inhibited, the agar without spice outside the watch-glass serves as a control plate to prove the viability of the spores used for inoculation. This double plate method is not recommended as ideal because there is some slight variation in results which may be due to the way in which the cover of the Petri dish fits the lower part. If the edge of the lower part of the dish is uneven, more of the volatile substance will escape and permit a better growth of the organism. Unless the plates are kept in a damp chamber, this unevenness may also allow considerable evaporation and the resulting drying of the agar may inhibit growth. It is desirable that the Petri dishes to be used should be carefully examined to have the covers fit well. With this precaution observed, the amount of variation in the sensitiveness toward spice of the organisms used for this study has been considered negligible.

In the study of several brands of spice the molds were grown in the double plates described above and also on spiced agar in test-tubes. The following tabulated results of mold growth are all from Petri dish cultures. It is seldom that the minimum dilution of a spice which permits growth is the same in a Petri dish and in a test-tube. The minimum dilution is usually somewhat less in a Petri dish. This is doubtless to be accounted for in the more shallow layer and the greater surface area of spiced agar in the Petri dish which results in a more rapid loss of volatile substances.

For the results given in this paper only the ground spices were used. Spices of different brands were obtained from various sources.

In the following table are given the results obtained after inoculating potato agar containing various amounts of cloves with suspension of mold spores in water. The different brands of cloves are given as A, B, C, D, and E. The results are from cultures on agar in Petri dishes as described above. In my former paper attention was called to the difference in sensitiveness to spice of the mycelial filaments and the spores of molds. This phenomenon has been frequently observed in the Petri dish cultures. There is often no growth on the spiced agar until the mold has grown abundantly on the agar without spice. When the filaments reach the sides of the watch-glass they grow upward and over the edge of the watch-glass and on the spiced agar. In the following table such a growth of the mycelium is indicated by the letter *m*. It may be observed that such mycelial growth is frequently given for *Rhizopus*, only once for *Alternaria*, and not at all for *Aspergillus* and *Penicillium*. This is not to be interpreted as an equal sensitiveness of the mycelium and spores of *Alternaria*, *Aspergillus*, and *Penicillium*. It is explained by the fact that in *Rhizopus* the mycelium produces a more vertical growth and thus is much above the surface. These filaments readily grow over the sides of the watch-glass embedded in the agar. In *Alternaria*, *Aspergillus*, and *Penicillium* the growth is more horizontal and close to the substratum. The same growth of the mycelium of *Alternaria*, *Penicillium*, and *Aspergillus* from non-spiced to spiced agar has been frequently observed in such plates as were described in my former paper[1] where the non-spiced and spiced agar is in contact.

It will be observed from Table I that cloves A and B are much more effective in inhibiting growth than C, D, and E. The species of *Rhizopus* which I have used appears to be more sensitive to cloves than any

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[1] Published by permission of Director of the Wisconsin Agricultural Experiment Station.

[2] Bachmann, "The Inhibiting Action of Certain Spices on the Growth of Microörganisms," This Journal, 8 (1916), 619.

[3] *Loc. cit.*

[1] *Loc. cit.*

TABLE I—EFFECT OF DIFFERENT BRANDS OF CLOVES ON MOLD GROWTH

MOLD	Dilution of spice	Brand A	Brand B	Brand C	Brand D	Brand E
Rhizopus nigricans	1 : 25	0	0	0	*m*	*m*
	50	0	0	*m*	+	+
	100	0	0	*m*	+	+
	200	*m*	*m*	+	+	+
	400	..	+	+	+	+
Alternaria tenuis	1 : 25	0	0	0	0	*m*
	50	0	0	0	+	+
	100	0	0	+	+	+
	200	+	+	+	+	+
	300	+	+	+	+	+
Penicillium glaucum	1 : 25	0	0	0	0	+
	50	0	0	+	+	+
	100	0	+	+	+	+
	200	+	+	+	+	+
	400	+	+	+	+	+
Aspergillus niger	1 : 25	0	0	+	+	+
	50	0	0	+	+	+
	100	0	+	+	+	+
	200	+	+	+	+	+
	400	+	+	+	+	+

of the other organisms and the *Alternaria* is more sensitive than the species of *Penicillium* and *Aspergillus*. This is in agreement with my earlier studies in which I found that *Rhizopus* and *Alternaria* were more sensitive to eugenol than *Penicillium* and *Aspergillus*.

Brands A and B inhibit growth in the *Rhizopus* in dilutions up to 1:300 or greater in Petri dish cultures. With Brand B a dilution of 1:300 greatly retards growth but after a mycelium is produced it grows fairly well. When grown in test-tubes a dilution of 1:400 is probably near its maximum tolerance for this brand of cloves. With this dilution I failed to get growth several times but at another time succeeded. With Brands D and E the organism always grows well in a dilution of 1:50 in Petri dish cultures.

In Table II are given the results of inoculating wort agar containing cloves in dilutions from 1 : 25 to 1 : 400 with yeast. There is very little variation in the sensitiveness to cloves in the different strains of yeast which I have used. Brand A is again the most effective in inhibiting growth and Brand E least effective.

TABLE II—EFFECT OF DIFFERENT BRANDS OF CLOVES ON YEAST GROWTH

YEAST	Dilution of spice	Brand A	Brand B	Brand C	Brand E
Yeast Foam—culture from	1 : 25	0	0	0	0
	50	0	0	+	+
	100	0	0	0	+
	200	0	+	+	+
	400	+	+	+	+
Fleischmann's compressed—culture from	1 : 25	0	0	0	0
	50	0	0	0	+
	100	0	0	+	+
	200	0	+	+	+
	400	0	+	+	+
Saccharomyces cerevisiae	1 : 25	0	0	0	0
	50	0	0	0	+
	100	0	0	0	+
	200	0	+	+	+
	400	0	+	+	+
Saccharomyces ellipsoideus	1 : 25	0	0	0	0
	50	0	0	0	+
	100	0	0	+	+
	200	0	+	+	+
	400	0	+	+	+
Saccharomyces anomalus	1 : 25	0	0	0	0
	50	0	0	0	+
	100	0	0	0	+
	200	0	+	+	+
	400	0	+	+	+

In Table III are given the results of inoculating beef broth agar containing cloves in dilutions from 1 : 50 to 1 : 400 with four species of bacteria. The brands of spice vary in their preservative value in the same way as they were found to vary when molds and yeasts were used. Brand A has the greatest preservative value, next Brands B and C, and Brand E least of all. As with molds, there is likewise considerable variation in sensitiveness among bacteria to any one brand of spice. In my former studies I found *B. subtilis* to be very sensitive to cloves. This fact has been frequently observed in the present work. *B. prodigiosus*, on the other hand, has considerable resistance to cloves.

TABLE III—EFFECT OF DIFFERENT BRANDS OF CLOVES ON BACTERIAL GROWTH

ORGANISM	Dilution of spice	Brand A	Brand B	Brand C	Brand E
B. subtilis	1 : 50	0	0	0	0
	100	0	0	0	+
	200	0	0	+	+
	300	0	0	+	+
	400	0	+	+	+
B. coli	1 : 50	0	0	0	0
	100	0	0	0	+
	200	0	+	+	+
	300	0	+	+	+
	400	+	+	+	+
B. prodigiosus	1 : 50	0	0	0	+
	100	0	0	+	+
	200	0	+	+	+
	300	+	+	+	+
	400	+	+	+	+
Sarcina lutea	1 : 50	0	0	0	+
	100	0	0	0	+
	200	0	0	+	+
	300	0	+	+	+
	400	+	+	+	+

A similar study has been made with a few brands of cinnamon and of allspice. In Tables IV and V are given the results of this study.

TABLE IV—EFFECT OF DIFFERENT BRANDS OF CINNAMON ON THE GROWTH OF MOLDS AND BACTERIA

ORGANISM	Dilution of spice	Brand A	Brand B	Brand C
Alternaria tenuis(?)	1 : 25	0	0	0
	50	0	0	0
	100	0	0	0
Penicillium glaucum	1 : 25	0	0	0
	50	0	0	0
	100	0	0	0
Rhizopus nigricans	1 : 25	0	+	+
	50	+	+	+
	100	+	+	+
Aspergillus niger	1 : 25	0	0	0
	50	0	0	0
	100	0	+	+
B. subtilis	1 : 25	0	0	..
	50	0	0	..
	100	0	+	..
B. coli	1 : 25	0	+	..
	50	+	+	..
	100	+	+	..
B. prodigiosus	1 : 25	0	+	..
	50	+	+	..
	100	+	+	..
Sarcina lutea	1 : 25	0	0	..
	50	0	+	..
	100	+	+	..

It may be seen from Tables IV and V that growth of the above species of molds, bacteria and yeasts has varied little on media containing different brands of allspice and cinnamon. Although the brands of allspice and cinnamon which I have used are thus more nearly equal in preservative value than the different brands of cloves, yet it is quite evident that Brand A of both cinnamon and allspice has considerably more preservative value than Brand B or Brand C.

It is desirable that the test organism used in determining the comparative antiseptic values of different brands of spice be fairly sensitive to the spice so that high dilutions of a strong spice could be used. Here again, as in my former studies, I have found that *Rhizopus nigricans* is not very sensitive to cinnamon. *Aspergillus niger* is somewhat more sensitive to this spice than the *Rhizopus* and shows nicely the difference in the preservative value of the different brands that I have used. *Rhizopus* grows so readily

Table V—Effect of Different Brands of Allspice on the Growth of Molds and Yeasts

Organism	Dilution of spice	Brand A	Brand B	Brand C
Alternaria tenuis(?)	1 : 25	0	0	..
	50	0	0	..
	100	0	+	..
Penicillium glaucum	1 : 25	0	0	0
	50	0	0	+
	100	+	+	+
Rhizopus nigricans	1 : 25	0	0	0
	50	0	0	+
	100	0	+	+
Aspergillus niger	1 : 25	0	0	+
	50	0	+	+
	100	+	+	+
Yeast Foam—culture from	1 : 25	0	0	..
	50	0	+	..
	100	+	+	..
Fleischmann's compressed—culture from	1 : 25	0	0	..
	50	0	+	..
	100	+	+	..
Saccharomyces cerevisiae	1 : 25	0	0	..
	50	0	+	..
	100	+	+	..
Saccharomyces ellipsoideus	1 : 25	0	0	..
	50	0	+	..
	100	+	+	..
Saccharomyces anomalus	1 : 25	0	0	..
	50	0	+	..
	100	+	+	..

on media containing cinnamon that it shows no difference at all between Brands B and C in the dilutions used. *Penicillium*, on the other hand, is very sensitive and in dilutions up to 1 : 100 showed no difference in the brands used for the results tabulated above. It may be that some species of *Aspergillus* would, by its growth on media containing cinnamon, show the comparative preservative value of many brands. It is highly probable that other organisms may be found which will serve the purpose even better than these which I have used.

Some observations have been made on the variation in sensitiveness of two other strains of *Rhizopus*. For the results given in this paper I have used a culture of *Rhizopus nigricans* which I have designated No. 4 in my cultures. It produces a very vigorous growth on culture media and grows much more readily on media containing some kinds of spice than a plus strain of *Rhizopus nigricans*. With a dilution of 1 : 7500 of cinnamic aldehyde, I found when I used *Rhizopus nigricans* No. 4 that there was scarcely any retardation of growth. With a minus strain of *Rhizopus nigricans* growth was somewhat delayed, but later quite vigorous, while with the plus strain there was no evidence of germination of the spores even after incubating the culture for a week. Using a 1 : 600 dilution of three brands of cloves, I found the plus strain of *Rhizopus nigricans* somewhat less sensitive to this spice than the minus strain.

It is again evident from the data recorded above that there is considerable difference in sensitiveness of any one organism to the different spices and also that no one spice has an equally inhibiting effect on the growth of different organisms. This makes it difficult to determine the minimum amount of spice necessary to preserve any food product. It is necessary that further data on the effect of spices on other organisms be obtained. That, of the different spices, cinnamon is the most generally effective as a preservative, as I stated earlier,[1] does not seem to have been proved. Results of further study with different brands of spice indicate that cloves may be just as effective as cinnamon. The best grades of these spices certainly exert a very considerable preservative effect, and although the amount used in flavoring a food product may not be sufficient to preserve it from spoilage, yet it may be a large factor in its preservation.

CONCLUSIONS

Molds, yeasts and bacteria show a marked variation in sensitiveness to different brands of spices. The amount of growth of such organisms in a given time on media containing spice may be used as a means of determining the relative preservative values of the different brands of the spice.

SUMMARY

Microörganisms have been used to determine the preservative value of different brands of spices. Spices of molds, yeasts and bacteria were grown on nutrient agar containing varying amounts of spice. Tabulated results of such a study using five brands of cloves, three of cinnamon, and three of allspice are given. The results show that there is considerable variation in the preservative value of the brands used and that the growth of microörganisms on a spiced medium may be used as a criterion of the preservative value of the brand of the spice.

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DISINFECTION WITH FORMALDEHYDE
A SUBSTITUTE FOR THE PERMANGANATE-FORMALIN METHOD

By C. G. Storm

Received December 7, 1917

The method proposed by H. D. Evans and J. P. Russell in 1904[1] for the rapid liberation of formaldehyde gas from its water solution, the "formalin" of commerce, in a condition suitable for practical disinfection, has been found by numerous investigators to be superior to most of the other known methods of formaldehyde disinfection, as regards simplicity, rapidity, cost and efficiency. This method consists in pouring the formalin quickly upon crystals of potassium permanganate contained in any suitable metallic vessel (for example, a water bucket), the oxidation of a part of the formaldehyde furnishing sufficient heat to cause rapid evaporation of the remainder of the formaldehyde together with the water.

The permanganate method has found very general application and is widely used in this country in practical disinfection work. Its use has, however, received a serious set-back by reason of the present scarcity of potassium permanganate and the resulting excessive cost of this chemical. Prior to the war in Europe, potassium permanganate was obtainable in this country at prices ranging usually from 9 to 10 cents per lb. It is now obtainable only at many

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[1] *Loc. cit.*

[1] H. D. Evans and J. P. Russell, "Formaldehyde Disinfection," *13th Ann. Rept.*, State Board of Health of Maine, and *J. Am. Chem. Soc.*, **27** (1905), 714. See also Daniel Base, "Formaldehyde Disinfection," *J. Am. Chem. Soc.*, **28** (1906), 964–96.

times its former price, having held at approximately \$4.00 to \$4.25 per lb. for the past six months.

This fact has impressed the writer with the desirability of publishing a note regarding an analogous method for generating formaldehyde, devised by him in October, 1911, which it is believed has never been proposed for practical use, which preliminary tests indicate to be safer, as rapid, and almost as simple of operation, and which will be much less expensive, owing to the relatively low price of the material used.

The new method depends upon the action between the water solution of formaldehyde and a soluble chlorate, and is apparently analogous to the permanganate method, in that the oxidation of a part of the formaldehyde furnishes a sudden evolution of heat which serves to vaporize the remainder of the formaldehyde. It was suggested to the writer in the course of analysis of a potassium chlorate explosive. On adding formalin to the water solution of the explosive and heating the mixture, a violent evolution of gas resulted, increasing in intensity even after the tube containing the mixture was removed from the flame of the burner. The gas evolved was largely formaldehyde, apparently liberated from its solution by the heat generated in the oxidation of a part of the formaldehyde by the chlorate.

An examination of the solution remaining after the reaction had subsided, showed the presence of large amounts of chloride which had resulted from reduction of the chlorate. Repeated trials showed that the violent evolution of gas resulted only from concentrated solutions, but that the reduction of the chlorate to chloride, with a corresponding oxidation of formaldehyde, took place even in the case of very dilute solutions of chlorate. It has been demonstrated that under proper conditions this reaction is quantitative, and the results of the study of this quantitative method for determining chlorates will shortly be published.

The object of this paper is merely to call attention to what it is hoped will be a satisfactory substitute for the permanganate method of disinfection, and to offer an opportunity for a more complete study of the method, the writer's investigation having necessarily been quite incomplete because of lack of time and facilities for conducting work of this nature.

Potassium permanganate reacts immediately on coming in contact with formalin at ordinary temperatures, and if the permanganate is finely powdered instead of crystalline, the reaction may be violently explosive in character.[1] If formalin is poured on crystals of sodium or potassium chlorate, no action results until the mixture is warmed by application of external heat to about 65° C. This may be considered as a disadvantage, but as a matter of fact the reaction may be started with very little difficulty. The chlorate and formalin are placed together in a suitable metal container, such as a water bucket, of sufficient size to prevent the reaction mixture from foaming over, and the bucket, properly weighted so it will not float, placed in a large shallow pan (an ordinary dish pan will answer the purpose) containing water heated to about the boiling point.

The mixture becomes heated to the reaction temperature in a few minutes, when bubbles of gas begin to be evolved, this evolution increasing rapidly until it is so violent that the mixture may foam over the top of the bucket. The action is completed in 2 or 3 min., and with the proper proportion of chlorate and formalin the residue remaining in the bucket is practically dry and consists chiefly of chloride together with some unreduced chlorate.

Sodium chlorate seems to give just as satisfactory results as potassium chlorate, and has the distinct advantage of costing less than one-half as much as the latter. Potassium chlorate is now quoted at 50 to 55 cents per lb., while sodium chlorate is listed at 24 to 25 cents.

Several investigators have attempted to determine what proportion of formaldehyde used in the permanganate process is liberated as gas and what proportion is oxidized by the reaction. Frankforter and West[1] obtained an evolution of 62 per cent to 75 per cent of the formaldehyde from formalin by this process in a long series of experiments under laboratory conditions, using glass apparatus and absorbing the evolved gas in water, the strength of the resulting solution being determined. D. Base,[2] in experiments with the process on a practical scale, used a specially prepared room of 2,000 cu. ft. capacity, determining the amount of formaldehyde gas in the room by drawing 5 to 10 liter samples through standard KCN solution, adding excess of standard $AgNO_3$ solution and titrating the excess of the latter with sulfocyanate. Base found that not over 40 per cent of the total amount of formaldehyde used as formalin was evolved in the state of gas in the room.

It has been suggested that the reaction between formaldehyde and potassium permanganate is probably as follows:

$$4KMnO_4 + 3HCHO + H_2O = 4Mn(OH)_2 + 2K_2CO_3 + CO_2$$

It is, however, likely that other reactions proceed at the same time, in which part of the formaldehyde is oxidized to formic acid. Assuming this reaction, however, it is calculated that with the proportions recommended by Evans and Russell (100 cc. of 40 per cent formalin to 37.5 g. $KMnO_4$) 5.34 g. of formaldehyde, or about 13.35 per cent of the formalin, would be oxidized by the $KMnO_4$. With the proportions recommended by Base (100 cc. of formalin to 50 g. $KMnO_4$) 7.12 g. HCHO or 17.8 per cent of the formalin would be destroyed. These figures are considerably lower than those found in the investigations mentioned above. In any event it is apparent that in the permanganate process a considerable part of the formaldehyde used as formalin is destroyed by oxidation, the reaction supplying the heat which causes the rapid volatilization of the remainder.

It may be assumed that the behavior of the chlorate with the formalin is entirely analogous to that of the

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[1] G. B. Frankforter and R. M. West, *J. Am. Chem. Soc.*, **28** (1906), 1234.

[1] *J. Am. Chem. Soc.*, **28** (1906), 1234.

[2] *Ibid.*, **28** (1906), 964.

permanganate, and that the reaction occurs according to the equation

$$2KClO_3 + 3HCHO = 2KCl + 3H_2O + 3CO_2.$$

Experiments have shown that the proportion of 25 g. of chlorate to 100 cc. of formalin is approximately the one giving best results, that is, with these proportions there is practically no liquid left in the residue after the reaction subsides, the formaldehyde being either driven off as gas or oxidized and the water evaporated at the same time.

A simple calculation shows that, according to the above reaction, 25 g. $KClO_3$ will theoretically oxidize 9.18 g. HCHO or nearly 23 per cent of the formaldehyde in the 100 cc. of formalin, leaving the remaining 77 per cent to be volatilized. It is probable, however, that other reactions occur, such as

$$KClO_3 + 3HCHO = KCl + 3HCOOH.$$

In fact, appreciable amounts of formic acid, as well as CO_2, are evolved by the reaction of formalin with either permanganate or chlorate.

In an attempt to determine in a simple manner the best proportions of formalin and chlorate, a series of roughly quantitative experiments were made, using varying proportions of the two materials. A weighed amount of powdered $KClO_3$ was treated in a beaker with a weighed amount of the 40 per cent formalin and the beaker immersed in hot water in order to start the reaction. The residue left in the beaker after the reaction had ceased was dissolved in water and titrated with standard solution of silver nitrate to determine the amount of chloride present. From the amount of KCl found, the weight of formalin representing the formaldehyde destroyed in the reduction of $KClO_3$ to KCl was then calculated from the reaction

$$2KClO_3 + 3HCHO = 2KCl + 3CO_2 + 3H_2O.$$

The results of these tests are shown in the following table. It is to be noted that if the reaction

$$KClO_3 + 3HCHO = KCl + 3HCOOH$$

is assumed, the calculated amounts of formaldehyde consumed will be just twice those given in the table.

TESTS OF RESIDUE REMAINING AFTER REACTION BETWEEN FORMALIN AND POTASSIUM CHLORATE

TEST No.	$KClO_3$ Used Grams	Formalin Used Grams	Calc. $KClO_3$ Reduced Grams	HCHO oxidized (equiv. to KCl found) Grams	Formalin (40%) equiv. to HCHO oxidized Grams
1	7	12	3.917	1.439	3.59
2	6	12	3.817	1.402	3.50
3	6	12	3.620	1.329	3.33
4	5	12	3.750	1.375	3.44
5	4	12	3.726	1.369	3.42
6	3	12	2.990	1.098	2.75
7	2	12	2.010	0.738	1.85

In Tests 5 and 6 a very small amount of liquid remained in the residue after the reaction; in Test 7 an appreciable amount of liquid remained and a determination of formaldehyde showed 1.104 g. HCHO, equal to 2.76 g. of 40 per cent formalin. In Tests 1 to 5, inclusive, where the weight of $KClO_3$ was at least one-third of the weight of the formalin, the amount of HCHO oxidized was fairly constant, the KCl found indicating that only part of the $KClO_3$ had been reduced. In Tests 6 and 7 the excess of formalin was such that practically complete reduction of the chlorate was effected and the amount of formalin oxidized much less.

A number of qualitative tests were made using formalin and sodium chlorate in proportions varying from 6 : 1 to 2 : 1, the maximum temperatures reached during the reaction being noted. With the ratios 2 : 1, 2.5 : 1, and 3 : 1, this temperature was 108–109° C., while with lower proportions of chlorate (4 : 1 and 6 : 1) the temperature was slightly less, 104° to 105° C. In each case the reaction started at 60–65° C., was violent at about 75° C., and lasted only about 30 seconds, the maximum temperature being indicated near the end of the reaction.

The writer hopes that comparisons of the actual disinfecting efficiencies of the permanganate and chlorate methods will be made by those who may be interested in the practical side of the question and that the chlorate method may be found to be of some use.

ORDNANCE DEPARTMENT, U. S. R.
WASHINGTON, D. C.

EFFECT OF FERTILIZERS ON HYDROGEN-ION CONCENTRATION IN SOILS[1]

By F. W. MORSE

Received September 29, 1917

Most of the fertilizer plots at the Massachusetts Agricultural Experiment Station have been continuously treated for more than 25 years, and there are marked differences in their crop-producing powers, which in some instances appear to be due to chemical or physical changes in the soil and not to a deficit of the usual constituents of a fertilizer.

Among methods of investigating these soils, the measurement of the hydrogen-ion concentration in water extracts of the soils has given some interesting results.

The method of procedure has been as follows: 25 grams of air-dry soil were weighed into an Erlenmeyer flask of 300 cc. capacity, and 250 cc. distilled water were added. The flask was repeatedly shaken during a period of an hour, and then the mixture was filtered through a dry paper filter. The first portions of the filtrate were usually cloudy and were returned to the soil flask. When the paper became well coated with soil, the filtrate would, as a rule, be clear, with the exception of some limed samples which would persistently retain a slight turbidity from clay. The soil and water were in contact for about 3 hours before filtration was completed.

The colorimetric method was used for determining the hydrogen-ion concentration. The range for the soils was found to be covered by the indicators methyl red, paranitro phenol and rosolic acid. The standard salt mixtures used were Walpole's[2] acetic-acid-sodium-acetate mixture, Sörensen's[3] mono- and dibasic phosphates, and Clark and Lubs'[4] mixture of monopotassium phosphate and sodium hydroxide. The last named covers practically the same range as Sörensen's and is much more convenient to prepare.

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[1] Presented before the Fertilizer Division, at the 55th Meeting of the American Chemical Society, Boston, September 10 to 13, 1917.

[2] *Biochem. J.*, **1914**; *J. Chem. Soc.*, **1914**.

[3] *Ergebnisse Physiol.*, **1912**.

[4] *J. Biol. Chem.*, **25** (1916), 504.

Ten cubic centimeters of the soil solution were compared with an equal volume of the standard mixture appropriate for the concentration of hydrogen ions in the former. Small porcelain dishes served the purpose for comparisons in nearly all cases, but tubes were used when necessary to check doubtful results.

The different fertilizers that had been used on the plots under investigation were acid phosphate, nitrate of soda, muriate of potash, sulfate of potash, double sulfates of potash and magnesia, sulfate of ammonia, land plaster and agricultural lime.

The range of H-ion concentrations was between P_H —4.5 and P_H —7.0.

Neutral salts of strong bases and strong acids, sodium nitrate, potassium chloride, potassium sulfate, calcium sulfate, produced little, if any, effect on the soil reaction, in comparison with unfertilized soil. The acid phosphate, a strong base with a moderately weak acid, behaved like the neutral salts just mentioned. Sulfate of ammonia behaved like a weakly ionized acid and carbonate of lime like a weakly ionized base, and the extremes of the range were always due to these two compounds.

When agricultural lime was used in conjunction with the other chemicals, it was noted that plots dressed with nitrate of soda or calcium sulfate retained the neutralizing effect of the carbonate of lime longer than the plots receiving potash salts, probably through a protective effect on the solution of the lime as bicarbonate. I have not yet demonstrated that point, however.

The effect of an application of 2,000 lbs. of hydrated lime per acre is perceptible on the crop and on the soil reaction for several years, but ultimately disappears, probably due to both leaching and transformation, but apparently due more to the former.

The comparative results obtained during this season's investigation of our plots are as follows:

	North	South		Field A
Acid phosphate....	P_H 5.2	P_H 6.15	Nitrate of soda.......	P_H 6.0
Nitrate of soda.....	5.22	6.5	Sulfate of ammonia...	4.9
Muriate of potash..	5.25	6.15		
Calcium sulfate....	5.0	6.65	No nitrogen..........	5.4
Calcium carbonate.	6.4	7.1		
Unfertilized.......	5.25	5.96	Lime................	6.0

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THE SEEDS OF THE ECHINOCYSTIS OREGANA

By MILO REASON DAUGHTERS

Received October 26, 1917

This investigation was made to determine the possible industrial value of the seeds of the plant called *Echinocystis oregana*, but more commonly known as the Old-Man-in-the-Ground or Wild Cucumber. It is a perennial plant, a remarkable feature of which is its gigantic root which penetrates to a depth of 1 to 2 meters and may weigh 30 or more kilograms. Heaney,[1] Manz,[2] and Young[3] examined the root of the *Megarrhiza californica*, a plant belonging to the same order as the *Echinocystis*, and reported on its pharmaceutical value. Heaney found a bitter glucoside, which he called megarrhizin. The root of the *Echinocystis* is decidedly bitter and is therefore unfit for food. The Indians are said to use it as a drastic purge in dropsy.

The fruit, which is borne on slender herbaceous stems varying in length from 3 to 9 meters, is egg-shaped. It varies from 25 to 50 mm. in the short diameter and is covered with soft green spines, a fact which explains the origin of the name "echinos" or hedgehog. It becomes lighter in color as the seeds reach maturity, and breaks open at times at the free end, leaving the seeds more or less exposed. Each fruit contains from one to several seeds, which are orbicular in shape, averaging 19 mm. in breadth and half as thick as broad. Fourteen to fifteen hundred of the seeds make a kilogram. The thin outer shell of the seed is readily broken and hence it is easily ground in a food chopper.

The *Echinocystis* is distributed along the Pacific slope from British Columbia to California, growing and thriving along railroad tracks, fence rows, in fields, along wooded ravines, and in the foothills. It is drouth-resistant, maturing its seeds under unfavorable conditions. So far as known no attempt has been made to grow this plant in quantity. Bearing in mind the character of the root it is readily seen why most farmers consider it a pest.

Seeds collected for three successive years had the following percentage composition:[1]

TABLE I

SAMPLE NO.	I	II	III
Date of Collection.........	1915	1916	1917
Ether Extract (Fat)........	30.10	34.92	35.45
Protein (N × 6.25).........	23.71	20.64	21.54
Starch....................	9.21	12.05	10.31
Crude Fiber...............	22.07	21.55	20.01
Moisture..................	4.04	3.90	4.54
Ash.......................	2.89	2.64	2.60

Samples of oil were prepared by extraction with petroleum ether, boiling point 44 to 65° C., and by expression in the cold from the whole seed previously ground in a food chopper. The expressed oil was thoroughly agitated with fuller's earth from which it was separated by means of a centrifugal machine. The constants of the oils thus obtained were:

TABLE II

	EXTRACTED OIL	EXPRESSED OIL
Color......................	Golden yellow	Olive-green
Specific Gravity (at 25° C.)..	0.9267	0.9166
Refractive Index (at 25° C.)	1.4722	1.4701
Solidifying Temperature......	+5 to —8	+5 to —8
Iodine Number.............	116.5	117.0
Saponification Number.......	193.4	189.1

Judged by these results, the oil from the seeds of the *Echinocystis* belongs to the cottonseed oil group. The oil tastes like olive oil. Both the extracted and expressed oils become turbid when cooled to a temperature of 5° C., but at —8° C. the former has the consistency of vaseline, whereas the latter is more solid.

Nearly 40 per cent of the oil content was expressed with the apparatus employed for this purpose. The pressure applied was approximately 83 kg. per square centimeter or nearly 1200 lbs. to the square inch. Freshly ground seeds gave an oil of olive-green color, which faded in a few days in bright light to a golden

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[1] *Am. J. Pharm.*, **48** (1876), 451.

[2] *Ibid.*, **53** (1881), 384.

[3] *Ibid.*, **55** (1883), 195.

[1] All analyses were made in August, 1917.

yellow. Seeds which were ground for two weeks before pressing gave an oil of greenish red color in reflected light and dark olive-green in transmitted light.

When subjected to hydrogenation at 220 to 240° C., with powdered nickel—prepared from nickel oxide just previous to its addition to the expressed oil—there was produced a bland yellowish white fat with a melting point of 29 to 36° C., a solidifying temperature of 25° C., and an iodine number of 76.6.

Feeding experiments with mice attested the nonpoisonous character of both the original oil and the hydrogenated fat.

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VARIATION IN THE ETHER EXTRACT OF SILAGE[1]

By L. D. Haigh

Received May 26, 1917

Having occasion to repeat the analysis of some samples of silage 10 months after the first analysis was made, considerable variation in the samples was noted. The ether extract in particular showed great variation, much less being found in the old than in the fresh silage. Table I shows the comparative results of the analyses of silage when new and also 10 months later. The results show that some factors entered into the determination when the silage was fresh which did not appear in the silage 10 months later.

Table I—Analysis of Air-Dry Silage—New and 10 Months Later

		A—Results in Percentages on Air-Dry Basis							B—Results in Percentages on Dry Basis					
Silage No.	Date Analyzed	Moisture	Ether Extract	Crude Fiber	Ash	Nitrogen	Protein N×6.25	Nitrogen-Free Extract	Ether Extract	Crude Fiber	Ash	Nitrogen	Protein N × 6.25	Nitrogen-Free Extract
1.......	3/16	5.54	3.61	21.07	5.20	1.32	8.25	56.33	3.82	22.31	5.51	1.40	8.73	59.63
	2/17	6.02	2.55	19.43	5.25	1.12	7.00	59.75	2.71	20.68	5.59	1.19	7.45	63.58
2.......	3/16	5.52	3.76	19.40	4.87	1.12	7.00	59.45	3.98	20.53	5.15	1.19	7.45	62.92
	2/17	6.51	2.99	17.74	4.88	1.08	6.75	61.13	3.20	18.98	5.22	1.16	7.22	65.39
3.......	4/16	7.85	4.81	20.05	5.72	1.24	7.75	53.82	5.22	21.76	6.21	1.35	8.41	58.40
	2/17	5.67	2.71	19.53	5.74	1.20	7.50	58.85	2.87	20.70	6.09	1.27	7.95	62.39
4.......	5/16	7.28	5.10	22.68	6.42	1.27	7.94	50.58	5.50	24.46	6.92	1.37	8.56	54.56
	2/17	6.09	2.75	21.67	6.53	1.25	7.81	55.15	2.93	23.07	6.95	1.33	8.32	58.73

Inasmuch as the sample must be dried before the ether extraction the effect of vacuum and oven drying on the percentages of moisture obtained was first studied. The results are shown in Table IIA. The effect of vacuum and oven drying on the results for ether extract are shown in Table IIB.

Table II—Results on Air-Dry Basis by Different Methods of Drying

A—Percentage of Moisture

Drying Method Used	Silage 1	2	3	4
Vacuum............................	6.02	6.51	5.67	6.09
Vacuum + Oven (15 min.)............	6.30	6.90	6.18	6.64
Oven (100 to 105° C.)................	8.73	9.31	8.71	9.13

B—Percentages of Ether Extract

Drying Method Used	Silage 1	2	3	4
Vacuum, before and after Extraction...	2.55	2.99	2.71	2.75
Vacuum+Oven, before and after Extrac.	2.01	2.11	1.90	2.12
Vacuum before, vacuum + oven after Extraction.........................	2.87	3.27	2.94	3.06

It was thought that the acidity of silage might have some part in the variation of the results for moisture and ether extract. The acidity of silage is due principally to the presence of acetic acid, and lactic acid. The former is volatile in a vacuum, the latter is not. Table III illustrates the determination of the acidity of silage based on this fact.

Table III—Acidity of Air-Dry Silage

	Total Acidity *As Lactic Acid*		Acidity Volatile *in Vacuo* Figured as		Total Acidity Recalculated as *Acetic and Lactic Acids*
Silage No.	Original Air-Dry Silage	After Drying *in Vacuo*	*Lactic*	*Acetic*	
1..............	4.43	4.13	0.30	0.20	4.33
2..............	4.78	4.40	0.38	0.26	4.66
3..............	4.66	4.25	0.41	0.27	4.52
4..............	3.93	3.56	0.37	0.25	3.81

Comparison was made of the acidity of the samples before and after the determination of moisture and before and after the determination of the ether extract, in order to study the effect of the acidity upon these two determinations. The results are shown in Tables IV and V. The last column in Table V shows that water will wash from silage not only the acid but also other substances soluble in ether.

No attempt is made in this report to explain the causes for the above variation. Further studies are being made with a view of ascertaining what these causes are. We only wish to indicate at this time that variations do occur in value for ether extract depending upon changes in the sample itself on standing and upon the drying operations employed.

It is evident that the analyses should be made as soon as possible after the feed is used and that a uniform method of drying be employed, if these variations are to be avoided.

Table IV—Effect of Acidity of Air-Dry Silage upon Moisture Determination

	Acidity of Silage				% Mixture	
Silage No.	Original Air-Dry *Acetic & Lactic*	After Drying at 100° *Lactic*	Acidity Volatilized *Acetic and Lactic*	% Loss on Drying at 100° *Moisture and Some Acidity*	After Deducting Volatilized Acidity	By *Vacuo* *Corrected*
1.....	4.33	1.80	2.53	8.78	6.25	5.82
		1.89	2.44	8.68	6.24	
2.....	4.66	1.89	2.77	9.39	6.62	6.25
		1.97	2.69	9.24	6.55	
3.....	4.52	1.88	2.64	8.68	6.04	5.40
		1.75	2.77	8.75	5.98	
4.....	3.81	1.61	2.20	9.11	6.91	5.84
		1.56	2.25	9.15	6.90	

Table V—Effect of Acidity of Air-Dry Silage upon the Determination of Ether Extract

	Acidity as Lactic Acid				Ether Extract	
Silage No.	Of Air-Dry Silage after Drying *in Vacuo*	Of Residue after Extraction with Ether	Extracted by Ether	Total Ether-Soluble Material Found	Less Ether-Soluble Acidity	After Washing with Water
1.....	4.13	3.47	0.66	2.53	1.87	1.76
		3.45	0.68	2.56	1.88	
2......	4.40	3.69	0.71	2.90	2.19	1.82
		3.63	0.77	3.08	2.31	
3......	4.25	3.62	0.63	2.88	2.25	1.50
		3.62	0.63	2.55	1.92	
4......	3.56	3.04	0.52	2.70	2.18	1.30
		3.06	0.50	2.80	2.30	

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[1] Presented at the 54th Meeting of the American Chemical Society, Kansas City, April 10 to 14, 1917.

LABORATORY AND PLANT

SOME METHODS OF ANALYSIS FOR NEBRASKA POTASH SALTS AND BRINES

By A. H. McDowell

Received October 29, 1917

The so-called Nebraska potash salts consist of alkali carbonates, sulfates, and chlorides in varying proportions, with a small amount of SiO_2 (usually less than 0.3 per cent), a small amount of organic matter, and moisture from 0 per cent up.

These salts are produced by evaporating to dryness the brines (salines) obtained from wells driven into the beds of certain alkaline lakes in the Sand Hills section of western Nebraska. They are largely used in the manufacture of fertilizers and are of value on account of the potash occurring in them, the price per unit K_2O (per cent per ton of 2000 lbs.) being the basis on which they are sold.

The determinations most frequently called for are K_2O and moisture at 120° C. and the potash content of the commercial product is rarely less than 20 per cent or more than 30 per cent. Complete analyses are not needed in connection with sales, but furnish useful information for investigations and development work.

The analysis of brines is essential principally in connection with prospecting for new sources of supply. In this connection it is of interest to note that the composition of mineral solids is not necessarily constant in different parts of the same lake, or in fact at different depths in the same well, and that the percentage of solids may vary widely in samples from wells driven in different parts of the same lake. The usual analysis includes specific gravity, mineral solids, and the content of the solids in K_2O, Cl, and alkalinity as Na_2CO_3. In general, the alkalinity will be found between 45 per cent and 80 per cent and in most cases a relatively high alkalinity is accompanied by relatively low K_2O and *vice versa.*

The details of the following methods of analysis have been developed during several months' experience with these materials. The platinum chloride method for K_2O is given preference because it is simple, accurate, and official with the A. O. A. C., and because of the ease with which both platinum and 80 per cent alcohol may be recovered.

METHODS FOR COMPLETE ANALYSIS OF SALTS

MOISTURE—A 10 g. sample (30 mesh) is dried at 120° C. to constant weight. Two hours are usually sufficient. Weigh to the nearest milligram.

K_2O (MODIFICATION OF THE OFFICIAL LINDO-GLADDING METHOD)—A 5 g. sample (30 mesh) is dissolved in 500 cc. of water and 20 cc. (0.20 g.) are taken for a determination. The portion for analysis is measured into a porcelain or platinum dish and 2 cc. of 1/1 sulfuric acid are added carefully to avoid spattering.

Evaporate the water on the steam bath and continue the evaporation over the flame very cautiously to drive off H_2SO_4, finally heating every part of the dish to redness to destroy all organic matter and decompose bisulfates.

Cool the dish and add a drop or so of concentrated HCl and about 10 cc. of water from a wash bottle, washing down the sides of the dish. Warm to complete solution of salts and filter through a small paper (to remove SiO_2) into another dish. Wash the paper thoroughly with hot water.

Add 2 cc. 10 per cent platinum solution for material carrying up to 20 per cent K_2O, or 3 cc. for material carrying up to 50 per cent. Evaporate on the steam bath to a pasty mass (that will solidify on cooling).

Wash the precipitate six times with 80 per cent alcohol, using 4 or 5 cc. each time and pouring the washings through a tared Gooch crucible. During each washing rub the precipitate hard with a policeman to break up lumps. This also aids in separating the sodium sulfate which is easily distinguished from the yellow crystals of K_2PtCl_6. Wash the Gooch crucible twice with alcohol.

Continue washing the precipitate exactly as above (six times by decantation and twice in the Gooch) but using 20 per cent NH_4Cl solution saturated with K_2PtCl_6 to remove impurities not soluble in alcohol.

Pipettes are used for measuring the wash solutions, insuring uniformity of procedure and doing away with the ammonium chloride wash bottle, which is generally messy.

Finally bring the precipitate on the Gooch crucible with alcohol and wash free from NH_4Cl. Taste the bottom of the Gooch. A sour taste indicates insufficient washing.

Dry at 110° C. one-half hour and cool in a desiccator one-half hour. Calculate $K_2PtCl_6 \times 0.1938 = K_2O$.

Keep alcohol washings separate so that platinum and alcohol may be recovered.

Cl—Use 100 cc. of the above solution. Acidify with HNO_3; render slightly alkaline with $NaHCO_3$; add a few drops of saturated potassium chromate solution and titrate with $N/10$ $AgNO_3$ solution.

CO_2 (ALKALINITY)—Titrate 40 cc. of the above solution with $N/5$ H_2SO_4, methyl orange indicator. Make deduction for soluble silica determined later.

SO_3—Determine asBa SO_4 in 100 cc. of the above solution.

LOSS ON IGNITION—Heat a 1 g. sample of salts in a tared platinum dish to quiet fusion and weigh after cooling in a desiccator. The weighing must be done quickly as the fused salts absorb moisture very rapidly from the air.

INSOLUBLE MATTER—Treat a 1 g. sample of salts with hot water and filter. Ignite the paper and weigh.

Na_2O AND SiO_2—Evaporate the filtered solution with an excess of H_2SO_4. Ignite and weigh the mixed sulfates and SiO_2, using a tared platinum dish. After weighing take up with hot water and filter to remove SiO_2. Deduct SiO_2 and calculated K_2SO_4 from the weight found for mixed sulfates and calculate the remaining Na_2SO_4 to Na_2O.

METHOD FOR THE ANALYSIS OF BRINES

SPECIFIC GRAVITY—Determine with the Westphal balance. Temperature correction is 40° F. = 0.010 sp. gr. The specific gravity at 60° F. is an approximate indication of the per cent solids. Thus 1.100 sp. gr. indicates about 10 per cent mineral solids.

SOLIDS—Use a pipette to measure into a tared platinum dish an amount of brine containing about 1 g. of solids. Evaporate to dryness and fuse. Cool and weigh rapidly to the nearest milligram.

Cl, ALKALINITY AND K_2O IN SOLIDS—Using the same pipette as in the determination of solids, measure out the same amount of brine for the determination of Cl. A similar sample is taken for alkalinity, and a third similar sample is made up to 100 cc. and 20 cc. taken for the determination of K_2O. The methods given on the preceding page under "Analysis of Salts" are used.

From the weight of solids found the size of sample taken for the other determinations and the percentages found can be calculated.

RECOVERY OF ALCOHOL AND PLATINUM

ALCOHOL—Most of the platinum in the alcohol washings will be found precipitated as $(NH_4)_2PtCl_6$ by the NH_4Cl in the final washings. The alcohol is decanted through paper to separate it from this precipitate. The filtered alcohol is then distilled on the steam bath. The residue not distilled over is treated as below for Pt recovery and any platinum precipitated in the distillation flask is dissolved in aqua regia and reprecipitated with the other platinum solutions for recovery.

PLATINUM—The contents of the Gooch crucibles containing K_2PtCl_6 are washed into a beaker and treated with hot distilled water slightly acidified with HCl. This will dissolve the K_2PtCl_6, leaving the asbestos, which may be used again. This solution is filtered (through the filter previously used for the alcohol washings) into the alcohol suction flask containing the bulk of the $(NH_4)_2PtCl_6$. Washing the asbestos is continued until all Pt salts are in solution. This solution is then transferred to a wide-mouth bottle, where the Pt is precipitated with aluminum. A piece of aluminum rod $^3/_8$ inch diameter and about $^3/_4$ inch long is convenient for this purpose. Precipitation is not complete until the solution is clear and colorless. It may be necessary to add more acid to complete the precipitation within a reasonable time.

The solution above the precipitated platinum is decanted and filtered through paper with or without suction. The remaining precipitated platinum is washed into a beaker, where it is heated with fairly strong HCl until effervescence entirely ceases. This is to remove any adhering Al or other metallic impurities. Stirring at this point will aid flocculation and make filtering easier. The Pt is then washed onto the filter previously used and washed with hot water until free from acid. It is then dried, ignited to destroy the paper, and weighed. It is then dissolved in aqua regia and evaporated to small volume several times to remove Cl and HNO_3. This solution is filtered through a tared Gooch crucible and the weight of insoluble matter is deducted from the amount weighed as Pt. The weight of insoluble matter is usually a few centigrams and probably consists of unburned carbon from the filter paper.

The platinum solution for use in analysis is of such strength that 10 cc. contain 1 g. of platinum.

THE HORD ALKALI PRODUCTS COMPANY
LAKESIDE, NEBRASKA

SUGGESTIONS ON SOME COMMON PRECIPITATIONS

By GEORGE H. BROTHER

Received October 20, 1917

A number of my friends engaged in analytical work have spoken to me about filtration difficulties they have encountered in some of the most common determinations. According to their statements, they are unable to get filters which will "hold" unless they resort to the very retentive, but comparatively slow papers. After considerable investigation of the subject, I have come to the conclusion that their blame is largely misplaced. The fault, in the great majority of cases, lies not so much in the paper used as in the method of precipitation. For this reason I am giving a few "tricks of the trade" which, I am sure, will be helpful to any analyst not already acquainted with them, if used in standard methods given in any reputable reference, such as Treadwell-Hall.

BARIUM SULFATE

The sulfate solution should be about 200 cc. in volume and weakly acid with hydrochloric acid (1 cc. 1.2 sp. gr. to a neutral solution). It should be heated to a temperature just below boiling,[1] and about half of the solution of barium chloride necessary for excess added drop by drop, stirring well meanwhile, and allowed to digest for about 5 minutes. The remainder of the precipitant is then added (not necessarily so slowly, though the solution should be stirred during the addition) and it is allowed to digest 10 or 15 minutes longer. It is then ready for filtration.

A precipitate formed in this way will be found quite crystalline and will be readily retained by a paper of moderately close texture. I have quite satisfactorily used Whatman 40, C. S. & S. 589 "White Ribbon," and Munktell's 0 instead of the slower Whatman 42, C. S. & S. 589 "Blue Ribbon," or Munktell's 00. In this way time may be saved in the filtration as well as in the much shorter period of digestion.

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[1] "Just below boiling" gives all the advantages of precipitation and digestion in hot solution and eliminates the risk of superheating and loss through frothing or bumping.

CALCIUM OXALATE

Heat the solution of the calcium salt to just below boiling. Add excess ammonium oxalate solution, then just enough hydrochloric acid (sp. gr. 1.2) to dissolve the precipitate. Add ammonium hydroxide solution drop by drop until distinctly ammoniacal, then run in a good excess. Digest at a temperature just below boiling for about half an hour. Filter while hot and wash precipitate with hot water.

The important point in this determination is the acid oxalate solution from which calcium oxalate is precipitated by the addition of ammonia. The formation of calcium hydroxide is in this way prevented and a crystalline precipitate of the oxalate insured. The method works out the same if the original calcium solution is made acid, the ammonium oxalate (or oxalic acid) added, then the ammonium hydroxide solution, as above. The objection to this procedure is, of course, the absence of an indicator to prevent the addition of an unnecessary excess of acid. For volumetric lime determinations, where an ashless paper is an unnecessary extravagance, Whatman 3 and 30 or Munktell 100 will be found quite satisfactory if the precipitation is done by this method.

AMMONIUM PHOSPHOMOLYBDATE

The principal difficulty with this precipitation is the adherence of many analysts to the old rule, *viz.*, heat the phosphate solution to about 70° C., precipitate and digest at no higher temperature. If this procedure is followed, digestion for several days is necessary to secure a filterable precipitate, and even then success is uncertain. I have found the method of Woy with modifications, as given in Treadwell-Hall's "Quantitative Analysis," (1915), p. 437, to be very satisfactory. The essential point of this method is precipitation and digestion at a temperature just below boiling. The phosphate solution should be made distinctly alkaline with ammonium hydroxide, then nitric acid added to slight excess. This is a convenient way to insure the presence of ammonium nitrate in the solution and prevents the addition of too great an excess of nitric acid. It should be heated to boiling, then, while stirring, add the ammonium molybdate solution drop by drop from a pipette. Digest on a hot plate at a temperature just below boiling until the supernatant liquid is clear and colorless (usually about 15 minutes). Decant, wash and filter as usual. Occasionally when the precipitant is added, no precipitate immediately forms, but instead the solution becomes colored yellow. Digestion, as described above, will bring about complete precipitation and conversion of the yellow solution to colorless, but in such cases more than 15 minutes' digestion is usually required. The precipitate thrown down in this way is coarse enough to be retained by quite open-textured papers, such as Whatman 1 and 31, C. S. & S. 595, or Munktell's OB.

MAGNESIUM AMMONIUM PHOSPHATE

Here, again, I think the difficulty lies in the use of old methods, which called for the addition of magnesia mixture to an *ammoniacal* solution of the phosphate *in the cold*. The method of B. Schmitz, as outlined in Treadwell-Hall (*Loc. cit.*), p. 434, gives much more satisfactory results. The phosphate solution is treated with excess magnesia mixture solution, hydrochloric acid added just to dissolve the precipitate and it is heated to boiling. Ammonium hydroxide solution is added slowly until a crystalline precipitate forms. If the precipitate is not crystalline, it should be redissolved by the addition of hydrochloric acid and reprecipitated with ammonia. When a distinctly crystalline precipitate has formed, the solution is made ammoniacal, it is removed from the hot plate and allowed to cool. When cold, add a volume of ammonia (sp. gr. 0.9) equivalent to about one-fifth the volume of the solution, and at the end of about 10 minutes it is ready to filter.

The reverse of these determinations, *i. e.*, the determination of magnesium by precipitating with a soluble phosphate, are carried out analogously. A number of chemists in brass work are having trouble with filtering tin dioxide. I have undertaken to investigate this determination, and hope to have some results on it before long.

OTTAWA, CANADA

A NEW PORTABLE HYDROGEN SULFIDE GENERATOR

By W. FAITOUTE MUNN

Received November 5, 1917

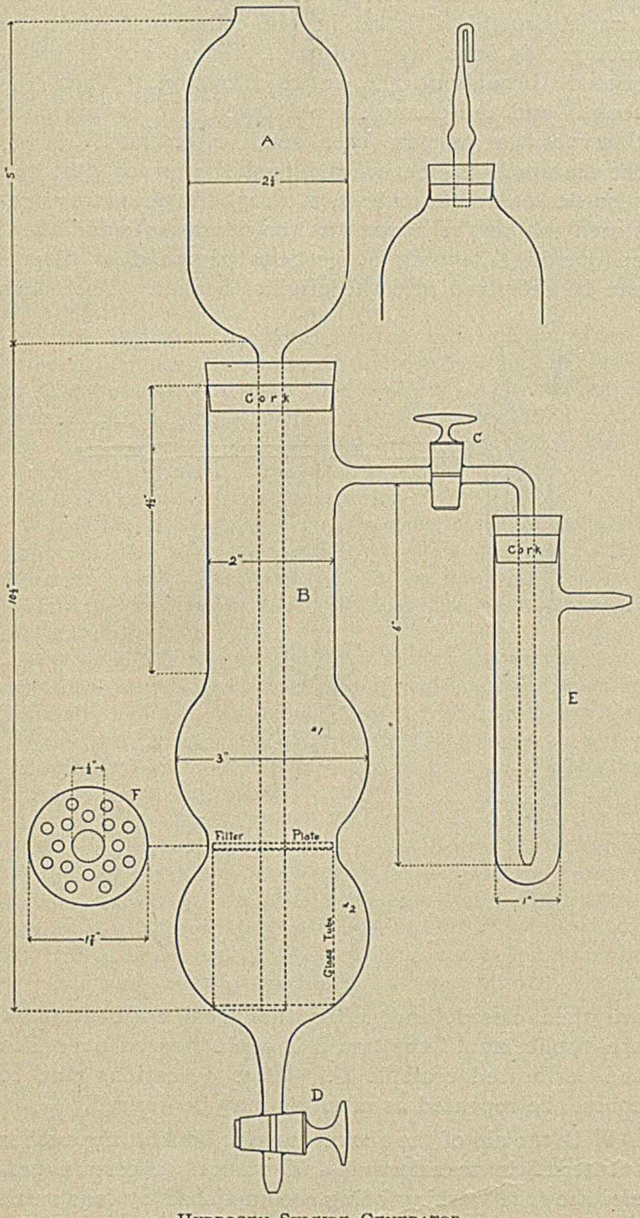
Because of the objections to hydrogen sulfide generators in general, namely, the renewal of the acid, the leaking of gas following the completion of the precipitation after the supply is not further desired, and bulkiness, the following apparatus is recommended.

The generator is quite light, practically in one piece, self-adjusting, made in a size adapted to most analytical work, and is supported by a condenser clamp to an iron support, thus enabling the chemist to carry it easily to all corners of the laboratory. Although designed for the preparation of H_2S, it may be used for preparing CO_2, H, or any of the other gases.

The fairly heavy glass tube *B*, which is the main part of the apparatus, has a stopcock, *C*, fused to it near the upper extremity. Two bulbs are blown on the lower half of the tube and below these bulbs a stopcock, *D*, with a fairly wide bore, is fused. A lead plate, with holes as shown in the drawing, is supported between the bulbs by means of a piece of tubing having about the same diameter as the plate and cut to a length such as is required to bring the plate support to a point between said bulbs. This glass support rests on the lower part of bulb 2. Both the plate and support should be of such diameter as to just allow them to easily clear the wall of tube *B*.

The tube *A* of the apparatus, and at least the capacity as given, acts as the reservoir for the acid. This consists of a $2^1/_2$ to 3 in. diameter tube, 5 to 6 in. long, with a $^3/_8$ in. diameter tube $10^1/_2$ in. long sealed to its lower extremity. This tube passes through a rubber stopper placed near its upper end, while the lower part passes through the filter plate and nearly to the bottom of bulb 2. The stopcock *C*, which has already been mentioned, has its tube bent as shown in the figure and supports a 1 in. diameter fairly heavy-walled test-tube, which in turn has a side tube fused to it near its upper end. This test-tube contains a small quantity of water and acts as a purifying chamber for the gas evolved. No support for this tube is required.

To fill the generator, the glass tube for supporting the lead plate is first carefully placed in position. The lead plate is then cautiously slid in. The tube sealed to the lower extremity of *A*, after putting the cork in position, is lowered into the bulb *B*, until the end has just passed through the center of the lead plate. (The center hole in this plate must be of a size which will allow the free passage of this tube.) The apparatus is then inclined and small lumps of iron sulfide cautiously rolled into tube *B*, until bulb 1 is filled. The tubes are then placed in a vertical position and tube *A* lowered until the lower extremity of the glass tube reaches the bottom of bulb 2. When the tube has reached this point the rubber stopper should be at the correct point for securely fastening in the neck of *B*. The apparatus is now fastened to a condenser clamp, at a point just above bulb 1, of tube *B*. Stopcocks *C* and *D* should be closed, and then dilute H_2SO_4 (1 : 8) poured into the top of *A*, until about full. The cock *C* is now opened until the acid has filled half of bulb 2. The cock *C* is then again closed and enough acid added to *A* to again fill it. The comparative volumes of tubes *A* and *B* should always be kept in mind because if *A* is made smaller than herein given, or bulbs 2 and 1 made larger, *A* will not be large enough to hold sufficient acid to supply bulbs 2 and 1, and still have space enough for the returning acid when the supply of gas is stopped. It is therefore advisable to make tube *A* about 6 in. in length to keep on the safe side.



Hydrogen Sulfide Generator

By opening the cock *D*, the lower portion of acid, which has been in contact with the iron sulfide and which finally becomes inactive, may be removed without disturbing the apparatus or causing the escape of gas. This arrangement allows one to use all of the acid added to the generator and permits complete renewal of acid in a very short time. The removal of spent acid through cock *D* also washes out the black sediment which is always left after the decomposition of the sulfide.

The firm of Eimer and Amend have made up one of these generators for exhibition purposes.

Research Department
Lederle Laboratories
New York City

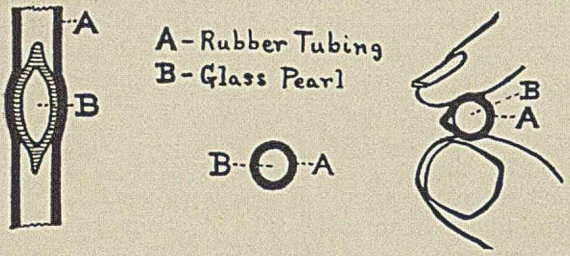
AN AUTOMATIC HYDROGEN SULFIDE STOPCOCK

By Carl H. Classen

Received November 15, 1917

In students' laboratories there has long been a need for a simple, efficient, and fool-proof hydrogen sulfide stopcock. Metal stopcocks, in general, are obviously impossible. Hard rubber or glass stopcocks are very useful so long as they do not stick or break, but in themselves they do not furnish any safeguard against being left open, or any easy control of the flow of gas. Rubber tubing with pinchcocks is also useful, but the prolonged squeeze of the pinchcocks rapidly weakens the rubber. Although screw pinchcocks give an excellent control of the flow of the gas, they, too, do not furnish any safeguard against being left open; also, the rubber is likely to be excessively squeezed, and even cut through with the consequent danger of hidden leaks. Except for the injurious squeeze and the loose adjustment, the ordinary pinchcock comes the nearest to the ideal stopcock. A satisfactory solution is particularly desirable where stopcocks are to be used in connection with numerous branch pipes from hydrogen sulfide mains.

Probably the nearest approach to a satisfactory solution is the device which is being used this year at the Rice Institute, Houston, Texas. To each small branch pipe of the hydrogen sulfide main, thin-walled, gum-rubber tubing one foot long and of convenient diameter is attached. Near the branch pipe, and inside the rubber tubing, is a glass "pearl" made from glass tubing of a diameter slightly larger than that of the rubber tubing—the rubber should not be stretched too much. The device is merely another application of a similar device that is used with ordinary Mohr burettes.



Longitudinal cross section *Closed* *In use*

The advantages of the device are obvious: the student must "be on his job;" open stopcocks are impossible; the flow of gas can be regulated with certainty and extreme ease; the device is not easily put out of order; it is easily and quickly replaced; there is no prolonged, excessive squeeze; the pearl may be moved to a new position when necessary; the cost is ridiculously low; the gas is not wasted.

The device has been in use only one month and, consequently, it is too early to say that it is an entire success where the durability of the parts is concerned. Where its use by the student is concerned, there can be no question of its entire success; its simplicity, and the ease and neatness of its manipulation make a "hit" with the student. Undoubtedly improvements in design and material are possible.

Rice Institute
Department of Chemistry
Houston, Texas

A SIMPLE AND EFFICIENT FILTERING TUBE[1]

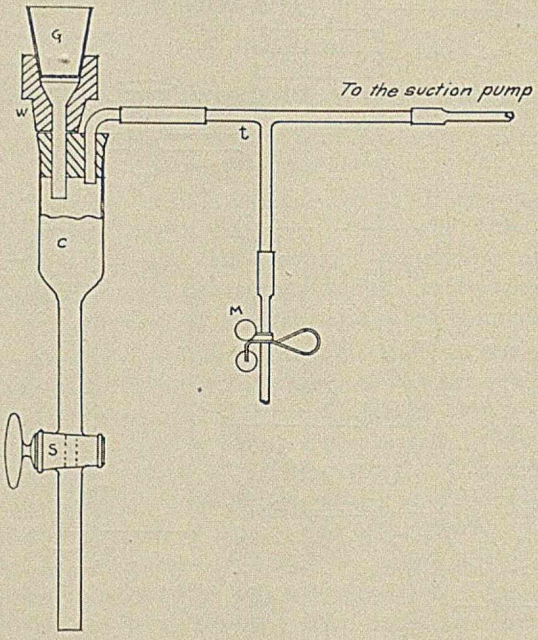
By William M. Thornton, Jr.

Received October 8, 1917

The apparatus here depicted was designed particularly for handling those precipitates whose solubilities are sufficient to necessitate great economy with the liquid used for transferring and washing. Although it resembles somewhat the filtering devices of Zöpfchen[2] and of Bailey,[3] it is not, however, identical with either. Moreover, the appliance can easily be put together from materials to be found in any well-equipped laboratory.

A straight glass tube, having a stopcock at its middle point, is sealed to a carbon filter tube. The latter is fitted with a two-hole rubber stopper. The stem of a Walter crucible holder passes through one hole of the stopper while the other contains a right-angled exit tube. The rubber hose leading to the suction pump is intercepted by an ordinary T-tube, the free end of which is joined to a short piece of rubber tubing. A Mohr clamp is kept in readiness. The whole is held in position by a stand with two clamps appropriately placed—one to grasp the main tube and the other to support the T-tube.

The manipulation is quite obvious. Having prepared the asbestos felt in the regular way, the perforated crucible *G* is set in the collar *W*. After the suction has once been adjusted, it need not be interrupted throughout the entire filtration. When the cock *S* and the clamp *M* are closed, the carbon tube *C* serves the purpose of a small filter flask. When the clamp *M* is opened and pushed upward past the shoulder on to the tube *t*, atmospheric pressure is restored within the apparatus (or nearly so); and then, on opening the cock *S*, portions of the clear filtrate can be delivered into the original beaker. The cocks are then closed, and, after the usual application of the "policeman," the liquid is again poured over into the perforated crucible *G*. These operations can, of course, be repeated as often as may be desired.



With the aid of the above-described outfit, the author succeeded in transferring and washing a certain precipitate (the washing being accomplished by four small portions of iced water successively applied) so that the sum total of the filtrate and washings did not exceed 27 cc. Furthermore, the apparatus is so convenient that filtrations can be very quickly performed with its help—thus reducing to small dimensions the losses incurred in handling those precipitates which suffer an increase in solubility on rise of temperature.

Johns Hopkins Chemical Laboratory
Baltimore, Maryland

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[1] Published by permission of the Chemical Director, E. I. du Pont de Nemours & Company.

[2] *Chem.-Ztg.*, **25** (1901), 1008.

[3] *J. Am. Chem. Soc.*, **31** (1909), 1144.

ADDRESSES

THE AUTOMATIC CONTROL AND MEASUREMENT OF HIGH TEMPERATURES[1]

By Richard P. Brown

Probably no employee causes the average works manager so many sleepless nights as does the furnace man, on whose shoulders rests the responsibility for the accurate heat treatment of the steel and the uniformity of the product. This is not only true of a steel plant, but is also equally true in the chemical industry, where the temperature of numerous processes must be accurately controlled; in the glass industry, where the melting of the glass and its annealing must be carried on within very narrow limits of temperature; in the brick industry, where the kiln firing is of the utmost importance, and I might continue the list throughout the whole industrial field.

The old furnace man, through years of practice, will endeavor to gauge the temperature of the furnace with his eye. Providing he has not been up all the previous night and his eye is clear, he will probably judge the temperature fairly accurately. If he is of a reasonably kind disposition, he may permit the manager to install pyrometers to guide him in controlling the temperature, and he may even in time use the pyrometers and rely on them.

But we can pardon the works manager or director for asking, "Suppose John dies, gets sick or quits his job, how am I to handle the output of these furnaces?" He would like to have an understudy for the old furnace man, but the latter does not like the idea. So he wonders why someone does not develop a device to automatically control the temperature of the furnaces, so that he can cease worrying about them.

This is one reason why a great amount of study has been given, not only to perfection of pyrometers, but also to the automatic control of temperature. It has, however, been only recently that real results have been accomplished in automatic temperature control.

First of all, it was necessary to perfect the temperature-measuring instrument in order that it could be relied upon to uniformly indicate the actual furnace temperature. It was then necessary to apply to the pyrometers attachments to throw the switches on the electric furnaces, or to open or close the valves on gas or oil furnaces.

My experience in the United States has shown that, for industrial service, an instrument actuated by the expansion of nitrogen gas is the most satisfactory for temperature measurements up to 800° F. or 425° C. The gas-expansion instrument consists of a bulb of copper which is inserted in the heat, and this bulb is connected by capillary tubing to an indicating or recording gauge containing a helical expansive spring.

The expansion of the gas in the bulb exerts a pressure in the capillary tubing which is conveyed to the spring in the instrument, and the pointer, attached directly to this spring, moves across the scale or chart.

It is essential in this instrument that the capacity of the bulb be some 50 times as great as the capacity of the capillary tubing and spring. This is essential to reduce to a minimum errors due to changes in atmospheric temperature along the capillary tubing or at the instrument. In consequence this instrument is not desirable for use where the gauge must be placed more then 100 feet from the bulb.

For use at moderate temperatures, where the measuring instrument must be placed at a considerable distance and for temperatures above the range of the gas-expansion instrument, the thermo-electric pyrometer has been almost universally adopted in the United States. A thermocouple of base metals, usually formed of one wire of nickel 90 per cent and chromium 10 per cent, and the other wire 98 per cent nickel and 2 per cent aluminum, is preferred for temperatures to 1800° F. or 1000° C. For temperatures above this, and as high as 2800° F. or 1500° C., thermo-electric pyrometers using a platinum-rhodium thermocouple are the most satisfactory. For still higher temperatures, a radiation type of pyrometer is available, consisting of a thermocouple in the focus of a reflector at the rear end of the tube, which is pointed at the door or opening of the furnace.

For measuring the voltage produced by a thermocouple, whether of base metal, platinum-rhodium, or the radiation type, high resistance millivoltmeters are available. We are producing in the United States such millivoltmeters of some 1000 ohms or more. This remarkably high resistance is naturally desirable to practically eliminate the errors due to changes in the resistance of the line or wiring connecting the thermocouples and the instrument, and also to nullify the effects of any changes in the resistance of the thermocouples due to heating.

Changes in resistance may be due to actual changes in length or changes in atmospheric temperature, which in turn affects the resistance of the line or wiring. We have been able to secure this exceedingly high resistance by reducing the weight of the moving element to a minimum, and I have a sample of this moving element for exhibition.

The total weight of the moving element in our high-resistance pyrometer, including pointer and springs, is 526 mg. This extreme lightness is secured by the use of an aluminum alloy wire, which we have succeeded in enameling. The enamel coating is much thinner than the silk insulation formerly used and more turns can be secured on a coil of a given width. Likewise, by the use of the aluminum wire, the weight has been reduced 66$^2/_3$ per cent as compared with copper wire which was formerly used for these moving elements. The aluminum wire is 0.003 inch in diameter and drawing this wire has been quite a mechanical problem.

The pointer tubing in this moving element is of aluminum with an inside diameter of 0.008 inch, an outside diameter of 0.012 inch, or a total thickness for the wall of the tubing of 0.002 inch. Even this weight for the pointer tubing could probably be reduced by the use of magnesium instead of aluminum, but to date we have been unable to satisfactorily draw magnesium.

I only cite these points regarding the construction of our present-day high-resistance pyrometer millivoltmeter to show what development work can produce. Instruments of this type made by Siemens and Halske, of Germany, were never developed to this extent, at least, prior to the outbreak of the war, and their moving element was several times as heavy as the sample I have here, and in consequence the resistance of their pivoted meter was several times less. Incidentally, I think we have about reached the limit of development along this particular line.

It will be understood that the same electrical system, such as I have described, can be used either to indicate the temperature, or, combined with the proper apparatus, to record the temperature constantly on a recording sheet. There are both portable and wall-type indicating pyrometers, and recording pyrometers are produced to make a record on a circular chart 8 inches in diameter, or to make a continuous record of the temperature on a roll of paper lasting two months or more. By the introduction of suitable switching mechanism a record of the temperature of quite a number of thermocouples can

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[1] Read before the Faraday Society, London, by the Secretary of the Society, November 7, 1917.

be made on the same record sheet. These temperature records are distinguished by using different colors for each record line, by using numbers corresponding to each thermocouple, or changing the form of line produced on the chart for identification.

For even greater precision in temperature measurements than is secured with the high-resistance millivoltmeter, we have developed a new instrument, which we call the Brown Precision Heat Meter. This instrument is suitable for either temperature measurement or automatic control of temperature, and brief description of this new instrument might be of interest.

My idea in the heat meter has been to eliminate all the bad features or drawbacks met with in using a millivoltmeter for temperature measurement.

Possible sources of error in the use of a millivoltmeter in temperature measurements, even one of high resistance, consist in changes in resistance of the circuit comprising the thermocouple and the leads or wiring, due to changes in length or changes in atmospheric temperature; also errors can occur due to temperature coefficient of the meter, that is, errors caused by changes in atmospheric temperature in the meter itself. Another source of error is a change in the actual indication of the instrument, due to spring fatigue, abuse or sticking, and to overcome these possible sources of error we have developed this rather interesting instrument.

Briefly, its operation is as follows:

With our standard millivoltmeter of high resistance, we supply an ordinary dry cell about $1^1/_4$ inches in diameter by $2^1/_2$ inches long, and furnish suitable rheostats to reduce the voltage of the dry cell from approximately $1^1/_2$ volts to a range from 0 to 60 millivolts, the maximum voltage produced by the thermocouples.

In our first operation, we oppose the voltage developed by the thermocouple to the reduced voltage of the dry cell, and when the pointer stands on zero, it indicates that the voltage from each source is the same. We now in operation No. 2 cut out with a switch the voltage of the thermocouple and read the voltage of the dry cell circuit by direct deflection. This eliminates the line resistance entirely as in a potentiometer.

We now have a deflection indicating the actual temperature developed by the thermocouple at the moment of reading the instrument, but fluctuations in temperature of the thermocouple will not be indicated, as we are reading the voltage from the dry cell. We have, however, incorporated other operations in this meter.

In operation No. 3 we connect the thermocouple with the meter instead of the dry cell circuit and we note whether the indications are the same. By switching back and forth quickly, the voltage from the thermocouple circuit or from the dry cell circuit can be noted. If excessive line resistance has caused the indications of the millivoltmeter to be lowered as compared with the dry cell circuit, a rheostat is operated to bring up the indications of the thermocouple circuit to that shown when we are reading the voltage of the dry cell circuit.

We now leave the instrument indicating on the thermocouple circuit and the errors, if any, which might be due to line resistance or changes in temperature of the line, have been eliminated, and we have a direct reading millivoltmeter, indicating the correct temperatures.

A rheostat of 15 ohms is supplied in the meter which permits of adjusting the indications for a total change of line resistance equivalent to 15 ohms, or a circuit of two copper wires almost a mile long.

We have eliminated the temperature coefficient of the meter by furnishing a copper resistor in the meter equivalent to the copper or aluminum of the coil; hence, in balancing the voltage from the dry cell against that of the thermocouple we also automatically eliminate errors due to the temperature coefficient of the meter itself.

There is now left only one possible source of error, the change in the actual indications of the meter due to sticking of the pointer, abuse of the instrument, spring fatigue, etc. To obviate this source of error we can supply with the instrument a standard cell with suitable resistors, and by the potentiometer method used in testing the meter we can check this meter. We supply three resistors, for example, where a meter is calibrated for 60 millivolts (we furnish resistors equivalent to a deflection of 20, 40 or 60 millivolts on the scale), and after balancing the standard cell against a part of the voltage of the dry cell, through these suitable resistors we can note whether the pointer swings to 20, 40 and 60 millivolts, respectively, on the scale. If it does not, the error can be noted and the actual error in calibration is detected.

Where the instrument is supplied with standard cell the temperature of the instrument should always be between 5° C. and 40° C., or 40° F. and 105° F.; in fact, standard cells of cadmium sulfate or zinc sulfate will be injured if the temperature rises or falls beyond these limits. This is true of any standard cell employed in instruments.

In this instrument we have all the good features of the potentiometer method of measuring temperature with the advantage that we have a direct reading instrument which can be adjusted once every day or oftener if desired, for the actual line resistance with which it is used and the surrounding atmospheric conditions. The meter will then indicate correctly throughout the whole scale range, and the furnace man has the instrument to guide him without hand manipulation, and an inspector can daily check the calibration of the instrument.

Naturally, this instrument is equally as suitable for automatic temperature control as the instruments previously described, when properly designed for this service.

AUTOMATIC TEMPERATURE CONTROL

Attempts have been made in the past to electrically operate switches and valves by permitting the pointer of the pyrometer to come in contact with adjustable contact arms on each side of the pointer. Unfortunately, the millivoltmeter, used with the thermo-electric pyrometer, has an exceedingly weak control for the pointer. One is easily able to blow the pointer across the scale with the breath.

In consequence, simply permitting the pointer of such a pyrometer to move into contact, is not sufficiently positive to be satisfactory for automatic control work.

The automatic control pyrometer exhibited here operates in the following manner: A thermocouple formed of a nickel-chromium alloy is installed in the electric furnace, the temperature of which is being controlled. The thermocouple actuates a high resistance millivoltmeter. Below the pointer and adjustable throughout the whole scale range, is a table carrying two contact pieces, separated by a thin piece of insulating material $^1/_{32}$ inch thick. The depressor arm driven by a small electric motor, or by a clock if preferred, depresses the pointer at regular intervals, usually every ten seconds, and in doing so the pointer forces together the two contact pieces below.

Let us assume the pyrometer controller is required to control the furnace at a temperature of exactly 1400° F. The knob on the left of the instrument is turned until the index in front of the scale stands at 1400° F. This index corresponds to the position of the thin insulating material which separates the high and low contact.

The switch connecting the furnace in the line is closed and the pointer slowly rises across the scale as the temperature of the furnace rises. As the switch is already closed, when the pointer is depressed on the low contact, the switch continues to remain closed, and no change occurs until the pointer passes over the neutral insulating piece and is depressed on the high contact. The switch indirectly operated by a solenoid and relay is now instantly actuated and the circuit opened. The temperature of the furnace begins to slowly fall, and when the pointer is again depressed on the low contact, the circuit is again closed. This operation continues as long as the furnace is to be operated.

When the switch opens and closes the main circuit, the current is either full on or off, and the fluctuations are continuous within narrow limits of some 10° to 20° F. These continuous risings and fallings of temperature can be largely reduced and closer control can be procured by the use of two rheostats in the furnace line. The solenoid-operated automatic switch is then used to simply cut in and out of circuit the second rheostat.

Assuming it is desirable to continually maintain 1400° F. in the electric furnace, irrespective of fluctuations of voltage, the two rheostats are set so that with only one rheostat in the circuit the temperature will rise to approximately 1500° F. With the second rheostat in the circuit the temperature drops to 1300° F.

When we now use the solenoid-operated switch to cut in and out the second rheostat, we naturally control the temperature only between 1500° and 1300° F., and we do not have the rapid surges or ups and downs in temperature, and thus maximum control is secured.

It is realized that the same form of switch can be used to operate a valve to control a gas or oil furnace. We have found it desirable to use an automatic valve in a by-pass so as to control simply a portion of the gas or oil supply, and in the same manner as in the electric furnace control, eliminate the maximum fluctuations caused by the complete opening and closing of the switch or valve.

Assuming that we have a 2-inch supply pipe for the gas to the furnace, it is customary for us to by-pass this and use a $^1/_2$-inch automatic valve, which gives us approximately 25 per cent control. This is sufficient to control the usual fluctuations in gas supply and secure very satisfactory control. This method also eliminates the difficulty which would occur where the gas is completely shut off and then turned on in full, as would occur without the by-pass control.

TEMPERATURE SIGNALING PYROMETER

In addition to an instrument to automatically control furnace temperatures, there has been a demand for an instrument to automatically signal by lights whether the temperature is too high, correct, or too low in any particular furnace.

It has been common practice in plants in the United States, where there are a number of heat-treating furnaces, to maintain an operator at a central pyrometer and by colored electric lights at the furnace to signal whether the temperatures are right or not. It is common practice to locate three lights above each furnace—red, white and green; the red light burns when the temperature is too low, the white light when the temperature is within certain limits, for example, within 20° F. of the correct temperature, and the green light burns when the temperature is too high. The fireman who operates the furnace is guided entirely by the lights and a central pyrometer is used to control the temperatures.

We have been able to develop an instrument to automatically signal by lights whether the temperature is correct or not, and in this way the services of the operator at the instrument are eliminated. The same form of instrument is used for this purpose as we use to automatically control the furnace temperatures, and the pointer is depressed at intervals of every ten seconds onto contacts corresponding to the red, white and green lights.

No special battery or other source of current than an ordinary service line, is required to operate these lights. The supply may be 110 or 220 volts, either a. c. or d. c. The current which lights the lamps does not follow through the instrument, but is made and broken by an auxiliary device containing the necessary mechanism. A high resistor is in series with the circuit connected with the pyrometer, which reduces the current flowing through the contractors within the instrument, to less than 0.07 amp. This prevents sparking at the contractors and errors due to the heating effect of a current of higher amperage. The lamps may be any reasonable distance from the pyrometer, in fact, they are operative at a mile or more if desired.

The various thermocouples in each furnace are connected successively to the instrument through switching mechanism, and at the same time a switching mechanism connects the various sets of lights at each furnace. We have constructed an instrument of this character to automatically take care of signal lights at 12 furnaces.

This form of equipment gives the fireman or operator of the furnace an indication by lights which he can easily understand, and he adjusts the valves or fires the furnaces accordingly. It is simple to instruct a man to keep the white lights burning and to explain what the red and green lights mean, and it requires a less experienced workman to control the furnaces in this manner than to read temperatures on a pyrometer scale. This newly developed instrument also eliminates the man required to read the temperatures at a central pyrometer.

The extensive use of pyrometers to measure or record high temperatures will serve to

(1) Eliminate guess work as to the temperature.

(2) Reduce fuel consumption through the maintenance of the correct temperature and not excessively high temperatures.

(3) Reduce time for heating of the product due to the maintenance of the correct temperature.

(4) Increase efficiency in operating a plant through the savings outlined above.

Instruments to automatically control the temperature, when properly constructed and applied, will eliminate entirely the personal element. The maintenance of the correct temperature in the furnace is automatic, and this is a step forward and an improvement over temperature control through pyrometers.

I do not doubt but that the next few years will see further improvements in pyrometers and temperature control. There will always be room for improvement, and the coöperation of the industrial works and the pyrometer manufacturers will largely hasten the development of practical instruments for the measurement and control of high temperatures.

The Brown Instrument Company
Philadelphia, Pennsylvania

AIRPLANE DOPES[1]

By Gustavus J. Esselen, Jr.

"Dope" is the term applied to the varnishes used on the wings of airplanes to render the fabric taut and waterproof. An airplane wing is made by covering a frame work of the proper size and shape with a linen or cotton fabric. There are then applied to the fabric several coats of a suitable varnish, of which the base is either cellulose acetate or cellulose nitrate. The term "dope" seems to have arisen in the slang of the factory workman, but is now firmly fixed and is used to distinguish the cellulose acetate or nitrate "dopes" from the spar varnishes made up of gums and oils, which are sometimes used as a finishing coat. The chief function of the dope is to tighten up the fabric and give a smooth, taut, waterproof surface, resistant to the weather,

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[1] Address presented before the Northeastern Section of the American Chemical Society, October 19, 1917.

and preferably also to oil and gasoline. It also adds to the tensile strength, the percentage increase depending somewhat upon the strength and character of the fabric, and upon how much the fabric was stretched before doping. The tentative specification of the Bureau of Standards requires an increase of at least 25 per cent in the tensile strength of linen fabrics, and of 15 per cent with cotton fabrics. In practice considerably greater increases are often obtained.

The fabric most in demand for airplane wings is linen, because of its strength and toughness and the difficulty with which it tears. Owing, however, to the present scarcity of linen, specially woven cotton fabrics are being developed. While some of the latest of these have a tensile strength as great as that of the best linen, they still seem to tear more readily. The average weight of an acceptable fabric is about $3^1/_2$ to 4 ozs. per sq. yd., and the tensile strength is in the neighborhood of 90 to 100 lbs., per inch width. In general, the dope, when dry, adds about 2 to $2^1/_2$ ozs. per sq. yd., but in certain types of war machines it is considered necessary not to let the combined weight of fabric and dope exceed 6 ozs. per sq. yd.

The dopes which are at present in use may be divided into two classes: first, those consisting essentially of a solution of cellulose nitrate or pyroxylin; and second, those made by dissolving cellulose acetate. The usual concentration is about 8 ozs. per gal. Among the more common solvents for the acetate are acetone and combinations consisting largely of tetrachlorethane and alcohol. The acetone may be either pure or some of the commercial grades containing methyl acetate and methyl alcohol. To dissolve the nitrate, the usual solvents such as amyl acetate and acetone are used, and the solution diluted to the proper strength with suitable non-solvent liquids. In addition, some substances are usually added to preserve the flexibility of the coating, or modify the shrinkage power, as, for example, castor oil, which is often present in nitrate dopes. It is possible in this way to get all gradations of shrinking power from almost none to an amount sufficient to twist the frames out of shape.

The great outstanding difference between the coatings given by cellulose acetate and cellulose nitrate dopes is the inflammability of the latter, a difference which will probably be emphasized more and more as the use of airplanes for peaceful purposes increases. Cellulose acetate dopes leave a non-inflammable finish. The relative behavior of the coatings left by the two types of dope is well illustrated by the fact that some gasoline can be poured on a piece of fabric coated with a good cellulose acetate dope and allowed to burn, and the fabric does not ignite. The same test applied to a pyroxylin-coated cloth results in the immediate ignition of the coating, and in a very short space of time there is nothing left of the fabric or coating but a puff of smoke.

The initial cost of pyroxylin dopes is somewhat less than that of cellulose acetate dopes, and on this account some manufacturers have tried to effect a compromise between expense and inflammability by applying three coats of pyroxylin first, and finishing with two or three coats of acetate. This gives a fire-resisting surface, even though the coating is not non-inflammable all the way through. In Europe only acetate dopes are used, and it seems to me only a matter of time when the users of airplanes in this country will demand that every possible factor of safety be taken advantage of, which will mean the use of a non-inflammable dope.

You perhaps remember that in one of the editorials of the September number of THIS JOURNAL, Mr. Leon Cammen, Vice-President of the *American Aeronautical Society*, is quoted as saying that the ideal dope "should make the fabric waterproof, air-proof, fire-proof, or at least slow-burning, should give low visibility, prevent deterioration, and be non-poisonous."[1] In these specifications, one or two essential points have been omitted, probably because they were taken for granted. One of these is smoothness and another adhesion. Smoothness is a quality which in this case can readily be measured by the wind resistance, and much attention has been and is now being given to such measurements.[1,2] An interesting calculation has been made by Gibbons[2] showing that in a moderately large machine the difference in wind resistance between a doped and an undoped fabric would correspond to a difference in lifting power of from 150 to 180 lbs., in other words, the weight of one man. As regards adhesion, a good dope should adhere well to the fabric, so that in case of an accident causing a break in the cloth, the coating will not be started and peel off. The dopes at present on the market vary considerably in this respect, but it sometimes happens that dopes are blamed when the real fault lies in the sizing of the cloth. One of the factors which seems to affect adhesion is the penetration. As a general thing, dopes do not penetrate well on sized fabrics. These are exceptions to this rule, and the explanation may be that in those cases where dopes do penetrate well on sized fabrics, the particular sizing involved is soluble in the solvent used, and blends with the dope. Another factor which probably enters in, is the character of the solution which constitutes the dope. While it has not been definitely established, the indications are that the more nearly the dope approaches being a true solution, the greater the penetration; whereas the more colloidal it is, the less it will penetrate. For any given dope, it has been demonstrated that it will penetrate better, adhere better, resist atmospheric conditions better, and wear better in every way when applied to an unsized fabric than it will on the same fabric sized. A reasonable way of accounting for this would be to assume that where there is no sizing the dope has a greater tendency to penetrate the fibers of the fabric, rather than merely between the fibers, and thus when dry becomes more nearly a part of the fabric. Since most sizing can readily be removed by boiling, either in soapy water or a weakly alkaline solution, it is a simple matter to insure good adhesion.

It may be interesting to know how near to the ideal the present-day dopes come. As to being waterproof and air-proof, all dopes which are used at all fulfill these requirements, because those are the first properties to be tested, but the extent to which they resist continued exposure to atmospheric conditions varies with the dope. The usual method of testing dopes is to try them out on linen stretched on frames about 12 to 15 in. square, and expose them on some convenient roof. This is much more severe treatment than airplanes ordinarily receive. The first desirable property to disappear is usually the flexibility, and the length of time before this happens can ordinarily be taken as a measure of the relative value of different dopes. The great difficulty with this test is that it varies so according to the season of the year, owing to the wide variation in atmospheric conditions. Another objection to the test is that it is not strictly parallel to service conditions, in that a wing in actual service is subjected to a very severe vibration to which the test panel is not. On the other hand, if a dope remains flexible, the vibration alone should not cause it to crack. The Bureau of Standards at present requires a dope to retain its flexibility at least 60 days when constantly exposed to the weather. There is a certain weakening of the fabric on exposure, and tests[3] have shown that this is much less with acetate than with nitrate dopes. Especially is this true of cotton and in a less marked degree of linen. This is what might be expected when one recalls that cellulose acetate is a much more stable compound than cellulose nitrate. In addition, any free nitric acid formed

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[1] THIS JOURNAL, 9 (1917), 826.

[1] Zahm, *Phil. Mag.*, 8 (1904), 58.

[2] Report of National Advisory Committee for Aeronautics for 1916, pp. 176ff.

[3] Gibbons and Smith, *Ibid.*, p. 168.

by the decomposition of the pyroxylin would have a much greater weakening effect on the fabric than would a similar amount of acetic acid, even if the rate of decomposition of the two cellulose esters were the same. In one instance the tensile strength of cotton fabric doped with a nitrate dope after 3 weeks' exposure was reduced to 48.5 per cent of the original, while linen coated with the same dope was reduced to 68 per cent. With an acetate dope under similar conditions, the tensile strength of the cotton was reduced to only 87.2 per cent and the linen to 84.8 per cent. All of these were taken in the direction of the warp.

In order to give a more waterproof surface, it is the general practice with some companies to apply one or two coats of spar varnish over the usual dope. The use of spar varnish has one disadvantage, however, and that is in the matter of patching. As a rule, a hole in a wing can be patched with a piece of fabric by using dope as a glue. On spar varnish, however, most dopes do not stick, so that in order to make a repair, the varnish first has to be removed.

There is one bane of the dope room which might be mentioned here, and that is the formation of white spots during the drying of the dope. They are due to moisture condensation during the drying and show up as a rule only on humid or rainy days. They can be removed by going over them with a little solvent, or better by applying a cellulose acetate dope designed for the purpose. Of course, the white spots can be prevented, even on damp days, by regulating the amount of moisture in the air of the dope room, but this, as a rule, requires a rather elaborate ventilating system.

Of Mr. Cammen's ideal properties, we have now considered all but the last two, *viz.*, that of being non-poisonous and that of low visibility. The biggest bone of contention as regards poisoning has been tetrachlorethane. Tetrachlorethane is one of the best solvents for cellulose acetate. Unfortunately, however, its vapors when inhaled affect the liver, and before its poisonous character was recognized, a few deaths had resulted abroad. These were the result of inadequate ventilation. As soon as suitable ventilation was provided, the trouble almost entirely ceased. Some argue for the total exclusion of tetrachlorethane as a solvent in cellulose acetate airplane dopes, but it gives the finished wing a certain resilience which nothing else does. Some of the British experts are very strong in their assertions that it is indispensable as a constituent of dopes for scout machines. However, the report is that for some months the use of tetrachlorethane has been forbidden in England. The policy for this country has not as yet been absolutely established, but should it prove desirable to use tetrachlorethane, it would seem possible to do this, if ample ventilation of the proper type were provided.

As regards low visibility, the dopes, being transparent and almost colorless, do not make the fabric appreciably more visible than if it were not doped, except possibly when the sun might be temporarily reflected from a glossy wing. The use of a dull surface would obviously remedy this. Should it be found that some color other than that of natural linen would blend better with the color of the sky background, either the dope or the fabric could be dyed. The goal of low visibility, however, is a transparent wing. Doubtless many of you saw a newspaper account a little over a year ago of a foreign airplane that did not have transparent wings. That no further reports of such machines have appeared would seem to indicate that as yet, at least, they are not very common. The wings of the machine in question were made of transparent cellulose acetate sheets, and it was claimed that at a height of a few thousand feet, the machine was almost invisible. It had the further advantage that the field of vision of the operator was much increased. Cellulose acetate sheets in a thickness of $^{10}/_{1000}$ of an inch, which thickness has the necessary strength, weigh about nine ounces per sq. yd., which is a little heavier than most doped fabric. The chief difficulty in using them is the fact that a tear once started spreads very rapidly. A patent has been taken out for remedying this difficulty by reinforcing the transparent sheet by means of a fabric such as silk woven rather loosely. As is readily seen, a wing of this sort would be waterproof, air-proof and non-inflammable, would have low visibility, would not affect the workmen applying it, and would give no trouble from stripping or peeling. In other words, there seems no doubt that if it can be obtained of the proper weight and strength, it will prove to be the ideal wing covering.

CHEMICAL PRODUCTS COMPANY
BOSTON, MASS.

THE COLLABORATION OF SCIENCE AND INDUSTRY[1]

By V. GRIGNARD

I am much honored that you have asked me to speak before you, but I must apologize for my ignorance of the English language which prevents my speaking at length without abusing your courteous attention.

I appreciate deeply the great honor done me in making me honorary member of the Mellon Institute and I wish to express my deep gratitude to the corps of professors and especially to the Director, Mr. R. Bacon.

My collaborator, Monsieur Engel, who speaks English fluently, will tell you better than I of the excellent impressions we have received in the course of our visit in your beautiful country; but I wish to emphasize myself all the gratitude which I feel toward your great industrial establishments and scientific institutions for the uniform kindness which we have received from them, for the courtesy with which they have facilitated our visits and have furnished us with literature.

I wish to express to you also all the admiration which I feel for the remarkable scope of your industry. You have made magnificent use of your immense natural resources, thanks to your will, your initiative, your faith in the future. These valuable traits the American race owes to the very conditions under which it has evolved: the first colonists, a small group of enterprising men constituting an élite because of their aptitude and their scientific knowledge which two centuries ago (and even less) was the lot only of the privileged few, found themselves struggling in a nearly virgin land with the many difficulties which social life has imposed upon humanity. They acquitted themselves with honor and if they did not always find the most scientific solutions, those which they did bring forward were marked for their daring and practical character.

And it happened sometimes that some of these solutions, obtained uniquely in the experimental way, proved superior to those of the European engineers; this was the case, for example, with the turbine.

But life becomes more and more difficult even for the most prosperous nations such as yours and the problem becomes singularly complicated when it is no longer simply a question of supplying a national market, largely open, but of entering into commercial competition, from within and from without, with new industries strongly organized. It becomes necessary then to reduce the net cost to the minimum, searching out the most rapid and most economical processes, allowing no loss of anything which represents any value.

Furthermore, the terrible war which has been imposed upon us and in which we are fighting side by side for the triumph of justice and of liberty, this terrible war has demonstrated that there exists for each large nation a certain number of vital industries which it cannot neglect without exposing itself to the danger of some day being at the mercy of its enemies. The nature of the problem changes. Scientific and technical in-

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[1] Translation of address delivered in French at Mellon Institute on December 8, 1917.

struction become necessary and in this your universities succeed wonderfully.

But this again does not suffice; it is necessary that the manufacturer should not allow himself to be elated by his earlier success and should understand the necessity of commencing, or rather, of continuing the contest, without waiting for this prosperity to be menaced by better-informed competitors. And that still is not all; when the manufacturer has comprehended his real concern it is not always possible for him to erect suitable laboratories, to endow them with the necessary equipment, and above all, with a specially trained personnel.

It is then that institutions like the Mellon Institute come into play, a production of genius which has solved in a manner truly American a problem already old but without satisfactory solution in Europe—that of direct collaboration between science and industry. In visiting it the day before yesterday I took account of its strong organization and of the incalculable service which it can render.

Between the too naïve disinterestedness of the majority of the French investigators and the German form, fruitful without doubt, but too materialistic, which has made of too many German scientific schools veritable industrial laboratories, for the glory and above all the profit of their directors, there was room for a solution considering the rights of the manufacturer but also considering the dignity of the university. This solution you have found and the success of Mellon Institute since its foundation proves that the way in which you have been doing it is the proper one.

Without doubt it will be necessary to struggle for a long time yet against acquired habits, as we say, against the routine, but further it will be necessary that the manufacturer have well impressed on his mind this truth: in the period of beginning the improvements to be realized are numerous, researches give results rapidly, but little by little the framework contracts, investigations require more and more specialized work, more time, and more money. And it will not be necessary that the manufacturer's interest should clash with that of research, the more difficult it becomes the more raison d'être will it have in the midst of the great economic struggle which, after the war, will array, on a new account, one part of the world against the other.

France, who has never considered herself behind in the march of progress, cannot fail to organize all her forces for this collaboration, too much neglected by her, of science and industry. She cannot be inspired by a better model than the Mellon Institute. Again, it will be necessary that our manufacturers accept the necessary sacrifices for the foundation of laboratories and of scholarships for research. But we can, I trust, have all confidence; the war has opened our eyes and demonstrated once again the truth of the proverb, "He who does not advance, falls behind."

I, therefore, salute with all my heart your magnificent Institute which I consider a wonderful instrument of scientific and industrial progress.

PERKIN MEDAL AWARD

The Perkin Medal for 1918 was conferred on Auguste J. Rossi, in recognition of his distinguished contributions to the metallurgy of titanium, at the meeting of the New York Section of the Society of Chemical Industry, held at the Chemists' Club, January 18, 1918. Introductory remarks by Mr. Jerome Alexander, Chairman of the Section, were followed by an address on "A. J. Rossi and His Work," by F. A. J. FitzGerald, Past President American Electrochemical Society. The presentation of the medal by Dr. William H. Nichols, Past President of the Society of Chemical Industry, was followed by an address of acknowledgment by Mr. Rossi. The addresses are printed in full herewith.

The usual informal dinner was held before the meeting in the dining hall of the Chemists' Club, giving the members the opportunity of meeting the recipient of the medal.—EDITOR.

INTRODUCTORY ADDRESS

By JEROME ALEXANDER

Nothing could be more illustrative of the cordial solidarity that unites us to our Allies, England and France, than this evening's meeting; for here before the New York Section of a British Society we are about to award the Perkin Medal to Auguste J. Rossi, a native of France.

France! How the heart of every true American, yes, of every true democrat throughout the world, leaps at the mention of that name! Fair are her fields and beautiful her cities; but France is much greater than all these—her artistic, literary, scientific and spiritual gifts to humanity and civilization have made us her debtors for all time; and to-day the civilized world is fighting under her slogan of "Liberty, equality, fraternity."

As a consequence of this dreadful war, and under the lash of its stern necessities, we have fortunately been brought in closer relationship with our French fellow chemists. For some months past we have been planning the formation of a New York Section of the French *Société de Chimie Industrielle*, the sister society of the British *Society of Chemical Industry;* and to-night, immediately following our meeting, it will be formally organized. We will, therefore, proceed with our program, and hear an account of Mr. Rossi's life and work from a gentleman well known to you all, Mr. Francis A. J. FitzGerald, Past President of the American Electrochemical Society.

NATIONAL GUM AND MICA COMPANY
NEW YORK CITY

MR. A. J. ROSSI AND HIS WORK

By F. A. J. FITZGERALD

It was, I think, in the year 1899 that I first made Mr. Rossi's acquaintance. There was much speculation in Niagara Falls at that time as to what he was doing. In those days there was always much gossip whenever some new work was started at Niagara Falls for it was still in the early period of power development before the MacFarlands and politicians got busy and we were all watching eagerly the rapid electrochemical developments,—aluminum, carborundum, caustic soda and chlorine, calcium carbide, etc. Wonderful stories would float about as to what newcomers were doing. When the Oldbury Chemical Company started, the most astonishing stories were afloat, caused I think largely by the high brick wall which surrounded the factory. It was told with bated breath that this works was engaged in the manufacture of cordite by a new and secret process and that you could not get inside the brick wall except over the body of an armed guard.

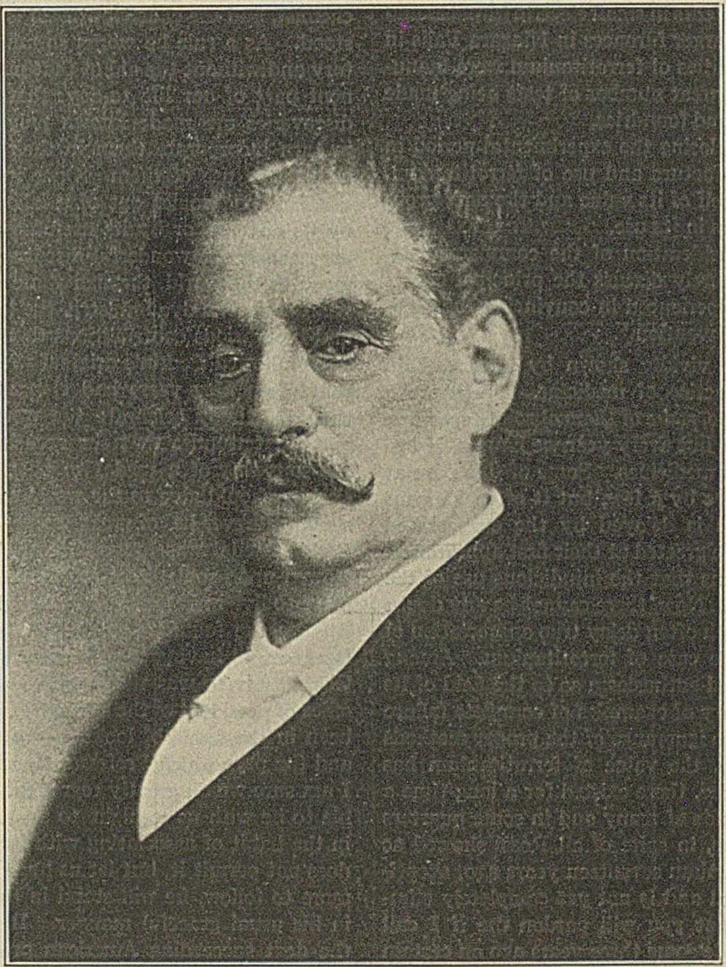
I cannot recall now what various stories were told about some remarkable work in the Porter house and the old stone barn which stood where the Acheson Graphite Company's plant now is, but these were sufficiently startling, and all agreed that it was a French chemist named Rossi who was working on some new invention.

I soon made Rossi's acquaintance, for in the electrochemical work of those days there was a fine spirit of coöperation, which indeed has become to a great extent permanently characteristic of Niagara Falls, and before long I got to know him very well. During visits to him I learned a great deal about his researches on the smelting of titaniferous ores, about the experiments he was then making on the manufacture and use of ferrotitanium and about many other chemical and engineering works with which he had been associated in his varied professional career. Although I may repeat some things which Mr. Rossi speaks of himself, I believe you will be interested in a brief history of his work.

In the year 1855 at the early age of 16 Rossi graduated from the University of France with the degree of Bachelier des Sciences et Lettres and four years later, after he had also graduated from the École Central of Arts and Manufactures, where the studies covered mechanical, civil and metallurgical engineering work, he came to New York. Here he soon obtained a position as assistant engineer with the Morris and Essex R. R., holding this until 1864 when the railway was taken over by the D. L. and W. After this he got a job which kept him busy for a year making the survey of a property in Boonton, N. J., which was to be converted into a park under the direction of Mr. Calvert Vaux, the architect of Central Park, New York. Next we find him engaged in building a railway to the Boonton Iron Works; but when Fuller, Lord Company of that concern found out that Rossi had studied chemistry and metallurgy at the École Central they put him in charge of a laboratory at the Iron Works.

It was here that Rossi's attention was first called to titanium, for the ores used in the blast furnaces at the Iron Works were magnetites from Morris County, N. J., containing in the best samples about one per cent titanic oxide and in others 2.5 per cent or more. Beyond noting the existence of titanium in the ores Rossi was not particularly interested in it until he met the work of Professor Cook, the State Geologist of New Jersey. This work called attention to the existence of titanium in nearly all the New Jersey ores, from fractions of one per cent to as high as 15 per cent titanic oxide. Professor Cook also established a relation between phosphorus and titanium in the ores, low phosphorus apparently going with high titanic oxide content. This set Rossi to work hunting up all the scanty, and incidentally very contradictory, literature he could find on the subject of titanium. None of this work was of any practical importance so far as the running of the Boonton Works was concerned, but several years after he had left his metallurgical work to devote his attention to tests on the pumping engines at Fall River, to calculations of the stability of the Beaver Bridge Dam, to work on refrigeration and the manufacture of ice-making machinery, his researches on titanium became of value because of a law suit in relation to his old firm of the Boonton Iron Works.

This law suit was of great importance so far as Rossi's future work was concerned as it no doubt established his reputation as an expert in the smelting of titaniferous iron ores. Thus, after Rossi had established an office in New York as a consulting engineer and when in 1890 Mr. James MacNaughton was interested in the development of the immense titaniferous ore deposits of the Adirondacks, it was to Rossi that he went for advice. Since that date Rossi has devoted most of his time to titanium.


AUGUSTE J. ROSSI, PERKIN MEDALIST, 1918

The problem presented in this case was a study of the feasibility of smelting the titaniferous ores of the Adirondacks. Rossi, as a result of his researches years before, was well acquainted with what had been done in Europe in this field and of special interest were the records he had of some blast furnaces near Stockton-on-Tyne, England, where ores running as high as 35 per cent titanic oxide had been smelted successfully by forming a slag that consisted of a silico-titanate of lime. This, of course, involved adding considerable quantities of lime and silica to the charge. Here lay the great objection to the Stockton-on-Tyne practice, for on account of the low iron content of the ore it was necessary to make about 4 tons of slag for 1 ton of iron.

Rossi told his client that success with the Adirondack ores could be obtained provided that the titanic oxide could be slagged off without using an enormous excess of fluxes and that so far as the pig iron produced was concerned, it would probably be of a very superior quality.

The upshot of the consultation was that Rossi went to examine the ore deposits and two small blast furnaces which had been run there by Mr. MacNaughton's grandfather from 1840 to 1858. In a curious old iron chest Rossi found the blast furnace records. Studying these and knowing the composition of the ore, the fluxes added and the amounts of these going into the charges of the old blast furnaces, Rossi found that the practice of the Stockton-on-Tyne furnaces had been anticipated. He also found that the iron produced was of such superior quality that a Gold Medal had been awarded to it in the great London Exhibition of 1851. Moreover, certain steel made from this iron won high commendation from the Navy Yards of Springfield and Washington, the report stating that it compared favorably with the best Swedish steel.

Rossi then began a series of experiments on slags with the object of replacing silica with titanic oxide in the normal blast furnace slag and, following his success in this direction, built what might be described as a laboratory blast furnace in which several hundred pounds of excellent pig iron were made from the titaniferous iron ores. Next followed a small blast furnace built at the New York Car Wheel Works in Buffalo, where the Mill

Pond ore running 16 to 17 per cent iron was successfully smelted.

In this way Rossi did the pioneer work in demonstrating the value of the great ore deposits of the Adirondacks in 1894. Twenty years later we find this work confirmed by Mr. Frank E. Bachman, General Manager of the MacIntyre Iron Company, Port Henry, N. Y., in an interesting paper, "The Use of Titaniferous Ore in the Blast Furnace," presented to the American Iron and Steel Institute in October, 1914.

Turning now to ferrotitanium—Rossi was convinced from what he had observed in the work on smelting titaniferous ores that titanium had a beneficial effect in the manufacture of iron. A natural deduction from this was that an alloy of iron and titanium would be of value for the treatment of iron and steel in the process of manufacture.

This led to a series of experiments in small electric furnaces and finally to the construction of larger furnaces in Niagara Falls in 1899 where considerable quantities of ferrotitanium were manufactured, thus permitting of a great number of tests being made on a large scale in steel works and foundries.

This may be considered as closing the experimental period in the development of the manufacture and use of ferrotitanium. Rossi had now convinced himself of its value and the next problem was the education of others in its use.

I shall not go into the consideration of the commercial development of titanium, but merely discuss the principal causes of the great difficulty Rossi experienced in convincing the technical world of the value of his invention.

Perhaps this may be most vividly shown by means of an analogy presented by the practice of medicine. There are a number of drugs of known value, some of them absolute specifics, in the treatment of human ills, but every year we have added to our drug list a number of new medicines. Some of these are at once recognized as of real value by a few, but it often happens that their general recognition is delayed by the exaggerated enthusiasm with which they are greeted at their first appearance; moreover, it frequently happens that the injudicious use of the new drug gives it a bad name. Now, there cannot be any doubt that the very same causes which often delay true appreciation of a new medicine existed in the case of ferrotitanium. Among some there was an unwarranted enthusiasm as to the field of its usefulness and there were plenty of examples of complete failure in its application due simply to ignorance of the proper methods of using it. While, therefore, the value of ferrotitanium has always been recognized by some, there existed for a long time a prejudice against it in the minds of many and in some quarters this exists even to-day. Thus, in spite of all Rossi showed as regards the possibilities of titanium seventeen years ago, skepticism was for long very general and is not yet completely eliminated. I hope, therefore, that you will pardon me if I call attention to a brief note I made some three years ago on certain statistics in regard to the use of titanium in rail steel, which I think very clearly disposes of the assertion frequently made at that time, that titanium has no effect on steel.

In this note are given the statistics of 155 heats representing the production of 9000 tons of rail steel, in some of which titanium was used while in others it was not. Where no titanium was used only 36 per cent of the steel came within the particular specification limit. Where 0.053 per cent of titanium was used 43 per cent passed, with 0.077 per cent titanium 84 per cent, and with 0.10 per cent titanium 100 per cent passed.

In the earlier days of the exploitation of titanium the chief drive was made on its use in rail steel and the data I have given show something of what can be done in that way. But of recent years much greater efforts have been expended in applying titanium to miscellaneous steels. Thus in the last ten years the ratio between titanium going into other kinds than rail steel has enormously increased, for while a decade ago the miscellaneous applications were only 12 per cent of the total, they are now 96 per cent and have increased in volume about 150 times.

Thus Mr. Rossi, after more than a quarter of a century of work followed with the greatest perseverence and undaunted by difficulties of all sorts, has not only shown what can be done with the titaniferous iron ores formerly regarded with distrust by the iron smelter, but has demonstrated that the very element supposed to make these valueless can actually be used for improving the manufacture of the metal they yield.

The FitzGerald Laboratories, Inc.
Niagara Falls, N. Y.

PRESENTATION ADDRESS

By William H. Nichols

The world is beginning to get a gleam of what it owes to the chemist, and the chemist himself is beginning to be better understood. As a rule he works so much in the quiet of his laboratory and without the aid of that publicity which is such a prominent part of our life to-day, that many of his most important discoveries are made known only to his fellow chemists, who in turn incorporate them in their own fund of knowledge and thus make use of them. Once in a while, however, something very striking comes out of a laboratory and attracts attention by its novelty, or by its usefulness, or both. Under this category comes the wonderful discovery of young Perkin which has given to the world, through those who have succeeded him, the great synthetic dye industry, the long list of synthetic remedies, and the various contributions to the "gentle art of war," which have followed Perkin's original work. What wonder, therefore, that when fifty years had elapsed, the chemists of the world should fittingly celebrate the work of the young Englishman, and what wonder that the occasion should be seized upon for the founding of a medal to be given to those who, following Perkin, should give to the world something of themselves which should forever be of great value to all mankind. Thus the Perkin Medal was founded, and thus it has annually been bestowed upon some great man who has made the world a better place to live in because he has lived in it himself.

Since the founding of the Medal, we have been exceedingly fortunate in having it presented annually by our own Professor Chandler, who was the first American selected to fill the great office of president of the Society of Chemical Industry. We have been in the habit of looking forward to these annual occasions and the part which Professor Chandler would take in them, and I am sure we all greatly regret that he has not found it practicable to be with us to-night to assume this duty which we are so in the habit of associating with him. Unfortunately, his health does not permit it, but let us hope that next year, and for many more to follow, he will stand in this place and perform this act in his usual graceful manner. It, therefore, falls to my lot as the next succeeding American president to present the Medal, and this task I undertake with great diffidence, feeling, as I do, the disappointment which you all sustain in not hearing from the grand old man himself. It is not, however, the first time in which I have undertaken this work, as I had the honor of presenting the first Perkin Medal to Perkin himself during the jubilee celebration in New York.

It is particularly appropriate that this Medal should be presented at one of the stated meetings of the New York Section of that great English Society of Chemical Industry which counted among its presidents so many great Englishmen, including Sir William himself. During the recent terrible years, the members of this Society have almost remade Great Britain from a chemical standpoint. Similar work of the highest grade has been accomplished by our French brethren. American chemists have taken great interest in the British society, and they are now to have an opportunity of doing similar work for the Société de Chimie Industrielle, the New York Section of which is to be formed to-night. We have always had a warm place in our hearts for our French friends and I am glad that we are going to have an opportunity of again proving this, if any proof be necessary.

You have heard from Mr. FitzGerald a statement of some of the work which Mr. Rossi has done and it is not my intention to add to that list a single statement. If Mr. FitzGerald has omitted anything, Mr. Rossi himself will please supply as much of the deficiency as his modesty will permit.

CONFERRING THE MEDAL

Mr. Rossi, there have been several candidates for the honor which is to be conferred upon you to-night. The claims of each have been carefully weighed by a committee, whose only desire has been to select the candidate best qualified to receive the Medal this year, when everything has been considered. You have been their choice, and are, after a long life full of hard work and the usual disappointments that come to every successful man, to receive the greatest reward which your fellow chemists can bestow upon you. You are joining a body of very brilliant men. You may well feel gratified that those who know best consider you worthy of the honor of that company, and in token of that selection I beg to express to you, what I am sure is the thought of every man in this room and of every other who knows of your work, our hearty congratulations on a life well spent, and our cordial "bon voyage" as you turn your face to the setting sun. Coupled with this is the hope that you have before you yet many days of usefulness, and that you will be able to return our confidence with something accomplished of even greater value than what has gone before. With the heartfelt respect of the entire chemical fraternity, and a hand-clasp as its token, it is my privilege to present to you the Perkin Medal of 1918.

New York City

ADDRESS OF ACCEPTANCE

By Auguste J. Rossi

I am not much used to making public speeches. True, once upon a time, I was asked by the president of the Polytechnic Department of the American Institute to deliver a lecture on "Ice and Refrigeration" at Cooper Institute. It seemed to be appreciated, at least so the president told me, but it was very long ago, so long that I am afraid I have forgotten about it. It was on a subject with which I was familiar, but to-night I have not only to speak on a definite subject of applied science, but to express my feelings for the distinction you have conferred on me and of which I appreciate all the honor. I have had occasion to notice that sometimes brilliancy of diction covers a multitude of sins, but to such brilliancy I cannot lay claim. However, there is another aspect to the question to-night—one which tells—it is the part which comes from the heart, the part which makes one say what he feels, not the way in which he expresses it, and that is precisely my case to-night, so I will let my feelings speak, not my oratory.

By your conferring on me this honor, I see an appreciation, a recognition of a continuous and continued work in lines which had not been much investigated before, so far, of course, I mean, as their industrial possibilities. I thank you heartily for this honor, personally, but also because it will prove an incentive for me and for others to devote to any researches they may pursue, this truthfulness, this interest that any professional chemist or technical investigator worthy of the name must give to his work. This I have tried to do and I see to-night with all my heartfelt thanks and appreciation that it is to this devotion to science, to this perseverance in working out a comparatively new problem, to the sincerity of the work done as much as to its value, that you have conferred this honorable reward to stand as a beacon to guide others, entering young in the career, to be as thorough, as devoted to an idea, as conscientious in recording results, as they can be. It is in part, I am sure, to this perseverance in trying to overcome such difficulties as have arisen, such criticisms and doubts as have been expressed that you have given your appreciative recognition by this honorable distinction you have conferred on me, and which can only encourage me, even at this stage of my life, to persevere to the end and develop certain possibilities of titanium compounds for other than metallurgical purposes, in other branches of industrial chemistry; and now that I have let my feelings speak and express how proud I am of the appreciation of my peers, I may be allowed, as an interesting history of the case, to explain to you, as briefly as possible, what little encouragement I could find in what had been written on the subject in a literature curiously contradictory and so many times misleading.

My special attention was called to the subject of the possibilities of titaniferous iron ores in 1890 by Mr. James MacNaughton, a graduate of Yale, interested in these immense Adirondack deposits, as an inheritance from his grandfather, Mr. MacIntyre—deposits of such a magnitude that Mr. Berkenbine, then president of the American Institute of Mining Engineers, at the Montreal meeting of the Institute in 1893, said of them, "these marvelous deposits seem to have been placed by Providence where most inviting, as they can be made available to tide water." I had been directed to Mr. J. MacNaughton by Professors Chandler and Rickett, as one who could give information on this subject as I had had occasion before to write a paper entitled "Titanium in Blast Furnace" for the *Journal of the American Chemical Society*.

Having been for some eleven years (1864–1876) technical director of the Boonton blast furnaces (Boonton Iron Works, Fuller, Lord Co., proprietors), I *knew* from actual practice that the presence of titanic dioxide (TiO_2), averaging 1.50 per cent in Morris County, New Jersey, ores which we were smelting and 2 to 2.50 per cent in our slags, had had absolutely no effect on the working of our furnaces, or the behavior of our slags as to fluidity and fusibility, so much so that I even ignored its presence, considering it as so much additional silica in the ores.

And still when the question of smelting these ores, alone or in mixture, arises, one can read in the proceedings of a well-known scientific technical society (A. I. M. E.) "that 1 per cent of TiO_2 in a slag was enough to make it pasty to impossibility of tapping." The advanced copy of the paper (in my hands) says, "the slags had to be pulled out with tongs." How can young men, new in the profession, be blamed for being skeptical about the smelting of this class of ores after such statements as I have quoted verbatim?

In 1876 the death of the two owners of the Boonton Iron Works and the legal complications it involved between the estates forced the closing of the works, involving rolling mills, plate mills, puddling and re-heating furnace, nails machines, etc. The furnaces were blown off, but not dismantled. Workmen and all had to leave and the small town was deserted for several years.

Later on in the 80's, Mr. Eckert of Reading, Pa., who was making there what is called stove iron, having decided to use our puddled cinders of which we had the accumulation of years, with some of our ores containing 2 per cent TiO_2, leased the two blast furnaces which were still standing with all the blowing apparatus and appurtenances. They made a failure of this run for technical reasons foreign to my subject and which could easily have been foreseen by one familiar with blast furnace work. Having stopped the furnaces after a very few months' run, they tried to get out of their contract binding them to use a stipulated number of tons of puddled cinders and ores by claiming that the presence of TiO_2 in our cinders and ores had been the cause of their non-success. I was retained as an expert by the estate and had no difficulty in proving by my books of analyses and statements of founder and keeper and others that the presence of TiO_2 to the extent their own analyses had shown in the materials of the charges was in no way greater than the amounts that I had found during an eleven years' practice to have had absolutely no effect on the working of the same furnaces, so they had to compromise and pay a heavy forfeit. I had to enter into these details to render intelligible what follows.

The suit came before the chancellor of New Jersey and I have on hand the brief submitted by the plaintiff, containing all the sworn testimonies of their experts.

No. 1—One of the experts, Mr. X, manager of one of the Durham furnaces (Cooper, Hewitt and Co.), testified, under oath, that 0.75 per cent of TiO_2 in an iron ore rendered it unfit for blast furnace purposes as it would be only a matter of a short time before the furnace would be "clogged up" if the use of the ore were not stopped.

No. 2—Also a manager of another Durham furnace, Mr. Y, testified, under oath, that not 0.75 per cent but 0.25 per cent of TiO_2 in an iron ore excluded its use in a blast furnace, it being merely the matter of a little longer time before the furnace should be clogged up.

No. 3—Mr. Eckert, of Reading, Pa., under oath, testified that from his 20 years' practice in iron making in blast furnace, not 0.25 per cent, but *traces* of TiO_2 were sufficient to produce the same result as above mentioned, ores containing *traces* of of TiO_2 being unfit for blast furnace uses.

This is only one of the many examples, if really a striking one, of the criticisms, prejudices and objections I met with in my fight for these much-abused ores. Such was the encouragement I found in the profession. When in 1894, at the New York Car Wheel Works, Buffalo, N. Y., in a small furnace of a capacity of three to four tons a day, which I planned and erected, at Buffalo, I melted, without mixture, titaniferous iron ores from the Adirondacks, containing 15 to 18 per cent TiO_2 and 55 to 56 per cent metallic iron, the slags as run from the furnace analyzed 25 to 30 per cent TiO_2, with some 15 to 18 per cent silica with lime, alumina, and magnesia as bases, magnesia having been recognized by me as an essential constituent of the slags to insure the best running. These ores, being inaccessible to a railroad, had to be transported on corduroy roads at that time to North Creek where they could be loaded on cars. It brought their cost to $15 per ton delivered at Buffalo. This excluded the idea of making a test on a larger scale, unless we could find a furnace small enough, one of some ten tons' capacity, which it proved impossible for us to do. Within the last two years, Mr. Bachman has smelted similar titaniferous iron ores in one of the large blast furnaces of Weatherbee-Sherman & Co., at Port Henry, making with these ores some 15,000 tons of a pig iron which he found in the tests for tensile or transverse strength of superior quality. In a very elaborate, comprehensive, scientific and technical report, Mr. Bachman has given the fusibility and fluidity of the titaniferous slags run in the furnace, analyses of all materials and results of tests for strength as compared with those of the average pig irons run without titaniferous iron ores in the charges. His conclusion as a whole is a confirmation of my own as drawn from this run in Buffalo in 1894–95 in my small furnace. One of them particularly is worth mentioning, as I had come to the same conclusion in Buffalo. He says, "This run with titaniferous ores was made with an economy of fuel per ton of pig metal smelted," and one can read in technical publications in 1894 that the smelting of titaniferous ores even if admissible meant such an excessive consumption of fuel per ton of iron, that on this score alone they are unsuitable, leaving aside titanium deposits in the furnace and pasty slags," an obvious contradiction. If the furnace is to be "clogged up," as claimed, by titanium deposits which remain in the furnace, they do not go into the slag and the latter need not be pasty on this score, or, if it goes into the slags making them pasty, it does not remain in the furnace as titanium deposit. These titaniferous ores of the Adirondacks are magnetic, associated with ilmenite ($FeTiO_3$), an iron titanate, just as iron silicate is found ($FeSiO_3$) in certain ores (ferruginous periodotite). They are like almost all titaniferous ores, low in phosphorus and sulfur, some as low in phosphorus as 0.017 per cent and sulfur 0.02 per cent, with 55 to 56 per cent metallic iron and little silica, 1.50 to 2 per cent. The iron smelted from them is an ideal open hearth steel and to-day open hearth steel has superseded Bessemer steel.

Tests made by me and in many foundries of such pig metal added to ordinary pig iron in certain proportions have shown an increase of strength over cast iron not treated, of 29.5 per cent in tests made and reported by Wm. Cramp and Co. and an increase of 4 per cent over cast iron treated with nickel.

These titaniferous iron ores, at least those found in the Adirondacks, are of plutonic origin. Professor Kemp told me they are not stratified or in veins but the metallic mass seems to have been pushed through the rock formation in fusion. He showed me photomicrographs in which you could see the rock, perfectly distinct and clear from ore, with big black patches of titaniferous ores free from rock. A corroboration of this fact is found by soundings made at the foot of a solid wall of ore, some 400 ft. wide and some 50 ft. high. The soundings at the foot of this ore wall went down 450 ft. of ore with only 10 per cent of rock, so the word inexhaustible seems not to be exaggerated as applied to these deposits as, on that property, the same kind of ore has been prospected on some 9000 acres. I have always claimed that these ores and similar ores would prove the resources of the future. Abundant, rich in iron, 50 per cent or more metallic iron without concentration, free from phosphorus and sulfur to such an extent as they are found to be, they are what were called Bessemer ores par excellence; and ores of this kind non-titaniferous, low in phosphorus, have been taxed to the utmost, so much so that what was called the Bessemer limit in phosphorus for steel making, had to be raised from a few hundredths of 1 per cent (0.08 or less) to 0.10 per cent.

If such work as I have done, confirmed by the very recent blast furnace runs on a large scale made by Mr. Bachman, has had and will have the result of calling the attention of the metallurgists to these much-abused ores, I will not have worked in vain.

But however much value these ores may have, alone or in mixture with other ores as blast furnace stock, their use for making alloys of iron or copper with titanium for the treatment of cast iron, steel, copper or copper alloys has opened a new and more important field of applications in metallurgy.

Checked by the difficulties I met in securing such large quantities of ore as are required for blast furnaces of the size and capacity of our present furnaces, as explained above, I proposed to Mr. MacNaughton in 1900 to make, so to speak, a concentrate of titanium which, added to ordinary cast iron or steel, would be likely to secure for the metal baths thus treated the same beneficial influence which tests had shown to be secured in pig metal smelted from these ores. TiO_2 not being reduced to any great extent by carbon at the temperatures prevailing in the blast furnace, I had to have recourse to electricity. And I may say here that Mr. MacNaughton was the one who supported me in these times of trial by his implicit confidence in me and in the possibilities of my work. He died in 1905.

About 1900 I started making ferrotitanium at Niagara Falls at the old Porter house by the electric current, the furnace itself, made of a masonry of graphitic materials, forming the cathode, and a carbon electrode, movable vertically in the cavity of the furnace by means of proper mechanical device or automatically by the current itself, forming the anode. It was an arc furnace. A mixture of the titaniferous material and of the carbon required for the reduction of both the TiO_2 and the oxide of iron, properly comminuted, was then charged gradually into the furnace as the current was turned on. Whenever the relative proportion of the TiO_2 and the oxide of iron of the titaniferous material were such as to produce an alloy higher in titanium than desired some scrap iron was charged with the mixture to dilute the alloy, so to speak. I made in this manner alloys containing from 10 to 25 per cent or more, but practice in steel and other works has shown that an alloy containing some 15 per cent of titanium, or thereabout, was best suited for most purposes.

Whenever the presence of carbon in the ferrocarbontitanium was considered objectionable for special applications, I reduced the titaniferous materials by my aluminum bath process. Instead of using a mixture of finely powdered aluminum and titaniferous materials as per Dr. Goldschmidt's Thermit process, the aluminum I used for the reduction of the oxides was melted in the electric furnace and the titaniferous materials (omitting the carbon) charged directly into the bath of aluminum. In such cases I used preferably TiO_2 as rutile, in order to avoid using the aluminum which would have been required for the reduction of the oxide of iron of the ore, charging scrap iron with the rutile to dilute the alloy to the content of titanium desired. In this manner I made ferrotitanium practically free from carbon, containing only from 0.18 to 0.50 per cent carbon more or less, and by reducing the amount of scrap iron added, I was able to make ferrotitanium containing as high as 80 to 85 per cent titanium. Titanium as a metal is as white and as shining as silver but very hard and brittle, so that, as such, it has no particular use. The high alloys of titanium, such as 75 to 85 per cent of titanium and even those with less titanium, are also white like silver and scratch glass and even quartz deeply.

This aluminum bath process is of a more general application. If into the aluminum bath a tungstic ore containing oxide of iron is charged, a ferrotungsten is obtained free from carbon. I have made thus 85 per cent tungsten alloy.

By using manganiferous ores I was able to make in the electric furnace 85 to 88 per cent ferromanganese free from carbon; by using chromic iron, a ferrochrome at about 80 per cent chrome, free from carbon.

The advantages of the electric furnace and of the use of baths of aluminum lie in the fact that if the reduction can be secured in a blast furnace as for ferromanganese, for ferrochrome, ferrotungsten, and other ferroes when high in the constituent metal, the ferroes obtained would be melted with difficulty, if at all, in the blast furnace in certain cases.

By adding to a bath of steel, for instance, or to cast iron a ferrocarbontitanium containing some 15 per cent of titanium, the bath of metal was cleansed of dissolved or occluded gases such as oxygen and nitrogen and also of such oxides of iron generally present in steel, especially in Bessemer steel made by the pneumatic process. When air is blown through the molten pig iron, the titanium of the alloy combines with the oxygen to form TiO_2, with the nitrogen to form titanium nitride, and the oxide of iron is reduced to iron by the titanium and the carbon of the alloy, the titanium combining with its oxygen to form TiO_2, and the slag, carrying with it the titanic dioxide and the titanium nitride, rises to the top of the ingot or ladle, the TiO_2 rendering the slag more fluid and fusible as experiments I have carried on for Dr. P. H. Dudley, the expert of the N. Y. C. R. R., have proved. A slag collected by him at the top of an ingot and containing 6.5 per cent of TiO_2 had a melting point of 1290° C., lower than or about the same as that of ordinary blast furnace slag. By adding enough TiO_2 to such a slag as to have in the slag some 13 per cent TiO_2, its melting point was lowered to 1190° C., as determined by Mr. FitzGerald. This infusibility of titanic slag was another objection I met to the use of these ores; it can be read in the Geological Survey of New Jersey in an article on the refractory properties of fireclay that 3.50 per cent of TiO_2, frequently met with in these clays, lowers their melting point two or three cones of Ziegler. "So," it is added in this report, "TiO_2 must be considered in these clays as a flux." As traces, or only a few hundredths of 1 per cent at most, of titanium are found in the steel treated, the titanium seems to act as a scavenger, as it has been called sometimes.

The presence of titanium in steel to the extent of 1 per cent or more imparts to the steel certain very special properties to which, later on, I may have occasion to call attention more particularly. The head of a crucible steel ingot treated with titanium, cast in 1900 at Atha & Illingworth's, East Newark, was drilled with five holes to obtain specimens for analysis. These holes are as bright now as they were when first drilled, though exposed to the air in my office. It contains 1 to 1.10 per cent titanium and the sheet was so hard that it was drilled with difficulty.

Copper, as is well known, absorbs, when melted and exposed to air, a large amount of gas and the bath contains oxide of copper. For the treatment of copper and its alloys such as bronzes, brass manganese or aluminum bronzes I have made a coppertitanium containing any amount of titanium, though 10 to 15 per cent appears best adapted. The titanium of such an alloy acts, as in steel, as a scavenger of the copper bath. I have also made aluminumtitanium containing 45 per cent titanium for seasoning aluminum bronzes. They are made in the same manner as ferrocarbontitanium or ferrotitanium in the electric furnace. For coppertitanium, TiO_2 and aluminum are charged in the furnace in a bath of copper. For aluminumtitanium, the TiO_2 is charged directly in the bath of aluminum. The reaction of aluminum on oxides being *exothermic*, much less current is required for the reduction proper.

As is well known the affinity of titanium for O and N is such that the metal burns at 800° C. or thereabout in an atmosphere of these gases (Wöhler and St. Claire Deville). It is such that when large cakes of alloy free from carbon, such as we make, are cast in the casting trough from the furnace at a dazzling white heat and broken while hot, the section exposed to the air colors itself with fine irisations presenting all the colors of the rainbow; blue (probably TiO), gold (probably nitride), copper color (probably cyanonitride formed by the small amount of carbon still present in the alloy) and combinations of these colors. I have been able to reproduce these irisations artificially on small pieces of alloy. These pieces look like gems, scratch glass deeply and even cut it. I had occasion to show samples of these at a meeting of this Society last year. All the details of these operations have been fully described in the patents which I have secured for our company, as also in many articles which I have written on this subject in technical publications here and abroad. In short, this modest beginning at the Porter house has developed into the manufacture of these alloys for iron and copper on a scale which requires the labor of several hundred men.

Some of my work has been in an entirely different line. I have used for purposes of artificial production of cold instead of liquefied gases such as sulfur dioxide, carbon dioxide, or ammonia, a binary liquid composed of one moderately volatile liquid holding in solution the vapors of a much more volatile liquid. In ice-making and refrigeration the volatile liquids are charged in a special vessel, the refrigerator being immersed in a bath of uncongealable liquid so called, such as a strong solution of sodium or magnesium chloride, or glycerine; or indirectly, by causing this liquid to circulate in a tank in which cans, containing the water to be frozen, are immersed, or circulated through pipes in the cellars of the brewery or storeroom. By relieving the pressure of the volatile liquid on itself, at ordinary temperature, by means of a double-acting pump run by any power (steam engine or other), the liquid boils and by its latent heat of evaporation cools the refrigerator contents. The vapors entering the pump are compressed in a condenser cooled by circulating water, are liquefied again under this pressure and cooling, and combined again, re-forming the original volatile liquid, which is returned by proper devices to the refrigerator, rendering the operation continuous.

One of the binary liquids I proposed and used for machines erected in breweries and cold storage plants was ordinary ether holding in solution sulfurous dioxide. Ether will absorb its own weight of SO_2, showing but a very few pounds of pressure (5 to 6 lbs.) per sq. in. at 90° F. By using such binary liquid instead of SO_2, for instance, when the mixed vapors enter the compressor to be compressed and discharged into the condenser, ether liquefies *first* being cooled by the circulation of water around the condenser, absorbing anew the SO_2 and causing the pressure in the compressor and condenser to reach the figures it would have reached had SO_2 alone been used, thus saving on the mechanical power required. I have a patent in collaboration with Mr. Tessié Du Motay for this system of refrigeration.

The other liquid I used was sulfurous dioxide which can absorb carbonic acid gas to the extent of some 5 to 10 per cent of its weight. In this case, the same advantages as with the ether binary are secured, but to a much greater extent. CO_2 gas boils at about 100° F., and the pressures required to liquefy its vapors are considerable. On this subject of artificial refrigeration and the thermodynamic questions it involves, I have contributed several articles to the technical paper *Ice and Refrigeration*, published in Chicago, and to other technical publications here and abroad.

In the domains of metallurgy I have written a paper which I read at the meeting of the Electrochemical Society at Lynn and Cambridge, Mass., on the utilization of the blast furnace waste gases for generating power in gas engines for electrical purposes or other. In districts in which pig iron is manufactured on a large scale, as in the Pittsburgh district for example, and even in others, the amount of these waste gases is far above what is necessary to heat the blast and I proposed to use this surplus in gas engines to generate power which could be utilized for the blast engine, leaving a large surplus for generating electricity for illumination, making alloys of metal, or any other purpose. In the paper I have written on this subject, assuming the most conservative figures for the volume of such waste gases and their calorific potentialities, it could be shown that after having utilized for the furnace itself a part of it, a surplus of some 1,000,000 H. P. could be obtained in the Pittsburgh district and some have placed this figure at nearly 2,000,000 H. P. But assuming even a possibility of only 500,000 H. P., what a waste of power easily saved with gas engines, at a time when waste counts for so much! Even in districts much less favored than the Pittsburgh and more or less isolated, the same possibility would exist for local purposes, and were electricity to be generated it could be transported and utilized through a very extensive radius. At the Falls we send electric power to Buffalo, some 23 miles distant, and even to Syracuse, over 200 miles away. A careful scientific study of this question appears to us to justify a more complete investigation.

Before dismissing this subject of titaniferous iron ore, I will say a few words on the results I have obtained in experiments made in smelting titaniferous ores in mixture with phosphoric ores even to the extent of obtaining a pig iron high in phosphorus. I used phosphoric ores containing as much as 1.50 to 2 per cent of phosphorus pentoxide (P_2O_5), even adding apatite (lime phosphate) to the mixture so as to obtain a pig iron as high in phosphorus as I could. Contrary to what might have been expected, the pig metal containing 0.40 per cent titanium and from 2.50 to 3.50 per cent phosphorus had the strength and the rating of fair No. 1 or No. 2 pig iron. This suggested treating phosphoric pig metal, generally weak and close grained, with ferrotitanium so as to incorporate a divided amount of titanium in it. The results were very encouraging and were published by me in a paper read before the A. I. M. E. at the Pittsburgh meeting in 1896. They were commented on in the *Engineering and Mining Journal.*

I have proposed using copper titanium to treat the copper which is used with gold and silver for coin or jewelry. The use of the alloy making the copper denser and harder, the gold and silver alloyed with such copper could be expected to be hardened, which for coins would prove quite a saving in circulation or in transit. The Philadelphia Mint considered this scheme very favorably and asked us to make such alloys for them to try. Obviously if there is anything in the scheme, such tests to have any value should be made officially by the Government, and I did not give any more special attention to it.

But if titanium alloyed with other metals has found important and increasing applications in metallurgy, if its ores have been shown to supply a valuable stock for blast furnace smelting, other of its compounds possess such special properties as to justify industrial application in other lines.

If a ferrotitanium, practically free from carbon, made by my aluminum bath method, preferably as high in titanium and low in iron as possible, is dissolved in hydrochloric acid, in which it readily dissolves at a gentle heat, a fine violet solution is obtained, a ferrous titanium chloride, and with such violet solution I have been able to bleach cotton, silk and wool. As is well known, silk and wool cannot be bleached in agents capable of generating free chlorine which injures and attacks these fabrics. Still I was able to bleach completely some 2000 yards of such fabrics by digesting them in this diluted violet solution at boiling temperature, for a shorter or longer time according to the intensity of the dye to be bleached or the yellowish tint of the white fabrics. Silk and wool, *colored* or yellowish, were thus bleached without injury to the fabrics. Such double chloride contained about 40 to 50 per cent titanous chloride, $TiCl_3$, the balance being ferrous chloride. During the operation, especially as the temperature approached the boiling point, TiO_2 precipitated, thus showing clearly that the chlorine must have been set free, and, as I explained it or tried to, must have combined with the ferrous chloride to form ferric chloride, the chlorine generated acting in the nascent state without even appearing as free chlorine in the solution. The experiment repeated several times with the same success was conclusive and suggests the possibilities of a very important application.

The fact that the TiO_2 was precipitated from one of its compounds by these organic matters suggested to me the possibilities of precipitating TiO_2 from other of its compounds by vegetable or animal organic matters and the results were very remarkable. If to a solution of titanic sulfate, $Ti(SO_4)_2$, is added a water extract of vegetable or animal substances and the liquid gently digested to boiling and boiled for a short time, TiO_2 is precipitated, and when calcined is obtained as a soft, smooth, flour-like, pure white powder requiring no mechanical pulverization. This is so characteristic of the action of these organic substances that a number of them, such as water extracts obtained from dry leaves, green leaves, sawdust, tannin, wood pulp, wood-pulp liquor, horsechestnuts, beans, docks, cranberries, radishes, etc., and urea itself, have given the same result, a TiO_2, smooth, soft, pure white. One of the interesting features of this process of obtaining TiO_2 as a pure white, smooth, and impalpable powder is that if this same titanic sulfate solution be precipitated by NH_3 or Na_2O and boiled, and the TiO_2 calcined, the product is granular and buff-colored. Even if this solution is freed by proper treatment of such metallic oxides as are found in the ores and remain in the solution during the process of extraction of the melt, the TiO_2 precipitated by alkalies is white but granular, requiring mechanical pulverization for special uses. As by addition of these organic extracts to the solution of crude titanic sulfate, the TiO_2 precipitates white and smooth by boiling, it must be that these metallic oxides are likely to form with these organic matters, compounds which are retained in the solution while the TiO_2 precipitates. This seems to be confirmed by experiments I have made, adding to the crude solution iron oxide, copper oxide, manganese oxide, dissolved in sulfuric acid. By boiling such solution with the same organic compounds as I had used with the crude titanic sulfate solution without these additions of metallic oxides, the TiO_2 precipitates as a white, smooth, impalpable powder. The patent for this method of obtaining TiO_2 *white* and *smooth* by means of organic substances has just been granted within the last two or three weeks.

This method finds a direct application in the production of a white titanic pigment, a product which our company, working under several other patented methods, is to manufacture on a large industrial scale in a special department of our works. TiO_2, calcined, possesses the remarkable property of covering when used as paint material alone or in mixture with such other pigments as are used in the trade for paints. Its superiority on this score when prepared with proper oils over white lead or zinc white is remarkable, and in addition, TiO_2 pigment is not attacked by sulfurous gases and is stable under climatic and atmospheric influences.

I have found that TiO_2, obtained as a gelatinous titanic acid, $TiO_2.2H_2O$, and dried at 212° F., has the property of fixing coloring matters like that of alumina. It can be prepared by known methods, but the one I have studied is much more economical, as I can obtain it from a waste product of some of our manufacture.

In that state it dissolves readily in oxalic acid yielding a titanium oxalate which can be used in the same manner and for the same purposes as potassium titanium oxalate which is much more difficult and costly to make.

Thanking you once more for the honor conferred on me, I will show you some photographs of historic interest in the case.

1—A photograph of the small furnace which I planned and erected at Buffalo in 1894–95 to smelt titaniferous ores without mixture.

2—The old Porter house where ferrotitanium was made on an industrial scale for the first time as early as 1900.

3—The transformer room.

4—My primitive laboratory.

5—The furnace room.

6—The furnace in operation.

7—Titanium jewels and other specimens.

And for the convenience of those who might be interested and cannot see them to-night I will leave them at the Chemists' Club temporarily.

NIAGARA FALLS, N. Y.

BRITISH PROGRESS IN DYESTUFF MANUFACTURE

BRITISH DYES LIMITED[1]

By JAMES FALCONER, M. P.

Gentlemen, I rise to move the adoption of the report which has been circulated to the shareholders, and before doing so I would like to state that Sir Gilbert Claughton, one of the members of our Board, is unavoidably prevented from being present by public business of an urgent nature, which makes it impossible for him to leave London to-day; and Dr. Forster, another member of our Board, is at present in America conducting some investigations. The circumstances under which this report has been issued have been explained. The fact that we have not been able to submit a balance sheet is due to its having been found impossible, for an indefinite time, to arrange the allowances and other amounts which have to be adjusted with the Ministry of Munitions and the Inland Revenue Department, with regard to the munitions levy and excess profits tax. You can understand it is as much a disappointment to us as it is to you that we have not been able to submit these figures to you in the ordinary course, but it seemed to us to be better to have our meeting, as many other companies have, and do our business, rather than wait indefinitely before meeting one another to discuss the work of the year.

Our subscribed share and loan capital, as on April 30, 1917, amounted to £2,084,138, as compared with £1,851,914 at the commencement of the year, being an increase of £232,224. The number of our shareholders is now 1,445.

As regards the financial results of the year, it is not possible to make any satisfactory estimate, and I shall, therefore, refrain from submitting any particulars, except to say that I am sure when we are in a position to submit them they will be generally regarded as satisfactory. Our policy has been to charge prices for our products which would enable us to build up a fund sufficient to meet the great extra cost of constructing our works during the period of the war, and with that fund to write off our plant so as to reduce it to a reasonably low figure. We are satisfied that the results of the year will enable us to go a long way in this direction, to the extent of a good many hundreds of thousands of pounds, but I do not want you to get any exaggerated estimate of the profit we have made. Apart from the amount which is necessary to enable us to meet the depreciation to which I have referred and to pay the interest and the limited dividend, it will be no object of this Company to make large profits, because, being a controlled establishment, we should simply hand them over to the Exchequer. We are assured and have the authority of the Auditors for being satisfied that we may safely pay the shareholders 6 per cent. The amount of the interest on the Government Loan has been paid, *viz.*, £40,615. The dividend at 6 per cent on the ordinary shares will amount to £30,945, making together £71,560. Whatever we may make, 6 per cent, of course, is the maximum we are allowed to distribute.

Now, in dealing with the work which we have carried out during the year, I would like the shareholders to realize that we have three departments of work, each of them of great importance. The first is for carrying out certain work of national importance for the Government; the second is for the supplying of dyes for the immediate needs of our shareholders; and the third is for the building up of a national industry for the permanent supply of dyes for this country.

First, as regards the Government work to which I have referred; the situation did not permit me to refer to this question in the previous year for reasons that no doubt will be obvious. The position is that when we acquired the business of Read Holliday & Sons, Ltd., there were certain arrangements in force with regard to the work and certain negotiations were taking place. A few months after we took over the business the arrangements were all settled and new contracts were entered into for different products, all of them of real importance. I am glad to be able to say that we have punctually fulfilled all our obligations and at present our deliveries are all months ahead of our contract dates. At that time our deliveries were of vital national consequence and when you cast your thoughts back to the winter of 1915–16 and realize the extent to which the fate of our armies and our country was dependent on such supplies, I am sure you will agree with me that it is a matter of great satisfaction that we were able to do the part we undertook to do. I have heard, from time to time, the criticism that other companies were confining themselves to manufacture of dyes and

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[1] Chairman's Address delivered at the Second Ordinary General Meeting of the Shareholders of British Dyes Ltd., Huddersfield, October 31, 1917.

we ought to have done the same. That has not been our view either of our duty or of the true interests of the Company.

With regard to the immediate supply, by which I mean the supply of the needs of our shareholders during the period of the war, our output during the year has been substantially increased. Now the output of colors is more than three times the amount of the pre-war output, and when you consider that we have, in addition, made most of our intermediate products and many of our raw materials to enable us to increase our output, I think you will agree that this is a very substantial effort to have accomplished. Not only is there the question of volume, but, as you gentlemen know, there has been an increase in the variety of dyes which we have supplied. In addition to our own production we have continued, as far as the means of transport would allow, the supply of materials and intermediates to Swiss manufacturers to enable them to produce dyes and to send them back to this country, either through us or directly. The result has been that by our efforts and the efforts of other dye manufacturers, the users of dyes in this country have been kept supplied, not with all they would like to have or would require in ordinary times, but sufficient to keep their works running, and I believe there has been no unemployment in any trade through want of a supply of dyes. When you recall the situation at the time when the Company was originally formed and consider the apprehension which existed that workpeople would be unable to find employment through the complete failure of the supply of dyes, I think you will agree that the fact that we have so far succeeded is a matter on which we may congratulate ourselves.

I come now to the third branch of our effort, the laying of the foundation of a permanent supply of dyes for the country. I have said we have produced already a considerable number of colors which we were not able to produce before. We are producing a series of colors which, although far from what ultimately has to be done, is nevertheless a substantial list. We have during the year increased the plants for intermediates, some of them are in operation, others are being pressed forward, and we must always keep in view, in estimating the amount of work involved in the manufacture of these intermediates, that they require for their operation general services on a very large scale, steam, power gas, electricity, water, compressed air and also supplies of acids and other raw materials. To provide these services requires plant on a scale which can only be properly realized by inspection, and I am to express the hope, on behalf of the Board, that as many shareholders as possible will take advantage of the invitation to see the works. Every building that we have is already allocated to plant, a good deal of which has been erected or is in course of erection. To some extent the allocation is in respect of plant for which arrangements for erection are being carried through. I have heard the criticism that we have built too much. As a matter of fact, it is quite the reverse. We made it our policy to press on the building as fast as we could, knowing that difficulties of labor were going to increase, but we find that we have not enough in the way of buildings, and in order to accommodate the plant for necessary intermediate products we must erect further buildings. When you look at the number of buildings we have put up, then I think you will appreciate the effort which must be made in order to fill these buildings with plant, and to supply them with all the services necessary to enable them to be successfully operated.

Our procedure with regard to the erection of plant is as follows: We must first get the process worked out in the laboratory—I am dealing now with some processes that we have not hitherto worked. Then we have an experimental laboratory containing plant on a small scale, but with everything working according to the ordinary commercial conditions, and the process is put through that experimental laboratory. Difficulties are thus discovered and remedied and then the plant is erected on a commercial scale. All this takes time, because these processes involve great care and considerable delicacy in their operation and a very small error will upset a chemical process. When you find, therefore, that a plant has been put up you will know that a great deal of study and experiment has been devoted to the task, from first to last, until the plant is completely erected and working successfully. In addition to these works to which I have referred, we have purchased a site suitable for the erection of dwelling houses, because we see that a real problem is coming in regard to the supply of dwelling houses for our workpeople. There is a greater scarcity of dwelling houses in Huddersfield than probably in any other part of the country, and dwelling houses are very scarce everywhere. We are also providing canteens in our old works and in our new works for the convenience of our workpeople, and we have established a club for our chemists.

Now that is a very sketchy outline of the work that we have done, but I think I have said sufficient to justify me in asking the shareholders to acknowledge the efforts which have been made by Mr. Turner, by the chemists, by the engineers, by our workpeople and by all our staff, in doing this work during the course of the year with which we are dealing. All of them are overworked, all of them are full of zeal and, so far as the Board are concerned, they have all our most sincere gratitude for their loyal efforts on behalf of this Company during a time of so much difficulty. And I think the country also owes to them an acknowledgment of the very special work they have been doing, and doing with such complete success, so far as the national requirements are concerned.

Now at the same time, while I am bound to recognize the work which has been done, I am bound also to say, in perfect frankness, that it is only the beginning of the work that has to be done before you can have a really adequate supply of dyes manufactured in this country. In the report there is mentioned the magnitude of the German concerns with a capital of £35,000,000; and I dare say you all must know something of the dimensions of the German works. While of course their output has been far in excess of what was required for Germany alone, still one can form some opinion of what will be required here in the way of chemists, in the way of scientific work, in the way of staff, if we are going to reach our goal. I am not in the least disposed to take a despondent view but, on the other hand, I am not at all disposed to underestimate the task we must perform, and I think it is well that the shareholders should realize its magnitude. The Germans have been working on this problem for over 30 years, with thousands of chemists and with almost tens of thousands of experienced workmen; and we cannot hope in a year or two to come up alongside of them. It will require earnest work for many a long day before this country reaches the position to which it ought to attain in providing a supply of dyes sufficient for the needs of this country.

I should like to refer to the necessity of research. There are two ways in which research can be carried out. There are the scientists who study the properties of substances or groups of substances, seeking to learn the laws which govern their action without any immediate practical purpose before them. They go out merely seeking for truth. In this field Britain has always been supreme. If you think of the great discoveries made in science you will almost invariably find they are the work of great British scientists. The discovery of the principle of synthetic aniline dyes is an illustration of it. That was not discovered by a man who was looking for a method of making a mauve dye by a synthetic process. He was looking for something else altogether, carrying out, no doubt, his research in the most scientific and careful manner possible. Many other discoveries of the first magnitude have been made by British scientists. My belief is, and my confidence in the industrial future of this country is largely based on this, that the people of this country have a genius for original discovery and invention which is not surpassed by any other nation, and indeed, so far as the history of the past shows, no other nation has attained to our position in the field of original research.

But there is another branch of research, and that is the study of the application of scientific principles to particular problems and to improvements in methods of operation. Take a great example, the following out with extraordinary zeal and at great expense of experiments with a view to discovering how to manufacture synthetic indigo commercially. In this and many other instances where the original principles have been discovered in this country by British scientists, the application of them to industry had been left to other people. Others have reaped where our scientific men had sown. Here, we must admit, in the past, has been our failing. I am not going to discuss the vexed question as to who is to blame most. You must have an enlightened far-seeing spirit of commercial enterprise, with a body of scientific men sufficient to do all the scientific work necessary to carry forward discoveries to practical operation and to overcome all difficulties that arise in practical operations. My belief is that the reason for which the aniline industry left this country was that, at that time, there was not available a sufficient number of chemists qualified to carry through the operations and to overcome the difficulties. There was much enterprise among the commercial people and if there had been a body of chemists, trained and qualified to carry out chemical processes, then, I think the aniline color industry would probably have been developed in this country as it has been in Germany. I do not enter upon the dispute as to whether it was the commercial men who were to blame for not encouraging the chemists, or whether the blame attached to the chemists in that they did not adapt themselves to the requirements of the commercial men. The really important point is that they must combine. I have dwelt upon this question because I want to say, and to say it with all the power of which I am capable, that it is essential to the success of an enterprise such as ours that a sufficient body of qualified chemists be provided for the carrying out of the work of the future.

We have established in different universities, colonies of research students who are in our employment, and who are acting under the guidance of Professor W. H. Perkin at Oxford; Professor A. G. Perkin at Leeds; Professor R. Robinson at Liverpool, and we are greatly indebted to these gentlemen for placing themselves at our disposal for supervising the work of these research laboratories. We have research work constantly going on in our new works laboratories and elsewhere among the chemists employed in our works since, when a man is carrying on practical work, you cannot prevent him, when he has got brains, from considering what better method could be devised for carrying out his operations. In order to encourage the supply of chemists, we have made an offer to the universities that, if they will send us a qualified chemist, we will find him a post at a satisfactory salary with an engagement for three years. It seems to me this is the best inducement we can offer to any young man thinking of working at chemistry. We are offering special facilities to the members of our staff of chemists to pursue their studies either in Huddersfield or in Leeds. In that way we are trying to make the most of all the material that exists. But there is a further difficulty, *viz.*, that of men, and we have made up our minds that we will take special steps to encourage promising young men to devote themselves to chemistry and to send them to technical schools and universities, and to do whatever is best for them. That, of course, will take time and cost money, but no figure that anyone can think of would be too much to pay for securing a really efficient staff of chemists for our work in the future, and we rely upon the support of the shareholders for any expenditure we may have to make in order to carry out that scheme. We have a long way to go. We welcome the help of all the experts that we can get. I do not say that we are anywhere near our goal, but we are earnestly and diligently pressing forward.

There are two questions to which I should like to refer. The first is that of the manufacture of indigo. You will remember last year I stated the steps which we had taken to place ourselves in a position to manufacture indigo and to tender to the Government for the purchase of Ellesmere Port Works, and that we were refused permission to tender. I said we had made every effort to ascertain the reason for which we were not allowed to tender, but had been unable to do so. In one quarter or another attempts were made to cast some doubt upon the statement I then made, and as the question is a matter of great consequence for the user of dyes I will tell the shareholders exactly how it stands. When we were asked to submit evidence of our ability to manufacture indigo by the process at Ellesmere Port, we appointed a committee consisting of Dr. Forster, chairman of our Technical Committee, Mr. Turner, Mr. Dean, chief chemist at the Turnbridge Works, and Mr. Robinson, an engineer of great experience and ability, one of the first mechanical engineers in the country, who was good enough to place himself at our disposal practically as soon as the Company was formed, and who has knowledge of our plant and premises. They went to Ellesmere Port and saw the plant there. They then went to France, to Creil, and saw a plant similar in every respect, in practically every respect—there were, I believe, some little details of no consequence—similar to that at Ellesmere Port. They made a report which was submitted on behalf of the Board to the Board of Trade. Here is a passage from their report which deals with our ability to manufacture: "The indigo plant at Creil is an exact duplicate of the Ellesmere Port plant. The plant was working on the occasion of our visit and we carefully investigated its operation at all the important stages. We also obtained from the chemist information with regard to the quantities, proportions, temperatures, times, etc., required in the operation. We entertain no doubt as to our ability to manufacture indigo with the plant at Ellesmere Port. Further, we have provisionally arranged with the Ministry of Commerce that if the Company should acquire the plant at Ellesmere Port, the services of the chemist, who is a French subject, and a mobilized soldier, should be made available to assist the Company in overcoming any difficulty which might present itself."

We sent that to the Board of Trade with the intimation that if there were any doubts we would be glad to supply further particulars upon any point. The answer we got was that, after considering the reports of the two independent referees, to whom the reports from Messrs. Levenstein and ourselves had been referred by the Board of Trade, they had decided that Messrs. Levenstein, and Messrs. Levenstein alone, should be allowed to tender. We asked them again and again to state the reason for which we were not allowed to tender. We failed to get an answer. We asked for an interview with the President of the Board of Trade and the whole of our Board of Directors attended. We repeated the question, but still without obtaining an answer.

There are two things for which we have pressed. We have asked whether the referees recommended that we should not be allowed to tender. We have got no answer to that, except that the decision of the Board of Trade was given after considering the report of the referees, and on further pressure we were told that it was not a matter which was referable. But I have got no answer to the question: "Did they or did they not in fact recommend that we should not be allowed to tender?" It would have been easy to answer.

I asked a further question: "In what respect did the referees consider that we were not able to manufacture by the Ellesmere Port process?" but I have got no answer to that. Now I have had a good deal to do with references of one kind and another. We were asked to submit our statement and Messrs. Levenstein were asked to submit theirs. We were asked to pay half the fee and I think we should have been entitled to know what was the award and why it has been kept back. My reason for pressing this is that neither of the referees was a practical man. They were both eminent scientific men—teachers of chemistry—but, so far as I am aware, neither of them had anything to do with the operation of any chemical process in connection with dye-making. The process so far as the scientific theory is concerned was well known and the only question was how to operate a particular plant, and in the face of a signed declaration and the statement of responsible practical men that they were perfectly satisfied they could manufacture indigo by that process—in the face of that particular declaration it was not for two professors to say offhand that these men could not do so. That does not seem to be the way in which business of the greatest importance to the textile industries and to the country should be dealt with. Until I am shown their report and told in what respect we are unable to manufacture indigo by the process, I decline to accept the view either of the Board of Trade or of the distinguished referees whom they appointed. I always make this reservation, I gravely doubt whether these referees made any recommendation which would justify the refusal to allow us to tender.

Now there is another feature in connection with this matter to which I think it right to refer. At the meeting of Messrs. Levenstein, Ltd., shortly after I made the statement, Sir John Lonsdale, who is the Chairman of Messrs. Levenstein, Ltd., referred to my statement and said he did not think we had anything to complain of. The reasons he gave were: that they had long and intricate negotiations with the Board of Trade on the subject of the Ellesmere Port Works so far back as February, 1916; that they were the first firm in the country to approach the Board of Trade intimating their desire to purchase; and that as soon as the Receiver or Controller told them the minimum price to be accepted they made an offer to purchase at that price. Now, in the first place, Sir John is misinformed in thinking that he made the first approach on the question of Ellesmere Port, because long before February, 1916, I had made enquiry with regard to it, and I was told that the Government proposed to continue working under a Controller. At the end of January I heard, incidentally, from the President of the Board of Trade, that the Committee which was dealing with the shutting down of German concerns in this country was going to consider the question of closing down the Ellesmere Port Works and of selling them. I then intimated that we should desire to purchase and I was told that the purchase was going to be by tender, and that the terms and conditions would be arranged by the Judge to whom application would have to be made. And I was under the impression that the sale was to be by tender, right down to July, when we got intimation that we were not to be allowed to tender. It, therefore, comes to me as a surprise that during that period there should have been long and intricate negotiations with one party, while the other was being held at arms length. As a matter of fact it was with the utmost difficulty that we could get the time extended to enable our deputation who had visited France to come back. A sale by tender conveys to my mind that everyone is to be treated in the same way and to have the same opportunity. And if there were negotiations with one party and if a price had been arranged I think it was unfair that we should not have been informed. In fact there should have been no such negotiations if there was to be a sale by tender. Everyone should have had a fair chance, and I suspect that these long and intricate negotiations which took place had practically settled the question before the referees were appointed, and before our memorandum went in. I raise this question now, not for the purpose of swelling upon any grievance, because everybody—every business man, at any rate—knows that the least valuable possession any man can have is a grievance. The best thing to do is to write it off and to go on with one's work. But I raise it for two reasons. I want to explain what has taken place in justice to our staff, and to the men who signed the memorandum. I do not think we could justly allow it to go out that we are not able to manufacture indigo by this process. In the second place, I want the shareholders to know the serious position in which users of indigo are placed. This Company was formed, and the capital was subscribed, on the invitation of the Government on the footing that users of dyes would have an undertaking which would control the supply of dyes as regards price and other terms, and that everyone would be treated alike. Now we find that this most important plant is given to a firm subject to no control as regards supply or price or conditions, such as making purchase of other dyes a condition of giving a supply of indigo. It would take a great deal to justify the action of the Government in this respect. It is said that this process, along with other processes, has been sold by Messrs. Levenstein, Ltd., to a large American firm who had hitherto nothing to do with dyes, and unless the conditions of sale submitted to us have been modified there is nothing in them to prevent it. No doubt there could be established in America, in that way, a very large undertaking for the manufacture of indigo. They would have power to supply all the markets of the world, whereas our hands are tied. But not only are Messrs. Levenstein, Ltd., allowed to do that, but they are entitled to give to anyone else their power to manufacture by this process without any responsibility to the users of dyes in this country. I venture to repeat that the transaction is one that has given us a shock. We have a process for the manufacture of indigo, a different process, and we shall do everything in our power to be placed in a position to manufacture and to supply anyone. But the difficulties of putting up large plants at the present time are undoubtedly very great. It will require all the support of the shareholders and the support of all the large users of indigo to enable us to carry out that program, and in any event there must be delay before it could be carried out even if we commenced it to-day.

Now there is only one other matter which I want to speak about, and that is the question of coöperation which is mentioned in the report. The position is this—that in July of last year arrangements were made for a meeting between all the dye manufacturers in this country for the purpose of endeavoring to bring about some arrangement which would prevent overlapping, and which would enable them to deal in the best way with the supply of the country. But at that time the question was taken out of our hands by the Board of Trade, who set on foot certain negotiations by committees and otherwise. Our attitude is this, and has been throughout, that we are in favor of any arrangements which will enable us to work together for providing a better supply. In the words of the report: "We would welcome any tangible proposals for coöperation with other manufacturers provided that the interests of the textile and other industries dependent upon the supply of dyes are safeguarded, and that the coöperation can be carried into effect in a manner consistent with the object for which this Company was established." Various suggestions have from time to time been made, and we have maintained that attitude throughout. We have been really anxious to bring about an arrangement but so far nothing definite has been reached. The difficulties have not been in the least of our making, but nothing has been arrived at, and we are to-day practically just where we were 12 months ago. Now let me say this. We still remain of the same opinion and we shall welcome any practical tangible proposals that comply with these conditions. But I want to say quite definitely that, if you are going to have combination, it is essential in the interest of the industries of this country that there should be adequate control to protect the textile industries, and that a monopoly of dye-making in the hands of one company without control would be a danger to which the industries of this country should not for a moment be exposed. I have had opportunities of studying the operations of some of the conventions made by the German dye-making combinations, and can assure you that there are many ways in which industries may be punished through the action of dye-making concerns which would probably never come to the surface, or would be imagined by those who have not had an opportunity of seeing the inner workings of those concerns. I therefore want shareholders to understand our view, *viz.*, that it is essential in any such combination that there should be adequate protection of the public interest and of the textile industries. We are all in favor of coöperation and combination not only at home but with the Swiss and with the French. Dr. Forster, at the present moment, is in America studying the question of how we may be able to supplement the supply of dyes to this country by coöperation with American manufacturers.

One thing I would say about this matter is, that it is no use talking of coöperation unless there is a genuine desire for people to work together, and I am bound to say that, as I study the atmosphere at the present time, I find, instead of an atmosphere which would lead towards coöperation, what seems to me to be an organized attempt to create hostility to this Company which does not promise very well for coöperation. We have taken no notice of general discussions on the merits of business men and of scientists. It is absurd to waste time on that, but there have been some definite statements made which do call for notice.

The first of them was made by Professor Pope, one of the impartial referees on the Ellesmere Port transaction, in which he says in the midst of a great deal of general discussion: "The Government organization (that is our Company) has proved to be not only a great failure, but has had the further effect of inhibiting the re-establishment of the coal-tar industry. That is to say, the organization apparently was to do everything that was necessary and consequently private effort was to a considerable extent hampered." Another professor repeats the statement. My object in referring to statements of this kind in regard to this Company is to say, in the first place, that they are not true. Further, the gentlemen who make them, so far as I am aware, have never been inside our new works and know nothing of the plant we have put down or of our program, nor of the research we have been carrying out, nor of our general policy. Why then these wild statements? As a rule you pay no attention to them, but when talking of coöperation, if you are going to coöperate, it can only be with people who are willing to work honorably and loyally with you, and not with people who write and publish statements like that in regard to you. The situation with which we are faced is far too grave, too important, for men of science or others, who may be able to give help, to indulge in recriminations or talk of this kind. And I want to make an appeal to them and to all dye manufacturers and to everybody engaged in the business each to carry on his business in healthy rivalry, and do everything he can to make it a success, but do not let us get into the old bad position of past days when it was the business of every man to try to do as much harm to his neighbor as he possibly could.

The task is one which is worthy of our best efforts. I can assure you we are only too anxious to get the coöperation and assistance of anybody and everybody able to help for the purpose of producing the dyes which it is our object to supply to the country.

LEVENSTEIN LIMITED

The following account appeared in the *Journal of Commerce and Commercial Bulletin* for Thursday, January 10, 1918:

London, Dec. 21—Instances of the progress made by the British dye industry were given yesterday by Sir John Lonsdale, presiding at the annual meeting of Levenstein's Ltd. That it is possible for Great Britain to produce all the dyestuffs it needs was one statement made by Sir John. On this point he said:

"If the Government is prepared to give the necessary financial assistance and special priority for the erection of plant, we, for our part, will guarantee to make this country independent of Germany or any other foreign source for dyestuffs. Let there be no misunderstanding on this point. In our organization the State has an asset of the greatest value, for we have the knowledge and experience to free the textile trade from German domination in dyes, and we shall undoubtedly produce the results required, given the necessary help from the State."

The original works of this company were erected by Germans solely to comply with the Patents Act of 1907; they were only designed to carry out the last stage of the manufacture of indigo, no provision being made for the manufacture of the all-important intermediate product without which it was impossible to obtain the finished dye. The war shut off the import of this intermediate material, and the present management, which purchased the German property, proceeded to erect the large plant necessary for the production of the essential intermediate.

PROGRESS OF DYE INDUSTRY

One of the many difficulties involved was the fact that the Government had already commandeered all the supplies of the raw material required. Accordingly, a new process from another raw material was developed, and with the remarkable speed extraordinary results were achieved. Ever since the outbreak of war the company has been by far the largest supplier of aniline dyestuffs to the War Office and Admiralty and to the Colonial and Allied armies, also shipping large supplies to the United States to cover the requirements of American textile manufacturers who had contracts with the Allied Governments. By March, 1915, the company had sufficient plant installed to meet the entire demands of the textile mills of the world for naval and military purposes outside the Central Empires.

"This achievement, effected without any financial assistance from the Government, entitles the chairman of the company to speak with the highest authority," says the *Financial News*. "The work done by Messrs. Levenstein at Blackley and Port Ellesmere in relieving the dye famine of the world and helping to break down the German monopoly of a key industry which had become a source of great economic power is of the highest national importance. But Sir John Lonsdale clearly demonstrates that much remains yet to be done if the British dye industry is to be placed in a position rendering it capable of resisting German competition after the war.

"Before the war the British textile industry and other dye users were dependent upon Germany for 80 per cent of the dyes employed—that is to say, British industries, representing £200,000,000 of capital, were practically at the mercy of German dye makers. Now the peace requirements of these industries are widely different from those of war time. The range of dyes manufactured in this country to-day has to be very widely extended. The manufacture of the peace requirements of the dye-using trades is limited (as far as Messrs. Levenstein, Limited, are concerned) not by their scientific knowledge, but by the extent of their plant.

AID OF GOVERNMENT SOUGHT

"The intention underlying the formation of the British Dyes Company and the investment of £2,000,000 of public money therein was excellent, says Sir John Lonsdale, but it has not solved the problem of securing adequate peace supplies of British-made dyes. Messrs. Levenstein, he declares, manufacture to-day more dyes and a wider range of dyes than all the other British makers of aniline dyes combined, but much more remains to be done. The company possesses the knowledge; what it requires is more plant, and the provision of such additional plant is governed solely by financial considerations.

"Sir John Lonsdale, on behalf of his company, therefore, throws out this challenge—or perhaps it should be termed invitation—to the nation: 'If the Government is prepared to give the necessary financial assistance and special priority for the erection of plant, we, for our part, will guarantee to make this country independent of Germany or any other foreign source of dyestuffs.'

"This is an undertaking which cannot be ignored by the Government. In the Levenstein organization the country has an asset of the highest economic value. After the war Germany's dye industry will be one of her strongest economic weapons. Tariffs will not exclude German dyes if the necessary plant does not exist in this country to meet trade requirements. British Dyes Limited, in which the Government is interested, has not yet displayed the capacity for production of the country's requirements, and it is essential that further steps should be taken. We hope that the fullest impartial consideration will be devoted to Sir John Lonsdale's statement and that prompt action will be taken, as the question admits of no delay."

CURRENT INDUSTRIAL NEWS

PLATINUM IN SPAIN

A memoir published by the Spanish Geological Survey gives some details of the platiniferous deposits of the Serania de Ronda. The region of Southern Spain, situated between Malaga and Gibralter, is of very complex structure. It has formed in recent years the object of investigations by mining engineers commissioned by the Spanish Government. Samples were taken of river sand and the gravel from a river the bed of which is dunite, and others from rivers in which dunite was absent. In the first case, the presence of platinum was revealed, but not in the second. On washing considerable quantities of sand and gravel, small, lucent grains of platiniferous ore were discovered, the platinum content of which varied from 78 to 82 per cent of pure ore. In some zones the ore contained from 2 to 3 grams of platinum per cubic meter of substance examined, while in others the yield was as low as 0.25 to 2 grams per cubic meter. From the economical point of view, the nature of the platiniferous sand or gravel is considered excellent, as it does not contain clay. The first river to be investigated systematically between February and June, 1916, was the Rio Verde over a stretch of 3$^1/_2$ kilometers. The platinum contained in this area gradually increased from 8 to 20 centigrams per cubic meter from the point at which prospecting was begun to the finishing point.—A. McMillan.

TUNGSTEN IN MALAYA

According to the *Mining Journal*, **119** (1917), 657, a rich deposit of mixed wolfram tin ore was discovered recently near the village of Changloon in Sungei Sintok. The discovery was made by Chinese who were working for tin on some small adjoining leases. The discovery caused a rush and as there were over twenty applications for the area, the Kedah Government decided to put the property up at auction. Subsequently, however, the area of 3000 orlongs was given to a local firm. The monthly output hitherto has been about 300 pikuls (1 pikul = 142.7 lbs.) but an increase to 800 pikuls is expected shortly. The rich discovery seems to be confined to this localized area and no further discoveries have been made outside. The ore occurs in quartz veins but as no regular prospecting work has been done upon it, no reliable idea can be formed as to its life.—M.

TUBULAR CYCLE COMPONENTS

A catalog issued by Messrs. Accles and Pollock of Birmingham, England, illustrates a wide range of tubular parts for the construction of cycles, motor-cycles, and aeroplanes, such as handle bars, seat pillars, seat-pillar laps, frame lugs, bridge pieces and loop struts, stays and front forks, and steering tubes. Full-size illustrations are given of 268 special sections in cold-drawn, weldless steel tubing, as well as of a number of sections from the Air Board's standard lists, and there is a description of an attachment called the "Apollo Mykarmo," which, when cramped on the thimble of standard micrometer calipers, at once converts them into a limit gauge having a tolerance of from 0.0001 in. to 0.022 in., with variations of 0.001 in.—M.

MAGNETO MACHINES FOR POCKET TORCHES

A recent issue of the *Elektrotechnische Zeitung* gives some particulars of a new type of pocket torch being developed in Germany and Austria in which the lamp is supplied with current from a small hand-driven magneto. The shortage of certain material is putting a limit to the manufacture of dry cells and small accumulators for public use and this is no doubt responsible for the tendency to utilize hand-driven sources of current for pocket lamps of various kinds. Lamps of this kind are more expensive than the ordinary kind but do not require refills or charging.

One of the types described depends on the release of energy from a series of springs put into tension by the pressure of the thumb on a lever. The whole arrangement weighs about 1 lb. and is so contained that the release of the spring supplies enough energy to keep the lamp alight for 3 min. In order to secure a light for 10 min. a heavier machine, weighing about 5 lbs. and requiring to be wound up with both hands, has been designed. In these lamps the armature is the rotating part but in another variety, due to O. Pletscher, the field revolves in ball-bearings round a T-shaped armature. This lamp is stated to weigh only about $^1/_2$ lb. The application of this principle of portable electric lamps seems quite simple and practicable.—M.

THERMIT WELDING

The British Board of Trade have now given formal sanction, says *Engineering*, to a new company with works in London and Liverpool and known as the British Barimar Thermit Welding Company to take up and exploit the Thermit-Welding process which prior to the war was exclusively in German hands. Thermit is especially applicable for tramway welding and for repair of heavy castings and machine parts. The registered offices of the new company are at 10 Poland St., London.

The Thermit Co., Ltd., of Commercial Rd., London, are not connected with this new company which has only rights to work certain of their patents. They continue their manufacture of various Thermit compounds and are at present especially engaged upon the manufacture of certain metals and alloys in connection with the war.—M.

REFRACTORY PROPERTIES OF MAGNESIA BRICKS

A contribution to the Proceedings of the Paris Academy of Sciences was recently made by MM. LeChatelier and B. Bogitch on the refractory properties of magnesia bricks, either made in the laboratory from pure magnesia or from commercial specimens. The resistance to crushing was measured at 15, 1,000, 1,300, 1,500 and 1,600° C. for two bricks and at 15, 1,500 and 1,600° C. for the remainder. All the magnesia bricks show a sudden fall of resistance to crushing at a temperature depending on the degree of purity, a fact which explains why, in practice, it has been found that magnesia bricks stand less well in furnaces than silica bricks, although their fusing points observed in the ordinary way without reference to resistance to crushing are higher than the silica bricks.—M.

PREVENTION OF SCALE IN BOILERS

The necessity of reducing to a minimum the formation of scale on the heating surface of steam boilers if efficiency is to be maintained was referred to in an article in the *Times Engineering Supplement* for September and in this connection Messrs. J. Dampney and Co., of Cardiff, Wales, draw attention to their "Apexior" compound as a remedy against both scale and the pitting of the plates. The compound consists of 98 per cent pure amorphous carbon combined with a neutral organic vehicle. It is painted over the internal surfaces of the boiler and well rubbed in, and, when dry, it presents a peculiar surface which is antagonistic to the building up of a hard scale of crystallization. Any deposit which may take place, the makers state, can generally be removed easily without the use of a chipping hammer and falls off readily over large areas by concussion. In cases where, through the use of soft water, the plates are attacked by pitting, the compound not only acts as a preventive but also arrests the progress of the pitting if it has already begun. The substance is not affected by boiling water or by steam under pressure, and, owing to its nature and the thinness of the film, the transference of the heat to the water is practically not interfered with.—M.

ELECTRIC HEAT STORAGE IN BOILERS

In a recent issue of *Engineering* an account is given of a new type of electrical generator invented by Col. Revel, an engineer in the Italian army. The essential idea in the apparatus is the direct conversion of electric energy into heat by making use of the resistance of the water to be evaporated. Alternating circuits of 200 to 3,600 volts can be applied and this apparatus is stated to be automatic and to require no regulation. Lack of feed water merely diminishes the production of steam until the supply of water is renewed. The efficiency is claimed to be 97 to 98 per cent. The Revel apparatus is constructed to work up to 14 atmospheres and can be connected up at any time to the steam pipes of an ordinary boiler. In this way, temporary use can be made of hydroelectric supplies and their utility can be judged by the fact that they were often used before the war when the price of coal in Italy did not exceed $8 per ton. An illustration is given of an installation of 8 generators taking three-phase current at 6,000 volts and each developing 900 to 1,000 kilograms of steam per hour. The production of steam varies according to the area of electrodes immersed. The sediment collecting at the base of the apparatus from water containing calcareous material can be released without interrupting the process.—M.

BRITISH BOARD OF TRADE

During the month of November the British Board of Trade received inquiries from firms in the United Kingdom and abroad regarding sources of supply of the following articles. Firms which may be able to supply information regarding these things are requested to communicate with the Director of the Commercial Intelligence Branch, Board of Trade, 73 Basinghall St., London, E. C.

- Artificial musk
- Boot buttons (manufacturers wanted)
- Camel-hair brushes
- CHEMICALS:
  - Calcium carbonate (precipitated)
  - Green stain for eye shades
  - Sulfonated castor oil (50 per cent volume)
  - Strontium carbonate
  - Bicarbonate of soda
  - Borate of sodium
  - Calcium silicide
  - London purple
  - Paris green
- Corrugated paper
- Commercial dried blood for pharmaceutical purposes
- Die-casting machines
- Disinfectant attachments for telephone mouthpieces
- Fiber tubes, $^3/_4$-in. diameter
- Flat handles for camel-hair brushes
- MACHINERY AND PLANT FOR:
  - Making felt wads for use with sport rifles
  - Production of clocks and parts thereof
  - Making electric light carbons
- Maple or hickory skewers, spoons and forks, cheap, tin and zinc
- Nichrome drawn wire
- Oiled cloth for electrical insulation
- Pumice powder (20 to 50 tons)
- Self-making stamp pads
- Spectacle and eye-glass frames, gold-filled
- Silver leaf for coating pills
- Sugar-cane wax
- Vegetable extract
- Vegetable parchment paper
- White chip oil

—M.

MINERAL PRODUCTION OF VICTORIA

The annual report for 1916 recently published gives the following yields of the various metals and minerals for the year:

Gold	276,168 oz.
Black Coal	417,183 tons
Brown Coal	2,915 tons
Antimony Ore	12,382 tons(*a*)
Tin Ore	122 tons
Manganese	85 tons
Gypsum	1,853 tons
Wolfram	314 tons
Magnesite	100 tons
Kaolin	810 tons

(*a*) Yielding 3,259 tons concentrates.

—M.

MANUFACTURE OF ELECTRODES

According to the *Ironmonger*, a company called the Norske Elektrodeverker is erecting a factory at Fredrikstad, Norway, for the manufacture of carbon and graphite electrodes. The capacity is 4,000 tons of carbon or 1,000 tons of graphite electrodes a year, and the works were expected to be in operation by the end of the year 1917. Carbon electrodes are expected to be the first article of manufacture. The machinery is almost exclusively of American make. The orders already in hand are said to ensure the profitable working of the factory for some time and it is stated that the factory is working in coöperation with the Norwegian Government. The company will use power from the great waterfall of Sarpsfall, but in anticipation of this being inadequate, it has purchased the rights to the entire power from the waterfall near Kristiansund.—M.

RECOVERY OF POTASH AND MAGNESIA FROM CANADIAN LAKE

The *Official Canada Gazette* of September 8, 1917, publishes an announcement to the effect that the Committee of the Privy Council have concurred in the recommendation of the Minister of the Interior that he be authorized to lease certain lands abutting upon Lake Muskiki in connection with the recovery and utilization of minerals from the bed and waters of the lake. These minerals, chief among which are potash and sulfate of magnesium, are intended for medicinal and other purposes and preliminary plans submitted by the company show that the proposed utilization of the waters of the lake will require the construction near the lake of a pumping plant, evaporating machinery, bottling works, etc. The name and address of the above-mentioned company may be obtained by manufacturers desirous of supplying plant, etc., on application to the Board of Trade, 73 Basinghall St., London. Reference No. 374 should be quoted.—M.

FLUXES

The fluxes commonly used in melting aluminum scrap, says the *Brass World*, are fluorspar, cryolite and salt. An excellent way of utilizing such material when a part of the scrap is small and not clean is first to melt a bath of aluminum using solid material and to allow it to reach a temperature of approximately 850° C., then to add the sweepings in such quantity that the bath will absorb them without losing its liquidity. The bath is then reheated and more scrap charged, the process of charging and reheating being alternated until the crucible is as full of metal as desired. The metal will most likely be pasty and a small piece of fused zinc chloride is added and the bath well stirred. The resulting action will free the mixed oxides and the metal will assume its natural fluidity. The crucible should be emptied immediately of aluminum before any reaction can occur between the metallic aluminum and the heated oxide on the one hand and the oxygen and nitrogen of the atmosphere on the other, as such action will consume metallic aluminum and greatly reduce the percentage recovered.—M.

WATERPROOF VARNISH FROM OIL

A French patent for the above has recently been published, in which oils vulcanized with sulfur chloride are dissolved in amyl acetate. The following method of preparation is used: One thousand parts of castor oil are mixed with 2,000 parts of amyl acetate and stirred up well with 250 parts of sulfur chloride. In a short time, the mixture sets to a fairly solid jelly and gives off large quantities of hydrochloric acid from the acetyl chloride formed. If, however, the product be left in a tightly closed vessel for several days it will be found to have become completely liquefied and dissolved. The acid is then neutralized with barium carbonate and, after the precipitate has been removed by decantation and filtering, a clear almost colorless liquid is left consisting of a perfect solution of the vulcanized oil, hitherto regarded as insoluble. This solution may be used for waterproofing fabrics, leather, paper, etc. On the other hand, if it be mixed with other solvents, *e. g.*, alcohol, benzene, acetone, acetic ether, and employed to dissolve a certain amount of nitrocellulose, there results an excellent varnish for glossy leather—the gloss resisting action of soap, friction, etc.—a leather polish, a varnish for oil cloth and when mixed with pigments, a waterproof, quick-drying paint which will stand washing and changes of temperature.—M.

SHELLAC DERIVATIVES

A paper on the "Investigation into the Inbibition Exhibited by Some Shellac Derivatives" by Messrs. A. P. Laurie and C. Ranken was read at the Royal Society, London. The paper dealt with experiments made on the substances obtained by boiling shellac with carbonate of soda or borax. The solid substances, very similar in consistency to gutta-percha, are found to expand rapidly when placed in water. The control of the expansion by the addition of soluble salts is not the same as in the case of gelatine, since, at any rate, in a large number of cases, it does not seem to depend upon the nature of the salt but simply upon the strength of the solution, and the amount of expansion increasing with the diminution of the strength of the solution. If the expansion is allowed to become complete in cold water, the mass cannot be contracted again, but if expansion takes place in a salt solution, then contraction will take place again if the mass is put into a stronger solution. Strong salt solutions are also found to precipitate the soluble portions of the shellac-borax compound.

As a result of the experiments described, the authors suggest that the facts can be best explained by supporting the shellac-borax mass to consist of a soluble organic nucleus surrounded by elastic diaphragms through which the organic nucleus cannot pass, but the salt molecules can pass, the organic nucleus being soluble in water but insoluble in strong solutions of salt.—M.

CELLULOSE TURPENTINE

During the treatment of wood for cellulose by the sulfite process there is obtained a considerable amount of a turpentine-like oil mixed with various impurities containing sulfur and having very objectionable odors. The amount of turpentine so obtained reaches as much as 22 lbs. per ton of wood treated where pines are used. The oil has recently been extensively examined, says a contemporary, the sulfur compounds being first removed by means of mercuric chloride dissolved in alcohol. The principal portion of the oil consists of alpha-pinene which is well known to be the main constituent of ordinary turpentine oil. It is, therefore, clear that this terpene is very stable or it could not stand the drastic treatment of the sulfite process. When the sulfite process is used, the pine is almost completely broken down to para-cymene. Beta-pinene is also present in the oil and probably di-pentene.—M.

SUBSTITUTE FOR OIL IN PAINT

According to the *Oil and Color Trade Journal*, a mixture of 100 parts of rosin, 20 of soda crystals and 50 of water melted over a fire and mixed with 250 parts of water containing 24 parts of liquid ammonia gives a syrupy liquid which can be used as a substitute for boiled oil or turps in the manufacture of paint. Such paint dries quickly without requiring any driers and has good covering power and withstands the influence of temperature, wet and dry. The substitute is improved in appearance and gloss by the addition of a mixture of 2 parts alcohol, $3^1/_2$ parts ordinary glycerine and 1 part of wax in proportions up to 10 per cent.—M.

DYE FROM SULFITE LYES

After chemical research succeeded in the useful application of sulfite lyes for the production of alcohol and of coal dust for heating purposes, an engineer in Finland, says the *World's Paper Trade Review*, claims the economical production of valuable color stuffs from the remarkable sulfite off lye. He claims especially the new production of methyl alcohol, cymol and furfurol for the transformation into coloring material as they are gained in Germany from coal tar. The inventor has claimed patents in the Scandanavian countries, Russia and Switzerland, and a color factory is being erected at Tammerfors, the centre of the Finnish textile industry, with a capital of 200,000 marks.

The inventor, Dr. Wiljanen, delivered an interesting lecture relating to his invention at the Technical Club, Tammerfors, and exhibited about ten different colors produced from cymol and numerous others containing cymol as a substantial part. He explained that about 300,000 kg. of cymol are obtainable in Scandanavia as a by-product wherefrom yellow and red cotton and wool colors could be produced in a simple manner.

Finland's largest paper mill association has installed apparatus in its factories for separating wood spirits, cymol and furfurol and investigations are being continued at the new Tammerfors factory with a view to obtaining new raw color material from home products. Preparations are being made to start, in the near future, the manufacture of cymol colors. —M.

ELECTRIC ARC WELDING

The welded fastening, reports the *Railway Mechanical Engineer*, has always been looked upon as a stronger fastening than the riveted or bolted joint. As a general proposition the riveted or bolted joint has the tensile strength of the original piece while the welded joint is as strong as the original section. There are two kinds of electric welding, known as the carbon electric welding and the metal electrode welding. In the former, an arc is drawn between a carbon electrode, the piece to be welded and the metal to be added are fed into the arc in the form of a "melt-box." The method is not used extensively in railway work due to the fact that the welding may only be done in the horizontal plane in this manner and that the work is in general inferior to that done with the metal electrode process. The metal electrode process as the name implies—a metal electrode—the arc being drawn between the electrode and the piece being welded. The heat of the arc melts the metal of the piece and the metal of the electrode simultaneously. As the metal of the electrode melts, it is drawn across the arc and a complete and homogeneous union is formed with the molten metal of the piece. With the exception of work with certain electrodes (manganese steel and slag-covered electrodes) the electrode is always made the cathode or negative, *i. e.*, the current flows from the piece being welded to the metal electrode. The voltage required for metal electrode welding is about 20 volts and direct current is necessary.—M.

SCIENTIFIC SOCIETIES

REDUCTION OF WASTE

The importance of reducing and preventing waste has been brought to the attention of the American Chemical Society by the following communications:

December 22, 1917

To the Chairmen and Secretaries of the Sections of the American Chemical Society:

The Alabama Section of the American Chemical Society early this autumn passed resolutions looking to the prevention of all wastes as far as possible and referred the question of a national movement in the American Chemical Society to the President of the Society. At a meeting of the Advisory Committee of the Society held in New York on December 8, it was decided to take up this movement as a most important one in which the National Society could be and should be active. It was decided that the emphasis should be placed not so much on a talk propaganda against wastefulness but, since the Society consists of experts in the field of recognizing and preventing waste, to ask the Sections to emphasize the need of pointing out specifically in as many instances as possible where waste occurs and how it is to be avoided. It is recognized that in many cases large problems of research would be involved in reaching recommendations as to how waste is to be avoided but chemists could be active in pointing out these problems and urging that the necessary investigations be undertaken by the concerns involved.

In accordance with this action, I would ask that you appoint a committee of your ablest men to take this matter in hand and to organize a campaign against waste in the most effective way possible. It might be desirable in given localities to appoint a very large committee to cover all the different kinds of waste or to appoint a number of committees each to take care of a definite field of effort. Such organization is left to the judgment of the local sections. In conclusion let me say that Past President Little pointed out to the Boston meeting that the saving of waste alone in this country would be sufficient to pay off the tremendous war debt which we are incurring in the space of a very few years.

In case any Local Section would like specific advice in regard to any matter connected with this movement it is urged to bring the matter up with the President of the Society.

Thanking you in advance for your coöperation in this most important movement, I am

Yours sincerely,
(Signed) J. Stieglitz,
President, American Chemical Society

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38 Albemarle Street
Rochester, N. Y.
December 21, 1917

Dr. Julius Stieglitz,
University of Chicago,
Chicago, Ill.

Dear Sir:

In reading an article in the July number of the *National Geographical Magazine* on rats and mice, I was very much impressed by the enormous economic loss to this country caused by this pest. The thought struck me that it is most absurd for the American people to strain at every point to conserve and increase their food supply and yet to ignore wholly a cause of loss which is estimated to cost the country from $100,000,000 to $200,000,000 per annum.

With this thought in mind I took up the matter with the Rochester Section and obtained their approval for me to act in this matter. Enclosed are copies of the correspondence to date.

My object in writing to you is to see if your views on the subject are in accord with mine and, if so, whether it would not be possible for you to help in this matter through your office and the Local Sections.

I realize that it is impossible for any one community to make a successful fight, for in the first place the rat, being migratory, will appear again shortly after being exterminated locally. In the second place, there is such an enormous indifference to be overcome such as, "Rats never bothered me, why should I worry?" that it is almost impossible to accomplish anything unless there is governmental authority behind it. Therefore, it appealed to me that there never could be a more fitting time to start a campaign against this pest than the present when nearly everybody is willing to do their bit. Besides the loss of food enormous damage is done to property and, by no means the least, there is the constant menace to health.

I would ask that you kindly give this matter your careful attention and let me have your opinion relative to it.

I received your letter of December 17, and will carry out the instructions given therein.

Sincerely yours,
(Signed) H. Le B. Gray,
Chairman, Rochester Section

....

Chicago, Ill.
December 27, 1917

Mr. H. Le B. Gray,
38 Albemarle St.,
Rochester, N. Y.

Dear Mr. Gray:

I have your letter of December 21 with the interesting correspondence in regard to the economic loss due to the rats and mice in this country. A few days ago I sent out letters to all the Sections of the American Chemical Society asking them to coöperate in the elimination of waste in this country and especially to indicate in specific cases where waste occurs and how it could be avoided. This letter no doubt has crossed your present letter. I think a movement to reduce the destruction by the rats and mice would be particularly desirable at this time, as it would not only save food and other important products but also be a safeguard in regard to the health of the community. I have no doubt that we shall have the same experience as they have had in Europe, notably in Germany, France and Italy, where the food shortage has been the most severe, and shall find that the necessity of saving of food will lead to reduced resistance towards disease and the increase of disease in this country. In view of that situation it would be especially desirable to offset this decreased resistance by such a positive element toward health as the reduction in rats and mice that you propose. I would recommend, therefore, that you take up this problem with each of the Sections of the American Chemical Society as timely and important.

Yours sincerely,
(Signed) Julius Stieglitz

....

38 Albemarle Street
Rochester, N. Y.
January 4, 1918

Dr. Chas. H. Herty,
Chairman, New York Section,
New York City.

Dear Sir:

Pursuant to the recommendation made by Dr. Stieglitz in his letter of December 27, 1917, replying to mine of December 21, 1917 (copies of which are enclosed), I am writing you with the hope that I may interest your Section in a matter which appeals to me as vitally affecting the country, especially at the present time. Local campaigns and those without official authority would be futile and it seemed to me that by the combined action of the Local Sections of the Society sufficient interest could be aroused to start a national campaign backed by governmental authority.

The matter has already been taken up with the U. S. Food Administration and in reply they state, "You are advised that the movement of your Society, as indicated in your letter, has our heartiest approval and support, and we trust your Society will do its utmost to aid in the elimination of these pests and the conservation of much needed food thereby"—and in a subsequent letter—"permit me to suggest that you take this matter up with Dr. Hayward, Chief of the Bureau of Insecticides and Fungicides, Department of Agriculture, who have their permanent representatives in all sections of the country and would, therefore, be in a better position to take charge of this matter, the importance of which we realize, than the food administration."

A letter together with a copy of this one will be sent by me to Dr. Hayward.

Any action which you or your Section may take to further this movement will be greatly appreciated.

Very truly yours,
(Signed) HARRY LE B. GRAY

SEVENTY-FIFTH ANNUAL MEETING AMERICAN ASSOCIATION FOR THE ADVANCEMENT OF SCIENCE, PITTSBURGH, PA., DECEMBER 28, 1917—JANUARY 2, 1918

The seventy-fifth annual meeting of the American Association for the Advancement of Science and the affiliated societies was held in Pittsburgh, Pa., from December 28, 1917, to January 2, 1918.

The first general session of the Association was held in the Music Hall of Carnegie Institute on Friday evening, December 28. President Charles R. Van Hise of the University of Wisconsin, retiring President of the Association, delivered an address on the subject "The Economic Effect of the World War in the United States."

A meeting of Section C, Chemistry, was held on Friday morning, December 28, presided over by the chairman, Professor Wm. A. Noyes. The feature of the evening was the informal but exceedingly enjoyable address of the retiring chairman, Professor Julius Stieglitz of the University of Chicago, upon the subject "The Electron Theory of Valence and Its Application to Problems of Inorganic and Organic Chemistry." After an extended discussion of the address, Doctor David Horn of Bryn Mawr presented a paper on "A Chemical Study of Formalin Fumigation." A paper by J. Davidson of the Bureau of Chemistry, Washington, D. C., entitled "Do Seedlings Reduce Nitrates?" was read by title.

On Friday afternoon the Section met with Section D, Engineering, and the Society for the Promotion of Engineering Education.

On December 29, a joint symposium was held with Section E, Geology and Geography.

Officers for 1918 were elected by Section C, as follows:

Vice-President and Chairman: Alexander Smith, Columbia University.

Secretary: A. H. Blanchard, Massachusetts Institute of Technology.

Member of Section Committee: Benjamin F. Lovelace, Johns Hopkins University.

AMERICAN METRIC ASSOCIATION

The second meeting of the American Metric Association was held in Pittsburgh in conjunction with the meeting of the American Association for the Advancement of Science on December 28 and 29, 1917.

The sessions on the afternoon of the 28th and the morning of the 29th were held in conjunction with the Section on Social and Economic Science of the A. A. A. S. and at these sittings papers on standardization were read by J. W. McEachren of the Crane Company, Chicago, and by F. O. Wells of the Greenfield Tap & Die Co., Greenfield, Mass. In his paper, Mr. Wells pointed out that he employed 1700 hands and that he calculated that he would save $100,000 by the introduction of the metric system. Other papers were read by W. C. Wells, of the Pan-American Union, who discussed measures of volume in metric and other measurements, and by H. T. Wade who pointed out the importance of the metric system as a means of international standardization.

The session held on the afternoon of the 29th was presided over by Dr. John H. Brashear, of Pittsburgh, and was devoted to reports from President George F. Kunz, Secretary Howard Richards, Jr., and Treasurer A. P. Williams, showing the healthy condition of the association. Fred R. Drake read the report of the executive committee and outlined the activities of the association in the way of publicity and of coöperation with other national bodies. Dr. H. D. Hubbard, of the Bureau of Standards, gave an interesting address in which he pointed out some of the fallacies of anti-metric arguments.

In the evening there was held a metric dinner with a menu based on war-time conditions, the calorie value of each viand being expressed in exact units. At the close of the meal impromptu addresses were made, followed by an election of officers resulting as follows:

President: G. F. Kunz, of New York.

Vice Presidents: William Jay Scheffelin, of New York; E. P. Albrecht, of Philadelphia; and H. V. Arny, of New York.

Secretary: Howard Richards, Jr., of New York.

Treasurer: A. P. Williams, of New York.

ANNUAL MEETING TECHNICAL ASSOCIATION OF THE PULP AND PAPER INDUSTRY, NEW YORK CITY FEBRUARY 5-7, 1918

The annual meeting of the Technical Association of the Pulp and Paper Industry will take place in New York City at the same time as the annual convention of the American Paper and Pulp Association, the Waldorf-Astoria Hotel being headquarters for both associations. The program will consist of a symposium on acid sulfite manufacture and a discussion of problems relating to engine sizing.

NEW YORK SECTION OF THE SOCIETE DE CHIMIE INDUSTRIELLE

Following the presentation of the Perkin Medal to Mr. A. J. Rossi on the evening of January 18th at the Chemists' Club in New York City, a New York Section of the Société de Chimie Industrielle was organized. The Secretary of the parent organization, Lieutenant René Engel, addressed the meeting in terms of grateful appreciation of the coöperation of the American chemists with those of France. He traced interestingly the origin of the new Society.

The following officers for the Section were then unanimously elected:

President, L. H. Baekeland; *Vice-President*, Jerome Alexander; *Secretary*, C. A. Doremus; *Treasurer*, G. F. Kunz; *Executive Committee*, Charles Baskerville, H. Blum, M. T. Bogert, C. F. Chandler, Ellwood Hendrick, W. H. Nichols, R. E. Orfila, G. E. Valabrèque, E. P. V. Vergé, Henri Enrique Viteaux.

The constitution and by-laws for the new Section were adopted and after felicitous remarks by Drs. Baekeland, Nichols and Baskerville, the meeting closed with a strong address by Prof. V. Grignard of the French Mission.

NOTES AND CORRESPONDENCE

TWO LETTERS ON THE CHEMICAL CONTROL OF AMMONIA OXIDATION

Editor of the Journal of Industrial and Engineering Chemistry:

In a paper on the "Analytical Control of the Ammonia Oxidation Process"[1] Messrs. Guy B. Taylor and Joseph D. Davis refer to the present writer's article[2] and a brief statement seems necessary by way of reply or further elucidation.

Taylor and Davis assert that the "statements of Schönbein, Weith and Weber are not to be taken to mean that ammonia is oxidized by hydrogen peroxide abundantly under all conditions." But there is nothing in our paper that says they are so to be taken. We said that hydrogen peroxide abundantly oxidizes ammonia—which is true. We did not say that it happened under all conditions. The course of this chemical reaction, and for that matter, all chemical reactions, is determined by the conditions of temperature, concentration, etc. This is a sort of Theorem I of chemical dynamics, and is presumed in any discussion. It may well be true that under the conditions of analysis as used by Taylor and Davis no such oxidation occurs; we should be the last to dispute it as we have no data to dispute it with. It does not appear to us, however, that the evidence brought forward to show absence of oxidation is sufficient to prove the case, under all conditions of analysis.

In the same paper the authors state (p. 1109) that "the reaction $2NO + O_2 = 2NO_2$ occurs in measurable time." That is, it requires a measurable time for the specified equilibrium to be reached. To prove this, they cite five journal articles, most of which are very long and full of data having no relevancy to the issue. They give, however, no particular references; in fact they might as well have cited the literature *en masse.* Let us see what the literature says. In the first paper cited,[3] on p. 2135, we find the statement: "these experiments show that two volumes of NO and one volume of oxygen of different origins (*i. e.*, made by different manufacturing processes) at atmospheric pressure are practically completely transformed into NO_2 and N_2O_4." On p. 2134 is given a curve of time against pressure decrease, which shows that the reaction practically runs its course in half a minute or less. Thus with only the theoretical amount of oxygen (which Holwech used) the reaction is practically complete in less time than would be required to go through our apparatus, but our method of course requires a decided excess of oxygen.

Perhaps the most important paper for the case on hand is that of Foerster and Blich,[4] and the issue amounts to this—when the mixture of air or oxygen and NO is run into a dilute caustic soda solution, does the reaction take place as follows?

$$2NO_2 + 2NaOH = NaNO_2 + NaNO_3 + H_2O$$

The answer is, that it all depends on Theorem I above, and there is nothing in the article bearing on our work because Foerster and Blich did not duplicate our conditions. However the results given in the table on p. 2019 "Versuchsreihe" Experiment 16 (where the gases are dilute) and in Experiment 42 on p. 2021 come as near as any in the paper to being comparable. In both instances the reaction runs practically as specified in the equation above. We do not base anything, however, on these statements from the literature. What is said in our paper was based on the fact that the amount of nitrite found in the first absorber was close enough to the reaction given to justify the calculation. The fact is, that so far from being in the sense of more nitrite as would be presumed from Taylor and Davis's supposition, there was invariably less, *i. e.*, more than half the acid was nitric. The writer thought there might be a little ozone in the oxygen but as the corrections involved were small, he did not think the point worth pursuing. Doubtless the amount of nitrite and nitrate are much influenced by the concentration of the alkaline solution into which they are led. In the writer's apparatus certainly all the NO yielded NO_2 and a little N_2O_5.

However, Taylor and Davis admit that by our procedure all the nitrous gases are absorbed, and all their contention would amount to, even if valid, would be that the formula would have to be modified. One does not adjust his testing to a mathematical formula, but calculates what he wants to know from the data he can get most conveniently and accurately.

Curiously enough, Taylor and Davis in their literature citations overlooked the only paper that could have been cited with any effect. We refer to that by Mandl and Russ[1] who found that with some kinds of oxygen (*e. g.*, that from electrolysis and also that from barium superoxide, bichromate and sulfuric acid, but not that from liquid air) the reaction between NO and O_2 did not go to completion.[2] The objection would apply of course to any methods requiring oxygen, including those of Taylor and Davis. However, we were fortunate enough not to get hold of any such oxygen. Mandl and Russ think that the differences in oxygen of different origin may explain the contradictory statements in the literature on the behavior of NO and oxygen.

Method I of Taylor and Davis amounts substantially to our method in that they have added oxygen to the gases before absorption, the difference being that they omit the precautions to prevent the oxidation of ammonia by the hydrogen peroxide. The main fact is that nitric oxide (NO) is not nearly completely absorbed by alkaline hydrogen peroxide. Sufficient oxygen must be present to convert it into $NO_2(N_2O_4)$. The writer proved this repeatedly, when nitric oxide would go through three absorbers, two of them filled with alkaline peroxide and beads, only to burst into brown fumes on coming into contact with the air. The essential features of our method are to insure by previous addition the presence of the necessary oxygen, and to avoid the oxidation of ammonia which takes place under not well-understood conditions. Now, Taylor and Davis have previously added the oxygen but there is no certainty that it would be enough; this difficulty is avoided by adding an additional dose in the large (1200 cc.) displacement vessel after the absorption is completed. This plan, while not specially appealing to us on account of the double titration and the necessity of getting the acid out of the large container, will doubtless get all the nitrous gases provided the mixture coming from the catalyzer contains enough oxygen already to oxidize practically all the NO to NO_2. In fact, with the saturators at 7 or 8 per cent ammonia (as mentioned in the article) we suspect that little or no oxygen would be necessary. Supposing, however, that the gases from the catalyzer contain all the combined nitrogen as NO, with little or no oxygen, then only limited absorption[3] will take place in K″, and, especially in a cool place, there would be ample opportunity in an hour for part of it to dissolve in the displacement water and get lost. We see then that the method is available only for a highly special manner of operating the catalyzer, and will be satisfactory only so long as there is sufficient excess of oxygen present. The writer was never able to get complete

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[1] THIS JOURNAL, **9** (1917), 1106.

[2] *Ibid.*, **9** (1917), 737.

[3] Holwech, "Über die Reaktion zwischen Stickoxyd und Sauerstoff," *Z. angew. Chem.*, **21** (1908), 2131.

[4] *Z. angew. Chem.*, **23** (1910), 2017.

[1] *Z. angew. Chem.*, **21** (1908), 486.

[2] These results have not been confirmed by later workers, without, however, disproving them. Holwech, *Z. angew Chem.*, **21** (1908), 2131.

[3] Probably also irregular. See the reference to Schönbein in the writer's original paper.

absorption with only one absorber, but possibly the absorber K″ is better than his. The use of a manometer is not very convenient.

The same criticism applies to the evacuated bottle method as described. A sufficiency of oxygen is not assured, except under special conditions. Moreover, a pump capable of evacuating to 2 mm. is necessary as well as connections and capillary stopcocks so well ground that they will retain the vacuum mentioned. Fortunately a concession is made in the matter of ground glass connections. What all these mean, it is unnecessary to state. The statement that it is the only method permitting the determination of ammonia escaping oxidation presumably applies only to the procedures described in the paper, for it is perfectly feasible by our method. We are convinced that anyone trying the vacuum bottle method will find it exceedingly elaborate. Presumably the plan could be modified by adding a measured amount of oxygen first to the vacuum bottle, but it then becomes even more complicated. A larger bottle will also be required to cover all cases of gas mixture.

Messrs. Taylor and Davis present a method for eliminating titrations because "the principle involved offers possibilities for development of a rapid method of works control." The essential novelty of the principle however—the running of a gas into a definite volume of liquid stained by an indicator until the indicator turns—had been stated already by the present writer in the second and third paragraphs of his paper. They have added an elaborated glass apparatus which would impress us as being a great deal more troublesome than half a dozen titrations.

Finally, and this a crucial point, catalyzers do not work uniformly and the taking of a sample should extend over considerable time, in fact should be continuous. It seems to the present writer that his is the only process which has this advantage; it is also free from limitations on the composition of the gases. He hopes to publish shortly an account of an improved and more compact form of his apparatus, together with a new method of approximate factory control requiring less skill than any of the proposed methods.

PAUL J. FOX

1605 E. CAPITOL STREET
WASHINGTON, D. C.
December 11, 1917

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Editor of the Journal of Industrial and Engineering Chemistry:

In reply to criticisms of Mr. Paul J. Fox, on our paper "Analytical Control of the Ammonia Oxidation Process."

In regard to the oxidation of ammonia by hydrogen peroxide in alkaline solutions, we are convinced that no such oxidation occurs in any method used by us and would not occur in the method proposed by Mr. Fox, even if hydrogen peroxide were contained in his first absorption vessel. No experimental evidence of such oxidation is presented by Mr. Fox, and certainly none in the reference quoted by him.[1] However, the question is relatively unimportant since little or no ammonia is allowed to pass the oxidizer in commercial operation.

In regard to the second point at issue, the completeness of the oxidation of NO to NO_2, the literature cited by us and confirmed by our own experiments shows that this reaction is not an instantaneous one and has a negative temperature coefficient. The latter is important. If the gas is not cooled to room temperature even with a large excess of oxygen, there is no assurance that the reaction will complete itself unless a large reaction space is provided before the gases enter the alkaline absorbing solution. The apparatus sketched by Mr. Fox shows that the only reaction space provided is the narrow tube conducting the gases to the bottom of the absorption vessel. Since the gases must be kept hot till they enter this tube to prevent moisture condensation, it appears likely to us that the reaction does not complete itself before the acid oxides are absorbed by the alkali. But if Mr. Fox obtained practically equal quantities of nitrate and nitrite in his absorbers from the *hot oxidizer gases*, we withdraw the objection in our original paper.

We quite agree that NO is not absorbed by alkali or alkali containing hydrogen peroxide. But a mixture of NO_2 and NO in any proportions such that NO does not exceed that of NO_2 is more readily absorbed by alkali than NO_2 alone.[1]

In our aspiration method a partial reaction of NO to NO_2 was all that was required. The use of oxygen was to clear absorption vessel K″ of air at the beginning of the test and of the oxidizer gases at the end of the test and *not* to assist in the absorption as assumed by Mr. Fox.

In our opinion no aspiration method is very satisfactory. We do not agree that it is desirable to draw continuous samples or samples over a period of time. In fact, to the authors' knowledge, a commercial plant using an aspiration method similar to that advocated by Mr. Fox, has recently discarded it in favor of the vacuum method which they have recommended. The vacuum bottle method has been in use over a year under all kinds of experimental conditions and in actual plant operation, where it has proved satisfactory. When high concentrations of ammonia are being oxidized it is necessary to introduce a little pure oxygen after taking the sample, but it is not necessary to measure it and is no trouble whatever. One man with a little experience can make a complete efficiency test including calculation of results in half an hour if determination of the free ammonia escaping oxidation be neglected.

GUY B. TAYLOR
J. D. DAVIS

BUREAU OF MINES
WASHINGTON, D. C.
January 3, 1918

AVOIDABLE WASTE IN THE PRODUCTION OF SULFURIC ACID BY THE CHAMBER PROCESS

Editor of the Journal of Industrial and Engineering Chemistry:

In connection with the subject of the increased production of sulfuric acid called for on account of explosives requirements, it is interesting to consider one phase which seems to have escaped general observation. In the United States we make some four million tons of acid by the chamber process each year. Very few chamber plants are run on a scientific basis; in fact, most of them operate by rule of thumb, this being particularly true of the acid plants attached to fertilizer factories. While there has never been a survey made of the average operating conditions in the chamber plant acid industry, I am reasonably sure from the data I have gathered during the past five years that the average chamber plant space obtained by combining all the plants in the country would be of the order of 13 cu. ft. per pound of sulfur burned. With proper analytical control of the gases, and with exact control of the volume and temperature of the acid circulated over the towers there is no reason why the chamber space used should not be cut down to 11 ft. per pound of sulfur burned per 24 hours. Suppose, however, the average improvement is no more than a reduction to 12 cu. ft., we would have an increased output, *without the construction of additional plant*, of some 300,000 tons of 50° Bé. sulfuric acid per year.

A questionnaire sent to the acid manufacturers covering chamber acid output and chamber space per pound of sulfur, would soon show the possibilities of increasing our acid production in the way I have indicated, but, of course, such a query to result in answers of real value would have to be sent out by some department of the Government.

A. E. MARSHALL
Chemical Engineer

BALTIMORE, MD.
January 4, 1918

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[1] *Ber.*, 7, p. 1745.

[1] Foerster and Blich, *Z. angew. Chem.*, **23** (1910), 2017–25.

BROMINE PROCESS DECISION

According to the *Oil, Paint and Drug Reporter* for December 24, 1917, the case of the Dow Chemical Company *vs.* the American Bromine Company and Arthur E. Schaefer, which has been heard in both Midland and Wayne counties, Michigan, and which was transferred to the Detroit district last June, has been decided in favor of the plaintiff, the Dow Chemical Company, by Circuit Judge Kelly S. Searl.

This case was probably the most striking of recent suits in the chemical industry based upon alleged disclosure of secret processes by former employees, and also involving the ownership of patents covering processes.

The plaintiff, the Dow Chemical Company, asked a permanent injunction restraining the defendants from making use of certain trade secrets, claimed to be the exclusive property of the Dow Company and unfairly obtained from certain of its employees and also asked that a certain patent be transferred to the Dow Company, since through contract with the defendant Schaefer the property right in the patent was held to belong to the Dow Company.

The taking of the testimony has consumed weeks, and in addition to models, marks and charts, the ordinary exhibits numbered several hundred and several thousand pages of testimony were taken.

JUSTICE SEARL'S FINDINGS

The patent and the processes in dispute had to do with the manufacture of bromine and bromides from brine by means of an electrolytic cell and by other combined methods held in strict secrecy by the Dow Company and, according to the ruling of Justice Searl, used and copied by the American Bromine Company in its plant. Justice Searl, in his opinion filed on December 14, 1917, said in conclusion:

> "The parties who subsequently incorporated under the name of the American Bromine Company had apparently never heard of the Kossuth cell. They did not undertake to build such a cell for their plant, but instead they copied the Dow cell and are now using one so near like as to require that they be restrained from using the same longer. The fact that a patent had been taken out years ago in Germany for this Kossuth cell is of very little importance in this case, and the same may be said of the other German and American patents. The existence of the majority of these patents was unknown to the defendants until after the commencement of this suit. They did not secure the right to use them nor to build up their business in reliance upon disclosures from them which the public by reason of the expiration of the patents were entitled to use.
>
> "On the contrary they set out, as admitted by them, to build a plant as near like the Dow plant as possible and not infringe the Dow patents. Whether they have infringed any of the Dow patents is not a question to be decided in this case. * * * but they did succeed in building and equipping a plant in all essential respects like the Dow plant at a time when the plaintiff was operating the only plant of that kind in this country—or anywhere for that matter.
>
> "Plaintiff, having by reason of the inventions of Dow and others kept its processes secret and built up an extensive and thriving business, is now entitled to the relief prayed for in the bill filed in this cause.
>
> "A decree may be entered accordingly, including a clause requiring defendant Schaefer to assign his said patent to the plaintiff. And, inasmuch as the American Bromine Company have under their contract with Schaefer an interest in said patent, such defendant will also be required to relinquish the same.
>
> "The court will not at this time undertake to state the exact terms of the decree, as counsel are better prepared than the court to frame and agree upon the terms thereof. Each party may propose a decree, and if the same cannot be agreed upon or settled by the court without doing so, a day will be set apart for hearing counsel on both sides upon the settlement thereof. Plaintiff will recover costs to be taxed as against the defendant."

COMPLAINT AS FILED

The bill of complaint set forth: That Herbert H. Dow, of Midland, Mich., discovered the presence of bromine in certain brine from natural gas wells, and that after investigations in 1888 and 1889 he discovered a new process by which the bromine was extracted by blowing air through the brine and then recovered by bringing in contact with certain chemicals, resulting in the formation of desired bromides; that during 1889 he manufactured bromide of iron by this process; that on October 28, 1889, he applied for a patent issued September 29, 1891 (No. 460,370), and reissued April 12, 1892 (No. 12,232), and that he began the construction of a plant in Midland, Mich., in August, 1890. That subsequent investigations resulted in the development of an electrolytic process, and that all processes were conveyed to the Midland Chemical Company and kept secret, the works enclosed with a barrier, and specially guarded to prevent the details from becoming public, the manufacture of bromides being carried on in a separate building, also guarded. That the two bromide plants of the plaintiff have a present capacity of 1,500,000 lbs. a year.

That on July 9, 1904, plaintiff employed Arthur E. Schaefer, one of the defendants, as a chemist in its laboratory; that later Schaefer had charge of the plaintiff's bromide plant and its manufacture of bromides and the processes used, and that he continued in that capacity until September 15, 1905; that about February 1, 1908, he returned to the employ of the Dow Company and became the superintendent of the bromide plant and continued in that capacity till July 1, 1910; that the Emerson Drug Company, of Baltimore, and the Dr. Miles Medical Company, of Elkhart, Ind., through their principal stockholders or stockholding interests undertook the organization of the American Bromine Company on December 1, 1915, and bought lands and erected a plant at Midland; that the plaintiff was advised and believes a complete apparatus for the manufacture of bromide identical with that used by the Dow Company and including all the Dow secret devices and apparatus was erected; that to acquire the knowledge of the processes the defendant employed Julius Burow, A. M. Douglas, former employees of the Dow Company, and sought to employ James C. Graves, formerly general superintendent for Dow, but he refused; that afterwards Arthur E. Schaefer was employed by the American Bromine Company as consulting engineer and expert adviser; that while Schaefer left the employ of the Dow Company July 1, 1910, he was continued on the payroll for three months under special agreement; that Schaefer obtained a patent, No. 1,085,944, on a method for the recovery of bromine by the aid and for the use of a solution of ferrous bromide therein, claiming he was the inventor and that the process was in use in the Dow plant at the time Schaefer was employed there.

ANSWER TO COMPLAINT

The American Bromine Company in answer to the bill of complaint filed affidavits to the effect that although it had employed a Dow employee, one Douglas, and he had collaborated on the erection of a cell, the cell was not successful, and that another cell was developed and also a process differing in essentials from the Dow process. An improvement on the Dow process of purifying the bromide brine as it passed through the electrolytic cells was especially commented upon. The defendant denied any intention to appropriate any secret of the Dow process, and denied any effort or steps to do this.

The suit was to compel the disclosure of sums paid by the American Bromine Company or any other person for right to use patent 1,085,944. the assignment of the patent by Schaefer, and perpetual injunction against the use of processes of Dow, or the employment of any of the former or present Dow employees or of disposing of the patent to anyone else, together with an accounting, and that the existing devices and apparatus in the American Bromine plant be ordered destroyed, together with payment to the Dow Company of all sums found due them in accounting as assessed by the court.

OPINION OF JUSTICE SEARL

The court traced the steps taken by Dow in perfecting an improved process of recovering bromine, in place of the old boiling-out process used previously, instancing the use of electricity and the blowing-out process more fully developed later, and both of which have been in use by the Midland Chemical Company and Dow Chemical Company practically continuously since 1892. The Dow Company had practically no competition in America, and although German manufacturers were shipping some bromine to the United States, these shipments ceased at the outbreak of the European war.

Justice Searl then proceeded: That the Emerson Drug Company and the Dr. Miles Medical Company had purchased bromides in considerable quantities, the former company as high as 100,000 pounds a year on contract. With the increased price demanded a controversy arose, the purchasing concerns alleging that the Dow Company was charging an exorbitant price because having a practical monopoly. In 1915 the Emerson Company considered the advisability of engaging in the manufacture of bromide, and learned of Douglas and of the fact that the Meyer Brothers plant at Midland was for sale. A copartnership was formed by the Emerson Drug Company and the Dr. Miles Medical Company and the American Bromine Company was incorporated under the laws of Michigan in December, 1915, with the Emerson Company holding 60 per cent and the Miles Company 40 per cent of the Bromine Company stock.

The court then reviewed the correspondence between Douglas and the Emerson Drug Company, the discussion of the process to be used and the fact that Dow was using an electrolytic process. After all the correspondence and conferences "in which the founders of the American Bromine Company were practically informed in so many words that the Dow Company were operating under secret processes in addition to its patented processes, Douglas was employed and set to work to build a plant to make bromines and bromides, not by the use of bittern waters as he recommended, but by the electrolytic process."

"The officials of the Emerson Drug Company," asserts the court, "must have had full notice and knowledge that it was proposed to duplicate the plant of the Dow Company in all its essential details, except possibly where it might conflict with the patents held by the Dow Company." Later, Schaefer was employed to take the place of Douglas.

The court also says: "I find that except in some minor details the plant of the American Bromine Company is in its essential characteristics a duplicate of portions of the plant of the Dow Chemical Company and that in the manufacture of bromine and bromides from raw brine the American Bromine Company are now using the same processes as were used by the Dow Company at the time Douglas and Schaefer were employed by the latter company."

UNITED STATES TARIFF COMMISSION INQUIRY IN REGARD TO CHEMICAL INDUSTRIES

The Tariff Commission is undertaking an inquiry into the significant developments that have taken place in the chemical industries since the passage of the tariff act of 1913. Changes which seem likely to alter permanently the conditions of international competition or the course or volume of foreign trade are to be special subjects of study.

All persons having direct knowledge of pertinent facts in regard to any particular industry or product are invited to submit a statement of the Tariff Commission. Among the matters on which the Commission desires as full and complete information as possible are:

1. The manufacture within the United States of articles formerly unavailable or obtained exclusively by importation, for example, phosgene.

2. In the case of industries previously established in the United States, the erection of new plants or increase in capacity of existing plants; for example, the increase in capacity of existing plants for making caustic soda and chlorine and the installation of such plants at textile and paper mills.

3. The future of industries or establishments newly created, or in which productive capacity has been greatly increased to meet a direct war demand. How can these plants be utilized when the war demand disappears? For example, the acetone industry.

4. Any general or significant differences in the prevailing method of manufacture in the United States and abroad, such as the relatively small use of the carbureted water-gas process in England compared to the process in the United States.

5. Differences in the organization of the industry in the United States and abroad.

6. The development or invention in the United States or abroad of new or improved processes which are likely to influence the conditions of international competition; for example, the hydrogenation of fatty oils or the flotation process for concentrating ores.

7. Significant changes in the conditions of international competition caused by the recent law-making patents owned by citizens of enemy countries available to American manufacturers; for example, the patents on salvarsan.

8. Industries which have been seriously hampered in their normal operations or in their development by difficulty in securing materials or supplies formerly imported; for example, the lack of potash for fertilizer or glass. If these difficulties have been met by the introduction of substitutes, it is expected that there will be a return to the old materials and methods when foreign supplies again become available, or will the changes be permanent?

9. Developments or changes in other industries which have created a new or greatly increased demand for chemical products; for example, the manufacture of new varieties of glass in the United States.

10. The discovery of new uses of materials, creating a new demand or furnishing a market for materials formerly wasted; for example, the use of aniline as an accelerator in the vulcanization of rubber.

11. Any governmental hindrances in the United States or abroad, either in manufacture or commerce; such as the export duty on nitrate from Chile.

The Commission will publish only general statements or summaries, which will not reveal the operation or plans of individual companies.

SPECIAL CHEMICALS AND APPARATUS AVAILABLE THROUGH THE CHEMISTRY COMMITTEE OF THE NATIONAL RESEARCH COUNCIL

The Chemistry Committee of the National Research Council will endeavor to locate for our chemical investigators chemicals and apparatus *where the need is definite and urgent, and where the article required is not obtainable in the open market.* Inquiries for chemicals should be addressed to Prof. Roger Adams, University of Illinois, Urbana, Ill., who has already published lists of certain of the products which are obtainable through his office. Requests for aid in locating apparatus should go to Mr. A. H. Thomas, W. Washington Square, Philadelphia, the Chairman of our Sub-Committee on Chemical Apparatus.

As these gentlemen are carrying out this work as a patriotic service for the welfare and security of our country and without any remuneration for the labor involved, it is requested that investigators in need of such supplies make sure that the material required is not available through the ordinary commercial channels before turning to these colleagues for aid, as the burden of correspondence is already heavy. When the desired material is located, the inquirer will be put in direct communication with the owner, so that a loan or sale may be arranged. It will assist these gentlemen in their labors if chemists and physicists having special or unusual chemicals or apparatus available for loan or sale will forward full details concerning the same.

WASHINGTON, D. C.
January 14, 1918

MARSTON TAYLOR BOGERT

AS TO PLATINUM

Editor of the Journal of Industrial and Engineering Chemistry:

Much has been written of late, and more said, regarding the use of platinum in jewelry, and it has been broadly intimated that the jewelers are not living up to their agreement of last April with the Government. It is worth while to repeat the terms of this pledge of the Jewelers' Vigilance Committee.

> "We pledge ourselves to discontinue and strongly recommend to all manufacturing and retail jewelers of the United States that they in a truly patriotic spirit discourage the manufacture, sale and use of platinum in all bulky and heavy pieces of jewelry.
>
> "During the period of the war, or until the present supplies of platinum shall be materially augmented, we pledge ourselves to discontinue and recommend that the jewelry trade discourage the use of all non-essential platinum findings or parts of jewelry, such as scarfpin stems, pin tongues, joints, catches, swivels, spring rings, ear backs, etc., where gold would satisfactorily serve.
>
> "Be it further resolved that the jewelry trade encourage by all means in its power, the use of gold in combination with platinum, wherever proper artistic results may be obtained."

Having been in close touch with the platinum situation I desire to state from personal knowledge my belief that the jewelers have fully lived up to their pledges and in many cases gone beyond them in efforts to conserve platinum.

It was the manufacturing jewelers who entered into this agreement, and it is not surprising that some retailers have been making great efforts to work off their stock on hand, and have thereby opened themselves to criticism.

Whether a metal, so limited in supply and so invaluable in scientific industry, ought to be used at all in jewelry is a fair question, but it is not the question in point. A large and legitimate platinum jewelry industry has sprung up in recent years, and the question is whether the exigencies of the present platinum situation demand the immediate wrecking of this industry by having the Government commandeer all platinum; personally, I do not believe that at present they do.

I hold no brief for the jewelers, but I think this statement should be made in fairness to them; and I may add that it is my conviction, that, should the Government be placed in straights from lack of platinum for the manufacture of war material, the jewelry trade can be relied on to find a way of furnishing all that is needed.

Jas. Lewis Howe
Special Committee on Platinum,
Chemistry Committee of the National
Research Council

Washington and Lee University
Lexington, Virginia

PLATINUM RESOLUTIONS

At the recent Pittsburgh meeting of Section C (Chemistry) of the American Association for the Advancement of Science, the following resolution was unanimously passed:

Whereas (1) The Government of the United States has purchased from Russia and safely brought to the United States twenty-one thousand (21,000) ounces of platinum, the largest amount that has ever been shipped to this country;

(2) The separation of so great an amount of platinum will offer scientific investigators an opportunity to study the chemical combinations and mineralogical associations of the platinum group of minerals,

Therefore it is *Resolved* (1) That Section C, the Section on Chemistry of the American Association for the Advancement of Science, respectfully *request* that the War Industries Board, the Bureau of Standards, the Bureau of Mines, the American Chemical Society, and others who are interested in chemistry, be offered the opportunity by the United States Government to coöperate with it in the separation of the platinum group of minerals of the above-mentioned material, and that the residue, of which there may be thousands of ounces, be loaned to such scientific investigators who can undoubtedly obtain interesting scientific results, and

(2) If necessary, that one platinum works be commandeered, with proper compensation, for a certain length of time so that the work of separation can be carried on with the greatest care and observation, and

(3) That as much as possible of this platinum be loaned to those who have need of platinum for chemical investigation, the platinum to belong to the Government of the United States and subject to the call of the Government when needed for raw or industrial purposes. As the expense of making crucibles or other utensils is small in comparison to the value of the platinum, this would offer a most unique opportunity to the chemists of the country.

To present this matter to the proper authorities a committee was appointed consisting of Dr. W. A. Noyes, *Chairman*, and Dr. W. F. Hillebrand.

New York City
January 5, 1918

George F. Kunz

FUEL FOR MANUFACTURE OF CHEMICALS

Editor of the Journal of Industrial and Engineering Chemistry:

In view of the shortage in fuel supply and the great demand for large supplies of cheap fuel for the manufacture of chemicals and other products necessary for the war and agricultural purposes, I am bringing to your attention our unusual supply of natural gas at Shreveport, which is available to large industrial consumers on a basis that makes it cheaper than water power or that derived from the use of coal.

There is now available at Shreveport about one billion cubic feet of gas daily, and on the basis of scientific estimate the content of the Shreveport field is two trillion cubic feet, and a very small amount of this gas is now being utilized. This estimate includes only the field as already defined and does not consider other fields which are being opened up near by in drilling for oil. A prominent geologist has recently stated that the Shreveport field has the largest supply of natural gas to be found in the United States.

In addition to our gas supply, we have an abundance of raw materials that are needed for war industries at this time. There is close at hand an abundance of iron, petroleum, lignite, limestone, sulfur, and salt, and generous supplies of asphalt, gypsum, kaolin, sand, gravel, clay, etc. It ought to be especially noted at this time that Louisiana has the largest deposits of sulfur and salt to be found in the United States.

Shreveport, Louisiana
November 21, 1917

Ellis Smith

A STUDY OF THE ESTIMATION OF FAT IN CONDENSED MILK, ETC.—CORRECTION

In our article printed under the above title [This Journal, 9 (1917), 1111] the following changes should be made:

Page 1113, 1st col., line 18—"0.4" should read "0.04;" Table V, 3rd col., No. 8—"2.5005" should read "2.0505."

C. H. Biesterfeld and O. L. Evenson

COMPOSITION OF LOGANBERRY JUICE AND PULP—CORRECTION

In the article under the above title [This Journal, 9 (1917), 1043] note the following rearrangement of 5th line, Table I:

		I	II	III
Per cent Ash	Alkalinity	0.4139	0.5785	0.4226
	K_2CO_3	0.4130	0.5075	0.288

should read:

	I	II	III
Per cent Ash	0.4139	0.5785	0.4226
Alkalinity (as K_2CO_3)	0.4130	0.5075	0.288

M. R. Daughters

CHEMISTS AND THE DRAFT

Editor of the Journal of Industrial and Engineering Chemistry:

The information contained in the editorial in the January number of the JOURNAL under the caption "The Chemical Service Section of the National Army" must indeed be gratifying to the members of the Society in that chemistry is now accorded, for the first time, a definite and official place in the organization of the War Department's activities.

Naturally, the organization of such a new branch of service must be at present in its infancy, and subject to such alteration and revision as experience may indicate. At the same time there comes up in the mind of the writer the question—and the same question must present itself to many others—of what will be the status of drafted chemists who may be assigned to this service; that is, whether a drafted chemist will be given the same rank as he might have been given had he sought a commission instead of waiting to be drafted.

Any distinction between the rank assigned a volunteer chemist and a drafted chemist of the same training, who does the same type of work, must be an artificial one. Many chemists have been deterred from seeking commissions by considerations such as were expressed in Dr. Parsons' recent circular letter to the members of the Society; they have had to face the dilemma presented, on the one hand, by the impulse to volunteer their services for work bearing immediately on the prosecution of the war, and, on the other hand, by the obvious desirability of a continuation in their usual work, which, though it did not deal with explosives or poison gases or gun-metal, was yet a necessary contribution to the public welfare.

Is the drafted chemist to be given the rank of private, irrespective of what rank his training might reasonably be expected to entitle him to, merely because he has waited for the draft, the selective principle of which may be expected to utilize his ability most efficiently? The question might appear premature, if not foolish, were it not for the fact that some chemists called in the first draft have been put on chemical research in the capacity of privates. One inevitably draws a comparison to the conditions obtaining with regard to physicians. To the writer's knowledge, physicians drawn in the first draft have been commissioned when they were assigned to medical work. There can be no essential difference between the two cases. To be sure, the term "chemist" (covering as it does everything from a routine analyst to a trained researcher) is a much more flexible one than the term "physician," which in general represents a more uniform, though not always more intensive, degree of training. Yet it seems almost too obvious to say that the Ph. D. (or in many cases a lower degree) in chemistry, with some years of experience in the practice of the profession, represents as high a degree of training as does the M.D., often without any experience to back it up. The raw M.D. has been getting and does get a commission, if he is a capable graduate of a reputable school. May not the chemist expect equal consideration of the value of his services?

The writer believes that some statement on these matters, derived either from information the Editor may have, or from additional information from the War Department, would be welcome and illuminating to many readers of THIS JOURNAL.

EDWIN C. WHITE

JAMES BUCHANAN BRADY UROLOGICAL INSTITUTE
BALTIMORE, MD., January 16, 1918

WASHINGTON LETTER

By PAUL WOOTON, Metropolitan Bank Building, Washington D. C.

The outstanding feature of the month in Washington was the "workless day" order of Dr. H. A. Garfield, the fuel administrator. Many manufacturers of chemicals joined in the protests against the order which descended almost in the volume of an avalanche upon official Washington. Owing to the shortage in most chemicals, reasons were presented why many manufacturers of chemicals should be included in the exemption list. At the time this is written, however, J. T. Lewis Bros. Co., Lafayette Building, Philadelphia, manufacturers of chrome green, C. W. H. Carter, 8 Ferry St., New York, manufacturers of linseed oil, and all manufacturers of optical glass, are the only chemical industries which have been granted exemption. Numerous others were under consideration, however, and it is anticipated that various manufacturing chemists will be added to the exemption list.

During the past two months, decided increases have been attained in the manufacture of many much-needed chemicals. In fact, the achievements in this direction have been so decided that much of the uncertainty expressed as late as two months ago has been dispelled.

Greatest concern just at this time is centered on sulfuric acid, arsenic and ammonia, but the situation in each of these cases has been relieved measurably. Many of the uncertainties, which entered into estimates of the requirements of sulfuric acid for 1918, have been removed, showing that many of the estimates were too high. In addition, it has been possible to increase the productive capacity of existing plants and it has been found that considerable restriction in the use of acid can be practiced without the serious unsettling of the industries affected. These conditions combine to make the immediate situation less serious, while the activities of the War Industries Board looking to the construction of new plants give reassurance for the future. In this latter work, M. F. Chase is prominent. His new duties made it necessary for him to relinquish his work with the chemical division of the Committee on Raw Materials. A. E. Wells, the superintendent of the Salt Lake City experiment station of the Bureau of Mines, has been assigned temporarily to the War Industries Board to look after the work on acids which heretofore has been handled by Mr. Chase. Mr. Wells has been specializing on sulfuric acid for some time and has just completed a personal visit to practically every acid-producing plant in the country.

Special steps have been taken by the Fuel Administration to insure a supply of coal for the sulfuric acid plants. In order that this may be done intelligently, each manufacturer of sulfuric acid has been asked to report the amount of coal on hand, his monthly requirements and the name of the company supplying the plant with coal.

Commendation for Charles W. Merrill has been forthcoming from all concerned in the arsenic industry as a result of the arrangements which he brought about with regard to the regulation of profits and the restriction of use so as to insure ample supplies for noxious gas manufacture and for insecticides.

The licensing system has been extended to all those engaged in importing, manufacturing, storing or distributing ammonia, ammoniacal liquor or ammonium sulfate. The enforcement of the regulations which have been drawn up to cover this trade will be in the hands of an interdepartmental committee headed by Mr. Merrill. The other members of the committee, each of whom is identified, directly or indirectly, with chemical industry, are M. L. Wilkinson and Carl L. Alsberg, Department of Agriculture; Maj. Backus, Bureau of Ordnance; Lieut.-Col. W. H. Walker, Chemical Service Section of the National Army; Maj. M. J. Whitson, Cantonment Division, Quartermaster Corps; Admiral Ralph Earl, Navy Department; Maj. J. T. Crabbs, Interior Department, and L. L. Summers, Council of National Defense.

In the campaign for the conservation of ammonia, a propaganda is being carried on looking to the harvesting of as much natural ice as is possible.

A price of $75.50 per ton has been placed on the Government's supply of nitrate of soda. Approximately 100,000 tons of nitrates were purchased by the Government in Chile and have been transported to several American ports. It is to be sold directly to farmers, who must agree not to re-sell and to use it on their own farms.

Exports of chemicals during the first eleven months of 1917 reached the unusual value of $171,942,221. This is more than

$20,000,000 greater than the value of chemical exports during the corresponding period of 1916. During November, 1917, chemical exports totaled $15,428,809 in value. This was slightly under exports in November of 1916, when they amounted to $17,153,625. Sulfuric acid in November, 1917, amounted to 3,823,898 pounds, as compared with 2,975,602 pounds in November of 1916. For the first eleven months of 1917, exports of sulfuric acid amounted to 57,311,684 pounds, or approximately the same amount exported during the corresponding period of 1916 when exports were 60,361,638 pounds. The more striking increases in exports were in acetate of lime, calcium carbide and glycerine. Increased amounts of chemicals were sent to France, Italy, Spain, United Kingdom, Brazil and Japan. Decreased amounts were sent to Russia, Mexico and Canada.

In order to prevent undue inconvenience to the public and to avoid the handling of large numbers of licenses, a very general rule is being adopted by the Government agency issuing licenses to exclude druggists, wholesalers and dealers handling only secondary products. Persons using prime products solely as ingredients in the manufacture of products not subject to license also are being excluded.

The Bureau of Standards is taking up a study of methods of analysis for molybdenum, tungsten and metallic products derived from them. Samples are being prepared which will be sent out to be analyzed by a number of experts. Thus conclusions will be reached as to the adequacy of the comparative values of metals employed by various analysts. Out of these returns, it is expected to show just where methods should be improved.

The absence of opposition to the Garabed invention was emphasized, when, in the midst of a busy day, no one in the Senate objected when unanimous consent was asked by Senator James for its consideration. The bill had been reported favorably by the committee on patents and was passed without opposition or discussion. The Senate Committee made a few corrections in the phraseology but did not alter the salient features of the measure, which had passed the House. It is believed now that the President will sign the bill. This will put the matter directly up to Franklin K. Lane, the Secretary of the Interior.

While there was some decrease in the amount of iron pyrites imported in 1917, as compared with that brought in in 1916, the general policy with regard to imports remained unchanged. Manganese imports in 1917 were somewhat in excess of those of the year preceding. The shipping question has grown more acute, but still there is no organized effort to promote domestic production of these two important war minerals. In order to meet this situation, it has been found necessary to attempt to secure legislation. As a result, the War Minerals Committee drafted a bill which would place in the hands of the President, for the handling of minerals, the same wide powers with which he already has been vested for the control of food. The bill has been considered by the House Committee on Mines and Mining. Some amendments have been made, but the bill, with its main features, which follow almost exactly those of the Lever Act, is on the point of being introduced by Representative Foster, the chairman of the House Committee on Mines and Mining.

PERSONAL NOTES

Assistant Professor Reston Stevenson, in charge of physical chemistry in the department of chemistry in the College of the City of New York, has been appointed Captain in the Sanitary Corps of the Medical Department of the National Army, and is at present in France.

Mr. Howard Adler, assistant tutor in physical chemistry in the department of chemistry in the College of the City of New York, was detailed to duty in Camp Upton, Yaphank, and subsequently placed in the chemical service of the army.

Mr. Arthur Davidson, assistant tutor in the department of chemistry in the College of the City of New York, has been appointed in the chemical branch of the United States Army.

Dr. Ernest E. Smith, of New York City, has been elected president of the New York Academy of Sciences.

Professor Frederick G. Keyes, of the Massachusetts Institute of Technology, who has been commissioned Captain in the chemical section, has been granted leave of absence and expects to go abroad soon. Dr. Duncan MacInnes, now research associate in physical chemistry, has been appointed to serve in place of Dr. Keyes.

The main laboratory of the United States Fisheries Biological Station at Fairport, Iowa, was destroyed by fire on December 20. The station is the center of most of the scientific work of the Bureau of Fisheries in the Mississippi Basin.

Professor Wilder D. Bancroft, of the department of chemistry of Cornell University, is serving as technical adviser in the U. S. Bureau of Mines, Washington, D. C.

On January 12, 1918, Professor H. C. Sherman, of the department of chemistry of Columbia University, lectured before the Harvey Society at the New York Academy of Medicine on "Food Chemistry in the Service of Human Nutrition."

Dr. R. L. Kahn, pathological chemist of the Montefiore Home and Hospital, New York City, has been commissioned First Lieutenant in the Sanitary Corps, stationed at the Department Laboratory, Southeastern Department, Atlanta, Ga.

Dr. Chas. E. Vanderkleed, formerly director of the chemical laboratories of the H. K. Mulford Company, is now engaged in the manufacture of synthetic chemicals. He is vice president of the Markleed Chemical Corporation, New York and Camden.

Dr. Joseph Price Remington, Dean of the Philadelphia College of Pharmacy since 1893, died on New Year's Day.

Dr. Charles T. P. Fennel, for fifteen years state chemist in Ohio and later professor of chemistry in the Cincinnati College of Pharmacy, has been appointed to the chair of materia medica at the University of Cincinnati, to fill the vacancy created by the death of Dr. Julius Eichberg.

Professor Arthur W. Browne, of the department of chemistry of Cornell University, has been appointed chemical expert of the Ordnance Department. He will continue his work at Cornell University.

A General Science Hall, erected at a cost of $60,000, is under construction at Defiance College, Defiance, Ohio. It is expected that it will be completed next July.

Mr. C. L. Brickman was recently appointed chief chemist by the Rex-Hide Rubber Manufacturing Company, East Brady, Pa. He was formerly in the research laboratories of the United States Rubber Company. He is a graduate of Defiance College and the department of chemical engineering of the Case School of Applied Science.

The Lenz Apparatus Company, New York City, announces that Dr. W. J. Lenz is no longer connected with or in any way interested in that company.

Dr. F. E. Carruth, formerly of the Chemical Division of the North Carolina Experiment Station, is now with the Schaefer Alkaloid Works, Maywood, N. J.

The Hon. Sir Charles Parsons, K.C.B., F.R.S., member of the Council of the Institute of Metals (London), is to give the 8th Annual May Lecture before the Institute this spring. The lecturer will speak on the subject of the formation of diamonds.

Dr. E. P. Wightman, research chemist of Parke, Davis & Co., of Detroit, Michigan, and Windsor, Ontario, has enlisted as a chemist in the Gas and Flame Division of the Thirtieth Engineers of the U. S. A.

Mr. Charles S. Purcell, formerly of the Bureau of Mines, Pittsburgh, Pa., has been transferred to the Boston Station of the Bureau of Chemistry.

Professor Charles H. LaWall delivered, on January 17, an illustrated lecture on "Some New and Interesting Vegetable Foods," at the Wagner Free Institute of Science, Philadelphia.

A society for the furtherance of chemical knowledge and current scientific data has been organized by the technical chemists and chemical engineers of Tacoma, Washington. The president is Paul Van Horst and the secretary, B. H. Bennetts.

The work of the National Research Council has expanded so rapidly that it has outgrown the space available in the Munsey Building, and therefore the Council has rented the entire building at the corner of 16th and L Streets, N. W., Washington, D. C., occupied until recently by the Fuel Administration. The office of the Chemistry Committee will be in Room 24, second floor front.

Dr. H. P. Corliss, former fellow in the Mellon Institute of Industrial Research, Pittsburgh, Pa., is now with the Metals Recovery Co. as research chemist and metallurgist, with headquarters at the General Engineering Co., Salt Lake City, Utah.

Mr. Samuel Ginsburg has resigned from the Drug Laboratory of the New York Station of the United States Bureau of Chemistry to accept a position as chemist in charge of laboratory with the National Gum & Mica Co. of New York City.

Mr. Roy Richard Denslow, assistant tutor in the department of chemistry, College of the City of New York, has been appointed instructor in Smith College.

Miss Grace MacLeod has accepted the position of Assistant Editor of THIS JOURNAL. Miss MacLeod holds the degree of S.B. from the Massachusetts Institute of Technology and of M.A. from Columbia University, and for the past seven years has been instructor in chemistry at Pratt Institute, Brooklyn, N. Y.

In spite of adverse industrial conditions brought about by the war and severe weather conditions which interfered greatly with traveling facilities, the Short Course in Ceramic Engineering is entering upon the second week of its session with a registration of twenty-one men coming from ten different states and representing eight different branches of the ceramic industries, including the manufacture of brick, sewer pipe, drain tile, glass, grinding wheels, terra cotta, and also including representatives in geological surveys and the general engineering firms.

Mr. Robert J. Anderson has resigned as chief chemist and metallurgist of the Cleveland Metal Products Company, Cleveland, Ohio, to take effect February 1, 1918.

Dr. Julius Stieglitz has been elected president of the Institute of Medicine of Chicago.

Mr. W. J. Suer has accepted a position as chemist with the Ault and Wiborg Company of Cincinnati.

The committee on the analysis of commercial fats and oils of the Division of Industrial Chemists and Chemical Engineers of the A. C. S. now consists of W. J. Gascoyne, H. J. Morrison, J. R. Powell, R. J. Quinn, W. D. Richardson, Paul Rudnick, L. M. Tolman and J. J. Vallertsen.

The Powdered Coal and Engineering Company, Chicago, Ill., announce the addition to their engineering staff of Mr. Alex L. Feild, formerly of the Pittsburgh Experiment Station of the Bureau of Mines, and Mr. A. R. Detweiler, formerly with the Tribullion Mining, Smelting and Development Company, at Kelly, New Mexico, from which company he resigned with the purpose of undertaking private investigations with a view to inventing a process of recovering clay and slag from used retorts.

Mr. Benedict Crowell, of the firm of Crowell and Murray, chemists, Cleveland, has been appointed Assistant Secretary of War with the rank of Major in the Engineer Reserve Corps. He has done a great deal of ore analysis work for firms manufacturing and selling Lake Superior ores, and with Mr. Murray he is joint author of "The Iron Ores of Lake Superior." Major Crowell was born in Cleveland in 1869 and was graduated from Yale in 1891. On the completion of his college course he became chemist for the Otis Steel Company of Cleveland. He left that company to engage in business for himself as a chemist and later also took up mining engineering in connection with his other work.

The fifth convention of the National Foreign Trade Council will be held at the Gibson Hotel, Cincinnati, on February 7, 8 and 9, 1918.

Mr. Victor Yngve has been engaged as research chemist by the Oldbury Electrochemical Company of Niagara Falls, N. Y., and will have charge of their research laboratory.

Professor Wilder D. Bancroft, of Cornell University, lectured before the District of Columbia Chapter of the Sigma Xi, December 20, on "Colloid Chemistry."

Mr. H. A. Baker, chief chemist of the American Can Company, has gone to Washington to act on the committee on conservation of tin plate.

Dr. K. L. Mark, head of the chemistry department and of the School of General Science at Simmons College, Boston, has been granted a leave of absence for the duration of the war to accept a commission as Captain in the Sanitary Corps of the Army.

Assistant Professor Frederick E. Breithut, in charge of municipal chemistry in the department of chemistry, College of the City of New York, has been appointed Director of Food Conservation by the United States Government Food Commission to cover the territory of Greater New York City.

Mr. F. F. Beverly, of Sears, Roebuck and Company, has enlisted in the Sanitary Corps, Gas Defense Service. He is now employed at Akron, Ohio, inspecting gas masks.

Mr. Albert King, of the laboratory of Swift and Company, has joined the Gas and Flame Regiment.

Dr. J. W. E. Glattfeld has been appointed a member of the committee on the supply of organic chemicals for research during the war.

The death is announced of Professor G. P. Girdwood, professor of chemistry in McGill University.

Dr. Lawrence J. Henderson, professor of biological chemistry in Harvard University, will give a series of lectures on food conservation at Smith College.

INDUSTRIAL NOTES

Mr. Arthur W. Kinney, Industrial Commissioner, Los Angeles Chamber of Commerce, in the following notes tells of the activity in the development of the mineral and chemical industries of Southern California:

The mineral output for the year 1916 was valued at $22,809,461, petroleum being the largest single product obtained. Second in importance of production was tungsten ore and concentrates which reached the astonishing figure of 1931 tons, valued at $3,915,434. The value of the mineral output for 1917 is not obtainable, but it will undoubtedly show a considerable gain owing to the prevailing high price of petroleum and the important increase in the output of potash products. Potash is now obtained in large quantities in this region from four sources: the Searles Lake deposits, kelp, sugar waste and from the various cement plants. One of the latter now employs a leaching system installed for preparing high-grade potash salts.

In connection with its oil refinery at Fillmore the Ventura Refining Company has expended several hundred thousand dollars in the construction and equipment of a wax extraction plant. Wax will be taken from lubricating stock and handled in large quantities as a by-product.

Oil refineries have shown a greatly increased output. Various refineries, notably the Standard Oil Company and the General Petroleum Company, have spent large sums of money in additions and equipment. The former company has expended over one million dollars at the El Segundo refinery alone and has shown a large increase in its output of lubricants. The latter company has installed furnaces for the cracking of low-grade distillate into synthetic gasoline.

Southern California iron ore deposits have been found to be extensive in quantity and of high quality. Experiments have demonstrated that this ore can be reduced through the medium of natural gas and electric furnaces. Six companies have introduced electric furnaces for various purposes during the year. The Union Tool Company, at Torrance, has installed an open-hearth steel furnace, making a total of three companies thus equipped. There is a great demand for the utilization of Southern Californian resources of iron ore and a great steel industry is promised in the not far distant future.

At Los Angeles harbor the Union Oil Company has commenced construction on the first unit of a two million dollar refinery for the manufacture of lubricating oils, gasoline, kerosene and other petroleum products. This company operates a fleet of tank steamers and not only assists in supplying the industries of the Pacific Coast, but is a large exporter of cargoes of refined and lubricating oils to Australia, New Zealand, and the Hawaiian Islands, and fuel oil to the west coast of Chili.

The Chemical Production Company, Los Angeles, is erecting a fire-proof plant at Owens Lake, Inyo County, for the manufacture of soda ash. This will make the fourth establishment of its kind now operating at this point and the second built there during the present year, the other being the California Alkali Co., at Cartego.

The Stauffer Chemical Co. is erecting a $100,000 plant on a 15-acre tract in Los Angeles. The main buildings will be of reinforced concrete, and hydrochloric acid will be the principal product manufactured.

In addition to the production of molybdic acid salts the Rose Chemical Company, Los Angeles, has made commercially during the year in its electric furnaces, ferrotungsten, ferromolybdenum and ferrochrome. This company will be soon operating three furnaces using electricity in the reduction on a large scale of California and Arizona ores. The American Alloy and Chemical Company has also installed an electric furnace for the making of ferrotungsten.

The Palau Metals Co. has erected a refinery in Los Angeles. Machinery has been installed for the reduction, by a new process, of platinum, palladium and other metals, the ore being brought from Nevada. This plant is said to be the only palladium refinery in America, and the only one in the world handling this ore in the rock.

The Pacific Refractories Company, at Vernon, is now manufacturing magnesite brick from magnesite produced in this region. For a long time California magnesite has been shipped east for the making of brick.

Another new chemical plant in the Vernon district is the California Chemical Company. This company, operating its own oil refinery, is manufacturing a full line of orchard sprays.

At Long Beach the Western Chemical Co. is successfully manufacturing strontium nitrate from celestite, a mineral found in Imperial County. F. G. Mortimer, of Imperial County, is also engaged in the making of this product, which is largely used by manufacturers of fireworks and railroad fuses.

At Searles Lake two potash companies, the American Trona Company and Solvay Process Company, have continued to produce large quantities of chloride of potash. The former company has made additions to its refinery at Los Angeles harbor, costing several hundreds of thousands of dollars, and is now shipping potash, soda and borax in large quantities.

The Western Calcium Chloride Syndicate has equipped buildings at 2472 Hunter Street, Los Angeles, and is producing calcium chloride.

The Southern Reduction Co. at Vernon is making chloride of lime and caustic soda.

At Santa Barbara the U. S. Department of Agriculture, under the direction of Dr. J. W. Turrentine, has erected at an expenditure of one hundred thousand dollars an experimental kelp potash plant. The principal product manufactured thus far is kelp ash.

White oxide of antimony, said to be the best product ever offered in this country and exceeding in solubility the highest grade of the French product, is now being made by the Western Metals Company at Harbor City.

The Crystal Hills Mine, Inc., has just completed a plant at Huntington Park for the making of salts of aluminum and magnesium sulfate, drawing on the extensive salt beds of Inyo County for raw material.

The oxygen plant of the Linde Air Products Company, completed early this year, is now in full operation. The California-Burdett Oxygen Company, making the same product at Vernon, has erected new buildings and greatly enlarged its facilities. The shipbuilding industry calls for a large supply of oxyacetylene gas.

An important industry, the manufacture of oil from soya beans, has been inaugurated at Vernon by the Globe Oil Mills Company. This company has recently received, direct from Russia, a cargo of 9,300 tons of these beans and is grinding the same at the rate of 120 tons a day. The oil is refined, washed, bleached and filtered and the material left after its extraction is made into oil meal cake. This industry in connection with the manufacture of cottonseed oil and cocoanut oil will enable the Globe Company to keep its plant busy every working day in the year and thereby give employment to a large number of people.

At San Diego the Lower California Chemical Co. is making orcein dyes, using as raw material the orchilla, a moss found growing in vast quantities along the western coast of lower California.

The Western Aniline Products Co. is installing machinery and equipment in a fire-proof building at Tropico for the purpose of manufacturing photographic developers and kindred coal-tar products. The former are to be used as a substitute for the metol which is no longer obtainable in the market. The new product has already been used satisfactorily by the motion picture film laboratories and others in Los Angeles.

At Pasadena the Rare Metals Refining Co. has erected a modern plant and is engaged in the refining of various precious metals.

The N. C. Ward Co., Los Angeles, has installed and is operating a complete plant for the manufacture of permanganate of potash.

At Inglewood the Graphite Products Company is equipping a factory for the making of crucibles and carbon electrodes.

At Corona, the United Chemical Company, recently organized, is equipping a factory for the manufacture of citric acid and other citrus by-products. This will be the second concern engaged in this business at this place, the Citrus By-Products Company, a coöperative organization composed of various members of the California Fruit Growers' Exchange, having opened a plant last year to handle cull lemons in that district.

We learn from the *Journal of Commerce* that the development of the quicksilver deposits and the sulfur beds of the upper border region of Texas has been greatly stimulated by the existing high prices of these minerals.

It is reported from Washington that the Federal plant at Summerland, Cal., started as an experiment to demonstrate the possibility of salvaging potash from giant kelp on a commercial scale, has already proved a success. Dr. F. W. Brown, in charge of the office of fertilizer investigations, who is father of the project, has returned from an inspection trip much pleased with the way the plant is working. As a result of the success attained in California, an effort will be made to establish a similar industry on the Atlantic coast. Mr. Brown expects to start preliminary work soon on the Florida coast, where the conditions are nearly the same as on the California coast.

A statement by the Bureau of Foreign and Domestic Commerce says the United States is now producing enough aniline dyes to meet all domestic demand and leave a surplus for export.

Announcement has been made of the formation of the Dixie Gas Company at Anniston, Alabama, under the laws of Delaware, with a capital stock of $2,500,000. This company will develop the oil and gas possibilities of Alabama on a huge scale.

The International Coal Products Corporation of Trenton has been incorporated under the laws of New Jersey with a capital of $11,000,000, by G. W. Bacon, C. L. Blair and J. B. Dennis, of New York.

It is reported from Germany that Prof. Thoms, of Berlin, has been making extensive experiments in poppy cultivation. It is stated that he has succeeded in obtaining from poppies opium containing no less than 22 per cent of morphine, and greatly superior to the Turkish and Bulgarian opium in morphine content.

Merck & Company have increased their capital from $250,000 to $1,000,000.

A company has been formed in Japan for the manufacture of fish grease used in the manufacture of soap and glycerin.

The U. S. Potash Products Company, capital $5,500,000, has filed a charter under the laws of Delaware. The company is organized to produce and market potash and alum. The incorporators are Dormann T. Connet, White Plains, N. Y., John F. Roach and Clarence E. Bohn, of New York City.

Amalgamated Dyestuff and Chemical Works of Manhattan has increased its capital from $50,000 to $500,000.

We learn from the *Textile World Journal* that the paper industry has found a new use for sulfonated castor or Turkey-red oil. This commodity is extensively used in cotton finishing and dyeing. It has been found in a practical application that a small quantity of an alcoholic solution of this oil prevents the formation of foam in the beaters.

A method of determining manila fiber in rope and twine has been discovered in the research department of Arthur D. Little, Inc., Cambridge, Mass. The method has been adopted by the United States Bureau of Standards. Briefly, it consists of freeing the rope from oil, soaking it for twenty seconds in a solution of bleaching powder acidulated with acetic acid, rinsing in water, then in alcohol, and finally exposing it for a minute to the fumes of ammonia. Manila fiber turns russet-brown while all other rope fibers turn cherry-red.

The Mallinckrodt Chemical Works of St. Louis on December 19 celebrated the fiftieth anniversary of the founding of its business by giving a banquet at the Mercantile Club, St. Louis. About one hundred and fifty guests were present, including the representatives of the various branches. An important feature of the evening was the presentation to Edward Mallinckrodt, Sr., president of the company, by the employees, of a beautiful bronze tablet or medallion prepared from one of his recent photographs.

An explosives manufacturing plant to require an investment of $60,000,000 will be built by the Government at Hadley's Bend, on the Cumberland River, near Nashville, Tennessee. From 4000 to 5000 acres of land will be utilized for the site and the buildings will cover about 2000 acres. It is planned to begin explosives manufacturing within ten months. This factory is one of the several big explosives manufacturing plants which the Government has decided to build in connection with its $90,000,000 expenditure for this purpose.

Secretary Lane has recommended to Congress an appropriation of $100,000 to investigate the commercial and economic practicability of utilizing lignite coal for producing fuel oil, gasoline substitutes, ammonia, coal tar and gas for power. There are immense quantities of lignite deposits in public land but the coal is of such character that it does not stand transportation in its natural state and is of small value for fuel except in the immediate vicinity of the mines.

The Carbo-Hydrogen Company of America, with offices at Pittsburgh, has increased its capital from $3,500,000 to $5,000,000.

Castor oil used in dyehouse preparations and for lubricating airplane engines has become so scarce that the Government, in view of the latter requirement, is arranging for the planting of 100,000 acres, 40,000 of which lie in Florida, with the India castor bean. Contracts will be made with farmers in suitable sections who will plant not less than 1000 acres, the Government undertaking to purchase the beans at a fixed price.

The United Chemical Company, Wilmington, Del., has been incorporated to manufacture chemicals and allied products. The capital is $3,000,000. Incorporators: M. M. Clancy, C. L. Rimlinger and C. M. Egner, Wilmington.

According to estimates of the U. S. Geological Survey, Department of the Interior, the production of magnesite in California in 1917 exceeded that of all former years, being estimated at 215,000 tons. This quantity and the magnesite produced in the recently discovered field in Stevens County, Washington, estimated at close to 100,000 tons, makes an output of 315,000 tons in 1917, or 15,000 tons more than the normal domestic demand.

Regarding the explosion at the extensive Griesheim-Electron chemical works in Germany recently, "Lloyd's List," in an article on the explosion, attributed to an authoritative source, says:

"It will be remembered that on November 22 it was reported that the large chemical factory of Griesheim-Electron, near Frankfort, had been destroyed as the result of an explosion. No details were obtainable at that time, and no great attention was paid to the report in this country, no doubt from desire to avoid exaggeration of what might after all prove to have been an affair of no great importance.

"But the extreme care taken to prevent fuller accounts from leaking out from Germany and the enforced silence of the German press on the subject are the best proof of the German Government's anxiety to conceal a very serious loss. The first telegram which managed to escape from Frankfort made a significant admission, which the subsequent silence only confirms. It announced that the excitement in Frankfort caused by the explosion was tremendous. Information which has since been obtained from perfectly trustworthy sources makes it clear that there was good reason for excitement. For it is now certain that the explosion caused the complete destruction of one of the greatest munition factories in the world, by which Germany has suffered a disaster comparable to a very serious military defeat in its effect on the issue of the war.

"Under these circumstances it is a matter of supreme interest to understand precisely to what extent German military equipment was dependent on the source of supply which was wiped out of existence a few weeks ago.

"The Griesheim factory was situated in the neighborhood of Frankfort with an extensive frontage on the river Main. It consisted of an enormous group of buildings covering an area of over 54 acres. Twenty-eight large chimneys, one of them over 200 feet high, gave the impression more of an industrial town than a single factory; and numerous piers abutting on the river, combined with an extensive railway system, enabled this huge concern to distribute its products among the world's markets economically and quickly. Before the war it ranked as fourth in importance of the great German chemical works, and was always a flourishing company, paying a pre-war dividend of 14 per cent, and worth, as a going concern, well over 60,000,000 marks.

"Its commanding position in the chemical world rested not only on its huge output, but on the extensive variety of its manufactures. These comprised, among other things, aniline dyes of every description, nitric, sulfuric and other acids, phosphorus, and alkali, with liquid chlorine, hydrogen and oxygen as important by-products. What it meant to Germany as a source of munitions of war can thus be readily understood. Moreover, as one of the uncommon instances among German chemical works possessing installations for electrochemical production, it was of prime importance as a source of synthetic nitrates; and its splendidly organized research laboratory enabled it to play a leading part in the production of poison gas, and the other more refined forms of frightfulness which Germany has introduced in the course of the war. That the Imperial Government has taken the fullest advantage of these facilities is shown by the rapid increase of the works both in extent and output since the beginning of the war, and by the fact that the company has recently decided to increase its share capital by 50 per cent, an increase in which the German Government is more than suspected of having a financial interest.

"With regard to the productive capacity of Griesheim some authoritative facts are available which cast an interesting light on the war activities of this concern. It has been producing saltpeter for the manufacture of black powders at the rate of 1,000 tons a day, and it is reputed to be the only factory turning out this article. To such an extent has its already impressive output of soda nitrate and concentrated sulfuric acid been developed that it supplied the whole demand of five nitroglycerine and dynamite factories, as well as two powder works, including that of Rottweil, one of the most important in Germany. Another explosive, which it manufactured in large quantities, was tonite, through its facilities for making synthetic phenol and consequently picric acid, from which acid this explosive is derived.

"Another circumstance of special interest to us is the fact that this factory supplied large quantities of electrolytic hydrogen for the inflation of Zeppelins, and possessed by way of a reserve three gasometers with a total capacity of over 300,000 cubic feet. So important was it in this respect that a Zeppelin shed, usually containing two or three airships, was erected in close proximity to the works. For the kite balloons at the front the gas was supplied in steel tubes in the liquefied state. Moreover, the extensive electrolytic plant was further utilized to produce asphyxiating gas and lachrymatory and poisonous shells. Indeed it was the greatest center of this manufacture in Germany, and in 1916 the output of poison gases reached the colossal figure of nearly 600,000 cu. ft. a day.

"The extent of the material loss which Germany has suffered by the destruction of the Griesheim factory can thus be easily comprehended. But the disaster is of still wider significance. The variety of the materials formerly produced means, in such a closely interlocking industry as chemical manufacture, that every concern in Germany is affected, both from the cutting off of supplies which many of them formerly drew from Griesheim and from the necessity of making the loss of these supplies good from plants already working to their maximum. The deaths of scores of trained workmen and specialists in the factory itself, and in the dwellings within its confines, will make the task of coping with this deficit all the more difficult.

"Even if the material loss can be successfully replaced, the problem of collecting miscellaneous quantities of explosives and acids from various quarters and conveying them over the greater distances thus made necessary is bound to complicate further the transport difficulty, already regarded as exceedingly pressing in Germany. It is impossible that the works can be reconstituted during the war, and sooner or later Germany must show on her fighting fronts the effects of this staggering blow which she has suffered within her own territory."

GOVERNMENT PUBLICATIONS

By R. S. McBride, Bureau of Standards, Washington

NOTICE—Publications for which price is indicated can be purchased from the Superintendent of Documents, Government Printing Office, Washington, D. C. Other publications can usually be supplied from the Bureau or Department from which they originate. Commerce Reports are received by all large libraries and may be consulted there, or single numbers can be secured by application to the Bureau of Foreign and Domestic Commerce, Department of Commerce, Washington. The regular subscription rate for these Commerce Reports mailed daily is $2.50 per year, payable in advance, to the Superintendent of Documents.

GEOLOGICAL SURVEY

Gypsum in 1916. R. W. Stone. Separate from Mineral Resources of the United States, 1916, Part II. 7 pp. Published October 30. In 1916 for the first time the total value of the gypsum products of the United States in a single year exceeded $7,000,000. Since 1912, inclusive, the annual output has been approximately 2,500,000 tons of raw material, but in 1916 the total production was over 2,750,000 short tons.

As might be expected from the general tendency of the times, the average price per ton for gypsum and most gypsum products shows a large increase over the price in 1915. The increase is least for crude gypsum sold to Portland cement mills and greatest for dental plaster. Dental plaster is reported as produced in six states and at prices ranging from $6 to $23.50 per ton, with an average price of $13.26 per ton. In former years it has ranged from $3 to $15, with an average price of about $5. This greatly increased average price in 1916 is due to the fact that the higher-priced material largely predominated in the output.

The increased average price per ton of all calcined plaster in 1916, due to higher wages and cost of all supplies, will doubtless be greatly exceeded in 1917.

Gypsum imported into the United States comes almost wholly from Nova Scotia and New Brunswick and enters the ports of the New England and North Atlantic States. The proportionate value of imports to domestic production is very small and decreasing. It was about one-seventeenth in 1915 and one-twenty-second in 1916.

Peat in 1916. J. S. Turp. Separate from Mineral Resources of the United States, 1916, Part II. 2 pp. Published November 19.

Peat was used in the United States in 1916 mainly for fertilizer, soil builder and as stock food. Small quantities were used for peat litter and fuel, and, as was predicted in the report for 1915, very little peat fuel was produced in this country in 1916.

During 1916, as in 1915, no new processes or machinery for preparing peat were reported to have been commercially tried in the United States, but several firms reported additions and improvements to their plants. The total number of firms reporting production in 1916 was 13, all but one of which sold peat for fertilizer. Two also sold peat for stock food. One sold a small quantity for fuel. The 9 firms that furnished data for 1915 furnished data also for 1916, and 4 firms reported that were not represented in 1915.

Peat Produced, Imported and Consumed in the United States in 1916

Use	Production Quantity Short Tons	Production Value	Imports Quantity Short Tons	Imports Value	Consumption Quantity Short Tons	Consumption Value
Fertilizer and fertilizer filler....	48,106	$336,004	...	.....	48,106	$336,004
Stock food.......	4,300	32,250	...	.....	4,300	32,250
Miscellaneous....	100	850	...	.....	100	850
Peat moss litter..	....	......	3,042	$27,859	3,042	27,859
	52,506	$369,104	3,042	$27,859	55,548	$396,963

Hydraulic Conversion Tables and Convenient Equivalents. Anonymous. Water Supply Paper 426-*C*, from Contributions to the Hydrology of the United States, 1917. 24 pp. Published October 31.

The Alaskan Mining Industry in 1916. A. H. Brooks. Bulletin 662-*A*, from Mineral Resources of Alaska, 1916-*A*. 62 pp. "This volume is the thirteenth of a series of annual bulletins treating of the mining industry of Alaska and summarizing the results achieved during the year in the investigation of the mineral resources of the Territory. In preparing these reports the aim is prompt publication of the most important economic results of the year.

"This volume, like those previously issued, contains both preliminary statements on investigations made during the year and summaries of the conditions of the mining industry, including statistics of mineral production. It is intended that this series of reports shall serve as convenient reference works on the mining industry for the years which they cover."

Mining in the Lower Copper River Basin and the Prince William Sound Region, Alaska. F. H. Moffit and B. L. Johnson. Bulletin 662-*C*, from Mineral Resources of Alaska, 1916-*C*. 66 pp.

Manganiferous Iron Ores. E. C. Harder. Bulletin 666-*EE*. 13 pp. The dependence of the United States on imported high-grade manganese ore and ferromanganese is well known to steel makers and other users of manganese. Normally the high-grade manganese ore produced in this country constitutes less than 2 per cent of the total amount of manganese ore consumed, not including the ore represented by the manganese imported in the form of the alloys—ferromanganese and spiegeleisen. During 1916 the domestic production was about three times that of 1915, largely on account of the high prices paid for ore. The exploration for new deposits has also been stimulated, and many discoveries of manganese ore are being reported. Even with this outlook for increased production, however, it is not unduly pessimistic to say that the deposits of high-grade manganese ore in the United States will probably never be able to supply the manganese consumed in domestic industries. If, therefore, the importation of high-grade manganese ore were discontinued, numerous industries would be vitally affected. Of these the steel industry consumes by far the largest quantity of manganese.

There are in the United States large quantities of manganiferous ores containing varying amounts of manganese. A very small proportion of these can be used in the production of high-grade iron-manganese alloys, but a large proportion can be used for lower-grade alloys, and nearly all can be used in making high-manganese pig iron. Compared with the manganiferous ores, the reserves of high-grade manganese ores in this country are insignificant. Hence, although a search for manganese ore is desirable, a more promising solution of the manganese problem would seem to lie in the direction of the utilization of low-grade manganiferous ores. Up to the present time the use of these ores has been very slight. Until a few years ago they were considered to have little value and were mined only incidentally. In the West manganiferous ores would not be mined were it not for their association with ores of other metals.

There are several ways in which the utilization of manganiferous ores may be brought about: (1) It has been suggested that by methods of concentration resulting in the elimination of iron, silica, or other constituents a product high in manganese might be derived from them. Such concentration has been attempted locally but with very little success, owing mainly to the intimate mixture which manganese generally forms with associated materials; (2) The steel-making practice might be changed so that more spiegeleisen and less ferromanganese would be used for deoxidizing. By the addition of small quantities of high-grade manganese ore much of the manganiferous iron ore could be used in the manufacture of spiegeleisen; (3) The most effective solution, however, as has previously been suggested, seems to be to so change the practice in the manufacture of basic open-hearth steel as to make possible the use of high-manganese pig iron. Experimentation along this line is extremely desirable. The successful application of such a change would make large reserves of manganiferous iron ore commercially available and would greatly decrease the quantity of high-grade manganese ore consumed.

BUREAU OF FISHERIES

The Menhaden Industry of the Atlantic Coast. R. L. GREER. Document 811, 30 pp. Issued October 18. Paper, 10 cents. "Descriptions of methods of conducting the fishery, vessels, factories, apparatus, and methods employed in converting the fish into oil and scrap, and includes statistics of the fishery for 1912 and selected bibliography of papers relating to the subject."

BUREAU OF STANDARDS

The Latent Heat of Pressure Variation of Liquid Ammonia. N. S. OSBORNE AND M. S. VAN DUSEN. Scientific Paper 314, 6 pp. Issued November 16. Paper, 5 cents.

COMMERCE REPORTS—DECEMBER, 1917

Lubricating oil is being made from the liquid rosin obtained in sulfite paper pulp factories in Sweden. (P. 849.)

Efforts are being made to develop **salt** beds in Holland, to replace salt hitherto imported. Salt brine and solid salt have been encountered in exploration work. (P. 853.)

Copper pyrites mines in Norway, formerly German owned, have been purchased by Swedish firms. (P. 871.)

A new type of hard **porcelain** has been developed at Stoke-on-Trent (England). It is made entirely of British materials, is cheaper than ordinary earthenware, and the glaze (which is leadless) is very satisfactory, and can be readily decorated. The ware can be fired in either oxidizing or reducing atmospheres, preferably the latter. (P. 872.)

American exports of **aniline dyes** are constantly increasing, being now equivalent to about $4,700,000 annually, *i. e.*, about twice the domestic production in 1914. A similar increase has occurred in the exports of vegetable colors. (P. 903.)

Efforts are being made to produce **paper** in Australia from native grasses. Newsprint paper is now very scarce, and costs four times the pre-war price. (P. 918.)

The **tin plate** situation in the United States is improving, as shown by increased imports of pig tin and palm oil (used in tinning), and by decreased exports of tin plate and terneplate. (P. 947.)

It is proposed to subsidize for three years any organization that will establish a **paper** factory in the Philippines. (P. 965.)

Exports of **manganese ore** from Brazil to the United States are five times those of 1913, and could be still more increased if the rich regions could be made more accessible. (Pp. 971 and 1155.)

The Japanese **steel** industry shows an increase of over 50 per cent above last year's production. (P. 975.)

To provide **steel** for shipbuilding and other industries, a large modern steel plant is to be erected in Holland at the entrance to the North Sea Canal; and will use imported iron ores. (P. 978.)

The conditions and methods in use in the Scottish **shale oil** industry are described in some detail. The principal products are naphtha, burning oil, gas oil (used as fuel and for enriching gas), lubricating oils, paraffin, coke, gas (illuminating), and sulfate of ammonia. (Pp. 990–1.)

The British Government has assumed control of all **vegetable oils** and oil seeds, and hydrogenated oils. (P. 993.)

Imports of **chemicals** into Great Britain in October showed a slight increase, while exports showed a marked decrease. (P. 1031.)

The **iron ore** and **steel** industry of the Biscayan Provinces of Spain is described in some detail. (P. 1128.)

Proposed **substitutes for gasoline** as motor fuel in England include denatured alcohol, coal gas and kerosene. The first is not possible with the present demand for alcohol, though efforts are being made to increase the cultivation of potatoes to be used for industrial alcohol. The use of coal gas, especially in busses, etc., is increasing. Containers are usually made of fabric made gas-tight by various coatings. Kerosene is not extensively used, as it requires special fittings, etc., and involves the use of gasoline for starting. (Pp. 1132–5.)

Experiments were performed in India upon three new **paper-making** materials: *E. monostachya* leaves from Western Australia, wood of the *N. macrocalyx* from East Africa, and bark of *brachyotegia* from Rhodesia, all with encouraging results. (Pp. 1203–5.)

Efforts are being made to increase the output of **asbestos** from Russia, in view of increased demands, especially from Japan. (P. 1139.)

Most of the **mineral products** of India show an increased output, the most important being coal, gold, manganese ore, petroleum, salt, saltpeter, tungsten ore, lead ore, mica, silver, tin ore, iron ore, and monazite. (P. 1175.)

To meet increased demands for **aluminum,** especially in the aircraft industry, large extensions of the plants in Scotland are planned. (P. 1191.)

Further exploration of the **manganese** mines in Mysore, India, has shown that the richness of the ore increases with the depth. Manufacture of ferromanganese in India is being considered. (P. 1192.)

Experiments are being conducted in the West Indies upon the concentration of **lime juice** by freezing, to reduce the cost of transportation. (P. 1219.)

Soya bean oil is obtained in Manchuria principally by the "expression" process, only one plant using the benzene extraction process. The oil by the latter process is inferior. (P. 1227.)

A firm in Scotland, experienced in the manufacture of gauge glasses, has extended its plant and force to include **chemical glassware** of high quality, including apparatus for HNO_3 condensation, miners' safety lamp globes, and special glass for airplanes and marine mines. Prohibition of imports of chemical and scientific glassware for seven years is urged as a necessary step for the establishment of a permanent industry in Great Britain.

SPECIAL SUPPLEMENTS ISSUED IN DECEMBER

SPAIN—15*d*
IRELAND—19*e*
CHINA—52*f* and *g*
STRAITS SETTLEMENTS—56*a*
MOROCCO—75*a*

STATISTICS OF EXPORTS TO THE UNITED STATES

HONGKONG—925
Antimony
Chemicals
Hides
Leather
Peanut oil
Aniseed oil
Cassia oil
Paper
Sugar
Tin

DARIEN, CHINA—Sup. 52*f*
Albumen
Bean oil
Bean cake
Linseed

TSINGTAU—Sup. 52*g*
Hides
Peanuts
Peanut oil

SINGAPORE—Sup. 56*a*
Cassia
Cubebs
Cutch
Dragon's blood
Gambier
Gamboge
Gum
Gum benjamin
Gum copal
Gum damar
Gutta joolatong
Gutta percha
Gutta siak
Hides
Mangrove bark
Cocoanut oil
Paraffin
Rubber
Tin

JAMAICA—1127
Annatto
Hides
Kola nuts
Logwood extract
Sugar
Fustic
Logwood

LONDON—1137
Rubber
Tin
Hides
Indigo

CANARY ISLANDS—Sup. 15*d*
Cochineal
Copper matte
Pumice

BOOK REVIEWS

Chemical Patents and Allied Patent Problems. By EDWARD THOMAS. John Byrne & Co., Washington, D. C. Price, $2.50.

Patent law is a wilderness of decisions, a jungle of entangled and interlacing precedents which, like most jungle vegetation, can be found running almost any old way. However, still pursuing the analogy, most of the main lianas pursue something the same general direction with only occasional divagations. Our usual legal custom, from which we have not departed in patent matters, is to enact statutes in a few brief paragraphs; and then leave it to the courts to find out what those paragraphs mean as applied to any individual case. And in the patent field, our judges have been reasoning from parallel case to parallel case for something over a century now, with awe-inspiring results considered volumetrically.

Every chemist is interested in patents at one time or another, whether it be because he wants one or is afraid of one or for both reasons together. Chiefly, he wants to know the law governing compositions and processes; and for his purpose the ordinary text books on patent law are more irritating than instructive. Naturally, such books seek to cover the whole field and do not specialize in these matters; the more so since in the United States invention and litigation have been much more concerned with matters of mechanics than with matters of chemistry.

Mr. Thomas' book is a specialization; it is, practically, confined to the law of process and composition. It is hardly a treatise on patent law; it is more what Mr. Thomas terms a "finding list" of the decisions relating to chemical things. In a way it is an attempt to do for chemical patent law what Beilstein or Richter did for organic chemistry, *viz.*, classify the literature with just enough running text to give the classification. The difference, and the difficulty, arise in that while Beilstein's running text can give facts and avoid theory or controversial matter, Mr. Thomas, in the nature of things, can only give opinions. And opinions do not lend themselves to verbal condensation as do boiling points and reactions. Any law suit represents a difference in opinion which generally spreads itself over many pages of testimony and argument with the court's opinion coming as a lengthy equilibration of the difference. This equilibration cannot well be compressed into a paragraph or a sentence; in most instances, anyhow.

As a finding list, Mr. Thomas' book is extremely good. The running text, considered purely as a classification, is also good. It is so short and compressed that little difficulty is found in glancing over it to catch a key word.

But because it is short and compressed, the running text is open to more objection if it is to be considered a "statement of the patent law." It says too positively that which is oftentimes a matter of opinion. This is something Mr. Thomas could not altogether avoid in a summary. To state the upshot of any decision in a single sentence, a world of qualifications and limitations must be omitted unless that sentence is to be, as Mark Twain said of Germanic wording, "long enough to have perspective." Nevertheless, Mr. Thomas might, perhaps, with advantage, have hedged a little in some of his statements. The law is proverbially a thing of glorious uncertainty; and where its conclusions are stated too categorically, the casual reader is apt to receive fixed impressions which may afterwards have to be unfixed. Or, Mr. Thomas might have inserted a brief warning that in general the dicta given are merely his opinion of what the courts' opinions were; and that wherever the matter is of importance the original decision should be consulted.

Sometimes, Mr. Thomas uses language which is a little confusing. It is not meant in the discussion in page 31, for example, that the courts and Patent Office are coming to the view that a thing demonstrably old can be re-patented merely because it is made by a new process; but that there are some things, like a fried egg, which can best be defined by a recital of the process of making. See also the reference to "fusible tubes of water" (page 6). And the collocation of sentences in the running text is often just a little bewildering; like the dictionary, the text changes the subject a bit too abruptly sometimes. For instance, in pages 24 and 25, one sentence is talking about new matter in re-issues while the next is devoted to infringement of an adrenalin patent.

But of course it is easier to criticize than to do things; and the fact is that Mr. Thomas has produced a very useful book; and one which represents a vast amount of labor. It fills, and fills well, a vacant place on the reference book shelf.

K. P. MCELROY

Explosives. By ARTHUR MARSHALL. Second Edition, Vol. II, Properties and Tests. 8vo, 386 pp. and 80 illustrations. P. Blakiston's Son & Co., Philadelphia, 1917. Price, $8.00 net.

The author's work, as evidenced in this second volume, is of the same high character as set forth in the review of Vol. I.[1] The extent of the revision is to a degree shown by the fact that Part IX, devoted to "Properties of Explosives," has been enlarged by twenty pages; Part X, "Special Explosives," by thirty-eight pages; Part XI, "Stability, etc.," by eleven pages; and Part XII, "Materials and Their Analysis," by twenty-two pages. Besides the enlargement of each of the former chapters there have been added one, of seventeen pages, on "Naval and Military Explosives" and another of twelve pages, on "Commercial High Explosives," while to the "Thermo-Chemical Tables" in Appendix II have been added tables of heats of formation of "Fulminates and Azides" and of "Aliphatic Nitrates and Nitro-Compounds." Novelties are found in the change of the legend for Chapter 35 from "Safety Explosives" to "Coal-Mine Explosives" which latter is a much more accurate title, and in Part XII, by placing in full-face type at the bottom of each page, in addition to the marginal legends, the name of the substance described and for which the methods of analysis are given on that page. This section of the book will be much used and this addition will undoubtedly prove very convenient and helpful.

The same degree of sensible frankness in treatment of devices and materials now used for war purposes is noticeable in this as in the first volume, and particularly in the chapter on "Naval and Military Explosives" introduced into this edition. As this information is without question already in the hands of the enemy it would be foolish to withhold it from friends who might be able to render useful service if supplied with the information. In the first edition the author gave the composition of the chlorate priming used by the U. S. in its small-arms ammunition. In this edition he says of potassium chlorate: "If made by the electrolytic method it always contains at least 0.05 per cent of potassium bromate, and may contain as much as 0.6 per cent. That manufactured by the old process does not contain this impurity. As bromate may have a deleterious effect on the stability of explosives made with it, especially if the explosive also contains sulfur, an official order has been passed in Germany that chlorate for the manufacture of blasting explosives must not contain more than 0.15 per cent of bromate, for fireworks not more than 0.10 per cent, and for cap composition none at all," and he gives methods for the detection and determination of the bromate.

As a rule, the author has covered each topic with fullness and has cited the original authority with accuracy. A notable

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[1] THIS JOURNAL, 9 (1917), 822.

exception, which therefore attracts attention, is that of the perforation of plates by hollow cartridges of detonating explosives which is referred to Neumann under dates of 1911 and 1914, though these cartridges were devised in this country, the method applied, and the results published so long ago as 1888 and repeatedly later. Also it is noted that the Bureau of Mines' Sand Test for detonators is attributed to Storm and Cope, though these authors, in Technical Paper No. 125 cited by Marshall, state that it had been devised by W. O. Snelling. These are, however, but minor defects in a very valuable work.

Unfortunately, the printers' work is more open to criticism and this is the more to be regretted as the author was so far distant from his publisher. Two flagrant errors are the numbering of chapter thirty-six in full-faced Roman as Chapter XXX, and the omission of a legend from the newly added table for the heats of formation of the fulminates and azides. In addition dropped letters, imperfect impressions, and uncouth formulas, due to wrong case type or signs, are not uncommon.

Charles E. Munroe

Tube Milling. A treatise on the practical application of the tube mill to metallurgical problems. By Algernon Del Mar. x + 159 pp. Illustrated. McGraw-Hill Book Co., New York, 1917. Price, $2.00 net.

This book is of what may be called the standard dimensions of page, the binding is good and the paper, type and illustrations excellent. The latter are numerous and are in general clear and well selected for their purposes.

The object of the work is shown in the statement in the author's preface that "it is important that the principles involved in their [tube mills] operation should be made common property. It is for this purpose that the author has compiled the present volume, hoping that it will meet the requirements of the engineer and millman."

Chapter I is headed "General Description," and in addition treats fully, more or less, what are called the essential factors governing capacity, together with feeding devices, power requirements, costs and foundations. Chapter II is entitled "Amalgamating with the Tube Mill," Chapter III "Grinding Ores with the Tube Mill for Flotation," Chapter IV "Crushing Efficiencies," and Chapter V "The Use of Wrought Iron and Alloy Steel."

There is also an appendix of six pages giving some useful data, tables and notes on slime density relations, and some information on assay methods, etc., for a cyanide mill laboratory.

There are a few evident typographical errors, among which are the following:

Page 13. "0.5316 tons of dry feed" should be "0.5297 tons of dry feed."
Page 32. "Guanajuanto" for "Guanajuato."
Page 51. In the formula $m = \frac{Sv(a - d)}{(s - d)}$ "s" should be "S"
Page 120. "Fig. 50" should be "Fig. 64."
Page 147. "Excising" should be "existing."

Criticism might be made as to some of the details of arrangement. For example, on pages 104 and 105 the figures given as "Cement Data" might have been more properly placed in the appendix. Also in the chapter headed "Crushing Efficiencies" there is a page and a half only on this subject, while the remainder of the chapter, six pages, treats of screen gauges, screen openings, etc.

In the reviewer's opinion the notes on "Cyanide Chemistry" in the appendix should either have been made very much fuller or else omitted altogether and replaced by references to the several complete treatises on the subject. These notes are condensed to such a degree that it would be very difficult for anyone not a chemist familiar with cyanide laboratory practice to use them to advantage.

These are all minor criticisms, of course. The main objection to the author's treatment of the subject is his insufficiently full and adequate handling of important divisions, with a resulting lack of balance. As referred to above, barely two pages are given to the very important subject of crushing efficiencies, although references are given to the literature. Over thirteen pages are given to a discussion of the use of wrought iron and alloy steels, with tables and diagrams. This chapter is not to be objected to in itself, but, in comparison, the three pages of text devoted to grinding ores with the tube mill for flotation are certainly very meagre. A considerable volume of very valuable data on this subject has either been published or could be secured easily.

For a treatise the complete collection and arrangement of important published information and data is always a very valuable feature. The danger usually is that very often this results in what is practically a compilation only. The author is to be congratulated in that the proportion of pure compilation in the present volume is so decidedly less than the average that the book is not open to this objection, nevertheless this proportion could have been considerably increased to advantage in some of the chapters.

G. D. VanArsdale

Everyman's Chemistry. By Ellwood Hendrick. Harper & Bros., Philadelphia. Price, $2.00.

During the past few years the public has come to realize, in a vague way, the tremendous importance of chemistry in its application to daily life. No book, however, that the average reader could comprehend has been written on this subject, and it is gratifying indeed to welcome such a volume.

In his book Mr. Hendrick discusses the great problems of the day in an interesting, instructive, and lucid manner, setting forth the general principles underlying the subject of chemistry in a language which is simple, direct and intelligible to all. The delightful manner in which the subject is presented makes the reader feel almost a personal contact with the author. The keen wit and sense of humor displayed throughout the text relieves the monotony of dry scientific facts. Thus like a sugar-coated pill one swallows the information without noticing the bitter taste.

Allen Rogers

Lubricating Engineer's Handbook. A reference book of data, tables and general information for the use of lubricating engineers, oil salesmen, operating engineers, mill and power plant superintendents and machinery designers, etc. By John Rome Battle, B.S. in Mechanical Engineering. 8vo. Pp. 333. J. B. Lippincott Co., Philadelphia and London, 1916. Second Impression. Price, $4,00.

After a careful perusal of the book, the reviewer has failed to find any phase of the oil business that has not been discussed: history, manufacture, tests, the varied uses, storage, oil house methods, specifications, etc., etc.

Two statements made are very interesting, one that the oil supply of this country is about one-third used up (in about fifty years), and, second, that the annual friction loss in the United States is nearly 200 million dollars. If the book helps to bring about a diminution of this, it will have served a useful purpose. This statement is made on p. 69, "The so-called gumming test for lubricating oils is of little practical value * * * * * and the same may be said of evaporation tests." On the other hand (p. 138), the statement is found, that "a lubricating oil for forced feed in a steam turbine should have a low gumming test." Speaking from an experience of more than thirty years, the reviewer has found both tests of great value: the first is an admirable sorting test, particularly for automobile oils, and the second is of cardinal importance to the insurance companies, having to do with textile mills.

The book was very much needed, and is eminently satisfactory, in fact, about as nearly perfect as is humanly possible to produce—but this might have been expected from the engineer of the Atlantic Refining Company, "the largest manufacturers of lubricants in the world."

A. H. Gill

NEW PUBLICATIONS

By Irene DeMatty, Librarian, Mellon Institute of Industrial Research, Pittsburgh

Antiseptics: A Handbook on Antiseptics. H. D. Dakin and E. K. Dunham. 129 pp. Price, $1.25. The Macmillan Co., New York.

Biochemical Catalysts in Life and Industry. Jean Effront. 8vo. 752 pp. Price, $5.00. John Wiley & Sons, New York.

Chemistry: A Course of Instruction in the General Principles of Chemistry. A. A. Noyes and M. S. Sherrill. 8vo. Price, $2.25. T. Todd Co., Boston.

Chemistry: An Elementary Study of Chemistry. W. McPherson and W. E. Henderson. 12mo. 576 pp. Price, $1.60. John Wiley & Sons, New York.

Colloids: The Chemistry of Colloids. R. Zsigmondy and E. B. Spear. 8vo. 288 pp. Price, $3.00. John Wiley & Sons, New York

Flotation: Testing for the Flotation Process. A. W. Fahrenwald. 16mo. 173 pp. Price, $1.50. John Wiley & Sons, New York.

Foods and Their Adulteration. H. W. Wiley. 3rd Ed. 8vo. 646 pp. Price, $4.00. P. Blakiston's Son & Co., Philadelphia.

Hydro-Electric Power Stations. E. A. Lof and D. B. Rushmore. 8vo. 822 pp. Price, $6.00. John Wiley & Sons, New York.

Metallurgy: A Practice Book in Elementary Metallurgy. E. E. Thum. 8vo. 313 pp. Price, $2.75. John Wiley & Sons, New York.

Minerals: Practical Instruction in the Search for and the Determination of the Useful Minerals, Including the Rare Ones. Alex. McLeod. 16mo. 254 pp. Price, $1.75. John Wiley & Sons, New York.

Non-Metals: Elementary Experiments on the Non-Metals. J. S. Long and D. S. Chamberlin. 8vo. 78 pp. Price, $1.25. W. S. Rhode Co., Kutztown, Pa.

Ore Mining Methods. W. R. Crane. 8vo. 277 pp. Price, $3.50. John Wiley & Sons, New York.

Refrigeration: Mechanical Refrigeration. H. Williams. 12mo. 406 pp. Price, $3.00. The Macmillan Co., New York.

Science: Experimental General Science. W. N. Clute. 12mo. 303 pp. Price, $1.00. P. Blakiston's Son & Co., Philadelphia.

Science: A Short History of Science. W. T. Sedgwick and H. W. Tyler. 8vo. 474 pp. Price, $2.50. The Macmillan Co., New York.

Soil Conditions and Plant Growth. E. J. Russell. 3rd Ed. 8vo. 243 pp. Price, $2.00. Longmans, Green & Co., New York.

Steam Power Plant Engineering. G. F. Gebhardt. 8vo. 347 pp. Price, $4.00. John Wiley & Sons, New York.

Sugar: A Handbook for Cane-Sugar Manufacturers and Their Chemists. G. L. Spencer. 6th Ed. 16mo. 561 pp. Price, $3.50. John Wiley & Sons, New York.

Technical Periodicals. National Carbon Co. 12mo. 22 pp. Gratis. National Carbon Co., Cleveland.

Wood: The Preservation of Wood. A. J. Wallis-Taylor. 8vo. 363 pp. Price, 10s. 6d. William Rider & Sons, London.

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Acetylene: New Exploits of Acetylene. J. M. Morehead. *Journal of Acetylene Lighting*, Vol. 19 (1918), No. 1, pp. 240–243.

Actinochemistry: Studies in Actinochemistry. H. H. Custis. *Journal of the Franklin Institute*, Vol. 184 (1917), No. 6, pp. 849–884.

Alcohol: Waste Sulfite Liquor and Its Conversion into Alcohol. Erik Haegglund. *Pulp and Paper Magazine*, Vol. 15 (1917), No. 51, pp. 1185–1192.

Babbitt: Physical Properties of Babbitts. W. G. Harris. *Pulp and Paper Magazine*, Vol. 15 (1917), No. 51, pp. 1183–1184.

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Compressed Air: Using Compressed Air in Forge Shops. C. A. Hirschberg. *The American Drop Forger*, Vol. 3 (1917), No. 12, pp. 391–397.

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Displacement-Tanks. W. S. Weeks. *Mining and Scientific Press*, Vol. 115 (1917), No. 24, pp. 855–856.

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Evaporation: New Method of Increasing the Evaporation in Boilers. Carl Hering. *Power*, Vol. 47 (1918), No. 1, pp. 10–13.

Flotation: The Effect of Addition Agents in Flotation. M. H. Thornberry and H. T. Mann. *Metallurgical and Chemical Engineering*, Vol. 17 (1917), No. 12, pp. 709–713.

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Forestry: A Financial Analysis of Forestry and Reforestation. Ellwood Wilson. *Pulp and Paper Magazine*, Vol. 16 (1918), No. 1, pp. 5–7.

Fuel Briquettes: Utilization of City Garbage for Fuel Briquettes and Other Products. W. B. Phillips. *Manufacturers Record*, Vol. 73 (1918), No. 1, p. 85.

Furnaces: The Action of Flame in Furnaces. A. D. Williams. *The Iron Trade Review*, Vol. 61 (1917), No. 25, pp. 1319–1323.

Fusion: Some Notes on Fusion Apparatus. Frederick Pope. *Metallurgical and Chemical Engineering*, Vol. 17 (1917), No. 12, pp. 704–709.

Gas Circulation in Regenerator Checkers. A. D. Williams. *The Blast Furnace and Steel Plant*, Vol. 6 (1918), No. 1, pp. 29–33.

Gas Explosions at Blast Furnaces. F. H. Willcox. *The Iron Trade Review*, Vol. 61 (1917), No. 26, pp. 1377–1380.

Gels: The Formation of Crystals in Gels. H. N. Holmes. *Journal of Physical Chemistry*, Vol. 21 (1917), No. 9, pp. 709–733.

Heat Hazards—An Industrial Waste. J. A. Watkins. *The Iron Trade Review*, Vol. 61 (1917), No. 26, pp. 1387–1388.

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Hydrosulfite of Soda: Preparing Hydrosulfite of Soda. *Textile World Journal*, Vol. 53 (1917), No. 26, pp. 23–25.

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Soils: Movement of Soluble Salts through Soils. M. M. McCool and L. C. Wheeting. *Journal of Agricultural Research*, Vol. 11 (1917), No. 11, pp. 531–547.

Steel Production by the Duplex Process. J. K. Furst. *The Blast Furnace and Steel Plant*, Vol. 6 (1918), No. 1, pp. 25–28.

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Ultraviolet Light. VIII. Types of Apparatus Used for Sterilization and Other Purposes. C. Ellis and A. A. Wells. *The Chemical Engineer*, Vol. 26 (1918), No. 1, pp. 28–34.

Wood: Economical Wood Splitting. H. G. Saecker. *Paper*, Vol. 21 (1917), No. 14, pp. 16–17.

MARKET REPORT—JANUARY, 1918

WHOLESALE PRICES PREVAILING IN THE NEW YORK MARKET ON JAN. 18

INORGANIC CHEMICALS

Acetate of Lime	100 Lbs.	nominal		
Alum, ammonia, lump	100 Lbs.	4.00	@	4.50
Aluminum Sulfate, high-grade	Lb.	2¾	@	3
Ammonium Carbonate, domestic	Lb.	11	@	11¼
Ammonium Chloride, white	Lb.	15	@	17
Aqua Ammonia, 26°, drums	Lb.	20	@	21
Arsenic, white	Lb.	16½	@	17
Barium Chloride	Ton	75	@	85
Barium Nitrate	Lb.	9½	@	11
Barytes, prime white, foreign	Ton	40.00	@	45.00
Bleaching Powder, 35 per cent	100 Lbs.	2.50	@	3.00
Blue Vitriol	Lb.	9	@	9½
Borax, crystals, in bags	Lb.	7¼	@	8
Boric Acid, powdered crystals	Lb.	13¼	@	15
Brimstone, crude, domestic	Long Ton	nominal		
Bromine, technical, bulk	Lb.	75	@	85
Calcium Chloride, lump, 70 to 75% fused	Ton	30.00	@	32.00
Caustic Soda, 76 per cent	100 Lbs.	6.00	@	6.25
Chalk, light precipitated	Lb.	4¾	@	5
China Clay, imported	Ton	18.00	@	30.00
Feldspar	Ton	8.00	@	15.00
Fuller's Earth, foreign, powdered	Ton	nominal		
Fuller's Earth, domestic	Ton	20	@	30
Glauber's Salt, in bbls	100 Lbs.	1.00	@	1.50
Green Vitriol, bulk	100 Lbs.	1.15	@	1.25
Hydrochloric Acid, commercial, 20°	Lb.	3½	@	4
Iodine, resublimed	Lb.	4.25	@	4.30
Lead Acetate, white crystals	Lb.	17	@	19
Lead Nitrate	Lb.	nominal		
Litharge, American	Lb	9½	@	10
Lithium Carbonate	Lb.	1.50		
Magnesium Carbonate, U. S. P.	Lb.	18	@	20
Magnesite, "Calcined"	Ton	60.00	@	65.00
Nitric Acid, 40°	Lb.	9	@	10
Nitric Acid, 42°	Lb.	9	@	10
Phosphoric Acid, 48/50%	Lb.	7½	@	8
Phosphorus, yellow	Lb.	1.70	@	1.90
Plaster of Paris	Bbl.	1.50	@	1.70
Potassium Bichromate, casks	Lb.	43	@	44
Potassium Bromide, granular	Lb.	1.45	@	1.46
Potassium Carbonate, calcined, 80 @ 85%	Lb.	45	@	55
Potassium Chlorate, crystals, spot	Lb.	40	@	42½
Potassium Cyanide, bulk, 98–99 per cent	Lb.	nominal		
Potassium Hydroxide, 88 @ 92%	Lb.	83½	@	84
Potassium Iodine, bulk	Lb.	3.75	@	4.00
Potassium Nitrate	Lb.	28	@	30
Potassium Permanganate, bulk	Lb.	4.10	@	4.15
Quicksilver, flask	75 Lbs.	135.00	@	140.00
Red Lead, American, dry	Lb.	10	@	10½
Salt Cake, glass makers'	Ton	30.00	@	35.00
Silver Nitrate	Oz.	53	@	56
Soapstone, in bags	Ton	10.00	@	12.50
Soda Ash, 58%, in bags	100 Lbs.	3.00	@	3.10
Sodium Acetate	Lb.	17	@	18
Sodium Bicarbonate, domestic	100 Lbs.	2.65	@	2.75
Sodium Bichromate	Lb.	20	@	22
Sodium Chlorate	Lb.	20	@	22
Sodium Cyanide	Lb.	44	@	45
Sodium Fluoride, commercial	Lb.	18	@	19
Sodium Hyposulfite	100 Lbs.	2.50	@	3.00
Sodium Nitrate, 95 per cent, spot	100 Lbs.	4.50	@	4.60
Sodium Silicate, liquid, 40° Bé	100 Lbs.	1.75	@	2.00
Sodium Sulfide, 60%, fused, in bbls	Lb.	4⅞	@	5
Sodium Bisulfite, powdered	Lb.	6¾	@	7
Strontium Nitrate	Lb.	22	@	30
Sulfur, flowers, sublimed	100 Lbs.	nominal		
Sulfur, roll	100 Lbs.	nominal		
Sulfuric Acid, chamber, 66° Bé	Ton	nominal		
Sulfuric Acid, oleum (fuming)	Ton	75.00	@	80.00
Talc, American white	Ton	15.00	@	18.00
Terra Alba, American, No. 1	100 Lbs.	85	@	90
Tin Bichloride, 50°	Lb.	55	@	56
Tin Oxide	Lb.	80	@	85
White Lead, American, dry	Lb.	9	@	9½
Zinc Carbonate	Lb.	25	@	30
Zinc Chloride, commercial	Lb.	10½	@	11
Zinc Oxide, American process XX	Lb.	13	@	14

ORGANIC CHEMICALS

Acetanilid, C. P., in bbls	Lb.	75	@	80
Acetic Acid, 56 per cent, in bbls	Lb.	11	@	11½
Acetic Acid, glacial, 99½%, in carboys	Lb.	34	@	35
Acetone, drums	Lb.	nominal		
Alcohol, denatured, 180 proof	Gal.	72	@	75
Alcohol, sugar cane, 188 proof	Gal.	5.05	@	5.25
Alcohol, wood, 95 per cent, refined	Gal.	nominal		
Amyl Acetate	Gal.	5.10	@	5.25
Aniline Oil, drums extra.	Lb.	26	@	28
Benzoic Acid, ex-toluol	Lb.	5.25	@	5.50
Benzol, Pure	Gal.	35	@	40
Camphor, refined in bulk, bbls	Lb.	85	@	86
Carbolic Acid, U. S. P., crystals, drums	Lb.	52	@	54
Carbon Bisulfide	Lb.	7½	@	8
Carbon Tetrachloride, drums, 100 gals	Lb.	15½	@	16
Chloroform	Lb.	63	@	65
Citric Acid, domestic, crystals	Lb.	75	@	78
Creosote, beechwood	Lb.	1.90	@	2.00
Cresol, U. S. P.	Lb.	18	@	20
Dextrine, corn (carloads, bags)	Lb.	7	@	8
Dextrine, imported potato	Lb.	18	@	20
Ether, U. S. P. 1900	Lb.	27	@	30
Formaldehyde, 40 per cent	Lb.	19½	@	20
Glycerine, dynamite, drums included	Lb.	64	@	65
Oxalic Acid, in casks	Lb.	45	@	46
Pyrogallic Acid, resublimed, bulk	Lb.	3.15	@	3.25
Salicylic Acid, U. S. P.	Lb.	1.10	@	1.15
Starch, cassava	Lb.		—	
Starch, corn (carloads, bags) pearl	100 Lbs.	6.30	@	6.45
Starch, potato, Japanese	Lb.	10½	@	11
Starch, rice	Lb.	10	@	12
Starch : sago flour	Lb.	6½	@	7½
Starch, wheat	Lb.	5¾	@	6½
Tannic Acid, commercial	Lb.	50	@	60
Tartaric Acid, crystals	Lb.	78½	@	79

OILS, WAXES, ETC.

Beeswax, pure, white	Lb.	55	@	60
Black Mineral Oil, 29 gravity	Gal.	19	@	20
Castor Oil, No. 3	Lb.	29	@	29½
Ceresin, yellow	Lb.	nominal		
Corn Oil, crude	100 Lbs.	18.65	@	18.75
Cottonseed Oil, crude, f. o. b. mill	Lb.	17½	@	
Cottonseed Oil, p. s. y.	100 Lbs.	20.25		
Menhaden Oil, crude (southern)	Gal.	—	@	—
Neat's-foot Oil, 20°	Gal.	2.70	@	2.80
Paraffin, crude, 118 to 120 m. p.	Lb.	11	@	11½
Paraffin Oil, high viscosity	Gal.	36	@	37
Rosin, "F" Grade, 280 lbs.	Bbl.	7.05	@	7.10
Rosin Oil, first run	Gal.	41	@	42
Shellac, T. N.	Lb.	58	@	59
Spermaceti, cake	Lb.	25		
Sperm Oil, bleached, winter, 38°	Gal.	2.08	@	2.10
Spindle Oil, No 200	Gal.	35	@	36
Stearic Acid, double-pressed	Lb.	24	@	25
Tallow, acidless	Gal.	1.57	@	1.62
Tar Oil, distilled	Gal.	30	@	35
Turpentine, spirits of	Gal.	50	@	$50^{1}/$

METALS

Aluminum, No. 1, ingots	Lb.	36	@	38
Antimony, ordinary	Lb.	14	@	14¼
Bismuth, N. Y.	Lb.	2.85	@	2.90
Copper, electrolytic	Lb.	23½	@	—
Copper, lake	Lb.	23½	@	—
Lead, N. Y.	Lb.	6¾	@	7
Nickel, electrolytic	Lb.	55	@	56
Platinum, refined, soft	Oz.	105.00		
Silver	Oz.	85⅝	@	90
Tin, Straits	Lb.	nominal		
Tungsten (WO_3)	Per Unit	23.00	@	25.00
Zinc, N. Y.	Lb.	8	@	8¼

FERTILIZER MATERIALS

Ammonium Sulfate	100 Lbs.	7.25	@	7.30
Blood, dried, f. o. b. Chicago	Unit	6.50	@	6.55
Bone, 3 and 50, ground, raw	Ton	31.00	@	33.00
Calcium Cyanamid	Unit of Ammonia	nominal		
Calcium Nitrate, Norwegian	100 Lbs.		—	
Castor Meal	Unit		—	
Fish Scrap, domestic, dried, f. o. b. works	Unit	nominal		
Phosphate, acid, 16 per cent	Ton	16.00	@	16.50
Phosphate rock, f. o. b. mine:		5.50	@	6.50
Florida land pebble, 68 per cent	Ton	2.25	@	2.50
Tennessee, 78–80 per cent	Ton	5.50	@	6.00
Potassium "muriate," basis 80 per cent	Ton	350.00	@	355.00
Pyrites, furnace size, imported	Unit	nominal		
Tankage, high-grade, f. o. b. Chicago	Unit	6.50	&	10