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EDITORIALS

THE PERKIN MEDAL

Davy cautioned Faraday not to indulge in immoderate expectations of the rewards of Science. True, her gifts are not, as a rule, redundant, and they are likely, moreover, to be sometimes capriciously bestowed; but Science gives more precious rewards for excellence or success than those which can be expressed as ponderable quantities, and, among these, few are valued more highly than medals.

We look upon a medal as tangible proof of preeminence, for it is an indication of individual accomplishments—a token cast, in artistic mold, from a fusion of the golden opinions won by illustrious work. It furnishes the individual with evidence as to the regard in which achievements are held by his co-workers and fellow men.

On October 6, 1906, at a banquet given to Sir William Henry Perkin, at Delmonico's, New York, Dr. William H. Nichols handed to Sir William the first impression of the Perkin Medal, in gold. Dr. Nichols announced that this medal would be given annually to that chemist residing in the United States who had accomplished the most valuable work in applied chemistry during his career, whether this had proved successful at the time of execution or publication, or had subsequently become valuable in the development of industry. The Perkin Medal is therefore an acknowledgment of conspicuous services to technical chemistry—a reward for the successful application of scientific methods to industry. It is associated with the name of one of the most distinguished chemists, the founder of the immense coal-tar industry, and the recipient of the highest marks of distinction possible for Science to award; and it is conferred in order to show high appreciation of and gratitude for the success of the recipient of the medal in advancing chemical industry in one or several of its branches. In this way the applicability of chemistry to the arts and manufactures is advanced, for someone eminent in the field of chemical technology is indicated for the benefit of the aspiring technologist, and chemistry applied to the industries receives further recognition. The lessons in methods which the labors of the medalists afford must prove invaluable to all ambitious chemists.

While the award of the Perkin Medal is made by the New York Section of the Society of Chemical Industry, the largest and most prominent of the American sections of a great Society which embraces in its membership all—among the English-speaking races—that is representative of enterprise and progress in relation to the chemical arts, engineering, and industries, yet the Perkin Medal Committee, which selects the medalist, is constituted of representatives of different chemical societies of this country in addition to a representation from the Society of Chemical Industry; and, in making the selection, this Committee shows how highly it appreciates the work of the one so honored. It is not required, as in the case of some medals, that any particular society enjoy the credit and distinction of disseminating the results of the enquiries to which his fruitful ideas give rise: the Perkin Medal is a medal of merit conferred on chemists and technologists residing in the United States for services done in technical chemistry, as determined by the scope, value and importance, and with no restrictions as to any definite field of endeavor. In being presented, therefore, with the Perkin Medal, the recipient is sensible of the great honor done him, and this sense of the honor is increased by the recollection of the eminence of the preceding medalists. Perkin, Herreshoff, Behr, Acheson, Hall, Frasch, and Gayley! How proud one must feel in being reckoned of their company!

THE ANNUAL MEETING AT MILWAUKEE

The forty-seventh annual meeting of the American Chemical Society is to be held at Milwaukee, March 25th to 28th. The general headquarters will be at the Hotel Pfister, and the meetings will be held at Marquette University, located in the business center of the city. The local committee is headed by Mr. C. H. Hall, as Chairman, and Mr. P. J. Weber, as Secretary.

The entertainment committee is planning an interesting program, and special attention is being paid to preparations for the entertainment of ladies, at such times as they cannot participate in the regular program. Many manufacturing plants will be visited. Milwaukee affords excellent opportunities to see the tanning and packing industries; the manufacture of iron and steel, engines—gasoline and kerosene, byproduct coke and gas, glue, refrigerating machinery, automobiles, automobile parts and tires; shops and breweries. Most of these will be open to inspection by our members.

President Van Hise has extended a special invitation to the Society to visit the University of Wisconsin.

The plans as outlined by the local committee clearly indicate that this is to be an unusually large and successful meeting. The social, business, and scientific value of these meetings is more and more a matter of importance to the profession, and it is to the profession, therefore, that we should look for support and coöperation. Every chemist should attend at least one meeting each year. Why not begin at Milwaukee?

THE BUREAU OF MINES

The crying need of the Bureau of Mines for buildings and equipment can be easily appreciated on reading Director J. A. Holmes' Second Annual Report. The amount and value of the work already accomplished in mine safety and rescue demonstrations, fuel investigations, explosive tests and standardization, and other field work is amazing when one considers that the Bureau has never had a permanent home. It is almost inconceivable that this important department should be housed in rented buildings. The delays and expense, already $150,000, incident to temporary locations, seriously handicap the work.

Thirty thousand men have been killed and nearly twenty thousand seriously injured in the coal mines within the last ten years. The Bureau's work within the past year has yielded a reduction of five hundred in this annual sacrifice of life, but our losses in all mines and quarries are so greatly in excess of those in other countries as to be a very discreditable commentary on our mining methods. In the face of the wonderful results so far accomplished, the mine rescue work of the Bureau has had to be curtailed and some important phases entirely omitted, due to lack of equipment. This one branch of the Bureau's work is of sufficient importance to merit the unlimited support of Congress.

The fact that the fuel testing done by the Bureau has saved the Government not less than $100,000 per year during the last three years, while laboring under the most unfavorable conditions imaginable, is an indication of what can be expected if the proper working conditions are provided.

The Bureau asks for the modest appropriation of $500,000 for buildings and proposes to use the "cheapest sort of construction that will be decent to have in a city." It wants nothing ornamental. It asks only for efficient working housing for the prosecution of its investigations, and a review of the valuable work already accomplished should serve to convince Congress of the urgent needs of the Bureau of Mines.

ORIGINAL PAPERS

NOTES ON MINE GAS PROBLEMS[1]

By George A. Burrell

The Bureau of Mines has accumulated considerable data regarding mine gases, as a result of the analysis of gas samples collected in different mines and as a result of experimental work performed in the laboratory. Some of the observations are presented here, regarding the explosibility and physiological effects of mine atmospheres, also regarding flame extinction and after-damp.

EXPLOSIBILITY OF METHANE

The limits of explosibility of methane have been worked out by different investigators and results, which in the main are in accord with each other, have been determined in the laboratory. Some differences have been published but these are mainly due to the fact that different experimenters have performed the work under different conditions. Sufficient emphasis has not always been placed upon the decided effect experimental conditions exert on the results. The size and shape of the vessel employed, nature of source of ignition, *i. e.*, whether flame, small electric spark or large electric flash; ignition of mixture from above or below; moisture in the mixture; and temperature and pressure, all may have some influence in determining the limits of explosibility of mixtures of combustible gases with air. A partial burning in a mixture of a combustible gas with air always takes place when the ignition temperature is reached, whether an explosive proportion of combustible gas be present or not. The extent of this burning will depend upon some of the conditions just referred to. But explosive limits in the sense the terms are ordinarily used have reference to those percentages of inflammable gas between which self-propagation of flame to all parts of a mixture occurs without help from the source of ignition other than the ignition of the mixture at one point. The smallest quantity of any combustible gas which when mixed with air (or oxygen) will enable self-propagation of flame to take place is termed the lower limit of explosibility of the gas. It is of vital importance to mining men of course, that the correct limit of explosibility of methane with air be known.

LOW EXPLOSIVE LIMIT OF METHANE AIR MIXTURES

More recent work has shown that this value is about 5.50 per cent. methane and not 6 per cent. as is sometimes stated. Even 0.50 per cent. is of significance. Modern ventilation aims to keep the methane in mine air as far removed from the explosive proportion as possible. One per cent. of methane in return air is considered a large quantity. One half per cent. is sometimes tolerated. If the explosive limit for methane is 5.5 per cent. and a mining management calls 6 per cent. the low limit of explosibility, they are 0.5 per cent. nearer the explosive proportion than they think.

Coquillon ignited methane-air mixtures in a closed vessel by means of an electric spark, and placed the lower limit of explosibility at 5.8 per cent. methane. Le Chatelier and Mallard and Boudouard have placed the limit at 6.0 per cent. Eitner determined the lower limit of explosibility of methane to be 6.1 per cent. Clowes obtained 5.0 per cent. as the low limit of explosibility when the gas was fired from below with a flame and 6.0 per cent. as the limit when the gas was fired from above. Teclu found the lower limit of explosibility to be between 3.20 and 3.67 per cent. Teclu's values are quite outside of the others mentioned here. The most recent values are those obtained by Burgess and Wheeler who place the lower limit of explosibility between 5.5 and 5.7 per cent. No discussion of the different methods of experimentation will be given here, the object being only to convey the idea that exact duplication of results by different experimenters has not always followed.

In the Bureau's laboratory an explosion could not be obtained when a mixture of methane and air containing less than 5.5 per cent. methane was subjected to the action of a small $^1/_8$ inch spark from an induction coil. The latter was driven by four dry cells. The mixture was placed in a 100 cc. spherical vessel over

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[1] Presented by permission of the Director of the Bureau of Mines at the winter meeting of the West Virginia Coal Mining Institute, Parkersburg, W. Va., December, 1912.

mercury. An explosion could not be obtained with 0.1 per cent. less methane when the pressure was increased from one atmosphere to two atmospheres. With 5.50 per cent. of methane, combustion was not quite complete. Analysis of the products of combustion showed, however, that only a trace of combustible gas remained unburnt. Pure methane was prepared by the action of alcohol and methyl iodide on a zinc-copper couple.

A number of experiments have been performed in the Bureau's laboratory in which a flame has been used as the source of ignition. In some of those experiments a spherical flask having a capacity of one liter has been used. Some burning followed under these conditions when as little as 4.75 per cent. methane was present. Ignition was effected from below. The burning extended as a cone about one-half of the way to the top of the flask. Inflammation in the mixture increased with increasing percentages of methane. All mining men have of course noticed this behavior in mixtures of combustible gases and air. With very small percentages the inflammation starts as a cap on the flame of the safety lamp. The inflammation spreads from the source of ignition as the percentage of combustible gas is increased. Finally conditions are right for a projection of flame throughout the mixture. At about 5.50 per cent. the low limit of explosibility, the flame extends to all parts of the mixture but travels comparatively slowly and can be followed by the eye. With increasing proportions of methane, the explosion becomes more violent. It was observed that the partial burning in a mixture containing less than 5.50 per cent. methane depended upon the several factors such as nature of ignition, shape of container, etc., but that complete explosion could not be obtained under several different conditions of experiment tried when less than 5.5 per cent. of methane was present. In determining the low explosive limit of methane-air mixtures, the products of combustion were examined to ascertain the completeness of the combustion. In the experiments that follow, this was not done unless otherwise stated. The term explosion will be retained to mean conditions when the flame filled the container as far as could be seen by the eye. Inflammation will mean only a partial filling of the container by the flash. The foregoing remarks are preliminary to a brief account of some experiments regarding explosibility of fire-damp, which are not so well known.

EFFECT OF CARBON DIOXIDE ON THE EXPLOSIBILITY OF METHANE

In explosions, as well as flame extinction and physiological effects, the influence of carbon dioxide in mine air has usually been overestimated. Carbon dioxide always occurs in mine gas mixtures in which explosive amounts of methane are present and there is always a greater oxygen deficiency than is produced when the methane is added to air for simple laboratory experiments. The following experiments show something about the explosibility of methane in the presence of excessive proportions of carbon dioxide and when the oxygen is considerably reduced.

In the Bureau's laboratory, when 2.5 per cent. of carbon dioxide was present in a mixture, an explosion followed when the methane was raised to 5.83 per cent.

With 5.0 per cent. carbon dioxide an explosion occurred when the methane was raised to 6.25 per cent. With 10.0 per cent. of carbon dioxide an explosion was obtained when the methane constituted 6.6 per cent. of the total. A small spark from an induction coil was the source of ignition. The presence of carbon dioxide narrows the explosive limits but it will be observed that even 10 per cent. only raised the low limit to 6.60 per cent.

EFFECT OF REDUCED OXYGEN ON THE EXPLOSIBILITY OF FIRE-DAMP

A spherical flask having a capacity of 1 liter was used in the following experiments: A mixture of the following composition was experimented with:

	Per cent.
Carbon dioxide	0.03
Oxygen	14.00
Methane	9.40
Nitrogen	76.57

When this mixture was exposed to a flame from above inflammation occurred. The flame spread downward to the middle of the flask, and out toward the sides where it died. The eye could easily follow the course of the wave. When the mixture was ignited with a flame from below, it exploded with considerable force. As far as the eye could see the flame filled the entire flask.

Further experiments were made in which the oxygen had been reduced to 13 per cent. This mixture had the following composition:

	Per cent.
Carbon dioxide	0.03
Oxygen	13.00
Methane	9.40
Nitrogen	77.57

The mixture was placed in a cylindrical vessel having a capacity of 2750 cc. Two copper terminals were quickly broken to produce the flash, which took place in the center of the vessel. A current of 7.5 amperes at a pressure of 220 volts was used. A flash about one-half inch long could be obtained. On the break of the contact the inflammation spread upward almost to the top of the jar.

With 15.1 per cent. oxygen and 9.4 per cent. methane, a quite violent explosion was obtained under the same conditions of experimentation. Experiments were also performed in which a small one-eighth inch spark from an induction coil was used as the source of ignition. A mixture having the following composition exploded:

	Per cent.
Carbon dioxide	3.92
Oxygen	16.25
Methane	9.43
Nitrogen	70.37

Under the same conditions a mixture having the following composition in which the oxygen was slightly diminished, did not inflame at all:

	Per cent.
Carbon dioxide	3.99
Oxygen	15.90
Methane	9.25
Nitrogen	70.86

It was observed in conducting these explosibility experiments that both the electric flash and flame produced inflammation under more severe conditions than the small spark; also, a flame when applied from below will produce inflammation, under more severe conditions, than when applied from above.

These are only a few of many experiments which have been made on the explosibility of gaseous mixtures at the Bureau's laboratory. Some experiments have been made in which black powder has been fired into mixtures of gas and air, in a gallery having a capacity of 600 cubic feet.

Much interest and criticism has followed Dr. John Harger's (England) proposal that the oxygen be decreased and carbon dioxide be increased in mines to prevent explosions. Briefly, Dr. Harger's proposition is this: A small reduction in the oxygen percentage and a small increase in the carbon dioxide percentage in mine air will suffice to produce an atmosphere incapable of supporting combustion and consequently an atmosphere in which explosions and gob fires cannot occur. For average mines it is suggested to start the experimental work on a large scale; that the oxygen should be reduced to 20 per cent. (*i. e.*, 1 per cent. below normal) and a half per cent. CO_2 added to it. Where the conditions were more dangerous the oxygen should be further reduced, say, to 19 per cent., and the CO_2 increased to three-quarters of 1 per cent.

Time will not be devoted now to a discussion of the many points involved in Dr. Harger's suggestion, unless it is the wish of the Institute. Experiments which have been performed by the Bureau, some of which are here presented, indicate that the oxygen percentage will have to be reduced much below the figures mentioned by Dr. Harger to prevent all explosions. Of course, as the oxygen percentage is decreased the mixture explodes less violently and less completely. As regards carbon dioxide, so much would have to be added to prevent fire-damp explosions that it appears that its use for that purpose would be entirely precluded.

MINE-GAS MIXTURES CONTAINING EXPLOSIVE AND OTHER PROPORTIONS OF METHANE

Below are given some analyses of mine-gas samples, some of which contain explosive proportions of methane. The accompanying carbon dioxide and oxygen content are of interest in connection with foregoing experiments. The samples were collected in mines wherein ventilation had been interrupted for some time. Explosions had occurred in these mines.

Sample number	CO_2	O_2	CH_4	N_2
1	8.28	10.50	4.49	76.73
2	0.48	17.49	9.20	72.83
3	5.81	13.95	5.07	75.17
4	1.75	17.63	7.60	73.02
5	0.34	19.46	5.79	74.41
6	0.35	18.81	7.25	73.59
7	2.00	15.64	7.37	74.99

Samples Nos. 2, 4, 5, 6 and 7 are explosive. There could be but very slight inflammation in mixtures represented by samples Nos. 1 and 3.

Knowledge of the explosibility of mixtures as they occur in mines has been especially useful to the Bureau in that such atmospheres are frequently encountered in exploration work following mine explosions and fires.

EXTINCTION OF ACETYLENE FLAME

The Bureau has already called attention to the tenacity to existence of the acetylene flame, in that it will exist in atmospheres in which the ordinary wick-fed flame is extinguished. It was found that a residual atmosphere in which an acetylene flame had been extinguished contained 11.70 per cent. oxygen and 6.30 per cent. carbon dioxide. More recent experiments have confirmed this finding. It was also found that when about one-half of the carbon dioxide was removed as it was formed the acetylene flame burned in a slightly greater oxygen deficiency. For these experiments the flame was placed in a gas-tight cabinet, having a capacity of 25 cubic feet. The residual atmosphere after the flame had been extinguished had the following decomposition:

	Sample from middle of chamber	Sample from bottom of chamber
Carbon dioxide	3.22	3.17
Oxygen	10.99	11.12

The Bureau has observed that in mines when the oxygen has decreased to 11 per cent., about 6 per cent. of carbon dioxide on the average will be present. An objection has been raised to the use of the acetylene flame in that it becomes extinguished only when there may be grave danger to men, because of the presence of much black damp. The acetylene flame, as is the case with other flames, burns less brightly as the proportions of oxygen in mine air diminishes. It was observed at the Bureau's laboratory that when the oxygen content of air decreased to about 16 or 16.5 per cent. the flame resembled the ordinary wick-fed flame when the latter burns in pure air, *i. e.*, air containing 21 per cent. oxygen. This indication can be used as a guide against men venturing into black-damp workings containing less oxygen than this proportion.

EFFECT OF CARBON DIOXIDE ON THE EXTINGUISHMENT OF THE FLAMES OF MINERS' LAMPS

The ordinary miners' lamp is extinguished when the oxygen falls to about 16.5 or 17 per cent. This extinguishment is almost entirely due to the oxygen deficiency and not to the carbon dioxide, since this constituent is rarely ever present in sufficient quantity when the oxygen in mine air drops to 16 or 17 per cent., to exert any appreciable effect. When a lighted candle was placed in a bell jar filled with ordinary air it went out when the oxygen percentage fell to 16.24 per cent. The carbon dioxide produced by the burning amounted to 2.95 per cent. When 3.22 per cent. of carbon dioxide was originally in the air the atmosphere, after the extinguishment of the flame, contained 16.68 per cent. When the atmosphere originally contained 13.52 per cent. carbon dioxide and 20.67 per cent. oxygen the flame went out when the oxygen had fallen to 17.39 per cent. These analyses are tabulated below:

	Composition of atmosphere at beginning of experiment		Composition of atmosphere in which flame went out	
	CO_2	O_2	CO_2	O_2
1..............	0.04	21.00	2.95	16.34
2..............	3.22	21.13	6.51	16.68
3..............	13.52	20.67	16.00	17.39

It will be observed that the initial presence of a large amount of carbon dioxide had little effect on the flame extinguishment. In all of these laboratory experiments the flame probably existed a trifle longer than would be the case in actual mining practice, because in mines a slight gust of air or sharp thrust of a lamp would puff out a diminishing flame.

EFFECT OF VITIATED AIR ON THE LUMINOSITY OF MINERS' LAMPS

J. S. Haldane[1] recently made an important contribution to mining literature when he determined the effect on the light of a safety lamp when burned in atmospheres deficient in oxygen. Mine air is always deficient in oxygen to some extent as compared with outside air. This deficiency may amount to from 0.1 to 0.2 per cent., to 2 or more per cent. under ordinary working conditions.

A table which J. S. Haldane prepared is of exceptional interest and is here presented:

Observed Percentage Diminution of Light with Observed Percentage Diminution of Oxygen

Light given	Oxygen percentage	Light percentage diminished	Oxygen percentage diminished
100	20.93	0	0.00
90	20.66	10	0.27
77	20.34	23	0.59
66	19.88	34	1.05
41	19.34	59	1.89
27	18.92	73	2.01
11	18.28	89	2.65
0	18.01	100	2.92

Dr. Haldane observed, roughly speaking, that every diminution of 0.1 per cent. in the oxygen caused a diminution of 3.5 per cent. of the value of the light in the pure air.

HIGH VELOCITY OF AIR CURRENTS IN MINES

As against many good reasons put forth for keeping miners plentifully supplied with fresh air, there has arisen the protest that with high velocities of air currents in mines and high oxygen content of mine air, explosions have increased in number. Low oxygen content as a preventive of explosions has been discussed in part in this paper; the question of high velocities is more completely wrapped up with the question of dust explosions, and will not be discussed here.

AFTER-DAMP

Two samples of after-damp atmospheres are here presented. They are of interest as showing the large amount of carbon monoxide (white-damp) present shortly after an explosion.

Sample No. 1 was collected 30 minutes after an explosion had occurred at the Experimental mine of the Bureau at Bruceton, Pa. It was taken in the main entry at the junction with the first right butt entry after ventilation had been restored in the main entry.

Sample No. 2 was collected at the face of the first right butt entry 100 feet from where Sample No. 1 had been collected. Ventilation had not been restored in this entry.

	Sample No. 1	Sample No. 2
Carbon dioxide.....	0.26	1.54
Oxygen...........	20.26	17.79
Carbon monoxide...	0.16	1.89
Methane..........	0.12	0.65
Nitrogen..........	79.20	78.13

Hydrogen present less than 0.20 per cent.

These analyses are instructive as showing that very dangerous atmospheres may exist after an explosion in a mine in close proximity to atmospheres wherein men would not soon feel distress. The Bureau found that in 0.16 per cent. carbon monoxide a mouse shows only slight signs of weakness at the end of one hour's time. In the same atmosphere a bird showed signs of distress in 3 minutes time and fell from its perch in 18 minutes.

In exploring a mine containing after-damp a person could, by disregarding the warning of a sensitive animal like a canary, travel in a very short time from an atmosphere that would not distress him rapidly into one where collapse would quickly follow.

AUTOMATIC GAS SAMPLER

The Bureau has lately developed an automatic mine gas sampler to a stage of completion where it is believed samples of after-gases can be trapped at the time an explosive wave goes by and at prearranged intervals thereafter in order that the chemistry of the explosions in the Experimental mine may be better studied.

An analysis of a sample collected at the Altoft Experimental station (England) $^1/_{20}$ of a second after the explosion had passed contained the following constituents:

	Percentages
Carbon dioxide......................	11.25
Oxygen...............................	1.15
Carbon monoxide....................	8.15
Hydrogen.............................	2.75
Methane..............................	2.95
Nitrogen..............................	73.75

The high percentage of carbon monoxide will be noticed. The British report calls attention to the presence of oxygen as showing the inrush of air even so short a time after the explosion wave had passed. They state that, at the instant of passage of the flame, the oxygen should have been entirely consumed. The gallery at Altoft's is above ground, however, and inrush of pure air would be quicker than in a mine.

DISTRIBUTION OF AFTER-DAMP

It is well known to mining men that after an explosion, parts of a mine may be quite unaffected by the explosion, and that in some places the products of the explosion (white-damp, etc.) may not penetrate. Men have lived for days in some mines after explosions until rescued. On the other hand, men rushing into the track of the explosion in the dash for safety have

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[1] *Colliery Guardian*, October 25, 1912.

frequently been overcome. As is generally recognized, the heaviest toll of life is almost invariably due to the white-damp and not to explosive violence.

USE OF BIRDS FOR CONTINUED EXPLORATION WORK

The Bureau has made experiments having to do with the use of canaries for continued exploration work, having in mind the fact that the same animal might be used and overcome several times in the same day. It was found that even after repeated exposures to carbon monoxide these animals, upon subsequent exposures, show distress, collapse and revive in about the same period of time as on the first exposure. Consequently these animals can be used again and again for all practical purposes with the knowledge that they will not be less efficient for exploration work even after repeated exposures. No parallel can be drawn regarding the effects on men of carbon monoxide from these experiments. Men have been months recovering from severe cases of poisoning, and the after-effects have been very formidable. To show how differently carbon monoxide affects men and animals the following experiments performed by the Bureau are here tabulated:

Quantity of CO Used = 0.25 Per Cent.

Effects on Canaries

Time exposed	Distress	Collapse	Recovery
3 minutes	2 minutes	3 minutes	7 minutes

Effects on Mice

Time exposed	Distress	Collapse	Recovery
12 minutes	6 minutes	12 minutes	25 minutes

Effect on Man

Time exposed	Distress	Collapse	Recovery
20 minutes	1 hour	....	8 hours

Whereas the animals were left in the atmospheres until they collapsed, the man left the atmosphere experiencing but little discomfort at the end of 20 minutes. One hour later he became very sick. Much might be written about the poisonous action of carbon monoxide. A further discussion will be reserved for a Bureau publication.

USE OF SPARROWS FOR RECOVERY WORK

The Bureau has attempted to use English sparrows for recovery work in mines with not much success, because those so far obtained have not survived captivity. Pigeons are not sensitive enough. At the end of 11 minutes in an atmosphere containing 0.25 per cent. of carbon monoxide, they showed but slight signs of distress. Mice are more sluggish and not so easily affected as canaries, but at the same time, useful. A mouse and a canary together would make a good combination. The usefulness of small animals for detecting bad air in mines has been so well recognized in England that they are kept at all mine safety stations and some collieries. The Bureau of Mines of this country keeps them at all of its stations.

INTRUSION OF NATURAL GAS INTO COAL MINES

Natural gas has intruded into some coal mines with disastrous consequences. Mining men are alert to the importance of the questions. Old abandoned wells are an especial menace in that nobody knows the whereabouts of some of them.

Some comparison between the explosibility of natural gas and fire-damp is interesting. Natural gases of the Appalachian fields contain other paraffin hydrocarbons than methane, principally ethane. The following analysis shows the composition of the natural gas used at Pittsburgh. This gas is drawn largely from West Virginia. Some comes from Western Pennsylvania.

Carbon dioxide.......	trace (less than 0.10 per cent.)
Oxygen..............	0.00
Methane.............	82.5
Ethane..............	16.0
Nitrogen............	1.5

Carbon monoxide, hydrogen or ethylene are not contained in the gas, not even in traces.

Below are shown the explosive limits of methane-air mixtures, and natural gas-air mixtures. Ignition was effected by a small electric spark from an induction coil.

	Methane	Natural gas
Low limit..........	5.50	4.92
High limit..........	12.50	12.00

Below are given the ignition temperatures of methane and ethane, determined by Dixon and Coward:[1]

Methane................	650°–750° C.
Ethane.................	520°–630° C.

It does not appear from the above that natural gas is so much more sensitive to inflammation as to produce an added menace from this cause upon intrusion into a mine. The great danger lies in the sudden inrush of a large body of inflammable gas which has not always been controlled in order to every disaster. The fact that carbon monoxide is not present in the gas is fortunate. Many published analyses show natural gas to contain this constituent. The statement is erroneous. Higher paraffin hydrocarbons than methane in natural gas give to the latter its characteristic odour.

INFLAMMABLE CHARACTER OF THE GASES PRESENT IN MINE AIR

The Bureau has almost ready for publication a report showing the exact character of the inflammable gases present in many samples of mine air. In many text books, the statement is made that hydrogen, ethylene, carbon monoxide, and ethane may be present in mine air under normal conditions of operation; as far as the authors of these books are concerned, they have been justified because occasionally analyses showing these constituents are reported, although, even so, the bulk of evidence shows them to be of rare occurrence under the conditions cited. Because of the reported presence of these gases in mine air the question has, at times, been raised regarding the application to mining conditions of experiments performed in the laboratory or testing galleries, in which methane or the nearly similar natural gas is used. In the Bureau's report, mine gas samples from about 50 mines are listed. Methods of examination are described. The great effect of very small errors which can hardly be avoided in the manipulation of many forms of gas analysis apparatus is shown in that these

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[1] *Chem. News*, **99**, 139 (1909).

small errors may result in the reporting of one or more per cent. of combustible gases other than methane. Of course, if a careful analyst reports other combustible gases than methane in even a few samples, then one is justified in believing that they may occur in other samples. The above statement excepts a few mines around the oil regions into which natural gas may have escaped.

A rather complete bulletin is also ready having to do with methods of examination of mine and natural gases. Apparatus is shown for research work, also simple machines that can be easily operated at mines. Chemical analyses are the basis from which safety lamps and other gas indicators are constructed and simple methods for these standard tests are desired. An abstract from the larger bulletin has also been completed which contains only those chemical apparatus suitable for use at mines.

USE OF BIRDS AND MICE FOR EXPLORATION PURPOSES

A publication has also been prepared containing further work regarding the use of canaries and mice. A short abstract of these findings is given in the preceding pages.

COÖPERATIVE WORK WITH ILLINOIS

In coöperation with the State of Illinois a large number of samples have been collected in that state by N. H. Darton, Geologist of the Bureau. This has also been done in the anthracite fields of Pennsylvania. The results of analyses of these samples are being shaped into a report, and will show the composition of the air in many different mines in those states. Mr. Darton also reports interesting findings having to do with geological conditions and methane liberation.

NORMAL MINE AIR SAMPLES

In conjunction with G. S. Rice, Chief Mining Engineer of the Bureau, a preliminary report is being assembled showing the composition of the mine air in many different coal mines, under normal conditions in different parts of the country. Almost all of the data has been assembled for this work.

MINE GASES AND EXPLOSIONS

Mine gas samples have also been collected by mining engineers of the Bureau following many mine explosions. Their significance will be discussed in the reports of those explosions.

USE OF GASOLINE LOCOMOTIVES

A large number of samples were collected having to do with use of gasoline motors in mines.

Exhaust samples have been collected, also samples of the air in the mines where motors were operated. Special investigations have been made for coal operators, inspectors, and motor manufacturers at their request. It seemed best to defer a report for publication, however, until results could be obtained connecting exhaust gases with power developed, gasoline consumption different carbureter arrangement, etc. This work is in progress and is under the direction of O. P. Hood, Chief Mechanical Engineer of the Bureau.

BLACK-DAMP IN MINE AIR

Enough date has also been collected for the issuance of a publication on black-damp in American mines. Also one on after-damp.

MINERS' CIRCULAR

A miners' circular is being prepared, having to do with a discussion of mine gases. No attempt, of course, is being made to replace those mine gas terms, with which miners are most familiar, such as black-damp, after-damp, after-gases, fire-damp, etc., but a circular showing the Bureau's observations on these subjects will be published, since in the performance of investigative work these names must, at times, be replaced by the chemical ones. Black-damp is still sometimes wrongfully used as meaning carbon dioxide instead of a mixture of nitrogen and carbon dioxide. The latter usually plays but a small part in the combustion effects produced by the mixture.

The deficiency of oxygen, which always accompanies excessive proportions of black-damp, is the main factor in flame extinguishment in mines. On the other hand, physiological effects, if produced in men when they are in atmospheres in which lamps do not burn, are frequently due to the carbon dioxide. This statement has reference to atmospheres in old workings, etc., and not those that have been vitiated by mine fires, explosions, etc., and wherein carbon monoxide might exist. Different meanings have been attributed to the word "fire-damp." To some it means methane, to others any mixture of methane and air. That other gases than methane may frequently be present is sometimes stated. Some persons define it as any inflammable mixture of methane and air.

Sufficient data are not at hand to completely describe the constituents present in after-damp. That the products of incomplete combustion of methane, coal dust and air can be quite complex, is shown by work already performed on single gases and air. This statement has reference to the immediate after-products of an explosion. The Bureau hopes to secure data on this point at the experimental mine.

After a stagnant mine atmosphere has been clarified of smoke particles and easily soluble gases, and gases that irritate, there can remain, for a long time, a clear atmosphere containing oxygen, carbon dioxide, nitrogen, methane, hydrogen and carbon monoxide; an atmosphere with only the characteristic odor of burnt coal and wood, yet one that can be fatal because of the presence of the carbon monoxide. Oxygen may be present in sufficient quantity to enable a lamp flame to burn fairly well, and absolutely no indication given to an exploring party of the presence of carbon monoxide, although the latter may be present in sufficient amount to produce fatal results. Deaths have been caused by men relying upon the lamp flame to warn them of carbon monoxide.

Bureau of Mines
Pittsburgh

THE DESTRUCTIVE DISTILLATION OF SEWAGE SLUDGE

By Augustus H. Fiske and Richard B. Earle

Received November 29, 1912

HISTORICAL

The problem of utilizing sewage sludge divides itself naturally into two divisions: First, the elimination of moisture, and second, the disposal of the solid material most advantageously. Experiments leading to the solution of these problems are constantly being tried, and in different places varying methods are in use. In England, filter presses and rotary driers, and in Germany, besides these, centrifugal driers have also been employed to bring the moisture in the sludge down to 60–80 per cent. Ruggles[1] estimates the cost of drying sludge to a moisture content of 8–10 per cent. at $1.66 per ton. This material is then sold as fertilizer. It is estimated[2] from analyses of the fertilizing constituents, that the substance should be worth $7.50 to $10.00 per ton, but it seems probable that the reason it has not come into wider use for this purpose is the fact that it contains grease.

Schweitzer[3] states that at the city of Elbing, the sludge contains 60–70 per cent. of water when it leaves the filter presses. It is then allowed to remain two to four weeks in the open, and then, either used for fertilizer (containing 2–4 per cent. nitrogen), or burnt in a suction gas producer. At Hannover[4] where centrifugal driers are employed, the sludge contains 50–70 per cent. of water, a slight improvement over the filter presses. In experiments at Brunn,[5] Austria, a rotary drier, heated artificially, was used, and the content of moisture in the sludge reduced to 25 per cent. before being utilized for the preparation of illuminating gas. For this purpose, 100 kg. of the sludge were mixed with 2 kg. of coal, and on distilling the mixture 23 cu. m. of gas were obtained per 100 kg. sludge.

Bayer and Fabre[6] gave the mean of 75 analyses of sludge as follows:

	Per cent.
C	31.04
H	3.95
N	2.87
O	20.73
Ash	41.41
P_2O_5	1.03
K	0.42

In their experiments 55 kg. of sludge were distilled at a time, the distillation lasting $2^1/_2$ hours, and the yield of gas was on a basis of 23.8 cu. m. gas per 100 kg. sludge. At Frankfort,[7] dried sludge used in a gas producer yielded 20 cu. m. gas per 100 kg. of dry sludge.

In England, Purcell[8] distilled dry sludge and obtained ammonia and gas. He also states that the residue from the distillation is valuable to the cement manufacturer. At Chorley,[9] illuminating gas is made in small amounts from the sludge. At Manchester, and Kingston-on-Thames, the sludge is dried and sold as fertilizer. At Norwich, grease is recovered from it. At Leeds a small plant for destructive distillation of the sludge has been used for the purpose of recovering ammonia. At Oldham, grease is recovered by treating the sludge in a retort with steam.

DESCRIPTIVE

The following experiments were tried with sewage sludge from the precipitation plant at Worcester, Massachusetts, where the treatment of the sewage is essentially as follows: Milk of lime is first added to crude sewage and the mixture run into large tanks to settle. The crude sewage contains large quantities of soluble iron salts, due to the iron works in the city. The lime precipitates the iron compounds which carry down with them most of the solid material in the sewage. After settling, the clear effluent is drawn off, and the dilute sludge pumped to filter presses, where its moisture content is reduced to about 75 per cent. It is then dumped away. The sludge from the press is a dark chocolate brown substance of a claylike consistency and sp. gr. about 1.2. It comes from the press in sheets about $^3/_4$ inch thick, which give a faint, rather unpleasant odor. The average content of water was found to be 72 per cent.

Experiments on the drying of this substance were tried, and it was found that it dried readily in a current of warm air leaving a brown friable residue of a sp. gr. 0.3. If the temperature was maintained about 70°–80° C., it dried in less than 10 hours. If the substance was merely exposed to the air of the room, about 16° C., it required four days for the substance to lose all but about 10 per cent. of its moisture, and thereafter the drying process was very slow.

The dried substance was next subjected to destructive distillation by two methods. In the first series of experiments, the dry sludge was distilled in a small iron retort directly connected to a condenser and suitable gas washing bottle. In the second series, the retort was connected directly with a coil of $^1/_8$ inch gas pipe about $2^1/_2$ m. long kept at a red heat, through which the products of distillation passed. The rest of the apparatus was essentially the same in both series. One of the gas washing bottles contained a dilute solution of sulfuric acid to absorb any ammonia which might be evolved.

In every experiment the retort was heated to a bright red, and kept in this condition until the evolution of gas was less than thirty bubbles per minute, experiments having shown that the distillation was practically complete when this condition had been reached. It was then allowed to cool, and on opening, it contained a black, friable charcoal weighing about 58 per cent. of the original sludge. This substance contained 18.8 per cent. of carbon, and the ash from burning it, contained Fe and Ca, as bases, and Cl, PO_4, SO_4, CO_3, as acids.

In the first series, where the red hot coil was not used, the liquid distillate was a brown oil smelling of pyridine bases and weighing about 20 per cent. of the weight of the original sludge. It was subjected to fractional distillation with the following results: 59 per cent. of the oil collected in the fraction up to 100° C., 17 per cent. at 100° to 115° C.; 10 per cent. at 240° to 285° C.; black tarry residue 13 per cent. left

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[1] *Eng. Record*, **63**, 79.

[2] *Ibid.*, **65**, 701.

[3] *J. Gasbel*, **54**, 231.

[4] *Eng. Record*, **58**, 445.

[5] *Rev. chim. Ind.*, **21**, 779.

[6] *Rev. gén. chim.*, **14**, 199.

[7] *Eng. Record*, **58**, 445.

[8] *Trans. Inst. Min. Eng.*, [4] **35**, 37; Brit. Pat., 18,199, Aug. 31, 1908.

[9] *Mun. J. Eng.*, **32**, 483.

in still. A few qualitative tests on these fractions showed thiophene, pyrrol, and possibly indol. In the second series, where the products of distillation passed through the red hot coil, the liquid distillate was only about 2/3 of the amount collected in the first series. It was black, and had the odor of ordinary gas works tar, probably containing similar constituents.

The evolution of gas in the first series, was on the basis of about 11.38 cu. m. per 100 kg. dry sludge. When burned in a fishtail burner, it gave a flame containing a comparatively large proportion of blue, and was distinctly poor in illuminating constituents.

In the second series the yield of gas was on a basis of 19.08 cu. m. per 100 kg. dry sludge, and was far superior in illuminating qualities, not only to the gas made in the first series, but also to the gas from the city main.

The following table is a comparison of the results obtained with the Worcester sludge and those of Bayer and Fabre, and others:

	Worcester sludge by direct distillation	Worcester sludge distilled with use of red hot coil	(Frankfort)	Bayer and Fabre
Gas per 100 kg..	11.4 cu. m.	19.1 cu. m.	20 cu. m. (common gas coal)	23.8 cu. m.
NH_3 per 100 kg..	....	0.27 kg.	0.13 kg. to 0.28 kg.	0.75 kg.
Tar............	20%	13%	3%	
Organic matter in sludge........	....	50%		60%
Yield of coke....	....	58.3%	60%	57.6%
Carbon in coke..	....	18.5%	90%	

Composition of gas evolved:

	CO_2	C_2H_4, etc.	O_2	CO	CH_4	H_2	N_2	Sp. gr.
Worcester sludge:								
First series......	24.6	5.0	0.6	9.6	13.9	18.8	27.8	0.733
Second series....	18.4	15.9	0.7	7.5	13.8	28.2	15.5	0.499
Bayer and Fabre:								
1907............	2.2	4.8	0.8	17.5	18.1	44.2	12.0	....
1908............	2.2	7.3	0.8	14.4	23.2	41.7	7.9	...
Coal gas (a)......	1.1–1.8	3–5.7	...	3–9	32–39	42–53	1.0–5.0	0.468

(a) "Gas Manufacture," by W. J. A. Butterfield, p. 76.

EXPERIMENTAL PORTION

To dry the sludge, two methods were tried, at the temperature of the room, and over a steam radiator where there was a good circulation of air. The temperature of the room was about 16° C., while the air above the radiator varied from 60°–80° C. The following figures are typical of many series of drying experiments:

	Original weight	After 10 hrs.	After 24 hrs.
At room temperature....	264 grams	207 grams	155 grams
On radiator............	172 grams	67 grams	65 grams
	After 48 hrs.	After 72 hrs.	After 12 days
At room temperature....	106 grams	92 grams	84 grams
On radiator............	No further loss		

Thus we see that the sludge on the radiator was practically dry in 10 hours, while the sludge at the room temperature lost 21 per cent. of its moisture in that time. After 24 hours it had lost 41 per cent., the next day 60 per cent., the following day 65 per cent., and thereafter the loss was very slow, its moisture content being reduced to about 3 per cent. at the end of 12 days. In each case the sludge was in the form of the filter press sheet.

The dry substance was light brown in color and almost odorless. It was found to contain approximately 50 per cent. of organic matter, while the sludge used by Bayer and Fabre[1] contained almost 60 per cent. This partly explains the smaller yield of gas from the Worcester sludge. When burnt to an ash, a brownish residue was left, the color being apparently due to iron. It had a strong alkaline reaction and contained phosphates, sulfates, chlorides, carbonates, calcium, iron and small amounts of the alkali metals. A quantitative analysis was not made, but it is probable that it contained about 1 per cent. of phosphoric acid and less than 0.5 per cent. of potassium, the results of Bayer and Fabre.

The dry sludge was heated in a small iron retort, 60 grams being taken for each charge. This yielded an average of 35 grams of a black friable residue containing 18–19 per cent. of carbon. In the first series of experiments, where the superheating coil was omitted, a brown oil was collected from the condenser, which had an odor resembling the tar of an old tobacco pipe. It averaged in weight 20 per cent. of the weight of the original charge of sludge. When fractionally distilled the following results were obtained:

Weight of oil taken 97 grams
Fraction (a) to 100° C. 57 grams
Fraction (b) 100–115° C. 17 grams
Fraction (c) 115–135° C. a few drops
Fraction (d) 135–240° C. nothing
Fraction (e) 240–285° C. 10 grams
Residue in still, 12 grams

There was a distinct break in the continuity of the distillation between 115 and 240°, and another between 265 and 284°. At this latter point much of the oil in the last fraction was distilled. The temperature then fell to 220°, whereupon the distillation was stopped. A thick, black tar remained in the still which would not distil with steam, and was not investigated further. Anderson[1] states that in the distillation of bone oil there was a break in the distillation after 2/5 of the oil had been distilled. This is like the break in the distillation of the sludge oil already described. He states that the temperature had to be considerably raised in order that the distillation might continue uniformly. No thermometric data are given.

A few qualitative tests on the fractions were made in order to get a general idea of their composition. Fraction (a) contained water, ammonia, gave a deep red with sulfuric acid, probably thiophene, and turned a pine shaving deep red, probably pyrrol. Fraction (b) was composed of two layers. The lower contained water and pyridine, the latter shown by a test with hydrochloric acid and chlorplatinic acid which gave a red insoluble precipitate. The upper layer was mostly pyrrol as its vapors colored a pine shaving moistened with hydrochloric acid a deep red. Fraction (c) gave no definite tests though indol was indicated by the action of $NaNO_2$ and H_2SO_4.

The gas which was freely evolved during distillation

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[1] *Trans. Royal Soc. Ed.*, **16**, 463.

measured 6–7.5 liters per charge of 60 grams of sludge, or on a basis of 11.38 cu. m. per 100 kg. It was collected over water and allowed to stand over night. It was then burnt in a fishtail burner and found to give about $^1/_2$ the illumination that the city gas supply gave. The flame contained a comparatively large porportion of blue. Analysis of the gas gave the following results, 100 cc. being taken for each analysis and the analyses being done in duplicate:

Vol. after KOH	74.9 cc.	75.9 cc.	24.6% CO_2
Vol. after fuming H_2SO_4	70.0 cc.	70.8 cc.	5.0% Hydrocarbons
Vol. after pyrogallol	69.4 cc.	70.2 cc.	0.6% O_2
Vol. after cuprous ammonium	59.8 cc.	61.2 cc.	9.6% CO

10 cc. of the residue were then mixed with 50 cc. of air and exploded by means of an electric spark.

Vol. before explosion	60 cc.		
Vol. after explosion	51.0 cc.	50.6 cc.	
Vol. of contraction	9.0 cc.	9.4 cc.	Mean, 9.2 cc.
Vol. after KOH	48.6 cc.	48.8 cc.	
Vol. of CO_2	2.4 cc.	1.8 cc.	Mean, 2.3 cc.
Vol. of CH_4, in 10 cc. residue			2.3 cc.
Contraction due to $H_2 + O_2$ in 10 cc. residue			4.6 cc.
Vol. of H_2 in 10 cc. residue			3.1 cc.

Results for CH_4, H_2 and N_2 on basis of original gas:

Vol. of H_2	18.8 per cent.
Vol. of CH_4	13.0 per cent.
Vol. of N_2	27.8 per cent. (by difference)

The density of the gas was found by Bunsen's diffusion method, as follows:

	Average time for series of experiments	Results
Air	26.85 seconds	1.000 sp. gr.
Sewage gas	22.99 seconds	0.733 sp. gr.
City gas	18.52 seconds	0.468 sp. gr.

In order to decompose the basic tarry distillate into more useful compounds, in the next series of experiments, the products of distillation were passed through a coil of $^1/_2$ inch gas pipe heated to bright redness. The coil was about 10 cm. in diameter and contained about 2.5 m. of pipe. It was expected that this would give a larger yield of gaseous products, and at the same time cause the decomposition of many of the complex organic bases into ammonia. Simultaneously the more valuable hydrocarbons of the benzol series ought to replace them in the tar. The only alteration, therefore, in the apparatus for this series of experiments, was the insertion of the superheating coil between the retort and the condenser. The procedure in the experiments remained the same as in the first series.

Under these conditions, the liquid distillate, which in the first series had the odor of pyridine and its homologues, now had the black color and characteristic odor of common coal tar. The amount, however, was less, the yield from 60 grams of sludge being on an average 8 grams. An aliquot part of the gas liquor was taken for the determination of ammonia, the gas being distilled off and titrated with standard acid. The yield was found to be on a basis of 0.27 kg. NH_3 per 100 kg. sludge.

The gas, as produced from the apparatus with the use of the superheater, was increased in quantity and improved in quality. A charge of 60 grams of sludge gave on an average 11.4–11.9 liters of gas, or on a basis of 19.08 cu. m. per 100 kg. sludge. When burnt in a fishtail burner it gave a flame far brighter than the flame of the city gas. Analysis gave the following results, the procedure being the same as in the analysis of the gas made without the superheated which has been already described.

Vol. after KOH	81.2 cc.	82.0 cc.	18.4% CO_2
Vol. after fuming H_2SO_4	65.2 cc.	66.2 cc.	15.9% Hydrocarbons
Vol. after pyrogallol	64.5 cc.	65.4 cc.	0.7% O_2
Vol. after cuprous ammonium	56.6 cc.	58.4 cc.	7.5% CO

10 cc. of the residue was then mixed with 40 cc. of air and exploded by means of an electric spark.

Vol. before explosion	50.0 cc.	....	
Vol. after explosion	37.2 cc.	38.4 cc.	
Vol. of contraction	12.8 cc.	11.6 cc.	Mean, 12.2 cc.
Vol. after KOH	34.8 cc.	36.0 cc.	
Vol. of CO_2	2.4 cc.	2.4 cc.	

Vol. of CH_4 in 10 cc. of residue	2.4 cc.
Contraction due to $H_2 + O_2$	7.4 cc.
Vol. of H_2 in 10 cc	4.9 cc.
Total vol. of CH_4 in original gas	13.8 cc.
Total vol. of H_2 in original gas	28.2 cc.
Total vol. N_2 in original gas	15.5 cc. (by difference)

The density of the gas was found by Bunsen's diffusion method, as follows:

	Average time for series of experiments	Results
Air	22.20 seconds	1.000 sp. gr.
Gas	20.06 seconds	0.499 sp. gr.

SUMMARY

We can briefly recapitulate the results obtained in this work as follows:

1. That the sludge will become almost dry at ordinary temperatures in four days, or in less than ten hours if exposed to a mild, artificial heat.
2. That the yield of gas from the heated sludge is comparable to the yield from a ton of gas coal, if the products of distillation are passed through a superheater.
3. That the quantity of illuminating hydrocarbons in this gas is almost three times as great as in coal gas.
4. That the superheating of the tarry distillate changes its character from a substance resembling bone oil to what appears to be coal tar.
5. That the amount of ammonia is fully equal to that obtained from coal.

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ALLOYS OF COBALT WITH CHROMIUM AND OTHER METALS[1]

By Elwood Haynes

As in organic nature, certain animal and vegetable forms have undergone modifications, and thus, as it were, fitted themselves to live in a new environment, so it has been found possible in certain instances to form new metallic combinations which are practically immune to the natural conditions that exist on the earth at the present time. A few of the rare metals, such as gold, silver, and the metals of the platinum group, are found native, but the commoner heavy metals are nearly always found in the form of oxides or sulfides.

For a number of years I have made experiments with a view to finding certain metallic combinations

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[1] Paper presented at the Eighth International Congress of Applied Chemistry, New York, September, 1912.

or alloys which would not only resist oxidation and other harmful influences, but would also possess valuable physical properties, which would render them fit for special services. The first decisive step made in this direction, was the discovery of an alloy of nickel and chromium in 1898. The properties of this alloy have already been described in a previous paper.[1] Immediately following the discovery of the nickel-chromium alloy, I produced an alloy of cobalt and chromium. This has likewise been described in the previous paper, but the range of proportion between the cobalt and chromium is so great, and the combination is modified to such a marked degree by the introduction of other substances, that I have felt justified in reading another paper on this subject, describing more fully my researches in this particular direction.

As early as 1907 and 1908, I made alloys or combinations of cobalt, chromium and tungsten; cobalt, chromium and molybdenum; and cobalt, chromium, tungsten and molybdenum. I have made alloys of cobalt and chromium containing zirconium, tantalum, thorium, titanium, vanadium, etc. I have also added to the cobalt-chromium alloys the non-metallic elements, carbon, silicon, and boron. Some exceedingly interesting results have been obtained from these various combinations, and while further investigation is necessary in order to fully determine their chemical and physical properties, a number of them have shown interesting economic possibilities.

The preliminary fusions were made in graphite crucibles by means of a furnace operated by natural gas. I was later obliged to use crucibles of a special composition, not only to avoid the contamination of the metal by carbon from the crucible, but also because they proved more reliable under long-continued heating, than those made of graphite.

The metal tungsten alloys readily with chromium and cobalt in all proportions. When added in small quantity to the cobalt-chromium alloy, it seems to have little influence on the properties of the combinations, but if the proportion rises to 2 per cent. or 3 per cent., a notable effect is produced. Generally speaking, the cobalt-chromium alloy becomes harder and more elastic, especially if it contains a small amount of carbon, boron, or silicon.

The following experiment shows the effect of melting the alloy in a graphite crucible: 90 grams of cobalt, 6.3 grams of tungsten, 18 grams of chromium, together with a small quantity of calcium silicide, were introduced into a graphite crucible. The resulting alloy was very hard, and the crucible much eroded on the inside. The bar could be slightly flattened at one end, and after being made into a cold chisel, showed remarkable qualities. It would not only scratch glass, but also quartz crystal. It was quite tough at ordinary temperatures, and would cut small chips or shavings from a piece of stellite. At a bright yellow heat it showed signs of fusion, and became covered with a skin of oxide.

An alloy was made by melting the following in a special crucible: Cobalt rondelles 80 grams, chromium 20 grams, tungsten 7 grams, calcium silicide 10 grams, calcium carbide 5 grams. As soon as the above were melted, the crucible cover was removed, and 15 grams of an alloy of cobalt and boron were introduced. The crucible lid was then replaced and the heating continued. A heavy, thick slag formed, which was removed before pouring the metal. The resulting bar was very hard and *elastic*, but drew only slightly under the hammer, and then broke. A cold chisel made from the cast metal cut iron readily. The bar was broken up and remelted with about one-third of its weight of an alloy of cobalt, chromium, tungsten and carbon. The result was a fine-grained alloy which was very *elastic*, and would draw out to a considerable degree under the hammer without checking. Its elastic limit must have been very high, since when it broke, the pieces were thrown violently.

Taking the alloy of chromium and cobalt as a basis, and holding the proportion of chromium at 15 per cent. of the entire mixture, it was found that the alloy gradually increased in hardness with the percentage of tungsten. When the quantity of tungsten rises to 5 per cent., the alloy becomes distinctly harder, particularly when forged under the hammer. When the tungsten reaches 10 per cent., the metal still forges readily, and a tool formed from the alloy takes a fine cutting edge. This alloy is suitable for both cold chisels and wood-working tools. When the tungsten rises to 15 per cent., the metal can still be forged, but great care is necessary in order to avoid checking. This alloy is considerably harder than that containing 10 per cent. of tungsten and is excellent for cold chisels. When the tungsten rises to 20 per cent., the alloy is still harder, and can be forged to a small extent. It makes good lathe tools for cutting steel and other metals at moderate speeds. When the tungsten rises to 25 per cent., a very hard alloy results, which cannot be forged to any extent, but casts readily into bars which may be ground to a suitable form for lathe tools. These tools have shown great capabilities, particularly for the turning of steel, since they are very strong, and retain their hardness at speeds which almost instantly destroy the cutting edge of a steel tool. The tungsten may be still further increased to 40 per cent., and the alloy will retain its cutting qualities, and for turning cast iron, this alloy answers even better than that containing 25 per cent. When the tungsten reaches 40 per cent. or more, the alloy becomes so hard that it will not only scratch glass, but will readily scratch quartz crystal. A small drill, made of this material, drilled a hole through the wall of a glass bottle without the addition of any liquid or other lubricant. A three-eighth inch square cast bar, when ground to a suitable edge, was set in a tool holder attached to a lathe. The workman who had operated the lathe, had been able to turn to form 26 cast iron wheels in 10 hours with a steel tool of the same size. The stellite tool turned 49 of these wheels to form in the same time. The steel tool was ground 50 times during the operation, while the edge of the stellite tool was dressed slightly by a carborundum whetstone, after its day's work was

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[1] This Journal, **2**, 397.

completed. A set of steel cutters, placed in the boring head of a cylinder-boring machine, were able to bore from 26 to 28 holes in 10 hours. These cutters were replaced by others made of stellite, which performed the work in 3 hours and 20 minutes, or a little more than one-third the time. Not only was the speed of the mill doubled, but the feed also, and notwithstanding this severe ordeal, the stellite cutters were only slightly worn, while it would have been necessary to regrind the steel cutters at least two or three times for the same service at slower speed. Some remarkable results were obtained in the turning of steel on the lathe. For example, a cylindrical bar of annealed nickel-chrome steel, about two and one-half inches in diameter, was placed in a lathe and turned with a steel tool at about as high a speed as the steel would permit without "burning." The steel tool was then replaced by one of stellite, and the speed at the same time increased to two and two-thirds its former speed. The stellite tool retained its edge under these severe conditions, and produced a shaving weighing one and two-tenths pounds in one-half minute. Just what the effect of the alloy will be in machine shop practice is at present somewhat difficult to determine. In my opinion, however, it will not fully supersede high-speed steel in the machine shop, but in cases where rapid work is the main consideration, it will doubtless replace high-speed steel.

When molybdenum is added to a 15 per cent. cobalt-chromium alloy, the alloy rapidly hardens as the molybdenum content increases, until the content of the latter metal reaches 40 per cent., when the alloy becomes exceedingly hard and brittle. It cuts keenly and deeply into glass, and scratches quartz crystal with ease. It takes a magnificent polish, which it retains under all conditions, and on account of its extreme hardness, its surface is not readily scratched. When 25 per cent. molybdenum is added to a 15 per cent. chromium alloy, a fine-grained metal results, which scratches glass rather readily, and takes a strong, keen edge. Its color and luster are magnificent, and it will doubtless find a wide application for fine, hard cutlery. It cannot be forged, but casts readily, and its melting point is not abnormally high.

If carbon, boron, or silicon be added to any of the above alloys, they are rendered much harder, though their effect is not always desirable, since they tend to render the alloys more brittle.

If either tungsten or molybdenum is added to a cobalt-chromium alloy containing 25 per cent. of the latter metal, the hardness of the alloy is rapidly increased. When the percentage of tungsten, for example, reaches 5 per cent., the alloy can be readily forged, and forms an excellent combination for wood-cutting tools, such as chisels, pocket knives, etc. When molybdenum is added to the same mixture of chromium and cobalt, much the same effect is produced, though, generally speaking, a smaller quantity of molybdenum is required to produce a given increase in hardness. In some instances I have found it advisable to add both molybdenum and tungsten to the cobalt-chromium alloys. Generally, the color and luster of these alloys, after polishing, are magnificent, and they seem to resist atmospheric influences equally as well as the binary alloy of cobalt and chromium.

KOKOMO, INDIANA

THE RELATION OF THE PRODUCTION OF ALUMINA TO THE FIXATION OF NITROGEN[1]

By Samuel A. Tucker

The connection of nitrogen fixation and the production of alumina is to be found in the Serpek process and probably in this process only. The Serpek process is primarily one for the fixation of nitrogen, but necessarily involves the production of alumina as a by-product and this by-product is all important to the success of the process commercially.

Briefly the Serpek process takes alumina and carbon in the proportions to form aluminium nitride in a strongly heated atmosphere of nitrogen, or $Al_2O_3 + 3C + N_2 \longrightarrow 2AlN + 3CO$.

The range of temperature necessary for this reaction is from 1600°–2000° C. according to Serpek. The product obtained contains about 30 per cent. nitrogen in the form of aluminum nitride, and is thus considerably richer in fixed nitrogen than calcium cyanamid which rarely runs over 20 per cent.

This reaction appeared so important to me that I thought it was worth while to investigate it and in conjunction with Mr. H. L. Read, the process was tried on a small scale. The results of this work have been read at the recent Congress of Applied Chemistry,[2] and it will be unnecessary to repeat them here, excepting that we found that the reaction worked satisfactorily on a small scale, the product contained 30 per cent. of nitrogen, and that bauxite was the best material with which to supply the aluminium. The temperature is important; below 1600° C. practically no nitrogen is fixed; above 2000° C. decomposition is likely to occur. I would like to know more as to the influence of temperature, and I had expected to conduct some experiments on this point so that I might give you this information now, but an accident to the electric furnace prevented it.

The commercial product must be nearly pure aluminium nitride, and at first sight seems to consist of two forms, the blue and the yellow. Professor Luquer has examined these specimens microscopically, and while his examination has not yet been completed he tells me that the internal structure is the same: both are strong double refracting and show uni-axial structure. With our present information, it would be difficult to figure the cost of production of this material, and even the power necessary for its manufacture is probably not generally known.

The question of the necessary time to convert the charge is important and in our experiments we found that it had a great influence on the nitrogen content of the product. It therefore seems necessary that

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[1] Presented at the Joint meeting of American Electrochemical Society, Society of Chemical Industry and American Chemical Society, Chemists' Club, New York City, February 7, 1913.

[2] See also *Met. Chem. Eng.*, 9, 745.

the mixture should be heated to a high temperature for a considerable time.

This is different from the Frank process for cyanamid, in that the temperature for the actual nitrogen fixation is relatively low. Granting that the process can be worked out on a satisfactory cost basis, we see that the production of alumina is an all-important consideration. By proper treatment of the Serpek product there seems to be no difficulty in obtaining the nitrogen as ammonia and the aluminum as alumina, and the latter should be practically pure Al_2O_3.

It might be supposed that the alumina so produced could be used over again in the process, but aside from its value in connection with the aluminum industry, pure alumina is not advantageous as a raw material, as it requires a higher temperature than bauxite. The process is therefore necessarily associated with the production of aluminium. If we might suppose that the Serpek process were to be conducted on the same scale as the Frank process for cyanamid manufacture, it is evident that it would involve the production of a large amount of alumina. From figures recently published regarding the Frank process, we find that the capacity output of all the plants covering various parts of the world for 1912 is roughly 200,000 tons. If we figure this as containing 18 per cent. nitrogen, it involves the fixation of 36,000 tons of nitrogen. With the Serpek product at 30 per cent. nitrogen content, it would require the production of 120,000 tons to equal this, and after decomposing to obtain the ammonia as follows,

$$2AlN + 3H_2O = 2NH_3 + Al_2O_3$$

we obtain 149,200 tons of alumina. This quantity of alumina should give 79,104 tons of metallic aluminum.

In the year 1911 we find that the capacity of all the plants producing aluminium was 62,100 tons, but that the estimated production actually was about 45,000 tons. We thus have raw material for the production of 34,000 tons of alumina in excess of present requirements. Of course the process will not be conducted on such a scale for some time to come, and by that time there might well be a market for the alumina. There appears to be no danger from the lack of bauxite as I find that in this country 155,618 tons were produced in 1911, and 196,000 tons in France for the year 1910.

As the Serpek process would necessarily come into competition with the other means of fixing nitrogen, and particularly with the cyanamid process, it might be well to consider for a moment a few points in connection with the latter.

Briefly the Frank-Caro process for making cyanamid is to subject finely ground calcium carbide to the action of pure nitrogen at a temperature of from 1000–1200° C. The energy necessary at this absorption stage is probably very small, as heat is evolved by the reaction

$$CaC_2 + N_2 \longrightarrow CaCN_2 + C$$

and the expenditure of energy is chiefly in the manufacture of the calcium carbide. Various conditions have to be observed in this absorption part of the process. The nitrogen must be practically free of oxygen and the temperature must not rise to the dissociation point of the cyanamid which is probably about 1370° C. The product so obtained contains anywhere from 15 to 22 per cent. of nitrogen. The nitrogen is obtained by passing air over copper to take up the oxygen, or by fractionally distilling liquid air. Both systems seem to be in use, but with the latter some treatment of the resulting nitrogen with copper would probably have to be employed as nitrogen obtained from liquid air contains upwards of 8 per cent. oxygen.

The process seems fairly complicated, but possesses the advantage over the high tension arc systems of fixation in that a better return of nitrogen is obtained for the power used. In the Frank process the chief expenditure for power is in the formation of calcium carbide. The average of several plants making carbide is 5.2 kg. of 80 per cent. carbide per kw. day of 24 hours. This gives 4.16 kg. of pure calcium carbide which corresponds to 173 grams per kw. hour. Now if this is converted to cyanamid having a 20 per cent. nitrogen content, we get 43.2 grams nitrogen per kw. hour.

The Birkeland-Eyde process obtains about 57.1 grams nitric acid per kw. hour and the nitrogen content is 12.7 grams. We thus have about three and one-half times the nitrogen fixed by the Frank process as cyanamid as we do for the Birkeland-Eyde process as nitric acid, and this is probably the reason for its being established on such a large scale.

As to the relative value of the fixed nitrogen fertilizers, we have no very precise information. Taking the various products adapted to this purpose in the different processes, we get calcium nitrate by the Birkeland-Eyde, calcium cyanamid by the Frank, and lastly ammonia by the Serpek process. These must all be compared with Chili sodium nitrate, and the general opinion seems to be that the latter is the most valuable.

Field experiments have been conducted with cyanamid and calcium nitrate, but precise information is lacking. It would seem, however, from what has so far been done, that we should put them in the following order:

(1) Chili saltpeter, (2) calcium nitrate, (3) cyanamid, (4) ammonia. The action of cyanamid is quite different from the others; it supplies a large amount of calcium which may or may not be an advantage. Its slow solubility is, however, advantageous. Ammonia in the form of sulfate is pretty well understood in its relation to fertilization and for that reason the Serpek product should have a ready market in this connection.

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ON THE RELATIONSHIP BETWEEN THE WEIGHT OF THE SUGAR BEET AND THE COMPOSITION OF ITS JUICE

By J. Arthur Harris and Ross Aiken Gortner

Received October 14, 1912

In the voluminous literature of sugar beet chemistry published from the Federal and State agricultural institutions, the weight of the beet, the per cent. of total solids and of sucrose in the juice, and the coëfficient of purity, *i. e.*, the ratio of sucrose to 100 parts of total solids, are usually given.

Much of this work has been done for the purpose of ascertaining where sugar beets can be profitably grown. Roots below a certain standard of sucrose content and purity are, from well known factory requirements, unworkable. Records for size of beets have apparently been added because yield is assumed to be, in some degree, dependent upon size, and because size has been suspected to have some relation to composition.[1]

This latter point has, however, been but little investigated, and we believe only by inadequate methods. It has seemed to us of importance to measure the intensity of this relationship on the —1 to +1 scale of the coëfficient of correlation; also to write the regression equations showing the absolute change in solids, sugar, or purity, associated with a unit change in weight (in grams or ounces) of the beet. The importance of doing this seems to lie in three considerations:

(*a*) The working out of such inter-dependences is from the standpoint of physiology and phytochemistry, a problem of considerable interest.

(*b*) If the composition of the beet depends at all closely upon its size, the analysis of small samples sent in to government departments are practically worthless as indicating the practicability of localities for sugar beet growing. Even if the samples are not consciously selected for size, the errors of random sampling are such as to render practically valueless any conclusions drawn upon analyses which are not corrected for the size factor.

(*c*) Finally, if composition be closely correlated with size, it may be possible to modify the rate of seeding, methods of fertilization, or cultivation, in such a way as to secure the sugar yield which is most profitable in relation to the cost of growing and handling.

Suitable published data seem all but wanting. In many series the weights given are averages, without specification as to the number of beets included. Analyses have been made by the thousands, and in some cases upon uniform material drawn from the same cultural conditions, but we have not been able to obtain such records, either published or in manuscript.[1] Hence, these notes serve merely to indicate the importance of the problem and the usefulness of the method of attack.

Table I gives the data slightly condensed from Wiley and Maxwell[2] for sixty-one Washington Klein Wanzlebener beets. Tables II–V give mean compositions for various weights in ounces[3] for four varieties from Nevada data.[4] The calculations have also been made from the total Nevada material.

The correlation coefficients[5] and the regression straight line equations are as follows, where w = weight,[6] t = total solids, s = sucrose, p = purity:

KLEIN WANZLEBENER, WASHINGTON DATA[7]—TABLE I

$$r_{wt} = -0.283 \pm 0.079,\quad t = 15.459 - 0.012\,w$$
$$r_{ws} = -0.224 \pm 0.082,\quad s = 10.903 - 0.011\,w$$
$$r_{wp} = -0.111 \pm 0.085,\quad p = 70.176 - 0.018\,w$$

IMPROVED KLEIN WANZLEBENER, NEVADA DATA[7]—TABLE II

$$r_{wt} = -0.538 \pm 0.060,\quad t = 18.864 - 0.068\,w$$
$$r_{ws} = -0.756 \pm 0.036,\quad s = 17.197 - 0.121\,w$$
$$r_{wp} = -0.499 \pm 0.063,\quad p = 89.996 - 0.300\,w$$

VESBESSERTEN KLEIN WANZLEBENER, NEVADA DATA—TABLE III

$$r_{wt} = -0.619 \pm 0.043,\quad t = 20.040 - 0.092\,w$$
$$r_{ws} = -0.641 \pm 0.041,\quad s = 16.819 - 0.106\,w$$
$$r_{wp} = -0.587 \pm 0.046,\quad p = 85.956 - 0.276\,w$$

VILMORIN AMELIORÉE, NEVADA DATA—TABLE IV

$$r_{wt} = -0.294 \pm 0.086,\quad t = 19.266 - 0.090\,w$$
$$r_{ws} = -0.580 \pm 0.063,\quad s = 18.054 - 0.223\,w$$
$$r_{wp} = -0.623 \pm 0.058,\quad p = 95.038 - 0.905\,w$$

DESPAREZ, NEVADA DATA—TABLE V

$$r_{wt} = -0.512 \pm 0.058,\quad t = 21.185 - 0.130\,w$$
$$r_{ws} = -0.472 \pm 0.061,\quad s = 18.691 - 0.131\,w$$
$$r_{wp} = -0.173 \pm 0.076,\quad p = 87.860 - 0.089\,w$$

ALL VARIETIES, NEVADA DATA, 475 BEETS

$$r_{wt} = -0.497 \pm 0.023,\quad t = 20.119 - 0.096\,w$$
$$r_{ws} = -0.576 \pm 0.021,\quad s = 17.644 - 0.122\,w$$
$$r_{wp} = -0.474 \pm 0.024,\quad p = 88.516 - 0.273\,w$$

Considering the shortness of the materials upon which they are based, these results are surprisingly consistent throughout. They show that composition and purity are very closely correlated with weight, and in such a way that as weight increases, total solids, sucrose and percentage purity fall rapidly. The rate of fall on the relative scale of —1 to +1 is shown by the coëfficient of correlation, r, the rate in an absolute scale by the second term of the regression equation. We have prepared graphs of most of these

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[1] We have examined fifty-eight papers on the sugar beet, comprising bulletins from the Bureau of Chemistry, U. S. Dept. of Agr. and from twenty-two state experiment stations. In only five of these is the possible relationship between the weight of the beet and the sucrose content and purity coëfficient considered. Two, *Bull.* **197**, Mich. Agr. Exp. Sta. and *Bull.* **68**, Purdue Univ. Exp. Sta., give experiments which indicate that the smaller the weight of the beet, the higher is the sugar content and coëfficient of purity. Of the other three, *Bulls.* **58** and **63** of the Colorado Exp. Sta., state that "as a rule medium-sized beets (1–2 pounds) are richer than either large or small beets," and *Bull.* **155**, N. Y. Agr. Exp. Sta. (Geneva) contains the statement that "between these limits (5–27 oz.) the increase in size did not apparently exercise any marked or definite influence upon either the percentage or the purity of sugar." Doubtless other suggestions might be found, especially in foreign publications. It is perhaps generally admitted that excessively large beets (4 pounds and over) are as a rule low in sugar and have a low coefficient of purity, but, as far as we know, there has been no thorough investigation of the general problem of weight and composition.

[1] We shall be grateful for the loan of any suitable series of data for more refined statistical analyses.

[2] H. W. Wiley and W. Maxwell, *Bull.* **39**, Bureau of Chem., U. S. Dept. Agr.

[3] The weights are grouped in units of 5-oz. range. The numbers given are the centers, *i. e.*, 3 = 1–5, 8 = 6–10, etc.

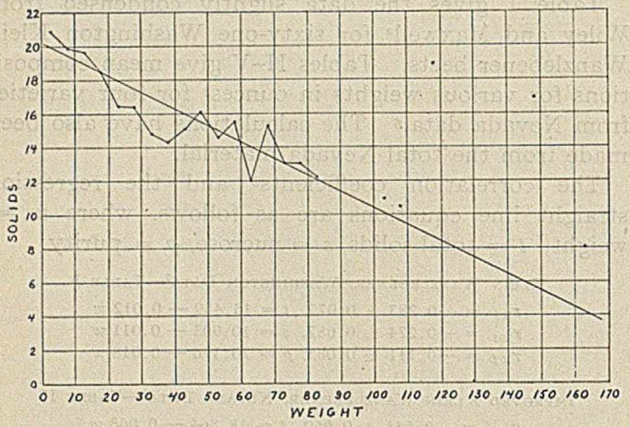
[4] N. E. Wilson, *Bull.* **32**, Nev. Agr. Exp. Sta.

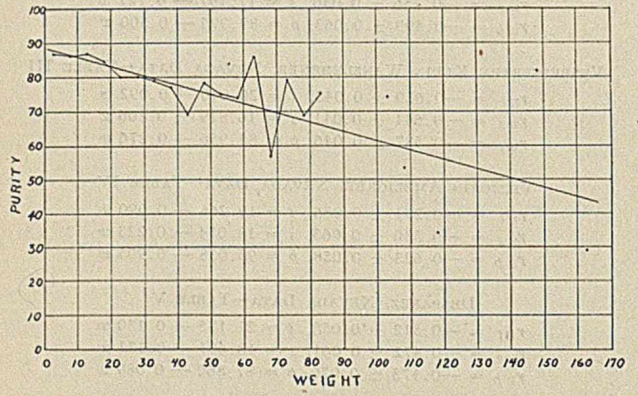
[5] The methods of calculating the correlation coëfficient and the regression equation can be found in various books on modern higher statistics. These for the first table have been worked out by the brute force method, summations, summations of squares, and summations of products being made directly. Those for Tables II–V have been made by the method described by Harris (*Amer. Nat.*, **44**, 693–699 (1910)) before the sums for each class weight were divided out to get the means, which show most clearly to the eye the changes in composition. Here again the means and standard deviations were obtained by the brute force method, the analyses being kept to one decimal place for solids and sucrose and to whole per cents. for purity.

[6] In the Washington data the rate of change in per cent. total solids, per cent. sucrose, and per cent. purity, are calculated on weight units of 10 grams each. In the Nevada data the weight units are ounces.

[7] *Loc. cit.*

equations and while the empirical means are very irregular there is no very clear evidence to show that regression is other than linear. The diagrams show the lines and empirical means of the (heterogeneous) Nevada total material. Those means which are based upon a fairly satisfactory number of beets are joined up.





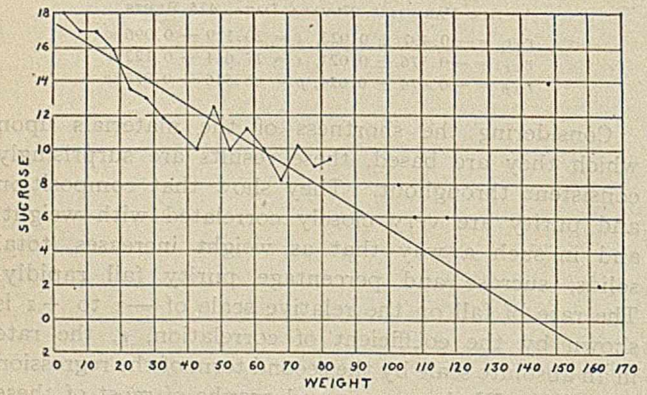


TABLE I—KLEIN WANZLEBENER

Key number	Weight in ten-gram units	Total solids	Sucrose in juice	Purity	Key number	Weight in ten-gram units	Total solids	Sucrose in juice	Purity
60	88	20.1	17.8	89	75	86	17.8	12.3	69
65	110	19.7	15.0	78	76	37	18.9	13.3	70
66	67	16.6	11.5	69	77	38	17.5	11.7	67
67	146	18.9	13.6	72	78	74	17.7	12.3	70
68	137	15.4	10.7	69	79	52	17.5	13.2	75
69	98	19.2	16.0	83	80	126	16.6	11.4	69
70	137	15.3	10.6	69	81	50	16.5	11.0	67
71	232	13.8	8.9	65	82	91	14.4	9.0	63
72	88	16.4	12.0	73	83	79	16.0	10.0	63
73	136	16.6	12.5	75	84	51	14.5	9.1	63

TABLE I—KLEIN WANZLEBENER (*Continued*)

Key number	Weight in ten-gram units	Total solids	Sucrose in juice	Purity	Key number	Weight in ten-gram units	Total solids	Sucrose in juice	Purity
85	164	14.8	9.8	66	138	109	13.6	9.4	69
86	120	15.0	10.0	67	139	86	14.0	9.8	70
97	102	17.0	11.9	70	140	140	13.7	8.5	62
98	88	16.8	11.8	70	141	54	14.1	8.0	57
107	98	17.9	14.3	80	142	74	15.1	11.0	73
108	88	16.0	11.5	72	143	84	15.0	11.3	75
110	61	17.1	11.6	68	144	92	16.5	11.6	70
111	50	15.9	11.2	70	145	74	16.3	11.5	71
112	48	15.2	9.7	64	146	55	13.5	9.6	71
113	33	17.0	11.8	69	147	82	15.2	11.9	78
114	54	17.9	13.2	74	148	109	15.5	10.7	69
116	33	17.7	11.0	62	149	54	16.7	12.4	74
117	38	18.5	13.9	75	150	72	16.4	11.4	70
118	91	16.9	13.6	81	151	65	15.8	10.6	67
120	85	17.7	14.5	82	152	47	17.0	13.4	79
121	72	16.5	12.8	78	153	37	19.3	13.3	69
123	40	16.5	12.6	76	154	50	18.8	15.4	82
130	157	16.2	12.0	74	155	58	14.3	10.3	72
131	65	17.5	13.7	78	156	95	17.5	13.1	75
135	45	16.5	14.8	90	157	127	15.7	11.2	71
137	126	15.2	10.0	66	..	..	..	..	..

TABLE II—IMPROVED KLEIN WANZLEBENER

Weight	N	Average solids	Average sucrose	Average purity
3	4	17.42	15.40	83.25
8	12	18.78	16.22	86.00
13	9	19.20	17.16	89.67
18	11	17.85	15.35	86.00
23	8	15.69	13.19	83.62
28	1	14.30	13.10	92.00
33	4	14.48	11.23	77.75
38	4	13.93	10.50	75.75
43	2	14.80	12.35	84.00
48	1	21.80	13.30	61.00
53	3	15.27	10.87	83.33
58	2	18.05	10.80	66.50
68	2	15.30	8.95	58.50
78	1	13.00	8.80	68.00
83	1	15.00	11.00	73.00

TABLE III—VERBESSETER KLEIN WANZLEBENER

Weights	N	Average solids	Average sucrose	Average purity
3	3	20.87	18.53	89.33
8	19	19.83	16.43	83.26
13	18	21.12	16.73	82.67
18	11	18.41	15.59	83.64
23	5	17.42	13.66	78.00
28	12	18.83	12.75	75.83
33	5	11.16	10.22	73.40
38	9	14.31	10.56	74.67
43	2	13.15	8.05	60.00
58	1	15.20	12.50	82.00
63	1	15.40	13.60	88.00
83	2	10.80	8.25	76.00
103	2	11.10	7.95	73.50
108	2	11.00	6.15	53.50
118	1	18.60	6.30	34.00
163	1	7.90	2.10	29.00

TABLE IV—VILMORIN AMELIORÉE

Weight	N	Average solids	Average sucrose	Average purity
3	2	19.60	17.00	86.50
8	3	17.60	12.56	90.00
13	6	19.57	17.13	87.16
18	13	17.70	13.97	79.08
23	11	16.65	11.60	70.25
28	8	16.06	11.38	70.37
33	6	17.73	12.11	68.50
43	1	14.40	8.00	54.00
53	1	15.60	7.00	45.00

Table V—Desparez

Weight	N	Average solids	Average sucrose	Average purity
3	4	18.80	16.55	88.00
8	19	20.79	18.16	86.38
13	21	19.92	17.36	87.10
18	8	19.31	17.20	88.75
23	6	15.78	13.15	83.33
28	7	11.67	14.14	84.86
33	2	14.55	12.45	85.50
38	1	16.20	13.40	83.00
43	3	16.67	14.13	84.67
48	2	16.80	14.55	86.50
58	1	15.20	11.80	78.00

We believe that these results show with sufficient force the necessity of fully taking into account the weight of the individual beets in all studies on composition.

Carnegie Institution of Washington

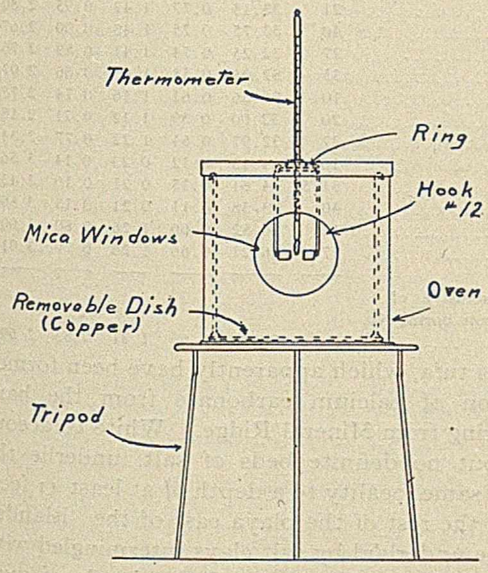
METHODS FOR TESTING COAL TAR, AND REFINED TARS, OILS AND PITCHES

By S. R. Church

Received November 20, 1912

Under this title, the writer has given[1] a description of the methods used in our laboratories for testing the raw materials and products of tar distillation. It was intimated that certain of these tests would undoubtedly be revised from time to time, in response to suggestions from our own chemists as well as from others engaged in the examination of these materials.

It is our present purpose to describe the tests that have been revised since that paper appeared. It may be well also to call to the attention of those interested in testing tars, oils and pitches, a paper by Arthur R. Warnes and W. B. Southerton, before the Midland Junior Gas Association,[2] entitled "Investigations on Coal Tar and Some of Its Products," and to "Methods for the Examination of Bituminous Road Materials," by Prévost Hubbard and Charles S. Reeve.[3]



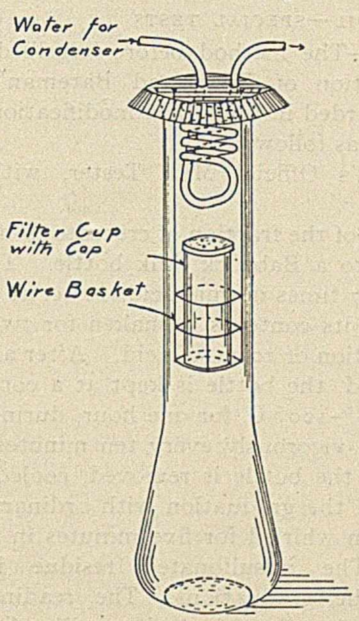
AIR BATH FOR MELTING POINT TEST.

Owing to the comparatively recent appearance of our first paper on this subject, no reference will be made at present to the methods in which no change has been made.

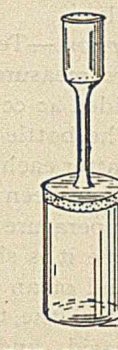
TAR

B. Specific Gravity.—The special Hubbard type specific gravity bottle, referred to in the previous paper, was not illustrated. It is now shown on the accompanying drawing No. 2.

C. Free Carbon.—In place of the Knorr extraction apparatus, we have adopted the extractor illustrated in the accompanying drawing No. 2. This apparatus the writer first saw in use in the Underwriters' Laboratories, Chicago, and it has already been described.[1] In it we use the same filter papers as described in our former article. The filter cup is suspended from two hooks, soldered on to the upper side of the flask cover.



EXTRACTOR FOR FREE CARBON



SPECIFIC GRAVITY BOTTLE

The advantages of this extractor over the Knorr or Soxhlet apparatus in making this determination are convenience, compactness, rapidity of operation.

E. Viscosity.—The time of flow of 100 cc. at 60° C. (140° F.) is taken, instead of 200 cc. as before. The quantity of material placed in the viscosimeter is the same, that is, sufficient to cover the fixed measuring points.

PITCH

C. Melting Point.—(3) For pitches from 77° C. (170° F.) upwards, an air bath, as shown in the accompanying drawing No. 3, is substituted for the bath of cottonseed oil formerly used. With this oven, shorter hooks are used, so that the pitch cube is suspended on a line running approximately through the center of the observation windows, the thermometer bulb being at the same level. The temperature of the oven is raised 5° C. (9° F.) each minute, as usual, and the temperature recorded by the thermometer at the instant the pitch drops to the bottom of the oven, is considered the melting point. To make

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[1] This Journal, **3**, 227.

[2] *Jour. Gas Lighting*, etc., Feb. 27, 1912.

[3] *Bull.* **38**, Office of Public Roads, U. S. Department of Agriculture, July 27, 1911.

[1] H. J. Cary-Curr, This Journal, **4**, No. 7.

results by this method comparable with results obtained in water or oil, 6.5° C. (12° F.) should be added to the observed melting point.

E. Evaporation Test.—Reference was made to the use of a circular oven (of the type E. and A., 2073 D) having double walls, circulating fan and self-contained burner. Results obtained with this oven have not been entirely satisfactory, and an ordinary drying oven, lagged with asbestos, as before described, is still used for this determination.

CREOSOTE OIL—SPECIAL TESTS

C. Sulfonation Test.—The method before described, which was a modification of Dean and Bateman's method,[1] has been discarded in favor of a modification described by Bateman[2] as follows:

Apparatus.—Babcock's Official Milk Tester, with four bottles.

Description.—Ten cc. of the fraction of creosote to be tested are measured into a Babcock milk bottle. To this is added 40 cc. of 37 times normal acid, 10 cc. at a time. The bottle with its contents is shaken for two minutes after each addition of 10 cc. of acid. After all the acid has been added, the bottle is kept at a constant temperature of 98°–100° C. for one hour, during which time it is shaken vigorously every ten minutes. At the end of an hour the bottle is removed, cooled, and filled to the top of the graduation with ordinary sulfuric acid, and then whirled for five minutes in a Babcock separator. The unsulfonated residue is then read off from the graduations. The reading multiplied by two, gives per cent. by volume directly. (Each graduation equals one two-hundredths of a cc.)

It is important that the acid be of the proper strength. Fuming sulfuric acid is too strong, and ordinary concentrated acid too weak, a mixture of the two being required to obtain the necessary 80.07 per cent. SO_3, which constitutes 37 times normal.

A steam bath or steam-jacketed oven should be used for maintaining the bottle at a temperature of 98°–100° C.

Research Department
Barrett Manufacturing Co.
New York City

THE COMPOSITION OF SALINES IN SILVER PEAK MARSH, NEVADA[3]

By R. B. Dole, with analyses by Walton Van Winkle and A. R. Merz

Received November 12, 1912

DESCRIPTION

Silver Peak Marsh, comprising the lowest part of Clayton Valley, lies in Esmeralda County, Nev., about 20 miles west of Goldfield and 25 miles southwest of Tonopah. It is about 10 miles long northeast and southwest and about 4 miles wide, its area being about 32 square miles. It is most readily reached by means of the Silver Peak Railroad, which connects with the Tonopah and Goldfield Railroad at Blair Junction and runs south to Blair, a small mining town near the western edge of the marsh. The marsh is a salt playa entirely devoid of vegetation and covered for the most part with a white crust of sodium chloride. Tracts 3 or 4 acres in extent rising gently 1 to 3 feet above the general level appear as rough brown sun-baked patches without a covering of salt. The most noticeable topographic features are Goat and Alcatraz "islands," two groups of steep limestone hills near the southwest end of the playa. The surface of this alkali flat is usually dry, though it is sometimes covered by a foot or more of water after excessively heavy rainfall. The ground-water plane is, however, always high, and holes a few feet deep anywhere on the flat enter mud, many parts of the marsh being too soft to bear the weight of a horse. The present drainage basin of the valley has an area of 570 square miles.

EXPLORATION

In the spring of 1912 fourteen borings 8 to 55 feet deep were put down in different parts of the flat by the author for the purpose of exploring the surficial deposits. The upper layer, 5 to 10 feet thick, consists of brown mud containing a great quantity of finely crystallized salt. The strong brines in it circulate very slowly as the mud contains a large proportion of clay. The mud along the west shore bears nodules of calcareous tufa, which apparently have been formed by deposition of calcium carbonate from the hard waters entering from Mineral Ridge. White tufaceous materials, but no definite beds of salt, underlie the muds in the same locality to a depth of at least 41 feet. Throughout the rest of the playa east of the "islands" the muds are underlaid by salt clays intermingled with well-defined beds of clay containing crystals of gypsum and beds of crystallized salt containing saturated brine.

Total Salts and Potassium in Brines from Silver Peak Marsh, Nev., June, 1912

Examinations by A. R. Merz. Quantities in grams per 100 cc. unless otherwise designated

Boring No.	Depth of sample Feet	Total solids at 105° C	Potassium expressed as			
			Potassium (K)	Potassium chloride (KCl)	Potassium oxide (K_2O)	Percentage of potassium oxide (K_2O) in saline residue
3	15.5	33.28	0.91	1.74	1.10	3.30
6	21	33.13	0.77	1.47	0.93	2.80
6	40	33.75	0.75	1.43	0.90	2.67
11	27	32.25	0.74	1.41	0.89	2.76
11	35	32.05	0.55	1.05	0.66	2.07
12	10	26.56	0.61	1.16	0.74	2.78
12	20	32.90	0.59	1.12	0.71	2.15
12	27	32.97	0.64	1.22	0.77	2.34
13	16	4.15	0.12	0.23	0.14	3.36
13	31.5	4.61	0.13	0.21	0.16	3.43
13	40	3.38	0.11	0.21	0.13	3.80
14	11	26.82	0.66	1.26	0.80	3.00
14	17	26.21	0.66	1.26	0.79	3.01
Average, exclusive of samples from boring No. 13	..	30.99	0.69	1.31	0.83	2.69

COMMERCIAL POSSIBILITIES

The records indicate that the northeastern two-thirds of the playa is underlain at a depth of about 20 feet by beds 5 to 15 feet thick of crystallized salt mixed with some clay. Besides these beds practically all other strata to a depth of 50 feet contain appreciable proportions of salt that readily dissolves in the waters percolating through them.

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[1] U. S. Dept. Agric., *Forest Service Circular* **112**.

[2] U. S. Dept. Agric., *Forest Service Circular* **191**.

[3] Published by permission of the Director, U. S. Geological Survey.

PARTIAL ANALYSES OF BRINES, SILVER PEAK MARSH, NEV.
Examinations by Walton Van Winkle. Milligrams per kilogram except where otherwise designated

Source	Depth	Date, June, 1912	Specific gravity at 20° C.	Total residue		Carbonate radicle (CO_3)	Bicarbonate radicle (HCO_3)	Sulfate radicle (SO_4)	Borate radicle (B_4O_7)
	Feet			Dried at 180° C.	After ignition				
Boring No. 1	6	1	1.0281	39,330	38,620	0	532	570	....
	27	4	1.0406	57,600	56,240	Trace	327	582	....
Boring No. 3	15.5	4	1.2103	276,800	271,600	0	38	2,475	0
Boring No. 6	21	7	1.2081	273,000	270,000	0	28	2,675	0
	40	10	1.2082	274,000	270,000	0	36	2,395	0
Boring No. 11	27	13	1.2105	274,000	271,700	0	74	4,955	0
	35	14	1.2089	272,000	270,000	0	122	4,600	0
Boring No. 12	10	15	1.1702	226,700	224,100	0	51	4,640	....
	20	17	1.2097	274,500	270,900	0	45	4,030	....
	27	17	1.2098	274,700	271,300	0	50	3,900	....
Boring No. 13	16	20	1.0284	39,950	38,860	0	803	476	....
	31.5	21	1.0321	44,900	44,300	0	840	491	....
	40	22	1.0297	41,510	40,900	0	792	477	....
Boring No. 14	11	24	1.1747	233,200	228,800	0	39	2,250	....
	17	24	1.1697	227,900	223,500	0	33	2,090	....
Hot spring at bathhouse, Silver Peak	..	8	1.0217	30,670	29,960	0	533	...	Trace(?)
Cold spring at bathhouse, Silver Peak	..	8	1.0226	31,980	31,530	Trace	530	...	Trace
Cold spring at northeast end of marsh	..	14	1.0124	16,830	16,620	0	274	...	0
Hot spring at northeast end of marsh	..	14	1.0177	24,430	24,180	Trace	515	...	....
Spring at pumping station, Silver Peak	..	28	..	1,920	1,500	0	144	...	....
30-foot well at power house, Silver Peak	..	28	..	1,520	1,220	0	132	...	....

ANALYSES OF WATER FROM SILVER PEAK MARSH, NEV.
Walton Van Winkle, analyst

Composition in milligrams per kilogram

No.	1	2	3	4	5	6	7	8	9	10	11	12
Specific gravity at 20° C.	1.2089	1.2019	1.0300	1.1722	1.0217	1.0226	1.0124	1.0177	1.0406	1.0281	...	...
Silica (SiO_2)	0	20	50	0	80	20	30	40	40	40	...	...
Iron (Fe)	Trace	Trace	Trace	Trace	Trace	Trace	Trace	Trace	Trace	Trace	Trace	Trace
Aluminum (Al)	Trace	Trace	Trace	Trace	Trace	Trace	Trace	Trace	Trace	Trace	Trace	Trace
Calcium (Ca)	1,800	940	790	2,800	580	420	130	220	470	490	170	150
Magnesium (Mg)	650	290	1,030	100	70	70	20	40	270	190	58	56
Sodium (Na)	97,180	95,190	13,620	77,480	9,650	10,110	5,770	8,370	19,210	13,030	352	195
Potassium (K)	7,290	5,890	1,290	6,590	930	930	500	800	2,060	1,180	21	15
Carbonate radicle (CO_3)	0	0	Trace	0	0	Trace	0	Trace	Trace	0	0	0
Bicarbonate radicle (HCO_3)	40	70	700	40	533	530	270	520	330	530	144	132
Sulfate radicle (SO_4)	2,360	4,420	540	2,210	410	480	580	690	610	610	95	160
Chlorine (Cl)	159,730	153,710	23,760	134,400	17,130	18,010	9,330	13,650	33,050	22,110	858(*a*)	548(*b*)
Total residue dried at 180° C.	278,760	264,030	42,820	233,440	30,670	31,980	16,830	24,430	57,600	39,330	1,868	1,379
Total residue after ignition	270,990	262,670	41,610	228,440	29,960	31,530	16,620	24,180	56,240	38,620	1,672	1,209
Anhydrous residue (*c*)	269,110	260,570	41,440	223,660	29,120	30,310	16,500	24,070	55,890	37,920	1,630	1,195
Percentage composition of anhydrous residues												
Silica (SiO_2)	0.00	0.01	0.12	0.00	0.28	0.07	0.18	0.17	0.07	0.11	...	...
Calcium (Ca)	0.67	0.36	1.92	1.25	1.99	1.38	0.79	0.91	0.84	1.29	10.43	12.56
Magnesium (Mg)	0.24	0.11	2.49	0.04	0.24	0.23	0.12	0.17	0.48	0.50	3.55	4.70
Sodium (Na)	36.11	36.53	32.86	34.64	33.14	33.35	34.97	34.77	34.37	34.36	21.58	16.32
Potassium (K)	2.71	2.26	3.12	2.95	3.19	3.07	3.03	3.33	3.69	3.11	1.28	1.26
Carbonate radicle (CO_3)	0.01	0.01	0.83	0.01	0.90	0.86	0.81	1.05	0.29	0.69	4.35	5.44
Sulfate radicle (SO_4)	0.88	1.70	1.30	0.99	1.41	1.58	3.52	2.87	1.09	1.61	5.83	13.40
Chlorine (Cl)	59.35	58.99	57.33	60.09	58.82	59.43	56.55	56.70	59.14	58.30	52.62 (*a*)	45.87(*b*)

(*a*) Nitrate radicle 5.6 mg. per kilogram, or 0.33 per cent. (*b*) Nitrate radicle 5.0 mg. per kilogram, or 0.42 per cent.

(*c*) Computed on the assumption that iron, aluminum, borates, and other radicles constitute 0.03 per cent. of the anhydrous residue.

1. Composite of samples from boring No. 3 at 15.5 feet and from No. 6 at 21 and 40 feet.
2. Composite of samples from boring No. 11 at 27 and 35 feet and from No. 12 at 10, 20, and 27 feet.
3. Composite of samples from boring No. 13 at 16, 31.5, and 40 feet.
4. Composite of samples from boring No. 14 at 11 and 17 feet.
5. Water from hot salt spring under bathhouse near Silver Peak, Nev., collected June 8, 1912.
6. Water from cold salt spring at bathhouse near Silver Peak, Nev., collected June 8, 1912.
7. Water from cold salt spring at northeast end of marsh, collected June 14, 1912.
8. Water from hot salt spring at northeast end of marsh, collected June 14, 1912.
9. Water from boring No. 1 at 6 feet, collected June 1, 1912.
10. Water from boring No. 1 at 27 feet, collected June 4, 1912.
11. Water from spring at pumping station, Silver Peak, Nev., collected June 28, 1912.
12. Water from 30-foot well of Nevada-California Power Co. at Silver Peak, Nev., collected June 29, 1912.

Practically the entire surface of the playa, 32 square miles, is covered with salt that averages in depth about one-quarter of an inch. The upper muds, averaging probably 10 feet thick, contain not less than 2 per cent. of salt. It is estimated that not less than 15 square miles of the northeastern part contains a 10-foot saline bed of which at least 60 per cent. is salt. It is calculated from these moderate estimates that 15,000,000 tons of salt lie within 40 feet of the surface. The high rate of evaporation, which would permit solar concentration of brines, the absence of long-continued rainfall to interfere with operations, the nearness of a railroad, and the high degree of purity of the product as indicated by analyses of the brines are extremely favorable features in considering the possibility of utilizing these deposits. Salt is now being produced on a small scale. Brines from pits in the upper muds or from furrows filled with rain water that has become saturated are concentrated and crystallized by solar evaporation in shallow vats dug in the playa, and the salt thus obtained is bagged for sale.

COMPOSITION OF THE BRINES

Samples of the brines were tested for potash by A. R. Merz at the Coöperative Laboratory, Mackay School of Mines, Reno, Nev. Partial analyses of the brines and more nearly complete analyses of composite samples of the brines also were made by Walton Van Winkle in the Coöperative Laboratory of the United States Geological Survey at Willamette University, Salem, Oregon.

The analyses show that the brines from the main salt body are essentially solutions of sodium chloride remarkably uniform in composition and concentration. Analyses Nos. 1, 2, and 4, of waters from borings Nos. 3, 6, 11, 12, and 14, indicate that the brines contain less than 2 per cent. of sulfate and 0.02 per cent. of carbonate. Only traces of borate were found. These brines on evaporation would yield a mass containing about 90 per cent. of sodium chloride and the character of the other ingredients makes it certain that a much purer product could be obtained by one crystallization. The differences in the amounts and proportions of the alkaline earths are especially noteworthy, as they indicate progressive steps in the concentration and deposition of those substances.

The content of potassium is too low to be of commercial importance. The anhydrous residues of the brines represented by analyses Nos. 1, 2, and 4 average 2.64 per cent. in content of potassium (K). The average potassium content of the same brines according to Merz is 2.23 per cent. of the saline residue dried at 105° C. This apparent discrepancy in estimates is, however, caused mostly by difference in unit of expression, the actual determinations of the radicle being equivalent respectively to 0.79 and 0.69 gram of potassium (K) per 100 cc. of brine.

SUMMARY

Silver Peak Marsh is a salt playa containing a high grade of sodium chloride. No extensive deposits of potash-bearing salts were found. To a depth of 50 feet the formations are chiefly salt clays and muds with layers of crystallized salt covered irregularly by gypsum-bearing clays. It is estimated that 15,000,000 tons of salt lie within 40 feet of the surface of the playa.

UNITED STATES GEOLOGICAL SURVEY
WASHINGTON, D. C.

COMPOSITION OF THE WATER OF CRATER LAKE, OREGON[1]

By WALTON VAN WINKLE AND N. M. FINKBINER

Received November 14, 1912

Crater Lake, in the heart of the Cascade Mountains of Oregon, is one of the most interesting spots in the United States, both to scientist and to tourist. Popular accounts of its beauties have been published in magazines and its chief features of scientific interest have been admirably set forth.[1] But though analyses of typical rocks are given in Diller and Patton's paper, no analysis of the water of the lake has yet appeared in print, and the subjoined analysis may, therefore, be useful to those interested in this remarkable body of water.

The following brief description of the lake, based on Diller's report and on personal observations by the senior author, makes the analyses more readily understandable. Crater Lake is situated in a geologically recent caldera occupying the site of a once lofty volcanic peak—Mount Mazama— in the midst of the Cascade Range, about 55 miles northeast of Medford, Oregon. The rim of the caldera is from 7,000 to 8,000 feet above the sea, and the lake surface was 6,175 feet, above sea level in 1908. The inner slope of the rim bears, in some places, a sparse growth of pine, but in many others its walls drop sheer to the water's edge, here and there fringed by steep talus slopes. The surficial area of the lake is approximately 21 square miles, and its drainage basin is only about 6 square miles larger. The greatest depth of water is 1,996 feet, and a cinder cone projects more than 760 feet above the surface at the western extremity of the lake to form Wizard Island. Precipitation is more than 70 inches a year, occurring chiefly as snow in winter. Evaporation is less than 55 inches, and this, with loss by percolation, almost completely balances the inflow, there being no surface outlet to the lake. Some of the water may find its way by percolation into Rogue River, but more of it probably goes southeastward appearing as springs in the drainage basin of Klamath Lake.

ANALYSES OF WATER OF CRATER LAKE AND WOOD AND ROGUE RIVERS, OREGON

	Milligrams per liter			Percentage of anhydrous residue			
	Crater Lake[1]	Wood River[2]	Rogue River[3]	Crater Lake	Wood River	Rogue River	
Total dissolved solids (heated to 180° C.)....	80	81	71	...	...	...	...
SiO_2............	18	37	26	22.36	49.82	38.86	SiO_2
Fe............	0.02	0.20	0.01	0.02	0.27	0.02	Fe
Ca............	7.1	5.7	5.7	8.82	7.68	8.51	Ca
Mg............	2.8	2.0	2.6	3.48	2.69	3.89	Mg
Na............	11	7.2	7.2	13.67	9.69	10.76	Na
K............	2.2			2.73			K
CO_3............	0.0	0.0	0.0	21.11	18.85	29.89	CO_3
HCO_3..........	34	28	40	*a*	*a*	*a*	HCO_3
SO_4............	11	7.6	3.8	13.67	10.23	5.68	SO_4
Cl............	11	0.6	1.5	13.66	0.67	2.24	Cl
NO_3............	0.38	0.06	0.10	0.47	0.10	0.15	NO_3
PO_4............	0.01	*b*	*b*	0.01	...	...	PO_4

[1] Collected August 27, 1912, by M. Mecklem about 1 mile from shore at depth of 6 ft. Analysis by N. M. Finkbiner.

[2] Collected August 26, 1912, by Walton Van Winkle at bridge near Fort Klamath, Oregon. Analysis by Walton Van Winkle and N. M. Finkbiner.

[3] Composite of daily samples Aug. 6–15, 1912, inc., collected at power house, near Tolo, Ore. Analysis by Walton Van Winkle and N. M. Finkbiner.

a HCO_3 computed to CO_3.

b Not determined.

As loss by percolation is only about one-third of the loss by evaporation, analyses of the water of the lake may be expected to give chemical evidence of slow

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[1] Published by permission of the Director, U. S. Geological Survey.

[1] J. S. Diller and H. B. Patton, "The Geology and Petrography of Crater Lake National Park," *Prof. Paper U. S. Geol. Survey*, 3 (1902); J. S. Diller, "Geological History of Crater Lake," Department of the Interior, 1912.

concentration. That the analysis really shows concentration almost identical with that of other surface waters of the region is explained, however, by the fact that no sedimentary materials are exposed, the andesites, dacites, and basalts forming the basin of the lake, being nearly insoluble in the cold water, and, therefore, incapable of rapidly increasing its content of mineral matter. Concentration of chlorides is great as compared with that of other materials, an indication of the concentrated character of the water. As the published analyses of rocks indicate that almost no chloride exists in these formations, it is possible that the high percentage of that radicle in the water is due almost entirely to accumulated "cyclic" chlorine precipitated with the rain and snow. The unexpectedly high percentage of sulfates is possibly caused by solution of sulfur that remained in the bottom of the caldera in a more or less oxidized condition at the cessation of active volcanism. No other features of the analysis seem unusual, when it is compared with the accompanying analysis of waters collected from Wood and Rogue rivers in the same season.

U. S. GEOLOGICAL SURVEY
SALEM, OREGON

ACTION OF FERMENTING MANURE ON REËNFORCING PHOSPHATES[1]

By W. E. TOTTINGHAM AND C. HOFFMAN

Received October 21, 1912

INTRODUCTION

For many years phosphorus[2] has held a position of importance in the established practice of applying fertilizers to the soil. Indeed, this particular element may be the limiting factor of fertility in some normal types of soil[3] or it may become such by depletion in excessively cropped soils.[4]

The chief raw materials supplying phosphorus for the fertilizer trade are bones and rock-phosphate. In finely divided condition these materials are known as bone meal and ground rock-phosphate or floats, respectively. They appear to consist essentially of basic phosphates of calcium and supply phosphorus in forms comparatively insoluble in water and in weak saline solutions.

The fertilizer industry furnishes more soluble forms of phosphorus derived from the crude phosphates by treating them with strong sulfuric acid. These products are monocalcium phosphate, soluble in water, and di-calcium phosphate, slowly acted upon by water and attacked by warm, neutral solution of ammonium citrate, which dissolves a considerable part of the phosphorus. Commercial acid phosphate is a mixture of these products with calcium sulfate, also formed in the reaction. The term "available phosphoric acid" as used in the fertilizer trade designates the combined amounts of phosphorus (expressed as P_2O_5) obtained in water extract and citrate extract successively from the same sample. It may include other forms than the mono- and dicalcium phosphates.

The increased cost of phosphorus in acid-phosphate as compared with the crude materials has been rather generally supposed to be justified by superior availability to plants, consequent upon its greater solubility. It is true that acid-phosphate has generally given results in the year of application superior to those from rock-phosphate. Yet it is generally recognized that the phosphorus supplied in this form is rapidly fixed in insoluble form by reaction with calcium carbonate and other soil minerals[1] or reverted in the dilute soil solution to more basic and less soluble phosphates of calcium.[2] The efficiency of acid phosphate is to be attributed, therefore, probably to the facility with which it permits phosphorus to be distributed through the soil in finely divided form, rather than to a permanent state of solubility. This important effect of fineness and thorough distribution in promoting availability has been recognized with reference to rock-phosphate and experiment stations have advised the farmer to purchase it under guarantee of fineness as well as of phosphorus content.

Considerable evidence has accumulated[3] which indicates advantage from the use of finely ground rock-phosphate directly as a fertilizer, especially when it is applied in conjunction with a liberal supply of organic matter turned into the soil. Thorne[4] and others have demonstrated considerable efficiency of rock-phosphate when applied to the soil as a mixture with farm manure, and this method of application for both rock-phosphate and acid-phosphate has been widely adopted in practice. The effect of decomposing organic matter upon rock-phosphate under these conditions, as observed in increased yield of crop, has been commonly attributed to organic acids. It has been stated that such acids produced in the fermentation of the organic matter liberate soluble phosphorus from the floats. Favorable results from this treatment have been observed to be generally most pronounced in seasons succeeding the year of application.

Experiments in which solvents commonly used for this purpose have been employed to determine any increase of available phosphorus in fermented mixtures of crude phosphates and organic matter have given, however, negative results. Fleischer[5] analyzed water extracts from fermented mixtures of moss turf and ground phosphorite in varying proportions and found no appreciable increase of soluble phosphorus during periods of 4$^1/_2$ to 9 months. In many cases the results showed decreases of water-soluble phosphorus. Pfeiffer and Thurman[6] found that a crude phosphate

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

[2] In the fertilizer industry and in agricultural practice phosphorus is commonly dealt with in terms of phosphoric acid anhydride, P_2O_5. The term "phosphoric acid" used in this paper signifies P_2O_5.

[3] Hopkins and Pettit, Ill. Expt. Sta., *Bull.* **123,** p. 239.

[4] Whitson and Stoddart, Wis. Expt. Sta., *Research Bull.* **2,** p. 45.

[1] See Farsky, Abstr. in *Bied. Centrlb.*, **1883,** S. 449, and Schreiner and Failyer, Bur. Soils, U. S. Dept. Agr., *Bull.* **32.**

[2] See Cameron and Bell, Bur. of Soils, U. S. Dept. Agr., *Bull.* **41,** pp. 21 and 35; also Cameron and Seidell, *J. Am. Chem. Soc.*, **26,** 1461.

[3] Some of the most extensive data have been obtained by Merrill (Maine Expt. Sta., *Rpt.* **1898,** p. 64), Hess (Penn. Expt. Sta., *Rpt.* **1895,** p. 157), Goessmann (Hatch Expt. Sta., *Rpt.* **1897,** p. 190), Jordan (Maine Expt. Sta., *Rpt.* **1894,** p. 23), Wheeler (R. I. Expt. Sta., *Bull.* **118**) and Patterson (Md. Expt. Sta., *Bull.* **114**).

[4] Ohio Expt. Sta., *Bull.* **183,** p. 206; *Circ.* **104,** p. 15.

[5] *Landw. Jahrb.*, Bd. **12,** S. 186.

[6] *Landw. Vers. Stat.*, Bd. **47,** S. 353.

after contact for three months with a fermenting mixture of peat meal and liquid manure yielded only a trace of water-soluble and a slight increase of citrate-soluble phosphorus. Lupton[1] fermented cotton-seed and cotton-seed-meal separately with ground South Carolina phosphate rock. Frequent analyses, during a period of three months, gave variable results and indicated little, if any, increase in the availability of phosphorus. McDowell[2] analyzed a mixture of cow and horse manure and crude Tennessee rock-phosphate which had fermented slightly over a year but found no increase of either water-soluble or citrate-soluble phosphorus as a result of fermentation. Egorov,[3] on the other hand, observed large decreases in the solubility of the phosphorus in fermenting mixtures of manure and rock-phosphate.

EXPERIMENTS WITH ROCK-PHOSPHATE AND MANURE

The work to be described in this paper included studies of the action of fermenting manure on rock-phosphate or floats conducted essentially after the method of McDowell.[4] The fresh manure free from litter, which was a mixture of one-third horse manure and two-thirds cow manure unless specified otherwise, was thoroughly hand-mixed with floats in the proportion of one pound of floats to fifty pounds of manure.[5] The samples were fermented in covered iron pails; and water was added occasionally in the earlier experiments to maintain a fairly even moisture content of the check and reënforced manures. Determinations were made as follows:

Moisture.—One hundred grams of fresh sample were dried to constant weight in a steam oven at 100° C.

Water-soluble Phosphoric Acid.—One hundred grams of fresh sample were extracted for five hours in a shaking machine at room temperature. The extract was then filtered and washed as rapidly as possible on a thick layer of paper pulp in a Büchner funnel, using ten successive 75 cc. portions of distilled water for the washings. Extract and washings were made to 2 liters and phosphoric acid determined in 200 cc. aliquots by the Neumann method.[6]

Citrate-soluble Phosphoric Acid.—The residue from the water extraction together with the filter pulp, sucked as free from water as possible, was transferred to a 1 liter Erlenmeyer flask with 800 cc. of neutral ammonium citrate solution[7] at 65° C. Extraction with this solvent was continued for one-half hour at 65° C., shaking at five-minute intervals. The extract was filtered as before and washed to a volume of 1500 cc. with water at 65° C. The solution was then made to 2 liters and phosphoric acid determined in 100 cc. aliquots by Neumann's method. The citrate-soluble added to the water-soluble gives the available phosphoric acid.

N/5 Nitric-Acid-soluble Phosphoric Acid.—The residue (with filter) from the citrate extraction was extracted for five hours with $N/5$ HNO_3 at room temperature (about 27° C. in all cases) in a shaking machine. The extract was filtered on paper pulp in the manner already described, washed with cold water to a volume of 1750 cc., and made to 2 liters. Aliquots of 200 cc. were analyzed for P_2O_5 by the Neumann method.

Total Phosphoric Acid.—Phosphoric acid was determined on the ground and mixed residue from the moisture determination, using the Neumann method.

In a preliminary experiment in which only cow manure was used the check manure showed a loss of 34.7 per cent. of the water-soluble phosphorus originally present and no loss of available phosphorus after 61 days of fermentation. In the floats-manure mixture the water-soluble phosphorus decreased by 65.4 per cent. of the original and the available phosphorus decreased by 39.6 per cent. of that originally available. After 96 days the check manure had lost 25.5 per cent. of the originally water-soluble phosphorus and 5.4 per cent. of the phosphorus originally available. During this time the mixture lost 63.6 per cent. of the water-soluble phosphorus and 25.8 per cent. of the available phosphorus, based on the respective original amounts of these constituents. There was, therefore, some recovery of water-soluble phosphorus in the check and of available phosphorus in the mixture during the later stages of fermentation. The losses of phosphorus appeared to warrant further investigation.

On August 17, 1909, a mixture of cow and horse manures was prepared and two 25-pound portions were fermented in the manner already described, one portion serving as a check and the other being mixed with one-half pound of rock-phosphate. A temperature of about 25° C. was maintained throughout the experiment. Liberation of ammonia was little evident after October 11th and by January 5th fermentation was progressing slowly. On the latter date sufficient water was added to the mixture to give it a percentage of moisture approximately equal to that of the check. Extractions and analyses were made by the methods already described and with the results given in Table I. By weighing the pails at samplings data were secured for calculating the losses of dry matter, due account being taken of rock-phosphate removed in sampling. In the columns succeeding the data for water-soluble, available and total-soluble phosphoric acid are given their respective values in percentages of the total P_2O_5.

The data of Table I show that the water-soluble phosphorus decreased during fermentation In the check manure the loss for the 92-day period amounted to 32.0 per cent. of the total and 36.9 per cent. of that originally soluble. The mixture suffered corresponding losses of 15.1 per cent. and 44.0 per cent., respectively. Citrate-soluble phosphorus increased in the check manure and remained practically constant in the mixture. Consequent decrease of available phosphorus in the check sample was 19.4 per cent. of the total and of the originally available phosphorus. The available phosphorus of the mixture decreased

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[1] Ala. Expt. Sta., *Bull.* **48.**

[2] Penn. Expt. Sta., *Rpt.* **1907–1908,** p. 177.

[3] Abstr. in *Expt. Sta. Record,* **26,** 2, 123.

[4] *Loc. cit.*

[5] The floats were furnished by the Michigan Carbon Works of Detroit and contained 27.58 per cent. total and 1.2 per cent. available P_2O_5.

[6] Throughout this investigation phosphorus was finally weighed as $Mg_2P_2O_7$ according to the standard gravimetric method.

[7] Bur. Chem., U. S. Dept. Agr., *Bull.* **107** (revised), p. 1.

by 13.7 per cent. of the total phosphorus and 25.9 per cent. of the phosphorus originally available. The losses in the respective samples and the proportional losses of check and mixture show general agreement with the results of the nearly equal period covered by the preliminary experiment. Added extraction with *N*/5 nitric acid recovered all but 14.0 per cent. of the total phosphorus in both samples after 92 days of fermentation. This extracting agent, which has been used for determining tricalcium phosphate in soils,[1] was used in the hope to detect marked changes of phosphorus other than simple reversion to tricalcium phosphate.

For the entire period of fermentation covering 190 days, the check sample lost water-soluble phosphorus equal to 21.1 per cent. of the total and 24.34 per cent. of that originally soluble. This was a recovery of solubility over that observed at 92 days and conforms with the results of the previous experiment. With the mixture there was a loss of 19.6 per cent. of the total or 57.14 per cent. of the originally water-soluble phosphorus. The available phosphorus in the check at the close of the experiment was less than at the beginning by an amount equal to 13.4 per cent. of the total and of the available phosphorus originally present. In the mixture the corresponding losses were 25.7 per cent. of the total and 48.7 per cent. of the available phosphorus.

TABLE I.—PERCENTAGES OF PHOSPHORIC ACID IN THE DRY MATTER OF CHECK MANURE AND MANURE-FLOATS MIXTURE

Sample and date of analysis	Period of fermentation, days	Dry matter in manures, Per cent.	Loss of dry matter, Per cent.	Total P_2O_5, Per cent.	Water-soluble P_2O_5		Citrate-soluble P_2O_5, Per cent.	Available P_2O_5		*N*/5 nitric acid-soluble P_2O_5, Per cent.	Total soluble P_2O_5	
					Per cent. of manure	Per cent. of total P_2O_5		Per cent. of manure	Per cent. of total P_2O_5		Per cent. of manure	Per cent. of total P_2O_5
Check												
Aug. 19..........	...	22.18	...	1.80	1.56	86.7	0.29	1.85	102.8	0.02	1.87	103.9
Nov. 19..........	92	17.60	31.20	2.54	1.39	54.7	0.73	2.12	83.4	0.08	2.20	86.6
Feb. 25..........	190	17.07	50.32	2.99	1.96	65.6	0.71	2.67	89.4	0.13	2.80	93.7
Mixture												
Aug. 19..........	...	23.65	...	4.70	1.61	34.3	0.87	2.48	52.8	1.78	4.26	90.7
Nov. 19..........	92	20.81	31.14	6.40	1.23	19.2	1.27	2.50	39.1	3.00	5.50	86.0
Feb. 25..........	190	21.30	50.97	7.67	1.13	14.7	0.95	2.08	27.1	3.67	5.75	74.7

The data agree with those of the previous experiment, indicating pronounced suppression of water-soluble and of available phosphorus as measured by the standard reagents used for estimating these constituents of manures. At the close of the experiment additional extraction with *N*/5 nitric acid recovered all but 6.3 per cent. and 25.3 of the total phosphorus in check and mixture, respectively. In the latter sample, at least, it appears that some change other than simple reversion to tricalcium phosphate occurred. Possible formation of organic compounds, especially of nuclein compounds by the action of bacteria, appears worthy of consideration as an explanation of the change.

Having found the phosphorus of fermented manure-floats mixtures depressed in availability as measured by solvents in common use it appeared that the natural solvent formed by the moisture of the fermenting manure might be a more efficient extractive agent. This solvent must contain considerable dissolved material, chief among which would be presumably ammonium carbonate. Having found the ammonia content of a rapidly fermenting sample of horse manure equivalent to a 0.07 per cent. solution of ammonium carbonate in the moisture of the fresh manure, extractions were made with this solvent at the two later extractions of the last experiment. Separate 100 gram portions of the sample were treated with 1 liter portions of the solvent, the work being carried out as already described for water extracts. On November 19th the water extract of the check sample contained 54.7 per cent. of the total phosphorus, while the ammonium carbonate extracted 59.4 per cent. of the total. These solvents extracted 19.2 per cent. and 20.1 per cent., respectively of the total phosphorus in the mixture. On February 25th, water and ammonium carbonate extracted 65.6 per cent. and 45.7 per cent., respectively of the total phosphorus in the check. In the case of the mixture, these solvents extracted 14.7 per cent. and 10.4 per cent., respectively, of the total phosphorus. The results do not indicate that ammonium carbonate, as a product of fermentation, is an especially effective factor in rendering phosphorus available in fermenting manure.

Carbon dioxide formed by the decay of crop residues and dissolved in the soil water has been recognized as an efficient solvent for soil phosphates since the time of Liebig. Many laboratory tests made to determine its solvent power for various phosphates also have demonstrated its efficiency. In the present investigation a solution of CO_2 saturated at room temperature proved about five times as efficient as water, or 0.07 per cent. ammonium carbonate solution in dissolving phosphorus from the rock phosphate. This extracting agent was applied also to the samples represented in Table I, after they had fermented 318 days. The results are given in Table II.

TABLE II.—PERCENTAGES OF PHOSPHORIC ACID IN DRY MATTER OF MANURE AND MANURE-FLOATS MIXTURE AFTER 318 DAYS OF FERMENTATION

Manure sample	Dry matter of manure Per cent.	Total P_2O_5 Per cent.	Water-soluble P_2O_5		P_2O_5 soluble in sat. solution of CO_2	
			Per cent. of manure	Per cent. of total P_2O_5	Per cent. of manure	Per cent. of total P_2O_5
Check..........	42.70	3.08	0.60	19.48	0.87	28.25
Mixture........	81.85	6.53	0.22	3.37	0.33	5.05

The data differ notably from those for the extraction after 190 days of fermentation (Table I, date of February 25th), since water recovered less than one-third of the phosphorus soluble in the check

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[1] Stoddart, Wis. Expt. Sta., *Research Bull.* 2, 59.

manure and only about one-fourth of that soluble in the mixture at the earlier date.

This seems to indicate a marked conversion of phosphorus to forms less soluble in water, the effect being especially marked where floats were present. Carbon dioxide was decidedly superior to water for it recovered about 50 per cent. more phosphorus from both samples than did the latter solvent. The results of this extraction are not comparable with those already reported for ammonium carbonate because of the great difference in condition of the manures. They present evidence of greatly depressed solubility of phosphorus in water at an extreme stage of fermentation. The superiority of the carbon dioxide solutions appears to indicate that some of the phosphorus previously involved in the process of fermentation and rendered insoluble in water, had been liberated finally as inorganic phosphates insoluble in water but soluble in carbon dioxide solution. The results would seem to indicate, therefore, that carbon dioxide may be the chief agent involved in finally placing insoluble phosphorus of manures at the disposal of growing crops.

The work already presented suggested two points upon which it would be important to direct further investigation:

Table III.—Percentages of Phosphoric Acid in the Dry Matter of Normal and Treated Manure-Floats Mixtures

Sample and date of analysis	Period of fermentation, days	Dry matter in manure, Per cent.	Loss of dry matter, Per cent.	Bacteriological count on gelatin,media,millions per gram dry manure	Total P_2O_5, Per cent.	Water-soluble P_2O_5		Citrate soluble P_2O_5, Per cent.	Available P_2O_5		$N/5$ nitric acid soluble P_2O_5, Per cent.	Total soluble P_2O_5		H_2CO_3 soluble P_2O_5	
						Per cent. in manure	Per cent. of total		Per cent. in manure	Per cent. of total		Per cent. in manure	Per cent. of total	Per cent. in manure	Per cent. of total
1. Normal															
Apr. 17	..	21.57	...	2190	4.21	1.40	33.26	0.63	2.03	48.21	..	..	..	1.26	29.89
May 12	25	19.12	14.90	218	4.71	0.98	20.81	0.88	1.86	39.55	..	..	..	1.40	29.77
July 6	80	16.95	38.81	4	5.69	1.00	17.57	1.11	2.11	37.08	3.27	5.38	94.62	1.08	18.98
2. With chloroform															
Apr. 17	..	21.72	...	344	4.21	1.37	32.54	a	..	...	..	..	..	1.25	29.66
May 12	25	22.05	...	1	3.91	1.32	33.76	..	..	...	..	..	..	1.47	37.57
July 6	80	21.82	1.13	0.1	4.13	1.16	28.11	0.48	1.64	39.73	2.15	3.79	91.77	1.05	25.42
3. With formalin															
Apr. 17	..	21.70	...	0	4.21	1.24	29.45	0.69	1.93	45.84	..	..	..	1.05	24.94
May 12	25	21.72	...	0.1	3.90	1.23	31.54	0.47	1.70	43.59	..	..	..	1.46	37.44
July 6	80	20.57	1.61	0	3.91	1.05	26.85	0.70	1.75	44.75	2.18	3.93	100.40	0.88	22.51

a Citrate extracts from chloroformed sample were lost at first and second extractions.

First, the cause of the decrease of water-soluble phosphorus which was observed with manures and manure-floats mixtures.

Second, the efficiency of carbonated water as a solvent for phosphorus of actively fermenting manure.

Behrens[1] states that microörganisms of manure cleave phosphoric acid and other ash constituents from organic compounds and use them in the elaboration of their own cells. Stoklasa[2] has shown also that approximately 25 per cent. of the phosphorus of insoluble phosphates added to media may be assimilated by bacterial cultures in the presence of carbohydrates. The latter author states that as much as 80 per cent. of the phosphorus of bacteria may occur in the form of nucleic acid. If these statements are reliable, the consumption of soluble phosphorus by bacteria might explain the depressions observed in the present work. This possibility is apparently strengthened by the work of Wüthrich and von Freudenreich,[1] who found great numbers of bacteria in the fresh feces of herbivora; and by the work of MacNeal and others,[2] who found that the bacterial cells of fresh human feces might amount to 50 per cent. or more of the total dry matter. It must be borne in mind also that bacterial counts upon fermenting manure by the usual plating method, while indicating a rapid increase of flora, do not measure the extensive accumulation of dead or inert bacterial cells which must occur.

In view of the preceding observations an experiment was planned in which the action of bacteria could be eliminated and the results compared with those of a normal fermentation. The usual extractions were supplemented by the use of a saturated solution of carbon dioxide (at room temperature) as a solvent to determine its efficiency with actively fermenting manure.

EXPERIMENT WITH ANTISEPTICS

On April 15, 1911, three 25-pound samples of the usual mixture of manures were prepared and mixed with floats[3] in the proportion and manner previously followed. One sample was allowed to ferment normally. To a second sample, 250 cc. of chloroform were added and thoroughly incorporated. The third sample was treated with a similar amount of 40 per cent. formalin. Care was taken to maintain an excess of these antiseptics in their respective samples, the purpose being to inhibit only multiplication of bacteria in the second sample while in the third sample the suppression of bacteria and probably of enzymes should be complete. The samples were stored in pails and weighed at samplings to follow the losses of dry matter. Bacterial counts were made as a check upon conditions favoring fermentation. $N/5$ nitric acid was used only at the last extraction. The samples for extraction with carbon dioxide were taken simultaneously with those for extraction with water, transferred to sealed Mason jars, treated with a few cc. of chloroform, refrigerated and extracted on the following day. The data of this experiment are given in Table III.

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[1] *Lafar's Handbuch d. Tech. Mykol.*, **3**, 429.

[2] *Centrlb. Bakt.*, etc., Abt. II, **29**, 400.

[1] *Centrlb. Bakt.* **(1895)**, Abt. II, **1**, 873.

[2] *Jour. Infect. Diseases*, **6**, No. 2, p. 123.

[3] The sample of floats used here contained 31.62 per cent. total P_2O_5.

A study of Table III shows that the water-soluble phosphorus of the normally fermenting mixture decreased during 80 days by 15.6 per cent. of the total and 47.2 per cent. of the amount soluble at the beginning. The corresponding losses in mixtures 2 and 3 were 4.5 per cent. and 2.6 per cent. of the total, and 13.8 per cent. and 8.8 per cent. of the originally soluble phosphorus of the respective samples. The data indicate that this readily soluble constituent of manure-floats mixtures is seriously reduced by the process of fermentation but remains practically constant when fermentation is inhibited. Samples Nos. 1 and 3 show losses of 23.1 per cent. and 2.4 per cent. of the phosphorus originally available in the respective cases. It would be difficult to explain why the single figure for availability in sample No. 2 fails to agree with the corresponding figure for sample No. 3, also treated with an antiseptic, but even the latter sample shows a decrease at the second analysis. The bacterial counts and losses of dry matter seem to indicate that this condition cannot be due to reactions accompanying the development of bacteria.

In sample No. 3 the water-soluble phosphorus was lower throughout than in sample No. 1 at the first stage and in sample No. 2 at corresponding dates of analysis. This condition may have been due to a coagulating or hardening action of the formalin upon the organic matter surrounding the otherwise soluble phosphorus compounds, which reduced the effectiveness of the solvent. $N/5$ nitric acid was more efficient than in earlier experiments, the added extraction with this solvent recovering all the phosphorus of sample No. 3 and all but 5.3 per cent. and 8.2 per cent. of the total phosphorus in samples Nos. 1 and 2, respectively, at the close of the experiment.

Comparing the efficiency of carbon dioxide solution with that of water, one finds the results less consistent for the former than for the latter solvent. The irregularities may have been due to changes occurring in the samples while standing previous to extracting. The fact that the efficiency was generally lower for the carbon dioxide than for the water seems to indicate that with these samples conditions were quite different from those obtaining where the solvents were compared upon floats alone or on manure-floats mixture at the extreme stage of fermentation represented in Table II. In harmony with the results from extraction with water, the data for carbon dioxide extracts give evidence of marked suppression of soluble phosphorus where fermentation and bacterial increase proceed freely, as in sample No. 1. These results agree with those of Egorov,[1] who found greater decreases of soluble phosphorus in normally fermenting manure than in manure treated with toluol. Considering the results of this experiment in relation to the two specific points which it was to cover, it appears that the development of bacteria is closely associated with a decrease of water-soluble phosphorus and that the phosphorus of fermenting manure-floats mixtures is not appreciably more soluble in carbon dioxide solution than in water alone.

EXPERIMENTS WITH BACTERIA

As a result of the preceding experiment attention was directed to the relation of bacteria to soluble phosphorus in culture media. Mixed flora of manure organisms were grown upon the usual standard agar medium (generally with 0.5 per cent. K_2HPO_4 added). After 2 to 5 days the growths were removed by means of a smooth-edged glass slide, oven-dried and pulverized. These bacterial cells contained 4 per cent. to 8 per cent. of P_2O_5. A water-extract of bacterial cells from one culture of this sort, obtained in the presence of chloroform and passed through a Pasteur-Chamberland porcelain filter, contained 60.9 per cent. of the total phosphorus of the cells. A similar analysis of the medium from which these cells were obtained showed that it contained 81.9 per cent. of its total phosphorus in water-soluble form. This indicated a depression of 21 per cent. in solubility of phosphorus when assimilated by bacteria from the medium.

Mixed bacteria from manure were grown upon agar media prepared from an extract of fresh manure-floats mixture obtained by squeezing the latter in a heavy press. The phosphorus supply was thus limited to the forms dealt with in the preceding fermentation experiments. Check portions of the media and five-day cultures representing equal amounts of the original media were extracted with water in the presence of chloroform and the extracts were filtered through porcelain. The treatment was thus essentially the same as that applied in the experiments with manures. Two experiments of this sort gave depressions of 40.6 per cent. and 26.5 per cent. of the soluble phosphorus of the media as a result of the growth of bacteria. Since the conditions attending these extractions closely simulated those attending the investigation of manure, it seems reasonable to conclude that bacteria are directly responsible for at least a part of the decrease of water-soluble phosphorus in fermenting manures.

STATUS OF PHOSPHORUS IN THE BACTERIAL CELL

In order to determine whether this effect was due to changes occurring in the medium or to withdrawal of soluble phosphorus from the medium by the bacteria, a study was made of the status of phosphorus in bacterial cells. A few pure cultures but mostly mixed organisms of manure were investigated. The growths were secured from standard media in the manner previously described and incubated 3 days. Water extracts were obtained from fresh intact cells, oven-dried cells and cells dried *in vacuo* over sulfuric acid at room temperature. Other samples of fresh cells were extracted after maceration with glass in a porcelain mortar until microscopic examination showed that practically all the cells had been crushed. This treatment must have eliminated, to a great extent, any protective action exerted against solvents by the cell wall.

Ten grams of fresh cells, or its equivalent in dry cells, were shaken by machine for three hours with 500 cc. of water at room temperature. The extracts were filtered immediately through porcelain. Care

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[1] *Loc. cit.*

was taken to have chloroform present throughout the work and the filtrates were refrigerated when not analyzed immediately. Phosphorus was determined in 100 cc. aliquots of the filtrate taken at the initial temperature of the solvent, using the Neumann method. It was found that an average of 48.9 per cent. of the total phosphorus in the fresh bacterial cells was soluble in water. Drying did not appear to exert a marked influence on the solubility of phosphorus. In one case water was found to be as efficient when acting upon the fresh, intact cells for 10 minutes as when applied for three hours. This seemed to indicate a possible concentration of soluble phosphorus at the surface of the cell as a result of adsorption from the medium. In view of this possibility determinations of inorganic phosphorus were made on water extracts of several cultures, using the following methods: Hart and Andrews',[1] Forbes',[2] Emmet and Grindley's[3] and Collisons'.[4]

On the whole, there was considerable variation in the results and it must be concluded that they were but rough approximations to the actual percentages of inorganic phosphorus. The average of the most consistent figures gave 31.5 per cent. of the total phosphorus as present in inorganic form. The average of water-soluble phosphorus recovered from these cultures was 37 per cent. of the total. From the rough agreement of these percentages it would seem that the greater part of the water-soluble phosphorus of bacterial cells as prepared in this work was derived from inorganic phosphates adsorbed from the medium and concentrated at the surface of the cells. The remaining phosphorus of the cells, which was partly soluble in dilute alkali, probably represented that actually assimilated by the bacteria and presumably existed chiefly as a constituent of nuclein compounds. This condition would be sufficient to account for an appreciable depressing influence of bacteria upon the soluble phosphorus of fermenting manures.

TABLE IV.—PERCENTAGES OF PHOSPHORIC ACID IN THE DRY MATTER OF CHECK MANURE AND ACID-PHOSPHATE MIXTURES

Sample number and date of analysis	Dry matter in manure, Per cent.	Loss of dry matter, Per cent.	Bacteriological count on gelatin media, millions per gram dry manure	Total P_2O_5, Per cent.	Water-soluble P_2O_5		Citrate-soluble P_2O_5, Per cent.	Available P_2O_5		$N/5$ nitric acid-soluble P_2O_5, Per cent.	Total soluble P_2O_5		Soluble in filtrate from porcelain	
					Per cent. in sample	Per cent. of total		Per cent. in sample	Per cent. of total		Per cent. in sample	Per cent. of total	Per cent. in sample	Per cent. of total
1. Check manure														
Apr. 6	21.70	..	6308	1.93	1.22	63.21	0.53	1.75	90.67	0.05	1.80	93.26	1.12	58.03
July 2 (a)	14.12	40.3	27	2.62	1.43	54.58	1.18	2.61	99.62	0.07	2.68	102.29	1.30	49.62
2. Acid-phosphate mixture														
Apr. 6	23.80	..	5380	3.70	2.03	54.87	1.07	3.10	83.77	0.07	3.17	85.66	1.77	47.80
July 2	18.10	32.3	60	4.43	1.47	33.18	2.89	4.36	98.42	0.08	4.44	100.23	1.22	27.54
3. Acid-phosphate mixture + $CHCl_3$														
Apr. 6	23.85	..	6740	3.62	2.10	58.01	1.09	3.19	88.11	0.04	3.23	89.21	1.79	49.40
July 2	23.50	0.19	0	3.71	1.26	33.94	2.00	3.26	87.85	0.12	3.38	91.08	1.14	30.73
4. Acid-phosphate mixture + $CaCO_3$														
Apr. 6	24.05	..	9145	3.42	1.30	38.01	1.91	3.21	93.86	0.10	3.31	96.78	1.11	32.50
July 2	16.47	44.5	34	4.82	0.95	19.71	4.02	4.97	103.11	0.14	5.11	106.01	0.91	18.88

(a) Period of fermentation = 87 days.

EXPERIMENT WITH ACID-PHOSPHATE

In view of the common use of acid-phosphate in the same manner as rock-phosphates for reënforcing farm manure, it seemed desirable to follow the changes of solubility of phosphorus in fermenting mixtures of manure and acid-phosphate and compare the results with those obtained from manure-floats mixtures. On April 6, 1912, a mixture of $^1/_3$ horse manure and $^2/_3$ cow manure was put up as follows:

Sample 1. 25 pounds of manure.

Sample 2. 25 pounds of manure + 390 grams acid-phosphate.[1]

Sample 3. 25 pounds of manure + 390 grams acid-phosphate + chloroform to saturation.

Sample 4. 25 pounds of manure + 390 grams acid-phosphate + 50 grams $CaCO_3$.[2]

Phosphorus was added in this experiment in a proportion roughly approximating that followed in the rock-phosphate experiments. On the dates indicated in Table IV, the samples were extracted and data obtained in the usual manner. An additional extract was prepared by passing a portion of the usual water extract through Pasteur-Chamberland filters to insure the removal of bacterial cells. Sample No. 3 was acid in reaction throughout the experiment, while sample No. 2 changed from weakly alkaline to weakly acid. The other samples were distinctly alkaline throughout. Mold growth was especially evident on the surface of sample No. 2 and in the bacterial cultures obtained from this manure. The analytical data are given in Table IV.

The losses of water-soluble phosphorus in this experiment were 8.63 per cent., 21.69 per cent., 24.07 per cent. and 18.30 per cent. of the total phosphorus of samples Nos. 1 to 4, respectively. These values are equivalent to losses of 13.65 per cent., 39.53 per cent., 41.49 per cent. and 48.15 per cent. of the originally water-soluble phosphorus of the respective samples. The phosphorus passing through porcelain filters showed decreases of 14.49 per cent., 43.40 per cent., 37.79 per cent. and 41.91 per cent. from that soluble at the start in the same respective samples. These latter results indicate the presence in the water ex-

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[1] *Am. Chem. Jour.*, **36**, 478.

[2] Ohio Expt. Sta., *Bull.* **215**, 488.

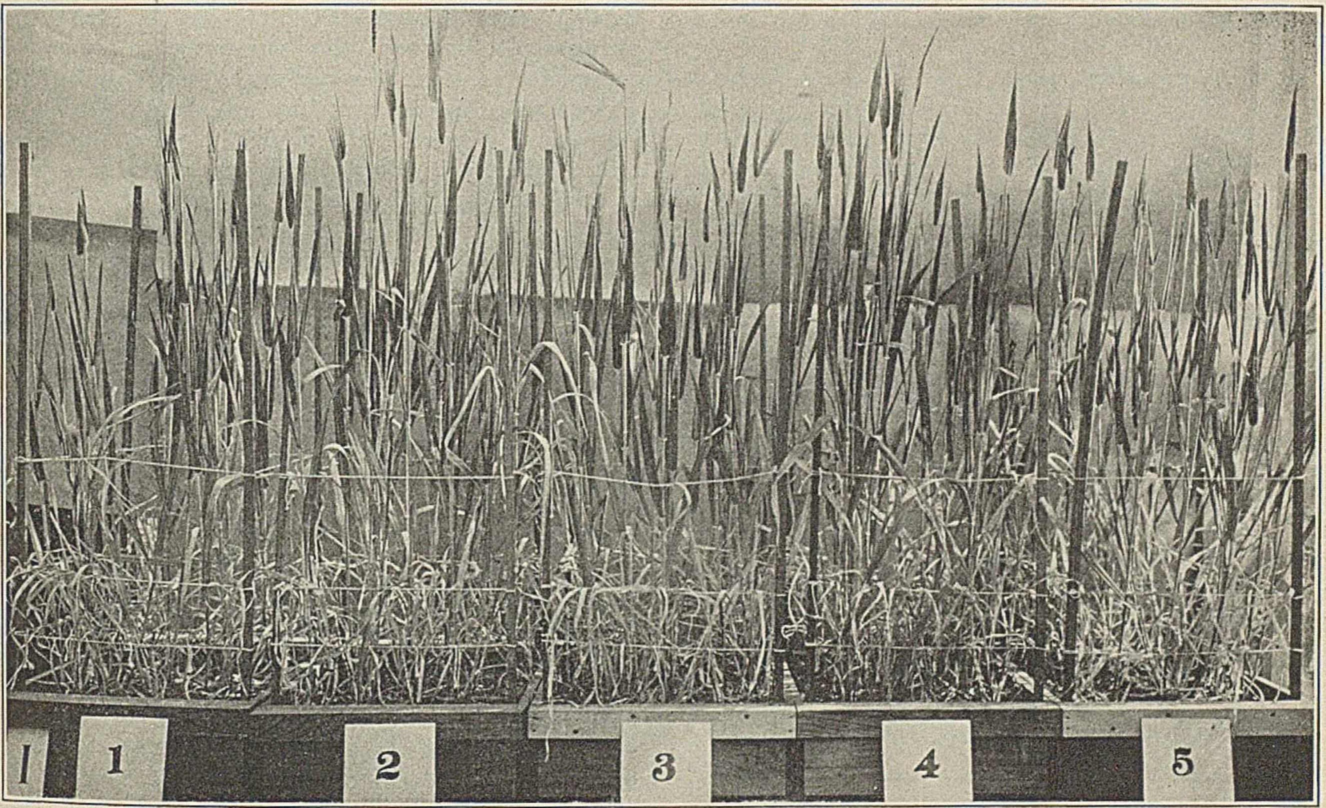
[3] *Jour. Am. Chem. Soc.*, **28**, 35.

[4] THIS JOURNAL, **4**, 606.

[1] The acid-phosphate was a commercial brand containing 17.7 per cent. moisture, 13.88 per cent. total P_2O_5 and 10.4 per cent. water-soluble P_2O_5.

[2] The calcium carbonate was about twice the amount required to neutralize the acidity of the phosphate as determined by titration.

tracts of only slight amounts of organic or other phosphorus compounds incapable of passing through porcelain filters. The experiment agrees with those described for rock-phosphate, which showed greater depression of solubility in the mixtures than in the check-manure. Moreover, the losses were of approximately the same magnitude as those observed during an equal period of fermentation in earlier work. Sample No. 3, which was subjected to extensive loss of soluble phosphorus in the presence of chloroform, appears to indicate that other factors than bacteria function in the depression of water-soluble phosphorus in fermenting mixtures of manure and acid-phosphate. An absorptive effect of the manure or chemical combination between the two would seem to be possible factors in the change. The high recovery of phosphorus in available form from the mixtures at the last extraction is a contrast to the results from rock-phosphate and further indicates the effects of such factors as absorption or conversion to compounds soluble in citrate solution. The change was presumably a conversion to dicalcium phosphate in sample No. 4, where an excess of calcium was present. The total soluble phosphorus underwent changes similar to those observed for available phosphorus and the results may be due to the causes suggested for the latter. While this single experiment cannot be considered as giving conclusive results it appears to justify the belief that conditions attending the presence of acid-phosphate in fermenting manure differ from those obtaining when rock-phosphate is present. In either case several factors may affect the status of phosphorus and the changes appear to be complex.



Check fermented manure Box 74

Fermented manure + tri-phosphate Box 66

Fermented manure + mono-phosphate Box 68

Fermented triphosphate mixture Box 70

Fermented monophosphate mixture Box 72

I. Barley Crop No. 1, Set 1, March 29, 1912

EXPERIMENTS WITH BARLEY CROPS

As a practical check upon the results already presented it was desirable to know the availability of phosphorus in fermented manure-phosphate mixtures as measured by growing crops. Greenhouse cultures of barley were grown, therefore, upon soil fertilized by manure to which Kahlbaum's c. p. tricalcium phosphate and monocalcium phosphate were added, with the purpose of comparing the influence of fermenting manure upon soluble and insoluble phosphates. On November 22, 1911, five samples based upon the usual mixture of manures were prepared as follows:

Sample 1. 10 pounds of manure.

Sample 2. 10 pounds of manure + 55 grams tricalcium phosphate.

Sample 3. 10 pounds of manure + 41.5 grams monocalcium phosphate.

Sample 4. 10 pounds of manure.

Sample 5. 10 pounds of manure.

The phosphates supplied equivalent amounts of phosphorus and corresponded to the proportions used in fermentation experiments. On December 22nd, after 30 days of fermentation, 55 grams of tricalcium phosphate were added to sample No. 4, and 41.5 grams of monocalcium phosphate to sample No. 5. The reenforced manures were mixed and analyzed for total and water-soluble phosphorus. Table V gives the data.

TABLE V.—PERCENTAGES OF PHOSPHORIC ACID IN DRY MATTER OF MANURE-PHOSPHATE MIXTURES APPLIED TO BARLEY

Sample	Percentage dry matter in manures	Total P_2O_5 per cent.	Water-soluble P_2O_5: Per cent. in manure	Water-soluble P_2O_5: Per cent. of total	P_2O_5 of H_2O ext. passing porcelain filter: Per cent. in manure	P_2O_5 of H_2O ext. passing porcelain filter: Per cent. of total	Bacteria in millions per gram of dry manure Gelatin media
1. Check manure	18.95	..	..	...	..	...	1098
2. Triphosphate mixture	18.77	5.50	1.22	22.18	0.88	16.00	724
3. Monophosphate mixture	20.82	4.98	2.44	49.00	2.13	42.77	5470
4. Manure + triphosphate	18.55	5.54	1.41	25.45	0.96	17.33	..
5. Manure + monophosphate	18.47	5.52	3.25	58.88	2.89	52.36	..

Detailed comment upon these results is unnecessary in view of the explanations of preceding tables. The depression of solubility by fermentation was much greater in case of the monophosphate than with the triphosphate; but the soluble phosphorus of the fermented monophosphate mixture was twice as great as that in either of the samples treated with triphosphate. These manures were applied in duplicate at the rate of 100 grams each to 15 kilograms of uniform clay loam[1] in wooden boxes. Applications were made as follows:

Box No.	
65 66	Fermented manure + tricalcium phosphate
67 68	Fermented manure + monocalcium phosphate
69 70	Fermented mixture of manure and tricalcium phosphate
71 72	Fermented mixture of manure and monocalcium phosphate
73 74	Check fermented manure

TABLE VI.—WEIGHT IN GRAMS OF AIR-DRY BARLEY OF HARVEST I FROM MANURES REËNFORCED WITH PHOSPHATES

Manure applied	Fermented manure + triphos.			Fermented manure + monophos.			Fermented mixture with triphos.			Fermented mixture with monophos.			Check fermented manure		
Box number	65	66	Average	67	68	Average	69	70	Average	71	72	Average	73	74	Average
Grain	20.92	24.82	22.87	26.42	29.92	28.17	19.13	26.96	23.05	17.06	24.02	20.54	21.62	21.16	21.39
Straw	35.38	42.18	38.78	42.88	44.18	43.53	33.17	41.84	37.51	35.44	34.18	34.81	39.68	36.84	38.26
Total	56.30	67.00	61.65	69.30	74.10	71.70	52.30	68.80	60.55	52.50	58.20	55.35	61.30	58.00	59.65

On December 23rd, the boxes were planted to barley of the variety Oderbrucker No. 6. The crops were thinned to sixteen per box on January 3rd. Several sprayings with atomic sulfur preparation were required to suppress mildew. Smut or stripe-disease rendered sterile 3 plants in box 66 and 2 plants each in boxes 72 and 74. On May 3, 1912, the mature crops were harvested, dried at about 50° C. and allowed one month to attain an air-dry condition. Seed and straw were then separated and weighed. The data are given in Table VI.

The difference in yield of duplicate boxes may have been due to slight differences in the greenhouse environment of the two sets of boxes. They run so nearly parallel, however, that the average yields, apparently, may be considered reliable data. The most striking result of the experiment is the superior yield from monophosphate when applied with fermented manure as compared with its application in the fermented mixture. The effect is more marked with the grain than with the straw.

The availability of the triphosphate, on the other hand, appears to have been influenced but slightly by the action of the fermenting manure. It was also as efficient as the monophosphate applied in the fermented mixture. The equal yields from triphosphate are in harmony with the approximately equal soluble phosphorus in the manures containing it. The monophosphate in the fermented mixture fails to give an increased yield corresponding to its excess of soluble phosphorus over that of the manures containing triphosphate. In the case of the monophosphate, however, the fermented manure plus phosphate contained 20 per cent. more soluble phosphorus than did the fermented mixture and gave an increase of 37 per cent. in yield of grain. The extensive bacterial flora in the fermented mixture of monophosphate would seem to be a possible cause of the low availability observed for that sample. Apparently the soluble phosphorus is retained, possibly by adsorption, in a form readily recovered by water and yet assimilated with difficulty by the roots of the barley plant. The yields of straw show the same general relations as those of grain.

A second crop of barley was planted in the soils of the previous experiment on May 8th. Chevalier barley was chosen on account of its resistance to mildew, and the selected seed was treated with formalin in the usual manner to destroy smut spores. The plants were thinned to 16 per box on May 21st. They were free from disease throughout growth. Nitrogen and potassium were liberally supplied to the odd-numbered set of boxes in the hope to make phosphorus the chief factor limiting fertility but these crops failed to produce seed and the yields are omitted. The normal crops were harvested on August 13th and cured as before. The data are given in Table VII.

TABLE VII.—WEIGHT IN GRAMS OF AIR-DRY BARLEY OF HARVEST II FROM MANURES REENFORCED WITH PHOSPHATES

Manure applied to crop of harvest I	Fermented manure + triphosphate	Fermented manure + monophosphate	Fermented mixture with triphosphate	Fer mented mixture with monophosphate	Check fermented manure
Box number	66	68	70	72	74
Grain	3.31	3.38	4.07	3.75	0.51
Straw	14.49	14.12	14.23	14.55	16.79
Total	17.80	17.50	18.30	18.30	17.30

In this second harvest from the original application of manures the triphosphate of the fermented

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[1] The soil contained 0.3 per cent. total and 0.15 per cent. HCl-soluble P_2O_5 on the air-dry basis.

mixture gave 23 per cent. more yield of grain than did triphosphate added to fermented manure. Monocalcium phosphate showed a complete reversal of efficiency from that recorded for the first harvest. In harvest I the freshly added phosphate gave an increase of 37 per cent. in yield of grain over the fermented mixture while in harvest II the yield of grain from the fermented mixture was 11 per cent. greater than from the fermented manure with freshly added phosphate. The results indicate increasing availability of the phosphorus in the fermented mixtures with duration of exposure in the soil.

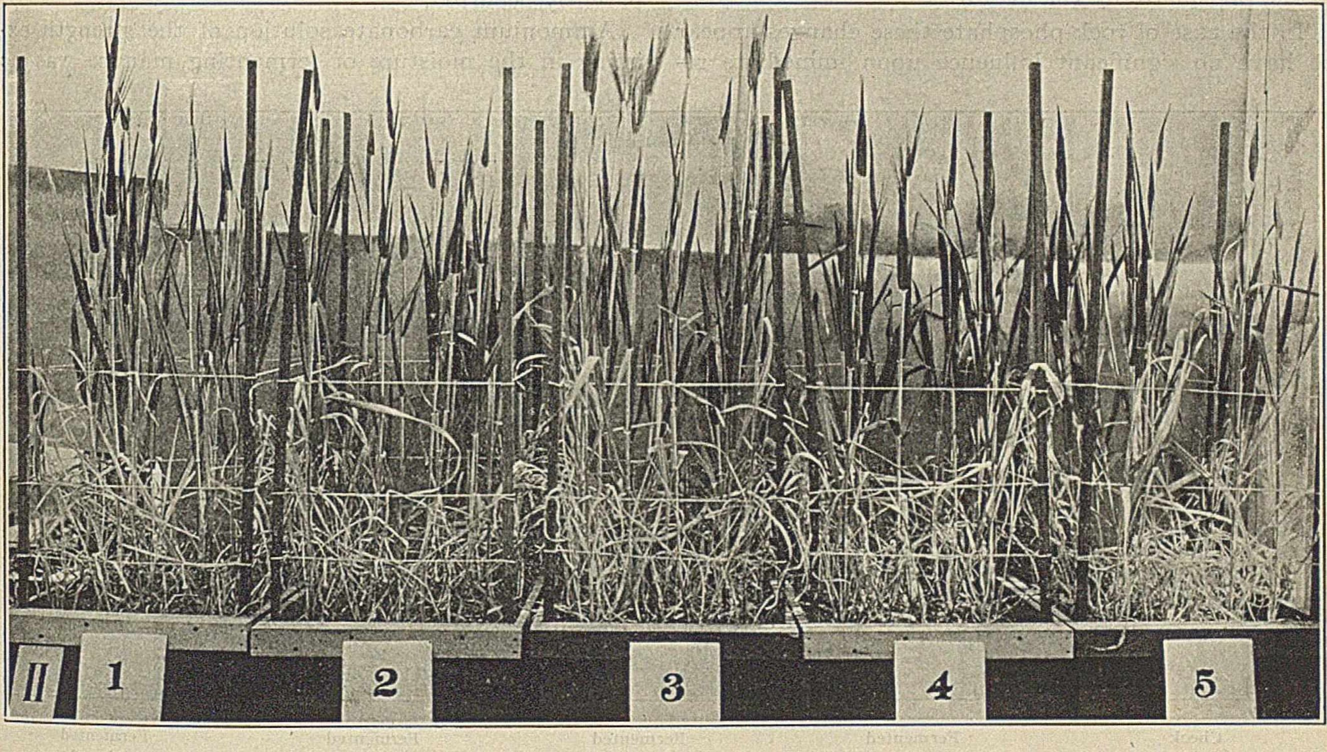
Determinations were made of the amounts of phosphorus removed from the odd-numbered set of boxes by harvest I, using the official method of incineration with $Mg(NO_3)_2$. The results showed only slight variations (from 0.9 per cent. to 1.08 per cent.) of P_2O_5 in the air-dry grain crops. The P_2O_5 content of the air-dry straw was 0.16 per cent. in the crops from check manure and fermented mixtures, 0.12 per cent. from fermented manure + triphosphate and 0.21 per cent. from fermented manure + monophosphate. Calculations based on yields and P_2O_5 content show that the following weights in milligrams of P_2O_5 were assimilated from the boxes indicated:

Check manure (box 73) 288; fermented manure + triphosphate (box 65) 229, + monophosphate (box 67) 354; fermented mixture with triphosphate (box 69) 246, with monophosphate (Box 71) 241.

According to Hall[1] barley straw shows variations of P_2O_5 content from which the availability of the soil supply can be interpreted. If this be true, the preceding results indicate that the phosphorus of soluble and insoluble calcium phosphates was equally available after prolonged contact with fermenting manure. Phosphorus of triphosphate appears to have been equally, if not more, available after contact with fermenting manure than when applied with manure previously fermented.

In the case of monophosphate higher availability was shown by the manure reënforced at the time of application than by the fermented mixture. The results agree with the yields of crop produced. On the basis of total P_2O_5 assimilated the manures showed increasing efficiency in the following order: fermented manure + triphosphate, fermented mixtures of mono- and of triphosphate, check manure and fermented manure + monophosphate.

It is manifestly unsafe to draw conclusions from the preceding experiments with pure phosphates and apply them to commercial materials, such as rock-phosphate and acid-phosphate. It would seem reasonable, however, to expect the same general conditions to obtain for both classes of phosphates and that, consequently, acid-phosphate will give the highest immediate returns when withheld from contact with fermenting manure until the latter is applied to the soil. Rock-phosphate may be expected, apparently, to give the same immediate returns whether applied in fermented mixture or simultaneously with fermented manure. Succeeding crops, however, ought to give superior results from fermented mixtures; and this condition



Check fermented manure Box 73

Fermented manure + triphosphate Box 65

Fermented manure + monophosphate Box 67

Fermented triphosphate mixture Box 69

Fermented monophosphate mixture Box 71

II. Barley Crop No. 1, Set 2, March 29, 1912

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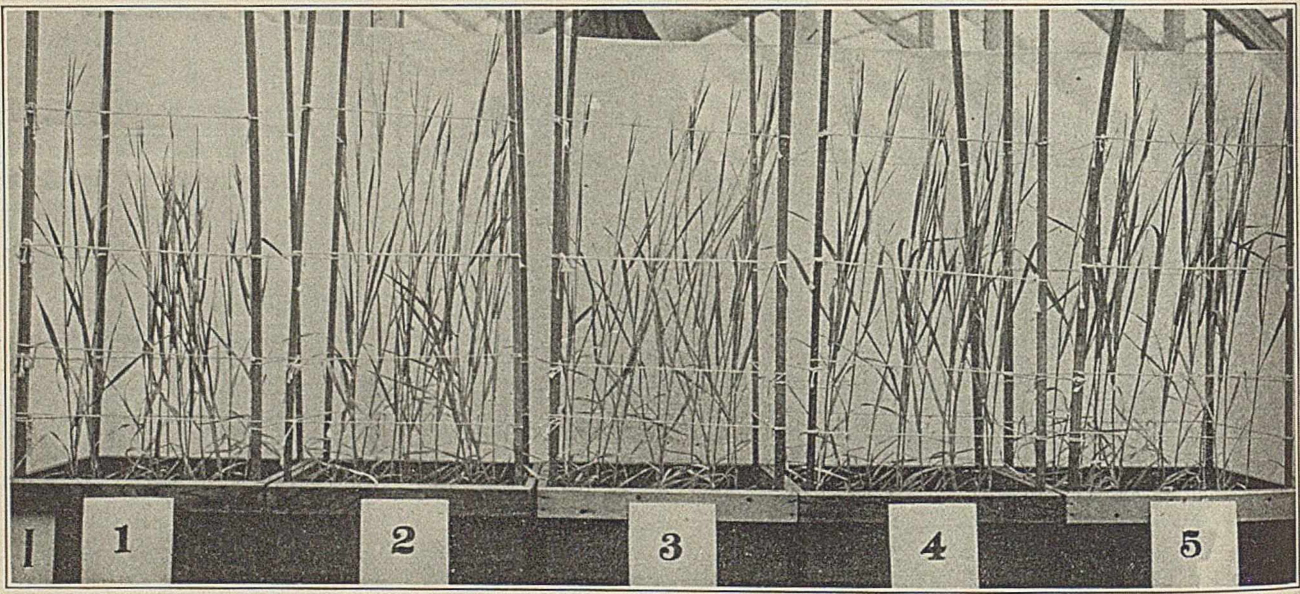
[1] "The Soil," second edition, p. 156.

agrees with the delayed action of rock-phosphate frequently observed in field practice.

While this investigation has indicated that complex relations exist between fermenting manure and reënforcing phosphates, it seems also to have indicated reasons which may explain the results and correlate them with observations from farm practice. It may be stated that, apparently, bacteria are a direct and important factor in depressing the soluble phosphorus of fermenting mixtures of manure and rock-phosphate. This effect seems to be due chiefly to the formation of insoluble nuclein compounds in the bacterial cell. When acid-phosphate is added to manure there is a depression of soluble phosphorus, even when the development of bacteria is practically inhibited. This result is apparently due to absorption of the phosphate by the manure.

In the case of rock-phosphate these changes appear to have no significant influence upon immediate returns of crop. Either the phosphorus affected by fermentation is as readily available as that of the phosphate, or the phosphate unaffected meets the demands of the crop as efficiently as the original supply. When a second crop is grown the phosphorus involved in fermentation of the mixture is apparently more available than that of the phosphate added to fermented manure. This condition may explain the delayed efficiency of rock-phosphate observed in field practice. It probably results from the action of soil organisms and other factors by which the bacterial cells of the manure are destroyed and the absorbed and assimilated phosphorus is liberated in forms available to the crop. The final result is apparently advantageous.



Check fermented manure Box 74

Fermented manure + triphosphate Box 66

Fermented manure + monophosphate Box 68

Fermented triphosphate mixture Box 70

Fermented monophosphate mixture Box 72

III. Barley Crop No. 2, Set 1, Unfertilized, July 19, 1912

In the case of acid-phosphate the apparent effect of contact with fermenting manure is to reduce seriously its efficiency for immediate returns of crop. The indications are that it is inadvisable to allow prolonged contact of this phosphate with manure in a fermenting mixture. For reënforcing purposes it seems best to add it to the manure when the latter is applied to the soil.

SUMMARY

Fermentation over periods of four to six months caused a decrease of water-soluble phosphorus in manures and mixtures of manures with rock-phosphate. The losses were greatest in the mixtures, where they amounted to more than one-half the soluble phosphorus originally present. Ammonium citrate solution and $N/5$ nitric acid did not recover this depressed phosphorus in all cases and hence other changes than simple reversion to insoluble phosphates apparently occurred.

Ammonium carbonate solution of the strength existing in the moisture of fermenting manure was no more efficient than water as a solvent for phosphorus in fermented manures and manure-phosphate mixtures.

A saturated solution of CO_2 was not superior to water as a solvent for phosphorus in manures and mixtures which had fermented about two and one-half months, and in which bacteria were active, but was superior when fermentation had covered ten months and the bacteria had become practically inert. It was five times as efficient as water or the ammonium carbonate solution in solvent power for phosphorus of rock-phosphate.

The addition of either chloroform or formaldehyde, which practically inhibited bacterial action, greatly reduced the losses of water-soluble phosphorus in mixtures of manure and rock-phosphate.

Dry bacterial cells of manure organisms grown on agar media contained four to eight per cent. P_2O_5. The growth of manure organisms upon media supplied with the soluble phosphorus of fresh manure-rock phosphate mixtures depressed the water-soluble phosphorus of the media by amounts which were comparable to the losses of water-soluble phosphorus observed in fermenting manures. About one-half the phosphorus of fresh intact bacterial cells was soluble in water. This phosphorus was apparently derived chiefly from inorganic phosphates adsorbed from the media. The remaining phosphorus of the cells was probably present in nuclein compounds.

Acid-phosphate incorporated with fermenting manure underwent essentially the same changes of solubility as did rock-phosphate. In the presence of chloroform, however, the acid-phosphate decreased markedly in solubility, while with rock-phosphate no decrease occurred.

Application of monocalcium phosphate to barley in pot experiments gave greater immediate returns when the phosphate was applied with previously fermented manure than when it was applied in a fermented mixture of manure and phosphate. The immediate returns from tricalcium phosphate were the same whether it was applied with previously fermented manure or in a fermented mixture. A second crop of barley from the original application of reënforced manures gave a superior yield from the fermented mixture for both phosphates.

The conclusions derived from this work require confirmation under field conditions before they can be applied to direct agricultural practice. However, so far as pot experiments indicate conditions in field practice, the final results from mixing rock-phosphate with fermenting manure appear to be advantageous. In the case of acid-phosphate it appears inadvisable to mix the material with fermenting manure. It is probably better practice to add it to the manure at the time of application to the field.

The authors desire to express appreciation to Professor E. B. Hart for helpful suggestions and criticism received during this investigation. The results will appear in detail as a technical bulletin from the Wisconsin Experiment Station.

Agricultural Chemistry and Bacteriology Laboratories
Wisconsin Experiment Station
Madison

THE GAY-LUSSAC METHOD OF SILVER DETERMINATION[1]

By Frederic P. Dewey[2]

Received November 19, 1912

This old and well-known method of determining silver is, in bullion work, so far superior to the furnace assay that it is looked upon with reverential awe by many, if not by most, users, and its ease of execution with proper equipment commends it highly where much commerical bullion work on silver is required.

The method is so thoroughly well known that a description of it here may seem to be unnecessary, but many thousand determinations are annually made by it in the United States Mint Service and its practice is there reduced to an art. This is necessary both for the sake of economy of time and because high-grade work with it requires constant practice. It is useless for a person who uses it only occasionally to expect to attain high accuracy with it, without spending more time upon a determination than is warranted in a busy commercial laboratory.

It is always employed in the Mint service whenever possible, but its chief field is in the determination of silver in standard silver (ingots and coin), which is 900 fine in silver and 100 fine in copper, and I shall first briefly outline its use on this metal and then take up various points in detail, especially as related to the accuracy of the results obtained.

For this determination 1115 milligrams of standard metal are weighed and transferred to a glass-stoppered bottle, the metal is dissolved in nitric acid and 100 cc. of a standard solution of NaCl run in. The bottle is then vigorously shaken and a measured portion of a decimal salt solution added. The bottle is again shaken, more decimal solution added, and again shaken, if necessary. These operations are repeated until the silver is precipitated.

Standard metal being 900 fine, 1115 mg. should carry 1003.5 mg. of silver, which is a convenient figure to work by. Legally, standard metal may vary 3 in fineness above or below 900. Practically, however, it seldom runs below 898 or above 901 fine. Therefore, the variation in actual silver present above or below 1003.5 mg. is not excessive. For testing the standard salt solution we weigh up 1004 mg. of proof silver. For convenience and accuracy we have single weights of 1115 mg. and 1004 mg.

The bottles should be of perfectly white glass, carefully made and well annealed; 8 oz. is a convenient size often used, the bottles are most conveniently handled in a circular frame or basket, holding ten. For dissolving the silver, both the amount and strength of nitric acid used may vary considerably without apparent effect upon the accuracy of the results, 8 cc. at 1.30 sp. gr. to 25 cc. at 1.20 sp. gr. being allowable. Some operators heat the bottles to remove nitrous fumes, others do not.

The standard salt solution is designed to have 100 cc. precipitate exactly 1 gram or 1000 mg. of silver, but it seldom "shows" this exact strength. The word "shows" is used instead of "is" because the equivalent of the solution depends upon other factors besides its composition.

In general, two proofs should be used in every set of 10 bottles, unless several sets are to be run in rapid succession, when one proof in each set may answer. Some operators run an independent proof occasionally during the day and omit the proofs in the sets, but this proceeding is objectionable.

The next step in the method is the addition of 100 cc. of the standard salt solution (often improperly called "normal" salt solution). This is a very simple operation, but it requires the utmost care and constant attention to details if high accuracy is desired in the results. Only a minute variation in the

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[1] Published by permission of the Director of the Mint and jointly with the American Institute of Mining Engineers.

[2] Assayer, Bureau of the Mint.

amount of solution added will make a serious variation in the fineness of silver shown.

The Stas pipette is the one universally used in the Mint service. It is a simple pipette open at both ends and securely mounted on a wall bracket. The upper end is drawn out to a fine opening and is provided with a collar-cup to catch the drip. The lower end is comparatively large and must have a free and smooth discharge. The lower end is connected through a removable rubber tube, provided with a pinchcock, with an elevated tank containing the salt solution. No stopcocks, floats, or graduations of any kind can be used on or in the pipette if rapid work is to be done. It is also questionable if any such arrangement can be as accurate as the simple filling of the Stas pipette and letting its contents run directly into the silver bottle.

In operating the Stas pipette the rubber tubing is slipped over the lower end and the pinchcock opened. As soon as the solution comes out of the upper end of the tube, it is closed by the first finger of the left hand and the pinchcock closed. The operator must now be sure that there are no air bubbles in the pipette. If such should appear they must be allowed to collect at the top of the pipette; the pinchcock must be opened, the finger momentarily removed from the upper end of the pipette, and the pinchcock closed again. When the pipette is full of solution and the pinchcock closed, the rubber tube is withdrawn from the lower end of the pipette. This end must now be carefully examined to see that there is no surplus solution adhering to it or that the air has not commenced to ascend the tube.

If the lower end of the tube is in proper condition the silver bottle is now placed directly under it, the finger removed from the upper end and the solution allowed to flow into the bottle. The solution should flow out rapidly in a smooth, solid stream. Just as soon as the flow stops, the bottle must be removed from under the pipette. It is absolutely fatal to accuracy to attempt any adjustment of the drip of the pipette. There should be only just easy clearance between the bottom of the pipette and the top of the bottle.

The pipette is supposed to contain 100 cc. and on various accounts it is desirable that it should be fairly accurate, but it is not at all necessary that it should be absolutely accurate. The absolutely essential point about it is that it should deliver exactly the same amount of solution to each one of the ten bottles composing a set. The amount delivered may be a trifle more or a trifle less than 100 cc. or it may vary slightly from a hot day in summer to a cold day in winter, but it should not vary between the first and last bottles of a set. It is a good plan to fill and empty the pipette a few times before beginning to fill the set. After withdrawing the bottle from under the pipette the stopper is dipped in distilled water and inserted into the neck of the bottle with care.

Having filled the set, the carefully stoppered bottles are placed in a shaking machine and agitated for 3 to 5 minutes to settle the precipitate. The bottles are next placed upon a black shelf, technically called a "board," with a black background about as high as the shoulder of the bottle and about 3 inches back of the bottle, the whole being installed in a window, preferably with a northern exposure.

Up to this point in the method, the procedure is substantially the same in all the laboratories, but from here on there are slight differences in the manipulations. In common with many volumetric methods, there is difficulty in this one in determining the end point. In many descriptions of the method the operator is directed to add decimal salt solution in small measured amounts, with agitation between the additions, until no more precipitate is formed on adding the salt solution. Too much salt has now been added and this excess must be determined by the addition of small measured amounts of decimal silver solution and the amount of silver present in the metal determined by balancing these amounts.

This method is open to two serious objections. When exactly the proper amount of salt has been added to precipitate the silver present, a condition of equilibrium in the solution results, which is disturbed by the addition of either reagent with the separation of a precipitate. This obscures the end reaction. These alternate dosings and shakings consume too much time for rapid work, and after many shakings the solution does not clear well.

In the Mint service, therefore, the operation known as "reading the cloud" has been substituted for these alternate dosings of the solution. This operation is far more rapid, but it requires a great deal of skill and constant practice to yield the best results. In reading the cloud, after the addition of the 100 cc. of standard salt solution and shaking, a measured amount, 1 or $^1/_2$ cc., of decimal salt solution is added to each bottle. The delivery end of the pipette is placed against the neck of the bottle as far down as possible and the solution allowed to flow gently down the side of the bottle so that it will remain on the surface of the solution in the bottle with the minimum amount of mixing; by a slight rotary motion of the hand the decimal solution is then mixed with the upper portion (about $^1/_3$) of the bottle solution. This produces a cloud of AgCl in the solution and the next step is based upon the appearance of this cloud. Here the skill and visual condition, together with the personal equation, of the operator are of the utmost importance.

If the cloud be very heavy two or more portions of decimal salt solution are added, the amount depending upon the density of the cloud, and the bottles shaken in the machine again. If the cloud is light, only one dose of decimal is used. If the cloud is very light the bottle is again shaken by hand to bring more of the solution into reaction and the cloud again examined. As the result of this treatment one dose of decimal may be used, or the bottle may be shaken by hand again to bring the balance of the solution above the precipitate into the reaction. Here again a dose of decimal may be used, or the final reading of the cloud may take place. In the final reading the operator estimates from the density of the cloud what portion of the dose of decimal solution was consumed in precipitating the silver. Many operators estimate to 0.25 cc., others claim to be able to estimate to 0.1. The results of the investigation given beyond indicate that on standard metal estimating to 0.1 is not profitable under present conditions.

All the bottles are eventually brought down to the final cloud, and at the end the amount of decimal solution added to each bottle is recorded. The records of the assays are then compared with the proofs and the fineness of the samples determined. The actual fineness of a sample is shown entirely by the amount of decimal solution used as compared with the amount of decimal required by the proof. It is entirely independent of the amount of standard solution used. Therefore in determining the fineness of a sample we simply compare the amount of decimal used with the amount used in the proof, making allowance for the 0.5 mg. more of silver in the proof above the silver in 1115 mg. of metal at the exact standard of 900, and taking into consideration the weight of the sample used. For instance, if the sample required 0.5 cc. less than the proof, the sample would be reported at 900. This is not strictly exact because 0.25 cc. of decimal salt solution equals 0.25 mg. silver, but 0.25 mg. is only 0.22 fine on 1115 mg. and 0.5 cc. would be only 0.44 fine. However, this variation is too slight for practical consideration, and in general the finenesses are read directly from the difference between the sample and proof. Generally also, the differences are read in quarters of a cc.

Practically we deduct $^2/_4$ from the amount of decimal solution used on the proof and call it standard. Then for each quarter's difference from standard we add or subtract, as the case may be, from 900 in accordance with the following tabulation:

For 1/4..............	0.2	For 6/4..............	1.3
For 2/4..............	0.4	For 7/4..............	1.5
For 3/4..............	0.7	For 8/4..............	1.8
For 4/4..............	0.9	For 9/4..............	2.0
For 5/4..............	1.1	For 10/4..............	2.2

A second method of determining the end point, which appears to be even more exact, but which requires more time, consists in adding only 0.25 cc. of the decimal solution after machine shaking, then estimating the number of quarters the solution will stand. These are added and the bottles shaken in the machine. This is continued until the quarter added after shaking produces no precipitate, in which case the last quarter is not counted, or else such a slight precipitate is produced that the quarter is counted but the bottle is not shaken again. The reading of the results is the same as before described.

In both these methods great care is exercised to avoid the addition of so much salt as to require back-titration with decimal silver. Back titration is regarded with much disfavor.

In the regular work of the Mint Bureau it became desirable to ascertain just how much reliance could be placed upon the results obtained by the Gay-Lussac method as ordinarily executed in the service and an extensive investigation was undertaken for this purpose.

Six samples, representing three melts of ingots, were distributed among four laboratories in the service without informing any of the institutions just what the samples were. Table I summarizes the results reported:

Table I—Assays of Three Ingot Melts

	No. 1		No. 2		No. 3	
Fineness	A	B	A	B	A	B
898.3			1			
898.4	2	1	2	2		1
898.6	2	4	2	2	3	2
898.7			1			
898.8			2	1		
898.9	5	5	5	5	6	6
899.0				2		
899.1	10	9	8	7	8	7
899.2		1			2	1
899.3	1	1				
899.4					1	1
899.5					1	1
899.6				2		
Total......	20	21	21	21	21	19

Notwithstanding the great preponderance of results from 898.9 to 899.1 shown here, the ingot melts were reported by the assayer of the mint where they were made as follows:

No. 1 898.5 fine in silver
No. 2 898.25 fine in silver
No. 3 898.5 fine in silver

This table was submitted to the various assayers for criticism and comment and suggestions for improvements in the method. Five reasons were advanced to explain, in part at least, the differences shown in the table, as follows:

(1) Difference in samples taken for assay.
(2) Non-homogeneity of the metal.
(3) Working on a sample on different days.
(4) The personal equation of the operator.
(5) Accidents.

Two assayers suggested that perhaps under the actual, every-day working conditions of our service, the method is not so accurate as it is supposed to be.

Table II—Assays of 22 Samples of a Small Bar of Standard Silver

3 assays showed 899.1
6 assays showed 899.3
6 assays showed 899.5
10 assays showed 899.6
1 assays showed 899.7
4 assays showed 899.8
7 assays showed 899.9
14 assays showed 900.0
2 assays showed 900.1
4 assays showed 900.2
8 assays showed 900.3
1 assay showed 900.4
fine in silver
—
66

It was quite impossible at this time to deal with the personal equation of the operator or the chapter of accidents, but, in order to meet the first two of these reasons, a small bar of standard silver was prepared in the Bureau laboratory with especial care to avoid segregation of the metal. It was then rolled out and cut into small oblongs, of which 30–35 were required to weigh 1115 mg. To meet the third reason, four samples of the oblongs, each weighing approximately 3.75 grams, sufficient for three determinations of the silver, were sent to each one of the same four laboratories at one time. Subsequently, one laboratory ran two more samples. In all 66 assays were made and practically all of the bar was used for these assays. Table II summarizes the results reported.

This table was also submitted to the four assayers for comment and criticism, and the investigation was continued upon six sets of ingot samples and six special samples. These samples were sent out to the four institutions for comparative test assays, in a long series. In no case was any institution informed as to the source of the metal, or given the results of any previous assays. The samples were sent out in lots of four in all cases but one. In several cases all four samples in a lot were the same and in two cases this fact was stated in sending them out. In many cases two or three samples in a lot would be the same, but this fact was not disclosed. In one case six samples, representing the three regular samples of a melt of ingots, were sent out with this statement, but it was not shown which were the duplicate samples.

Table III—Assays of Three Melts of Ingots

Melt No. 132

	Original assays		Test assays				
	Mint of origin		Mint of origin		Independent		
Fineness	A	B	A	B	A	B	Total
898.3			2	1	1	1	5
898.4					2		2
898.5						1	1
898.6			1	2	3	4	10
898.7							
898.8							
898.9					3	9	12
899.0							
899.1	1	1	3	1	2	2	8
899.2							
899.3						3	3
899.4							
899.5	1	1		2	2	2	6
Total....	2	2	6	6	13	22	47
Report 899.3							
			Melt No. 133				
898.3				1	2	1	4
898.4					3	1	4
898.5					2	1	3
898.6	1		6	2	8	5	21
898.7							
898.8							
898.9					3	4	7
899.0						1	1
899.1	1	1		2		4	6
899.2							
899.3						1	1
899.4							
899.5				1			1
900.0		1					
Total....	2	2	6	6	18	18	48
Report 899.1							
			Melt No. 138				
898.6					9	2	11
898.7							
898.8					1		1
898.9					4	7	11
899.0					2	2	4
899.1	1		6	6	2	5	19
899.2						1	1
899.3							
899.4							
899.5	1	2					
Total....	2	2	6	6	18	17	47
Report 899.5							

In the ordinary operations of melting ingots and taking samples from the melts, it is quite possible that there might be some slight difference in the samples themselves. The two following tables, however, indicate that in the present condition of the method such differences would be masked by differences in the results due to the method itself.

The regular samples from a melt of ingots are generally too small to allow a sufficient number of test assays to be made. From three melts one mint supplied larger samples and they were sent to the four laboratories for test assays and Table III summarizes the results reported, together with the original assays made at the mint of origin of the samples.

This table indicates that there was a very decided tendency at this mint to report their ingots high.

Another mint supplied two sets of ingot samples that were sent out to the four laboratories in the general series of samples, and Table IV summarizes the results reported and gives the original assays made at the mint:

Table IV

Melt No. 175

	Original assays			Test assays						
	Mint of origin			Mint of origin			Independent			
Fineness	A	B	C	A	B	C	A	B	C	Total
898.4							1			1
898.5										
898.6				1						1
898.7										
898.8										
898.9				5	1					6
899.0							1			1
899.1		1			2			2	1	5
899.2							1			1
899.3	1	1	1		3	1	3		2	9
899.4										
899.5						1	8	7	1	17
899.6	1		1						3	3
899.7						2	1	2		5
899.8							3	3	4	10
899.9						2				2
900.0								1	5	6
900.1										
900.2										
900.3										
900.4								2	2	4
Total....	2	2	2	6	6	6	18	17	18	71
Report 899.3										
					Melt No. 176					
898.9				3	3	3				9
899.0										
899.1				1		3			1	5
899.2										
899.3			2	1	1					2
899.4										
899.5							6	2	11	19
899.6	2	2		1	2		1		2	6
899.7							2	3	1	6
899.8							2	1	1	4
899.9							1	1		2
900.0							5	11	2	18
900.1										
900.2							1			1
Total....	2	2	2	6	6	6	18	18	18	72
Report 899.5										

A third set of samples from the second mint was sent out in a set of six and the institutions informed that they represented a melt of ingots, but the duplicates were not indicated; Table V summarizes the results reported, together with the original mint assays:

TABLE V—SAMPLES OF AN INGOT MELT ASSAYED AT ONE TIME
Melt No. 180

	Original assays			Test assays						
	Mint of origin			Mint of origin			Independent			
Fineness	A	B	C	A	B	C	A	B	C	Total
899.3		1	1				1			1
899.4								1	2	3
899.5							6	5	9	20
899.6	2	1	1					1		1
899.7								1	1	2
899.8					2	1	4	5	4	16
899.9							3	2		5
900.0				6	4	5	3	2	2	22
900.1							1	1		2
	—	—	—	—	—	—	—	—	—	—
Total....	2	2	2	6	6	6	18	18	18	72
Report 899.5										

The general tendency of the original reports on these three melts appears to be a trifle low, and this is supported by the low test assays on melt No. 176, made at the mint of origin.

In the last three tables all the results reported have been given. Undoubtedly, some of these were vitiated by accidents. These tables also indicate that there is a general laboratory equation similar to the personal equation of an operator.

In order to eliminate differences in the samples as a source of difference in the assay results, one mint prepared two special samples in regard to which the assayer wrote: "I have prepared two samples of standard silver (6 oz. each). I used coin ingot melted a number of times with stirring; finally cast in a closed mold that had been chilled with ice; poured silver at as low temperature as possible. Resulting bars were very free from oxidation. They were, however, thoroughly cleaned, and rolled into strips and cut."

The samples cut into oblongs, of which it took about 30 to weigh 1115 mg., were forwarded to the Bureau, where they were further mixed and portions of 3.75 grams weighed out into small envelopes, giving ample material for three determinations of silver in each envelope. Ten envelopes were sent at various times to each one of the four institutions. Each laboratory made 30 determinations of silver on each of the larger samples, making a total of 120 assays on each, but in tabulating the results reported a certain amount of discretion has been exercised. In general, results at either extreme in fineness have not been included in the table unless they were reported at least twice by two laboratories, or five times by one laboratory; Table VI summarizes the results, together with the original assays at the mint of origin.

TABLE VI—ASSAYS OF TWO SPECIAL BARS

	Original assays	Test assays				
Fineness	Mint of origin	Mint of origin	Lab. No. 1	Lab. No. 2	Lab. No. 3	Total
			Bar No. 1.			
898.4		6	3			9
898.5			2			2
898.6	10	18	11	19	20	68
898.7			8			8
898.8			1			1
898.9		6		11		17
899.0						
899.1			2		6	8
Eliminated			3		4	7
	—	—	—	—	—	—
Total.....	10	30	30	30	30	120
			Bar No. 2			
898.4	1	5	4			9
898.5			4			4
898.6	5	14	12	14	12	52
898.7			2			2
898.8			3			3
898.9	4	7	1	13		21
899.0			1			1
899.1				3	15	18
Eliminated		4	3		3	10
	—	—	—	—	—	—
Total.....	10	30	30	30	30	120

Another mint (Lab. No. 3 above) prepared two special granulation samples in regard to which the assayer wrote: "I am submitting two samples of six ounces each of the most homogeneous standard silver that I have been able to prepare. These samples were prepared as follows: Standard silver ingot granulations assaying from 899.1 to 899.8 and weighing in the aggregate a little over 30 ounces were melted with frequent stirring and the entire melt granulated. Two samples of about 8 ounces each were then segregated and 18 assays made from different portions of the sample. Seventeen of these assays went 899.5."

These samples were divided into portions weighing 3.75 grams each, at the Bureau. It was not noticed at first that they were really only one sample so that 11 envelopes of No. 1 and 9 envelopes of No. 2 were sent to each one of the four institutions. Table VII summarizes the results reported, together with the original assays at the mint of origin.

TABLE VII—ASSAYS OF SPECIAL GRANULATIONS

	Original assays	Test assays				
Fineness	Mint of origin	Mint of origin	Lab. No. 1	Lab. No. 2	Lab. No. 3	Total
			Portion No. 1			
898.6			8	8	1	17
898.7			6			6
898.8			5			5
898.9			3	13	13	29
899.0			3			3
899.1		10	1	12	12	35
899.2			2			2
899.3			2		6	8
899.4						
899.5	17	23	1		1	25
Eliminated	1		2			2
	—	—	—	—	—	—
Total.....	18	33	33	33	33	132
			Portion No. 2			
898.6		2	11	5		18
898.7			5			5
898.8			7			7
898.9			3	7	12	22
899.0						
899.1		9		15	14	38
899.2						
899.3					1	1
899.4						
899.5	17	16	1?			17
Eliminated	1					
	—	—	—	—	—	—
Total.....	18	27	27	27	27	108

In this case Lab. No. 3 is the one that supplied the special bar samples. The laboratory supplying these special granulation samples is the same one that forwarded ingot samples Nos. 132–3–8, and the same tendency to obtain high results is again shown here.

More than 15 years ago, in an article entitled "The Actual Accuracy of Chemical Analysis,[1] in speaking

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[1] *Trans. A. I. M. E.*, **26**, 370 (1896); *Jour. Am. Chem. Soc.*, **18**, No. 9, p. 808 (Sept., 1896).

of the "degree of accuracy exhibited in actual everyday practice" I said: "In estimating this, little or no weight will be given to the evidence afforded by the agreement of duplicate or multiple determinations by the same chemist, for I am convinced that such agreement is a delusion and a snare." The results reported on these granulation samples afford a striking illustration of the soundness of this position. This metal was assayed 257 times. The laboratory preparing it reported it 56 times at 899.5 fine, but the other laboratories reported this figure only 3 times and one of these determinations was questioned by the laboratory reporting it. On the other hand the work of the other laboratories shows that this metal could not have been over 899 fine.

Another small bar was prepared in the Bureau laboratory with especial care, and cut up into oblongs requiring 30–35 to weigh 1115 mg. These oblongs were thoroughly mixed and small samples of 3.75 grams put into envelopes. Ten of these envelopes were sent to each one of the four institutions and Table VIII summarizes the results reported:

TABLE VIII—ASSAYS BUREAU SPECIAL SAMPLE NO. 2

Fineness	Lab. No. 1	Lab. No. 2	Lab. No. 3	Lab. No. 4	Total
899.1		6			6
899.2					
899.3		6	3		9
899.4					
899.5	5		6		11
899.6	4				4
899.7	12				12
899.8			9		9
899.9	6				6
900.0	2	13	9	17	41
900.1					
900.2		5	3		8
900.3					
900.4	1?			13	14
Total......	30	30	30	30	120

The laboratories here are numbered the same as in Table VII, and No. 4 is the mint of Table VII.

A final sample was made up at the Bureau by uniting the oblongs left over from the regular coin work of the Bureau, which were of widely varying finenesses, and, without any special mixing, weighing up samples of 3.75 grams. Ten envelopes were sent to each one of the institutions and Table IX summarizes the results reported. As this sample had no claim to uniformity all the results reported are given:

TABLE IX—ASSAYS OF BUREAU COIN SCRAP SAMPLE

Fineness	Lab. No. 1	Lab. No. 2	Lab. No. 3	Lab. No. 4	Total
898.6		4			4
898.7					
898.8					
898.9		7	2		9
899.0	1				1
899.1		1	12	8	21
899.2	4				4
899.3		6	6		12
899.4	2				2
899.5	9	12	6	20	47
899.6	6				6
899.7	3				3
899.8	1		4		5
899.9					
900.0				2	2
Lost	4				4
Total......	30	30	30	30	120

The laboratory numbers here are the same as in Table VIII.

As a final test of the method when practically applied to ingot work, three large samples were taken from each one of nine melts of ingots and assayed in various laboratories. Each sample was assayed in duplicate five times and in triplicate once, making a total of 39 determinations on each melt. Table X summarizes the results reported.

TABLE X—ASSAYS OF NINE COIN INGOT MELTS

	Melts									
Fineness	1	2	3	4	5	6	7	8	9	Total
897.7	1									1
898.0	1	1	1							3
898.2	1	2	1							4
898.4			1							1
898.6	7	5	7	3	1					23
898.7	1									1
898.8	3		3			1				7
898.9	7	1	3	1	3					15
899.0						2	2	1		5
899.1	12	4	10	15	10	7	2	3	5	68
899.2						2				2
899.3	1	4	4	6	7			2	8	32
899.4						2		5		7
899.5	5	8	8	13	14	15	16	19	17	115
899.6		2	1			4	3	5		15
899.7		3				3	3	1		10
899.8					1	1	4	1	1	8
900.0		7		1	3	1	5	2	8	27
900.1						1	4			5
900.4		2								2
Totals...	39	39	39	39	39	39	39	39	39	351

As a general conclusion from the elaborate tests herein given, it may be stated that two operators, working upon identical samples of standard silver and making four determinations each, may differ as much as 1 fine in their reports. Having thus established the capacity of the method as at present carried on as a commercial operation, attention is now being given to improving the method so as to reduce the allowable limit of difference.

It may seem unnecessary to many of my readers, but my experience shows that there is much confusion and uncertainty in stating the composition of precious metal bullion. This is generally done in parts per thousand or fineness. It is similar to stating composition by percentage, but in bullion work there is much misunderstanding regarding the decimal point. Intelligent people who would never think of expressing fifty per cent. by ".50%" habitually write five hundred fine as ".500 fine" and incorrectly use the decimal point before the numerals in stating fineness. Whenever "fine" or "fineness" or an equivalent expression is used in stating the composition of bullion the decimal point should not be used unless the figures following it express a quantity less than 1 fine. Again, some people incorrectly use a preliminary "0" when the fineness is less than 100. They would not think of writing "05 per cent." but habitually do write "052 fine." Again, some people mistake fineness as being the exact equivalent of percentage, but it is only $^{1}/_{10}$ of percentage, and in considering the accuracy here shown by the Gay-Lussac method it should be noted that 1 fine is the equivalent of 0.1 per cent.

WASHINGTON, D. C.

THE SULFOCYANATE-PERMANGANATE METHOD FOR COPPER IN ORES

By D. J. Demorest

Received November 12, 1912

Under the proper conditions copper may be precipitated practically completely as cuprous sulfocyanate. It is true that a qualitative test for copper may be obtained in the filtrate from the cuprous sulfocyanate when potassium ferrocyanide is added but this is due to the presence of only about 0.0001 gram of copper, as the writer has found by colorimetric measurements. Furthermore, the writer has found that practically all the copper added to the solution before precipitation may be recovered from the precipitate by electrolysis.

In Guess's method for copper the cuprous sulfocyanate is dissolved on the filter with sodium hydroxide, leaving the cuprous hydroxide on the filter while sodium sulfocyanate goes through. The filtrate is then made acid with sulfuric acid and is titrated with permanganate. Theoretically, the titration reaction is

$$5HCNS + 6KMnO_4 + 4H_2SO_4 = 5HCN + 3K_2SO_4 + 6MnSO_4 + 4H_2O$$

Practically, however, the oxidation in acid solution is never complete and an empirical factor must be used amounting to about 5 per cent. of the copper present, according to Guess's tables of correction given in Low's "Technical Methods of Ore Analysis." This is also in accord with the writer's experience and with that of Grossman and Hölter.[1]

It occurred to the writer that if the permanganate were added to the sulfocyanate in alkaline solution until about as much was added as would be required to titrate the sulfocyanate in acid solution and if then the solution were made acid, the titration might be finished by a few drops, and in this way complete oxidation might be brought about and no empirical factor would be needed.

It is easy to tell when enough permanganate has been added to the alkaline solution by testing a drop or so on a white plate with a drop of an acid solution of ferric chloride. If not enough permanganate has been added a red color develops but if enough has been added no red appears. It is not necessary to run in permanganate until no red is obtained on testing. When the red color of the test is very faint the solution may be made acid and the titration finished in the usual way.

PROCESS OF ANALYSIS

Weigh out enough of the ore to have from 0.05 gram to 0.30 gram of copper present. Transfer the sample to a 200 cc. beaker, add 5 cc. of strong hydrochloric acid and heat for several minutes; then add 10 cc. of nitric acid and digest on a hot plate until the ore is completely decomposed. Then add 10 cc. of 1 : 1 sulfuric acid and boil down until fumes of SO_3 appear. Cool and add 50 cc. of water containing 3 grams of tartaric acid and heat until all soluble salts are in solution. Cool and add ammonia until the solution turns a deep blue, then add sulfuric acid until the liquid becomes acid, then add one cc. more. Now add one gram of sodium sulfite dissolved in 20 cc. of water, heat nearly to boiling and add slowly and with vigorous stirring one gram of potassium sulfocyanate dissolved in 20 cc. of water. Heat at a nearly boiling temperature for several minutes to coagulate the precipitate and dissolve out of it all tartaric acid. Cool somewhat and filter while still hot through, preferably, an asbestos mat on a Gooch filter. Wash well with water and rinse out the suction flask. Then pour through the crucible 30 cc. of a hot 10 per cent. sodium hydroxide solution and wash well with water.

Warm the filtrate to about 50° and proceed to titrate. Run in slowly at first, while the flask is vigorously shaken, the standard permanganate solution. The solution in the flask turns green, due to the K_2MnO_4 formed. After about 10 cc. have been run in take out a drop of the solution and place it in a drop of strong hydrochloric acid on a paraffined white plate; then add a drop of a 10 per cent. solution of ferric chloride. If a red color appears continue to add the permanganate solution, testing after each 5 cc. until the red becomes weak, then test more frequently until the red color of a test becomes quite faint. Then add 30 cc. of 1 : 1 sulfuric acid and shake the flask until the MnO_2 all dissolves and finish the titration to the usual permanganate end point. This will require perhaps one cc. of permanganate.

NOTES ON THE PROCESS

Fairly close results may be obtained by titrating carefully in the alkaline solution until five drops of the solution give no red color with ferric chloride. But it is easier and more accurate to titrate only to an approximate end in the alkaline solution and finish in an acid solution. It is important that the precipitation be made as above directed with vigorous stirring and that the solution be digested on the hot plate after precipitation.

Under the conditions of titration the permanganate is reduced in the alkaline solution thus: $2KMnO_4 = K_2MnO_4 + MnO_2 + 2O$. The oxygen possibly oxidizes the sulfocyanate to tetrathionate, but of this the writer is not sure. He has proved by filtering off the MnO_2 and weighing it and by titrating the manganate in the filtrate that the permanganate is reduced as above written. However, it makes no difference what reaction takes place in the oxidation of the sulfur in the alkaline solution, since the test for sulfocyanic acid is made on the plate in a strongly acid solution and the reaction between the sulfocyanate and the manganate and manganese dioxide completes itself there. The manganese dioxide does not settle out of the solution unless it stands a long time.

According to the titration reaction given in the second paragraph of this article 5 atoms of copper are equivalent to 6 molecules of $KMnO_4$ or 30 atoms of iron. Therefore, the iron value of the permanganate multiplied by Cu/6Fe or 0.1897 gives the copper value of the permanganate. The permanganate used

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[1] Grossman and Hölter, *Chem. Ztg.*, **33**, 348.

by the writer equals 0.01 gram Fe, or 0.001897 gram Cu per cc.

After the filtrate has cooled, a crystalline compound of tartaric acid may separate. Hence the solution should be filtered hot.

The following are some results obtained by the above method:

Copper		Copper	
Present	Found	Present	Found
0.1870	0.1890	0.0600	0.0596
0.1503	0.1511	0.0605	0.0603
0.1117	0.1125	0.1610	0.1630
0.1666	0.1668	0.0663	0.0667
0.0940	0.0930	0.1103	0.1109
0.2005	0.2017	0.1140	0.1147
0.2220	0.2224	0.1000	0.1001
0.2220	0.2210	0.1000	0.0999

The later results were the most accurate and were obtained after the necessity of digesting the precipitate in the hot solution was learned.

No elements interfere except, according to Low, silver. This is easily removed. If there is any lead present, the lead sulfate obtained after evaporating to fumes of SO_3 should be filtered off after the soluble salts are dissolved.

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ELECTROLYTIC DETERMINATION OF COPPER IN ORES, CONTAINING ARSENIC, ANTIMONY OR BISMUTH

By D. J. Demorest

Received November 1, 1912

Ores containing arsenic, antimony or bismuth have been best analyzed for copper by the iodide method, for the copper must be separated from these elements before it can be accurately determined electrolytically. To separate the copper from these interfering elements, by the old method of precipitating the sulfides, dissolving the arsenic and antimony sulfides in sodium sulfide and then the copper sulfide in potassium cyanide is tedious and troublesome. The following method greatly reduces the time required and gives very accurate results.

It has long been known that under the proper circumstances copper can be precipitated free from arsenic, antimony or bismuth as CuCNS. Guess's permanganate method is based upon this precipitation. However, owing to the solubility of the CuCNS as ordinarily precipitated, a considerable correction factor was necessary.

While working on the analysis of Babbitt metals high in antimony and tin, the writer observed that the copper was apparently entirely precipitated as CuCNS from a solution containing ammonium sulfate, ammonium tartrate and tartaric acid. When the method was applied to the analysis of impure ores, it proved successful. It is as follows:

One gram, or more if the sample is low in copper, is treated with 7 cc. of HCl and 7 cc. of HNO_3. The solution is heated until the ore is decomposed, then 5 cc. of sulfuric acid are added and the liquid evaporated to copious fumes of sulfuric acid. Now 30 cc. of water containing 3 grams of tartaric acid are added, and the solution is heated until all soluble salts are dissolved; then it is cooled somewhat and ammonia is added until alkalinity is reached. Now H_2SO_4 is added until the solution becomes acid, and then one cc. more. To the solution, which will now be hot, is added 2 grams of sodium sulfite, and the solution is stirred until it is all dissolved. Then 1 gram of KCNS dissolved in a few cc. of water is poured in. The copper precipitates immediately as CuCNS. The solution is stirred vigorously and heated to boiling; then the precipitate is allowed to settle for a few minutes. The precipitate is very dense. The solution is filtered through a dense filter and washed several times with a solution containing about 1 per cent. of KCNS and the same amount of tartaric acid.

Onto the filter paper, which should be held in a rather large funnel, is poured 18 cc. of 1 : 2 nitric acid and the funnel is immediately covered with a watch glass to prevent loss by spraying, due to the vigorous action of the nitric acid on the CuCNS. When the acid has run through, the filtrate is heated to boiling and poured through again; the paper, funnel and watch glass are washed well with water, care being taken to keep the volume of the filtrate as small as possible. The solution is now heated to vigorous boiling for five minutes to destroy the HCNS, water is added to make the volume about 125 cc., and the copper is precipitated by electrolysis, using a gauze cathode, a drop of potential of three volts and a current of three amperes. Precipitation should be complete in from thirty minutes to an hour. The copper should precipitate in a beautiful and crystalline condition. If it does not the solution was not boiled long enough.

If the ore contains lead, some will precipitate when the sodium sulfite is added, but this does no harm. The tartaric acid prevents the precipitation of antimonic acid.

The sulfocyanic acid must be completely destroyed before electrolysis or the copper will come down in a spongy and non-adherent form. Instead of dissolving the CuCNS in nitric acid, the precipitate and filter paper may be ignited in a porcelain crucible until all carbon is burned off and the CuO dissolved in 5 cc. of nitric acid, diluted and electrolyzed.

The following are some results obtained with the method:

Grams of Metals Present						Grams Copper found
Copper	Arsenic	Antimony	Bismuth	Iron	Lead	
0.1000	0.07	0.07	0.100	0.100	0.100	0.0997
0.1000	0.07	0.07	0.100	0.100	0.100	0.0999
0.1000	0.07	0.07	0.100	0.100	0.100	0.0995
0.1000	0.07	0.07	0.100	0.100	0.100	0.1000
0.1500	0.07	0.07	0.100	0.100	0.100	0.1495
0.1500	0.07	0.07	0.100	0.100	0.100	0.1495
0.1500	0.07	0.07	0.100	0.100	0.100	0.1500
0.1500	0.07	0.07	0.100	0.100	0.100	0.1500
0.1500	0.07	0.07	0.100	0.100	0.100	0.1499

It is the writer's belief that this is the most accurate and usable electrolytic method for copper on ores containing arsenic, antimony or bismuth.

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AN ELECTROLYTIC METHOD FOR THE DETERMINATION OF TIN IN CANNED FOOD PRODUCTS

By Allerton S. Cushman and Everett B. Wettengel

Received November 18, 1912

The issuance by the United States Department of Agriculture under date of September 30, 1910, of Food Inspection Decision, No. 126, governing salts of tin in food, which allows no more than 300 mg. of tin to the kilogram of material, has been the occasion of a number of laboratories being called upon to make a special study of the subject. These investigations have necessitated thousands of determinations of tin in every conceivable form of canned food. The usual analytical method for estimating very small quantities of tin contained in foods is difficult, tedious, and requires the evaporation of from 150 to 200 cc. of strong acids. When a considerable number of determinations have to be made, the question of a hood and draft, which will take care of such large quantities of acid fumes, presents a very difficult problem.

The gravimetric method as used in the laboratories of this Institute is briefly as follows: The entire contents of a can is pulped by maceration in a large porcelain mortar: 50 grams of the sample are transferred to a 9-inch evaporating dish. From 50–150 cc. of strong nitric, and from 20–50 cc. strong sulfuric acid are added, according to the nature of the sample. (Beets, spinach, asparagus, etc., require less acid than beans, sweet potatoes, corn, or fruit, to which a large amount of sugar has been added.) The mixture in the evaporating dish is covered with a watch glass and heated over a strong Bunsen flame until sulfuric acid fumes begin to come off. Nitric acid is added repeatedly until the solution is colorless. The solution is diluted with a large volume of water and neutralized with strong ammonium hydroxide, making a total volume of 300–500 cc. The solution is made slightly acid with HCl, transferred to a flask and a slow stream of H_2S passed through it for two hours. The flask is heated on the water bath for an hour or allowed to stand over night to settle the tin sulfide. The solution is then filtered and the precipitate washed alternately with a solution of ammonium acetate and water. The tin sulfide is dissolved through the filter with three successive portions of yellow ammonium sulfide and reprecipitated with glacial acetic acid. The tin sulfide together with some precipitated sulfur is filtered on a weighed gooch, dried in a water oven, ignited over a Méker burner, and weighed as stannic oxide, SnO_2. Factor for metallic tin, 0.7882.

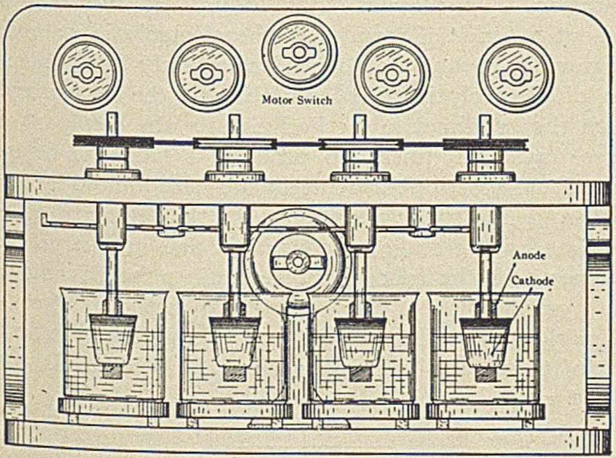

Fig. 1—Front Elevation

If occasion requires, a single determination can be rushed through by the above method in one working day, but in the usual routine in which the samples are worked in batches, from two to three days are consumed in getting out results. The hoods and flues suffer great damage from the acid fumes and the cost of the acids consumed in total destruction of the organic matter is very high.

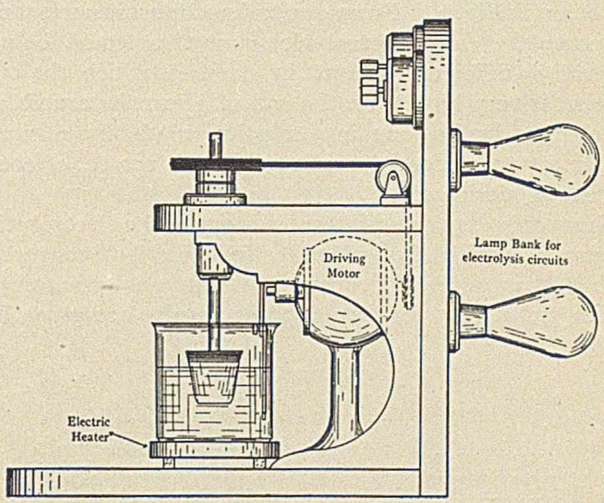

Fig. 2—Side Elevation

These objections to the current method of analysis led us to investigate all possible ways of shortening and improving the method, with the principal object in view of avoiding the destruction of the organic matter by long digestion in nitro-sulfuric acid. In the preliminary experiments, the expedient was tried of cooking the slightly acid sample in an aluminum pan with the object of separating the tin from the organic matter preliminary to its re-solution in stronger acid and preparation for separation by electrolysis. It was believed that this method would aid in the separation of tin from the organic colloids in which it was held, but subsequent experiment showed that the preliminary cooking in an aluminum utensil was unnecessary and the simple method as finally worked out is as follows: 50 grams of the pulped material are placed in a 600 cc. beaker and brought to a slow boil with 50 cc. of concentrated hydrochloric and 25 cc. of nitric acid. The mixture is stirred continuously and the boiling continued five minutes unless there is danger of foaming, in which case the flame is removed and the material allowed to digest for ten minutes. The solution is then diluted with about an equal quantity of water, made alkaline with strong ammonia and 25 cc. of saturated ammonium sulfide. The mixture is digested for a few minutes with thorough stirring, and all insoluble organic matter filtered out on a ribbed filter. The washing is done with boiling water containing a little ammonium sulfide. About 150 cc. of wash water are used in five separate washings, making the total solution to 400 cc. The solution is then electrolyzed hot, using 1.5 amperes at 6 volts. A rotating cathode is used. The end of the revolving spindle carries a rubber stopper over which a clean weighed platinum crucible is slipped. The apparatus which has been found convenient for carrying out the electrolysis of a number of determinations at the same time is shown in front elevation in Fig. 1 and in side elevation in Fig. 2. From one to four hours is necessary to complete an electrolytic run, two hours being generally sufficient, except in cases in which the tin content is very high. At the end of a run the crucibles are cleaned by heating in a solution made by mixing 100 cc. of 10 per cent. oxalic acid with 100 cc. of concentrated nitric acid.

Numerous experiments were made with the electrolytic apparatus which showed that tin could be recovered with a fair degree of accuracy from an ammonium sulfide solution. Some of these results are given in Table I.

TABLE I

Sample No.	Tin added mg	Tin found mg	Time taken
1.............	1.0	1.1	1 hour
2.............	1.0	1.0	1 hour
3.............	10.0	9.8	$2^1/_4$ hours
4.............	10.0	9.9	$2^1/_4$ hours
5.............	10.0	10.0	$2^1/_3$ hours
6.............	10.0	10.0	$2^1/_3$ hours
7.............	25.0	24.6	$2^3/_4$ hours
8.............	50.0	50.5	4 hours

The next step was to study the effect of organic food material on the method. Tomatoes were put into glass bottles with 50 cc. of a ten per cent. sodium chloride solution, and 10 mg. of tin in solution were then added to each bottle. The bottles were corked and autoclaved under pressure in a manner similar to the processing of canned foods. The results of analysis are given in Table II.

TABLE II

No.	Tin added mg.	Tin recovered mg.	Time of electrolysis
1..............	10	9.9	2 hours
2..............	10	9.2	2 hours
3..............	10	10.1	2 hours
4..............	10	9.9	2 hours

In run No. 2 the cork blew out of the bottle during processing and it was known that a slight amount of the contents had been lost. These results were very encouraging as the determination of small amounts of tin in the presence of an excess of organic food material must be but a fair approximation of the truth at the best. The next step was to try the method on a large variety of food products. The results of a number of duplicate runs are given in Table III.

TABLE III

MATERIAL	Tin found Mg. per kilo		Time, hours
	Run I	Run II	
Apple butter A........................	178	180	3
Apple butter B........................	142	142	2.5
Apple butter A........................	168	168	2.5
Apple butter B........................	142	142	2.5
Blackberries........................	28	26	2.5
Rhubarb A........................	170	174	2.5
String beans........................	72	64	2.5
Beef soup(a) (solid)...................	18	22	2.5
Tomato soup........................	74	74	2.5
Bouillon soup........................	28	26	2.5
Oxtail soup (solid)...................	66	76	2.5
Vegetable soup (solid)................	22	36	2.5
Mock turtle soup (solid)...............	20	20	2.5
Baked beans with tomato sauce..........	40	42	2.5
Baked beans without tomato sauce.......	8	10	2.5

(a) All the soups except the bouillon and tomato were solid and contained large amounts of meat and vegetables.

The results given in Table III show that it is possible to produce satisfactory check results when working in duplicate by the method, and, in fact, the checks are generally much closer than can be expected when working with the longer gravimetric method. In each analysis the insoluble residue, consisting of food pulp, was carefully examined for tin, but if any was present it was in such small amount that it could not be found by qualitative tests.

In Table IV the results are given of a series of analyses of various foods when the gravimetric and electrolytic methods were both used.

TABLE IV

MATERIAL	Tin found Mg. per kilo	
	Electrolytic method	Gravimetric method
Tomato soup........................	145	130
Rhubarb........................	112	102
Squash........................	316	314
Spinach........................	35	22
String beans (liquid portion)...........	94	70
String beans (solid portion)............	340	356
String beans (total)..................	229	226
Beets (solid portion)..................	70	27(a)
Cuthbert raspberries..................	248	222
Sweet potatoes........................	40	39
Pork and beans........................	64	73
Sauer-kraut........................	12	13
Apple-butter........................	142	112
Blackberries........................	27	17

(a) Result probably low.

An inspection of Table IV shows a generally satisfactory agreement between the results obtained by the two methods, and it should be stated that these are not selected results but are given directly in the order in which they were obtained by two different workmen. Only about three hours are required to get out a series of results by the electrolytic method, the number in the batch being limited only by the number of electrolytic cells available in the laboratory. With the gravimetric method usually the best part of three days is required to produce a batch of determinations, the number being in this case limited by the hood and flue space available. In the saving of time, acids and other reagents consumed and in destruction to flues and the laboratory generally, the advantage is all on the side of the electrolytic method. If considerable work of this kind has to be done, the first cost of the electrolytic apparatus is fully justified.

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WASHINGTON, D. C.

POTASSIUM PERMANGANATE IN THE QUANTITATIVE ESTIMATION OF SOME ORGANIC COMPOUNDS[1]

By C. M. PENCE

Potassium permanganate has been most generally used in the volumetric estimation of iron. Some uncertainties formerly existed since it was impossible to obtain a chemically pure article and insufficient data were

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[1] Read before the Indiana Section of the American Chemical Society, May 10, 1912.

at hand as to proper methods of preparation and standardization of its solutions.

At present, these objections have been largely overcome and almost all of our text-books on quantitative analysis contain an extended treatise on proper means of preparation and standardization of volumetric permanganate solutions.

One of the most commonly known organic compounds that is quantitatively determined by the use of volumetric permanganate is oxalic acid. Now oxalic acid and iron are determined in acid solution, but the procedure most applicable for the oxidation of all types of aromatic compounds as well as carbohydrates and hydrocarbons is with alkaline permanganate. Oxidations in acid solution are less energetic than those with alkaline $KMnO_4$ and in the latter case the final product of a completed decomposition of the organic compound is oxalic acid instead of CO_2 and H_2O.

Among the substances mentioned in the literature[1] as being oxidized to oxalic acid are propylene, isobutylene, amylene, acetone, fatty acids; butyric, lactic, succinic, and tartaric acids; dextrose, sucrose, glycerol and phenol. Now when an organic compound is oxidized to oxalic acid, a further oxidation to CO_2 and H_2O readily follows upon acidifying and warming the solution. Such a procedure forms the nucleus of a method for the determination of the compounds previously enumerated. Tocher made use of this method and found that phenol could be determined. His method was substantially as follows: Dissolve 1 gram phenol in 1000 cc. distilled water and take 10 cc. for titration. Add 3–4 grams $NaHCO_3$ together with a little distilled water. Then add 50 cc. $KMnO_4$ and boil for five minutes. Set aside to cool and gradually add dilute H_2SO_4 to decided excess; warm to 60° C. and titrate the excess of $N/10$ $KMnO_4$ with $N/10$ oxalic acid.

This method was found to be open to the following objection: that the manganese dioxide formed as a result of the action of $N/10$ $KMnO_4$ upon the phenol did not reduce readily enough with consequent solution upon direct titration with $N/10$ oxalic acid. Thus the solution was full of oxide which not only obscured but rendered the end point of little value, in that the oxide was not completely reduced before the permanganate end point was obtained.

The following modification of Tocher's method was found to give good results:

Dissolve 0.4 gram phenol in 1000 cc. distilled water. Place 50 cc. $N/10$ $KMnO_4$ and 3 to 4 grams $NaHCO_3$ in a 500 cc. glass-stoppered Erlenmeyer flask. Add 25 cc. of the phenol solution with gentle rotation. Boil 5–10 minutes (with stopper removed). Cool flask to about 60° C. Acidify with dilute H_2SO_4, let stand about 2 minutes; cool to room temperature. Dilute with distilled water, add 5 cc. 20 per cent. KI and titrate the liberated iodine with $N/10$ thiosulfate solution, using starch as indicator. The number of cc. of $N/10$ thiosulfate subtracted from the number of cc. $KMnO_4$ originally added = no. cc. of $KMnO_4$ consumed by the phenol.

1 cc. $N/10$ $KMnO_4$ = 0.000336 gram phenol.

If a glass-stoppered Erlenmeyer flask is not available an ordinary Erlenmeyer may be used and its contents transferred to a glass-stoppered bottle before acidifying. Any oxide adhering to the Erlenmeyer is easily removed by the addition of a little distilled water acidified with H_2SO_4 and containing a few drops of 20 per cent. KI.

In considering the nature of the oxidation with $KMnO_4$ in acid and alkaline solutions it is observed that each molecule of $KMnO_4$ in acid solution liberates 2.5 atoms of oxygen according to following equation:

$$2KMnO_4 = K_2O + 2MnO + 5O$$

or

$$2KMnO_4 + 3H_2SO_4 = K_2SO_4 + 2MnSO_4 + 3H_2O + 5O$$

Now in alkaline solutions the two molecules of MnO are immediately oxidized to 2 MnO_2 at the expense of 2 atoms of oxygen, so that we actually have

$$2KMnO_4 = K_2O + 2MnO_2 + 3O$$

Hence, for each molecule of $KMnO_4$ used, only $1^1/_2$ atoms of oxygen are available for our oxidation process. This fact must be recognized in providing sufficient $KMnO_4$ to readily complete the oxidation process, and it would necessarily enter into a calculation of the value of $N/10$ $KMnO_4$ in terms of phenol if the MnO_2, or rather its hydrated form, were filtered from the solution before acidifying and adding the KI. But since the procedure is not lengthened by a filtration the MnO_2 is reduced to its manganous form with the liberation of free iodine, and we must calculate our factor by considering that reaction proceeds as in acid solution with 2.5 atoms of oxygen available per molecule of $KMnO_4$ although such is not literally the truth.

To completely oxidize phenol, 14 atoms of oxygen are required, according to the following equation:

$$C_6H_5OH + 14O = 6CO_2 + 3H_2O$$

Since only 2.5 atoms of oxygen are available per molecule of $KMnO_4$ then 5.6 molecules of $KMnO_4$ would be required for every molecule of phenol and the factor for $N/10$ $KMnO_4$ in terms of phenol becomes:

$$\frac{\text{Mol. wt. phenol}}{2.5 \times 2 \times 10 \times 5.6 \times 1000}$$

Experiments with a phenol solution containing 0.0005 gram of phenol, per cc. as determined by the Koppeschaar bromine method, resulted as follows:

Exp. No.	Grams phenol taken	Per cent. phenol found
1	0.0050	99.65
2	0.0050	100.18
3	0.0075	99.65
4	0.0076	99.86
5	0.0100	99.86
6	0.0100	99.72

Now when the cresols were run in the same manner as phenol it was found that they were not completely

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[1] "Oxidation of Organic Compounds with Alkaline Permanganate," Eduard Donath and Hugo Ditz, *J. prakt. Chem.*, [2] **60**, 566–576 (1899; through *J. Chem. Soc.*, [1] **78**, 197 (1900). "Contribution to the Knowledge and Determination of the Carbohydrates," J. König, W. Greifenhagen and A. Scholl, *Z. Nahr. Genussm.*, **22**, 705–723; through Abstr. *J. Am. Chem. Soc.*, **6**, 901 (1912). "Volumetric Determination of Phenol," Jas. F. Tocher, *Pharm. J.*, **66**, 360.

oxidized and that they varied slightly as to the rate with which oxidation proceeded; hence, any permanganate method for their accurate determination must depend upon definitely fixed conditions.

Likewise, it was obvious that commercial creosote and guaiacol could not be determined by this procedure, since they are mixtures of several more or less related phenols that are not present in like proportion in different specimens. However, with single solutions of several common phenols and closely related compounds, fairly gratifying results were obtained. Pyrogallol, pyrocatechin, resorcinol and hydroquinone, from all of which the CH_3 group is absent, were readily and completely oxidized.

Benzoic acid was very slightly attacked while under similar conditions salicylic acid and salol were completely oxidized. Thus it would seem that the phenolic OH group predisposes towards a complete oxidation and that many uninvestigated phenols and closely related compounds would give analogous reactions. In making up solutions of the several phenols, sufficient $N/2$ NaOH was added when necessary, to insure ready solution.

The following table is self-explanatory:

SUBSTANCE	PERCENTAGE FOUND	SUBSTANCE	PERCENTAGE FOUND
Pyrogallol	100.4 100.1	Hydroquinone	99.49 99.56
Pyrocatechin	100.2 99.9	Salicylic acid	99.79 100.2
Resorcinol	100.5 100.4	Salol	99.77 99.91

The alkaline permanganate method is especially applicable for the quantitative estimation of the above compounds when they occur individually in very small amounts in single solutions or in conjunction with substances not readily oxidized.

DEPARTMENT OF CHEMICAL RESEARCH
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SOME PROPERTIES OF KOJI-DIASTASE

By G. KITA

Received September 18, 1912

The properties of the saccharifying enzyms of koji (a culture of aspergillus oryzae on steamed rice) have been studied by many investigators and are supposed to be comparatively well understood. There are, however, certain obscurities which exist in regard to the identification and classification of these enzyms. They must be cleared up on account of their scientific and practical importance.

I have lately been occupied in a series of investigations relative to Soya or Shoyu (Japanese sauce fermented from beans, wheat and salt) making, and incidentally cleared up certain of the above points.

1. IS SACCHARIFICATION OF STARCH BY KOJI-DIASTASE CARRIED OUT BY TWO DIFFERENT ENZYMS, DIASTASE (AMYLASE) AND GLUCASE, SUCCESSIVELY?

It has been generally thought that koji contains two kinds of saccharifying enzyms, *viz.*, diastase (amylase) and glucase, and it is supposed that the glucose present in a liquid saccharified by means of koji is produced by the two enzyms above mentioned. It has been affirmed by some investigators that while the glucase present in koji does not act rapidly, yet, the sugar finally produced in the saccharification of starchy matters with koji is invariably glucose, and that maltose is present only in a negligible quantity. Evidence in support of the above assertions is still meager and I was led to think that there may be present in koji another new enzym, which is altogether different from glucase, and which can convert starch directly to glucose without intermediate steps. The following experiments were made:

Experiment 1.—To each 200 cc. of a 2 per cent. solution of maltose (moisture = 4.25 per cent.) and also starch (moisture = 16.99 per cent.), 20 cc. of the solution of enzyms (corresponding to 2 grams of koji) were added. After allowing the enzyms to act for three hours at 50° C., the reducing action of the digested liquid on Fehling's solution was tested and a rough estimation of glucose was made by means of the glucosazone reaction.

In the determination of the osazone, 100 cc. of each liquid were boiled $1^1/_2$ hours with 4 grams of phenylhydrazine hydrochloride and 6 grams of sodium acetate, filtered and washed with 100 cc. boiling water. This, of course, cannot be said to be a perfect method either for the isolation or determination of glucosazone, but is sufficiently accurate for comparison of results.

Solution acted upon by enzyms	Starch	Maltose
Cc. saccharified solution required to reduce 50 cc. Fehling's solution	18.6	21.0
Grams of glucosazone obtained from 100 cc. of saccharified solution	0.2575	0.1164

Experiment 2.—A like experiment was performed with Taka-diastase. In this case each 100 cc. of a 2 per cent. solution of maltose and starch, respectively, was hydrolyzed with 20 cc. of a 1 per cent. solution of the enzym. For the determination of glucosazone 50 cc. of each solution were taken:

Kind of solution acted upon by enzyms	Starch	Maltose
Cc. saccharified solution required to reduce 50 cc. Fehling's solution	30.0	23.6
Grams of glucosazone obtained from 100 cc. of saccharified solution	0.2088	0.0772

As will be seen in the above experiment, the reducing power of the starch solution after digestion is so strong that we can hardly compare the rate of hydrolysis of maltose to that of starch. Moreover, the quantity of glucosazone from the starch solution is very much higher. The presence of dextrine in the production of glucosazone does not seem to possess remarkable influence, but the presence of maltose has a tendency to give an increased quantity of glucosazone when phenylhydrazine hydrochloride is used in excess. Therefore, in the hydrolysis of starch by means of koji-diastase, it is evident that glucose is not produced principally by the action of glucase, but directly without the aid of it. Dr. Nishisaki[1] asserts that in the case of saccharifying starch by means of koji, the sugar produced thereby is composed in its first stage, of maltose, on account of the weak activity

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[1] *J. Chem. Soc., Tokyo*, **29**, 325.

of glucase. But this must have arisen from an error because of his presumption of the presence of malt-diastase in koji.

II. INFLUENCE OF SALT ON ENZYMS IN KOJI

It is a well-known fact that enzymic activity is lost by heating, but so far as I know the effect of heating it in the presence of salt has not yet been investigated. In the soya mash, enzyms act in salt solution and it is, therefore, necessary to find out the relation of enzyms toward heat in the presence of salt. With this object in view, I made the following experiments which have fortunately proved that NaCl has a protecting influence on koji-diastase but not on malt-diastase; while Na_2HPO_4, asparagin, and H_2SO_4 have an injurious effect even on koji-diastase.

Experiment 3.—One hundred cc. of a 2 per cent. starch solution, taken in each of three different vessels, were digested for two hours at 50° C. with 5 cc. solution of enzyms (100 grams koji or malt macerated in 1,000 cc. water for 10 hours), into two of which 20 grams of NaCl were added in two ways: one from the beginning and the other after digesting for three hours. The remaining one was kept blank for control.

No. of cc. of the Saccharified Solution Used for the Reduction of 50 cc. Fehling's Solution

No.	Kind of digestion	Enzymic solution from koji	Dried malt	Green malt
1	Not heated	50.2	41.4	25.2
2	Heated 3 hrs. at 50° C. and added NaCl	67.6	90.0	25.2
3	Heated 3 hrs. at 50° C. after addition of NaCl	55.6	over 200.0	57.4

Experiment 4.—The experiment was carried as in Experiment 3, except that Na_2HPO_4, asparagine and H_2SO_4 were used instead of NaCl. For 100 cc. koji extract, 5 grams Na_2HPO_4, 5 grams asparagine or 14 cc. of 1 per cent. H_2SO_4 solution were added.

No. of cc. of the Saccharified Solution used for the Reduction of 50 cc. Fehling's Solution

No.	Kind of digestion	Enzymic solution with asparagine	Na_2HPO_4	H_2SO_4
1	Not heated	35.2	57.0	87.5
2	Heated before addition	44.5	65.4	100.0
3	Heated after addition	92.5	112.4	140.0

III. THE INFLUENCE OF SALT ON SACCHARIFYING ACTIVITY OF KOJI-DIASTASE

The results obtained by previous investigators with regard to the influence of salt, acid, etc., on the saccharification by malt- and koji-diastase agree on the point that a very small quantity of these substances acts favorably, but that increase of the same is injurious.

Among various authors, Dr. Saito[1] says that the activity of koji-diastase is injured by the presence of salt, for instance, in a ten per cent. salt solution its activity is lessened to 11.3% and in twenty per cent. solution to 7.6 per cent. S. Mori,[2] however, says that a large quantity of NaCl gives no inhibitory influence on koji, but he attributed this observed influence to a peculiarity of some kinds of koji. His result is inaccurate since a development of organisms took place in his experiment. At that time, I[1] also observed the similar curious phenomenon which led me at once to entertain an opinion like Mori. Men employed in soya-making alleged that in practice where a considerable quantity of salt is present in mash, no retarding of enzymic action is noticed. Such diversity of opinion was found lately to have originated in the fact that when the enzymic solution is concentrated a large quantity of sodium chloride does not apparently exert an inhibitory influence, while in a less concentrated enzymic solution even a smaller quantity of salt will have a very injurious action. In general, the influence of salt on diastase is related to the concentration of the enzym solution. The following experiments show this relation clearly:

Experiment 5.—One hundred cc. of a 20 per cent. soluble starch solution with or without 40 grams NaCl were saccharified with 100 cc. koji extract (200 grams wheat koji extracted in 1000 cc. water) and were filtered after 16 hours standing at 50° C. The iodine reaction of the solution without salt was purple or light red after 10 or 30 min., respectively, while that of the salt-containing solution was somewhat deeper in color. After 30 min. 5 cc. concentrated caustic alkali solution were added in order to inhibit further diastatic action and then sugar was determined after diluting the solutions to five times their own volume. To the digestion which did not contain NaCl the same amount of NaCl was added as was in the other, so that the conditions might be similar in conducting the sugar determinations.

No. cc. of the Diluted Sugar Solution to Reduce 25 cc. Fehling's Solution

Digestion with NaCl	Digestion without NaCl
16.5	15.0

Experiment 6.—One hundred cc. of a 2 per cent. or 20 per cent. soluble starch solution were saccharified with 10 cc. of the koji extract at 50° C. wherein 20 grams NaCl were added before or after digestion. After the digestion was over, NaCl was added to the one which did not contain the same, as was done in the previous experiment.

The reaction of iodine toward these liquids was tested and the following result was obtained:

Duration of digestion in minutes	2 per cent. starch solution		20 per cent. starch solution	
	Digestion with NaCl	Digestion without NaCl	Digestion with NaCl	Digestion without NaCl
20	purple	red	blue	blue
30	red	red	blue	blue
60			purple	purple
120			red	red

Similar digestion was repeated after 30 minutes; 5 cc. caustic alkali solution were added in one case, and in the other after 120 minutes, so that further diastatic action is entirely arrested. Then, diluting the digestion in case of 30 minutes duration to five times and in case of 120 minutes duration to ten times its original volume, it was tested how many cc. of such dilution can completely reduce 25 cc. of Fehling's solution.

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[1] *Centrbl. Bakt., II Abt.*, **17**, 102.

[2] *J. Pharm. Soc., Tokyo*, **311**, 1–10.

[1] *J. Chem. Soc., Tokyo*, **13**, No. 145, pp. 221–235.

No. cc. of the Digestion to Reduce 25 cc. Fehling's Solution

30 minutes digestion		120 minutes digestion	
with NaCl	without NaCl	with NaCl	without NaCl
23.2	17.5	45.5	30.3

Experiment 7.—Two grams soluble starch in 500 cc. water with different quantities of NaCl were saccharified with 10 cc. koji extract at 50° C. After 30 min., 10 cc. alkali solution and NaCl were added so that each solution contained 100 grams NaCl in total solution.

Reactions toward Iodine

No. of solution	No. of grams of NaCl added before reaction	After 10 min.	After 30 min.
1	0	red	red
2	20	purple	red
3	100	purple	purple

No. of solution	No. Cc. of sugar solution to reduce 5 cc. Fehling's solution
1	17.0
2	26.0
3	45.0

In Nos. 5 and 6 we see that the influence of NaCl depends upon the concentration and not on the ratio of starch to the enzymic quantity. The more dilute is an enzymic solution the stronger is the inhibitory action of sodium chloride.

From Exps. 6 and 7 it is clear that the inhibitory action of NaCl does not relate to dilution when the proportion of NaCl to enzym is constant.

IV. CAN KOJI-DIASTASE BE PRESERVED IN BRINE?

The stability of the koji-diastase in brine is not yet well known and it is a very important factor in soya making, where a long period of activity is necessarily maintained. I have kept diastase in brine for a month in summer and compared it with that kept in a dried condition in a desiccator. The result was as follows:

Experiment 8.—One hundred cc. of a 2 per cent. starch solution was saccharified for 3.5 hours at 50° C. with an enzymic solution (1 gram Taka-diastase, 15 grams NaCl in 100 cc. water), and its reducing power was determined.

No.	Preservation of Diastase	No. cc. of saccharified solution to reduce 25 cc. Fehling's solution
1	In brine....................................	30.8
2	In dry condition with envelope of black glazed paper	28.3
3	In dry condition in thin layer exposed to sunlight....	33.4

It will be seen from these results that the diastatic power of the enzym in brine was stronger than that which was exposed to sunlight. The gelatine liquifying power of the same diastase was in the same order. From this and other data I have, I believe the strong diastatic power exerted in the old mash which was kept about a year was principally due to the koji originally used.

SUMMARY

I. Though koji may contain two different saccharifying enzyms, *viz.*, amylase and glucase, the total quantity of the glucose in a saccharified solution could not be produced from maltose by the action of glucase alone; hence, koji must contain some other kind of diastase which produces glucose directly from starch without the aid of glucase.

II. Common salt has a protecting action on koji-diastase under heating but not on malt-diastase, while Na_2HPO_4, asparagine and H_2SO_4 impair its activity more quickly.

III. The inhibitory action of salt on koji-diastase has a certain relation to the concentration of diastase. In a dilute enzymic solution it is very strong but not in a concentrated enzymic solution. It is therefore necessary in every case to note concentration of diastase when we consider the influence of some salt upon it.

IV. The activity of koji-diastase is conserved in brine for a long period.

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APPLICATION OF THE AMMONIUM CARBONATE METHOD FOR THE DETERMINATION OF HUMUS TO HAWAIIAN SOILS[1]

By J. B. Rather

Received November 12, 1912

The ammonium carbonate method for the determination of humus in soils[2] has been used on a number of samples of Texas soils, of different types met with in the United States, including the Travis, Orangeburg Houston, Laredo, Norfolk, Lufkin and Cameron series, and in every case the results have been entirely satisfactory, the "ash" averaging less than 0.40 per cent. While this method has proved to be reliable in this laboratory, it has been shown by Kelley and McGeorge[3] that on certain soils of Hawaii the method does not give good results. This is due to the nature of the clay and not to the amount of clay present, for we have precipitated the clay completely in a solution containing 33.45 per cent. "ash" as determined by the official method.

Kelley and McGeorge modified the ammonium carbonate method by adding 4 grams ammonium carbonate per hundred cubic centimeters instead of 0.5 gram. This coagulated the clay in most cases, but they claim that the organic matter was partially precipitated.

These investigators propose a method for the removal of the clay by filtration by means of reduced pressure through a Pasteur-Chamberland tube and washing with ammonia as was done by Alway, *et al.*[4] In connection with this it might be of interest to state that the filtration of humus solutions by reduced pressure has been done by Fraps and Hamner,[5] who themselves do not claim originality for the process. Fraps and Hamner state that the method is very tedious and that organic matter may not pass through the filter. Neither Cameron, who first used the porcelain tube, Fraps and Hamner, nor Alway, *et al.*, recommended the method.

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[1] Under the general direction of G. S. Fraps, chemist. Read at the meeting of the Association of Official Agricultural Chemists, Washington, D. C., September, 1912.

[2] This Journal, **3**, 660.

[3] *Ibid.*, **4**, 9, 604.

[4] Nebraska Exp. Sta., *Bull.* **115**.

[5] Texas Exp. Sta., *Bull.* **129**.

Although the types of soil such as are found in Hawaii may not occur in this country, on account of the simplicity and convenience of the ammonium carbonate method it was thought desirable to secure such a modification as would include such soils.

The soils used in this study were Hawaiian soils kindly furnished by Mr. McGeorge. After a number of trials to ascertain the most suitable conditions, the following method was adopted:

Prepare the solution as described in the official method, but decant the solution instead of filtering. Place 100 cc. in a graduated flask, add 2 grams ammonium carbonate, and heat on the steam bath for one hour. Allow to stand over night, pipette off 50 cc. of the supernatant solution, and evaporate to dryness in a tared dish. Dry three hours at 100° C., weigh, ignite, and weigh again. The loss in weight represents the humus.

The results for humus and humus ash are shown in the table along with the humus and ash by the official and original ammonium carbonate methods. All determinations here reported were made on the same solutions.

	No. 6185		No. 6187	
Method	Humus	Ash	Humus	Ash
Official	5.44	7.90	7.31	34.80
Ammonium carbonate	5.16	6.68	5.13	17.20
This modification	3.80	0.18	2.70	0.10
	3.68	0.40	2.75	0.18

The original ammonium carbonate method reduced the ash by about 15 per cent. in No. 6185 and by about 50 per cent. in No. 6187. This modification of the ammonium carbonate method reduced the ash in sample No. 6185 from 7.90 to 0.29 per cent., and in No. 6187 from 34.80 to 0.14 per cent.

The humus which was 5.44 per cent. in No. 6185 by the official method was reduced to 3.74 per cent. by this modification of the ammonium carbonate method, and in No. 6187 was reduced from 7.31 to 2.73 per cent. The differences do not represent humus, but are probably entirely due to the loss of water on ignition of the hydrated clay and oxides of iron and aluminum.

The solutions obtained by this modification were free from all turbidity. It was found that with these soils the clay could not be filtered off and the aliquots were therefore removed with a pipette. The introduction of the ammonium carbonate increases the volume of the solution, and after standing over night, it is about 100.5 cc. This would make the results about 0.01 per cent. too low (in percentage of humus in soil). Since this is well within the limit of error with these soils, it is disregarded.

The Hawaiian soils studied are said to be of volcanic origin and are very rich in iron. Although the original ammonium carbonate method gives unsatisfactory results with these soils, we believe soils of this type will rarely be met with in the United States, since as stated before the method has given entire satisfaction on a number of the soil types of this country.

SUMMARY

The ammonium carbonate method for the removal of clay from humus solutions has given uniformly good results on a number of the soil types of the United States, but on exceptional soils, like some of those of Hawaii, a slight modification of the method is necessary to remove the clay. The modification consists essentially in increasing the amount of ammonium carbonate to 2 grams per hundred cubic centimeters, and heating for one hour.

Texas Agricultural Experiment Station
College Station, Texas

LABORATORY AND PLANT

THE NEW HIGH FORM OF SULFURIC ACID CHAMBER

By F. J. Falding and W. R. Cathcart

Received January 20, 1913

Since the Sulfuric Acid Chamber-process has existed, the two important factors which have been uppermost in the minds of manufacturers, engineers, and chemists, are: *First*, economy of chamber space, which of course means economy of lead; and *Second*, economy of nitration. These economies have been accomplished in a very high degree and in a most satisfactory manner, by the use of a new form of lead chamber, which embodies a scientific principle heretofore ignored on account of a misconception of its importance.

HISTORICAL OUTLINE OF THE LEAD CHAMBER

The evolution of the Chamber-process is well known. At first intermittent—then continuous but without recovery of nitrogen oxides—next the addition of Gay-Lussac towers, and finally the Glover tower.

The chambers, however, have been always simply a series of enlargements of the flue or gas conduit connecting, at first, the sulfur burners with the outer air or exit, and later the Glover and Gay-Lussac towers. These enlargements of the flue retarded the speed of the gases, permitting them to mingle and react.

As the chambers have been almost universally framed with timber construction, the height and width have varied principally in conformity with considerations of carpentry, a width of thirty feet and a height of twenty-four feet having been rarely exceeded; whereas a length of one hundred to two hundred feet or over, for each chamber, used to be quite common. Lead chambers have been constructed empirically, and of many shapes and sizes. Sometimes the gas would pass through from four to ten chambers in series having a total length of from four hundred to one thousand feet in all.

The principal dimension of the chamber space has been always the horizontal axis in the same direction as the travel of the gas.

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[1] Covered by U. S. Patent No. 932,771 and Canadian Patent No. 124,132; owned by the Chemical Improvement Company, 220 Broadway, New York City. Also patented in England, Germany, France, Belgium and other countries.

It is an admitted observation that, irrespective of the length of the first chamber of a series, all the active work of such a chamber is performed within the first thirty or forty feet of its length. The remainder of the chamber is practically inactive, and reaction is not resumed until the gas has been again mixed by passing through a comparatively small flue or special apparatus in the nature of a surface condenser, when a new zone of activity is induced in a succeeding chamber. The condition existing in the first chamber is reproduced, but in a lesser degree of intensity, due to the diminished percentage of the sulfur dioxide content of the gas, as evidenced by a reduced temperature of the active zone. The same conditions with increasing reduction of activity apply to all subsequent chambers of the series, until the absence of sulfur dioxide in the final chambers sometimes actually permits the conversion of the nitrous gases into nitric acid and nitrogen peroxide, causing severe losses of niter and rapid destruction of the lead. It has even been the practice in some cases to admit a fresh supply of burner gas into the last chamber to counteract such conditions. (Benker.)

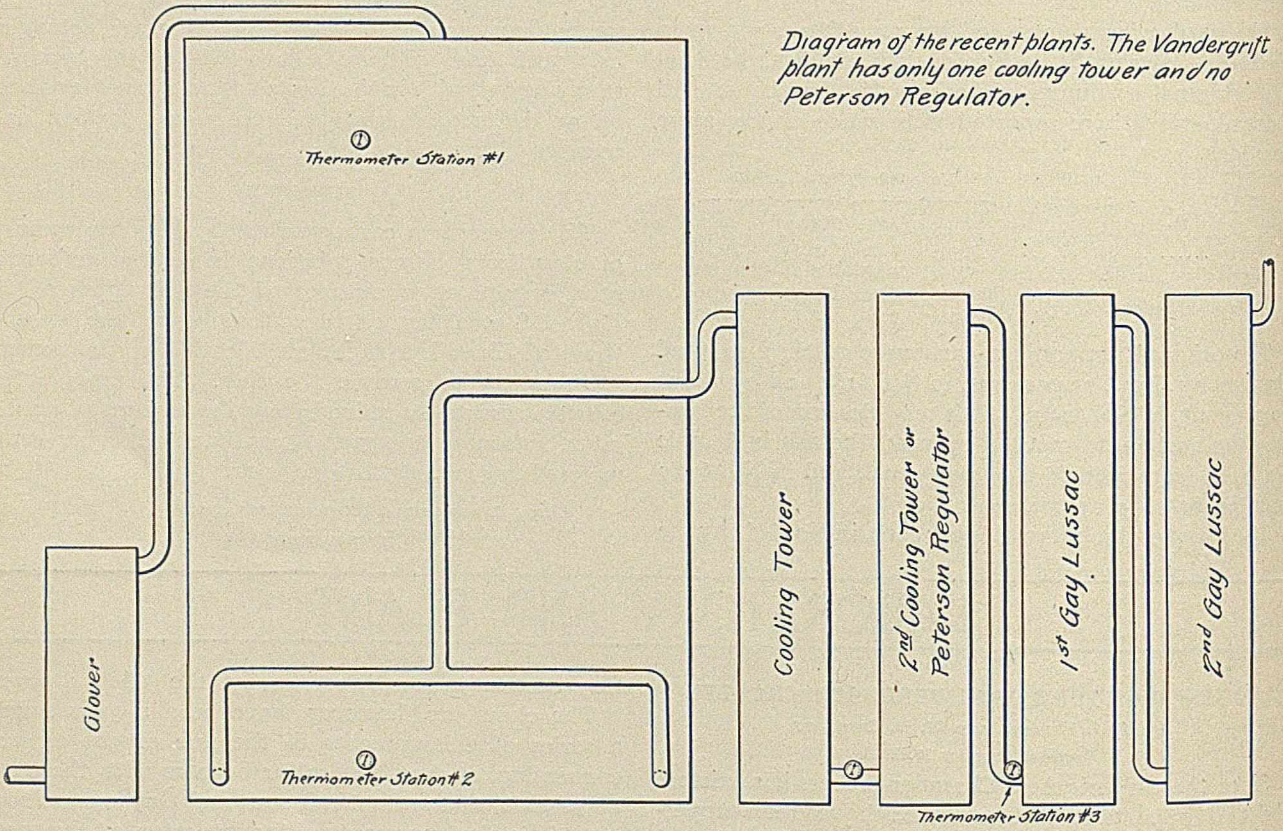

Diagram of the recent plants. The Vandergrift plant has only one cooling tower and no Peterson Regulator.

Mention should be made of the fact that, in earlier days, the chamber space consisted in some instances of a single, long, horizontal chamber, and, that the reaction was completed in this chamber, and to that extent was quantitative. This was accomplished, however, at so great an expense of chamber space relatively to the volume of gas reacting, that it was long since discontinued and a series of chambers adopted.

In recent years various attempts have been made to secure a quantitative reaction, or at least an intensive reaction in the first part of the chamber space. These attempts have been known as efforts "to reduce the chamber space." Among the schemes tried are the following:

(1) The French "systême intense," the principle of which is to increase the proportion of nitrous oxides to the sulfur dioxide in circulation—thereby increasing the speed of the initial reaction.

(2) Shortening the first chamber and passing the gas through surface or spray condensers before admission to succeeding chambers so as to increase the intensity of secondary reactions (Lunge plate columns, Gilchrist pipe columns, Hoffmann intensifiers, Pratt converters, etc.).

(3) Division of the initial volume of gas into two or more chambers in parallel instead of in series with a reduction of the secondary chamber space.

(4) The Meyer tangential admission of the gas into cylindrical chambers working on the assumption that the spiral passage of the gas near to the cool chamber sides would accelerate the reaction.

Most of these propositions have produced considerable economies but none have been entirely successful. The first reaction has not been quantitative and the inert gas leaving the first zone of reaction has contained sufficient unoxidized sulfur dioxide to make succeeding chambers, with further zones of reaction of decreasing intensity necessary.

The first considerable alteration in the shape of the lead chamber was introduced by Dr. Theodor Meyer in January, 1900. To use his own words, quoting from a pamphlet issued by him at Halle in 1907: "A tangent chamber is a hollow cylinder of approximately the same diameter and height, whose feed pipe is attached near the top in the direction of a tangent, and whose exhaust pipe is in the center of the floor of the chamber. An important factor in a large capacity is the height of the chambers (as I considered probably as early as 1900), for the highest capacity is shown by those two plants, whose chambers are highest: Griffith & Boyd, Baltimore, and Guilini Works, Ludwigshafen on Rh. (with 35 and 42 feet height). The height of the old oblong chambers has also been increased lately. Hartman & Benker built theirs up to 32.8 feet high. While this figure, however, is near the limit of profitable use of chamber-room (for oblong chambers), there is no such limit to the tangent chambers, for it is only necessary to keep width and height in proper proportion. If a chamber were to be changed into a tower the consequence would probably be a gradual retardation of the spiral-movement toward the lower part. The best ratio is an approximate equality of diameter and height."

ALGOMA STEEL CORPORATION, LIMITED—COKE PLANT DEPT., SAULT STE. MARIE, ONT.

Daily Report of Sulfuric Acid Plant, for 24 hours ending 7 A.M. Sunday, Dec. 29, 1912

Hour ending	Furnace Gas Per cent. SO_2 Station 1	Niter ($NaNO_3$) Pounds	Glover Bottom		Peterson regulator Percent. N_2O_3	Gay-Lussacs Percentage N_2O_3		Chamber						
								Temps. at stations, ° F.			Difference, ° F.			Drips
			° F.	° B.		No. 1	No. 2	1	2	3	1–2	2–3	1–3	° B.
8 A.M.	7.5	4	232	58	5.00	2.00	0.87	167	147	65	20	92	102	46½
9 A.M.		4	232	58	5.00	2.00	0.87	165	147	67	18	80	98	46½
10 A.M.	7.5	4	232	58	5.00	2.00	0.50	167	149	69	18	80	98	47
11 A.M.		4	236	58	5.00	2.00	0.50	168	150	69	18	81	99	47^2
12 Noon	7.5	4	237	58^2	5.00	2.00	0.50	170	153	69	17	84	101	47^2
1 P.M.		4	237	58^2	5.00	2.00	0.50	175	156	69	19	87	106	48
2 P.M.	7.5	4	237	58^2	5.00	1.86	0.50	176	158	69	18	89	107	48
3 P.M.		4	237	58^2	5.00	1.86	0.50	179	160	69	19	90	109	50
4 P.M.	7.5	4	237	58^2	5.00	1.86	0.50	180	160	70	19	90	109	50
5 P.M.		4	236	58^2	4.60	1.86	0.50	181	162	71	19	90	109	51
6 P.M.	7.5	4	236	58^2	4.60	2.00	0.45	183	162	72	21	90	111	49
7 P.M.		4	236	58^2	4.60	2.15	0.50	183	163	72	20	91	111	48
8 P.M.	7.5	4	236	58^2	4.60	2.15	0.50	183	163	72	20	91	111	48
9 P.M.		4	236	58	4.60	2.15	0.50	183	163	72	20	91	111	48
10 P.M.	7.5	4	237	58	4.60	2.15	0.50	184	164	73	20	91	111	48
11 P.M.		4	237	58	4.60	2.15	0.50	185	166	73	19	93	112	48½
12 Midnight	7.5	4	237	58	4.60	2.15	0.50	186	167	74	19	93	112	48½
1 A.M.		4	237	58	4.60	2.15	0.50	186	167	74	19	93	112	48½
2 A.M.	7.5	4	237	58	5.00	2.00	0.50	186	166	74	20	92	112	48½
3 A.M.		4	237	58	5.00	2.00	0.63	184	165	74	19	91	110	48½
4 A.M.	7.5	4	236	58	5.00	2.00	0.63	182	163	73	19	90	109	49
5 A.M.		4	235	58	5.00	2.00	0.63	175	159	72	16	87	103	48½
6 A.M.	7.5	4	235	58	5.00	2.00	0.63	174	158	72	16	86	102	48
7 A.M.		4	235	58	5.00	1.86	0.61	175	158	72	17	86	103	47
Total		96												
Average, 24 hours	7.5			58	4.85	2.02	0.60	178	159	71	19	88	107	48+

RAW MATERIAL USED

Name of ore—"Helen Mine."	Per cent. S—40.34
Pounds charged in 24 hours—20,700	Per cent. S available—40
Per cent. $NaNO_3$ on S—burnt, 1.16	Per cent. S in cinder—0.36 A.M., 0.4 P.M.

This proportion has been observed in all his installations. In the United States the Griffith & Boyd chambers are 33 feet diameter and 35 feet high and the chambers of the Mountain Copper Company in California are 39.4 feet wide by 42.6 feet high.

Again to quote from Dr. Meyer: "Aside from the small end chambers the most rational shape and size of a tangent chamber is a cylinder of 32 ft. to 48 ft. height and with a capacity of 28,000 to 72,000 cubic feet." This would give respectively a diameter of 33.5 feet and 44 feet.

As regards the Benker chambers, Lunge[1] quotes from a personal communication from Benker, who states he "employs only narrow chambers, say 18 to 20 feet wide, and from 25 to 33 feet high. Such chambers are, in the first instance, better adapted for water spraying, but they also afford a better mixture of the gases, especially if the sides are cooled, by employing an open bratticing for the side passages ($2^1/_2$ in. laths with $1^1/_2$ in. clear space) and a roof-rider. The cold gases descending along the sides must rise again in the center; but in the case of very wide chambers a dead space remains where the velocity is very slight, and where the mist of nitrous vitriol sinks down without acting on the gaseous constituents. Benker objects to Meyer's tangential chambers that there is no question of tangential action, that in the center of these circular chambers gases of different concentrations get mixed up, and that they would be too expensive if the only proper way was followed, *viz.*, building many small circular chambers in a set."

Neither in the case of Meyer or Benker does the question of the convection currents seem to have been seriously considered. Benker's chambers are of the long horizontal type 75 to 85 feet long and are arranged in a series of three chambers.

Meyer evidently considered that the question of height is merely a question of chamber space and says that "If a chamber were changed into a tower the consequence would probably be a gradual retarda-

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[1] "Sulfuric Acid and Alkali," Vol. I, 3rd Ed., page 459.

tion of the spiral movement toward the lower part." He therefore increases the diameter in proportion with the height.

In this short history of the lead chamber, it has not, of course, been possible to mention all the forms of chambers which have been used at different times, but it is submitted that they all contain the same defect, in that they practically ignore the convection currents of the reacting gases, that they are all virtually enlargements of the horizontal axis of the flue and that they all involve a series of reaction zones of decreasing intensity.

The practical ignoring of the laws of convection is responsible for the comparative failure of the above described forms of chamber construction.

THE THEORY OF THE HIGH CHAMBER

The technical gas mixture admitted to a chamber sulfuric acid system is diluted with a large percentage of nitrogen. This nitrogen takes no active part in the chemical reactions, but absorbs and carries away a large amount of the heat of the exothermic reactions taking place in the chamber.

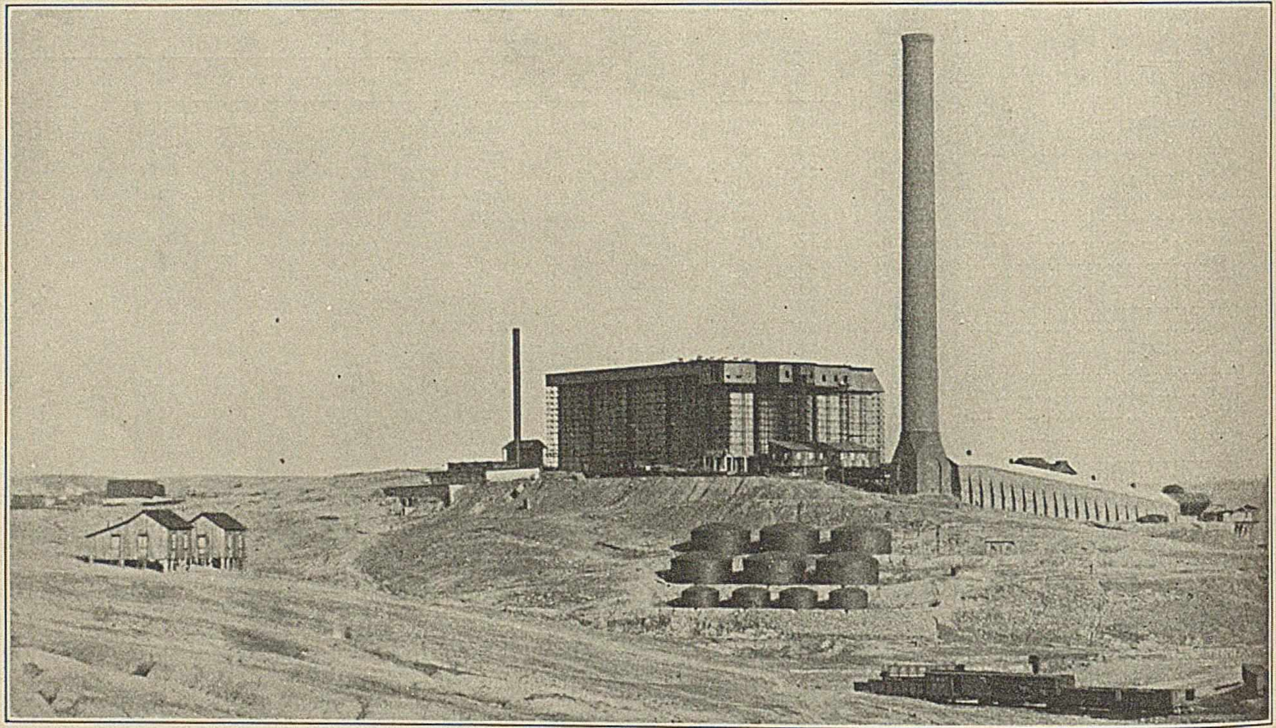
A good technical gas mixture, when produced from the oxidation of pyrite, will contain approximately, omitting minor impurities:

	Per cent. volume
Sulfur dioxide	7.5
Oxygen	9.0
Nitrogen	83.5
	100.0

Such a gas mixture will contain oxygen sufficient for the oxidation of the sulfur dioxide to the trioxide, and also provide the necessary excess of 5 per cent. of oxygen for the satisfactory operation of the chamber process. This 5 per cent. excess oxygen will be found in the exit gases.

During the passage of this gas mixture through the Glover tower the percentage of sulfur dioxide it contains is further reduced by partial oxidation to the trioxide (SO_3) and partly by the addition of nitrous oxides, and partly by water vapor derived from the evaporation taking place in the Glover.

The gas on leaving the Glover tower enters the lead chambers, and just at this point it is better fitted in every respect to react rapidly and quantitatively, than it will be at any subsequent stage of its passage toward the exit; and any means that can be devised for holding the active zone at this point, until the reaction is completed, and securing a rapid separation of the inactive from the active gas, permitting only the inactive gas to go forward to the Gay-Lussac towers and exit, will surely be advantageous.



TENNESSEE COPPER CO. ACID CHAMBERS, SHOWING STORAGE TANKS (7000 TONS) AND GAS FLUE

Remembering that the gas mixture under consideration is dilute in active constituents and contains a large percentage of nitrogen which does not enter into the reaction at all, and that the reactions entered into by the active gases are strongly exothermic, it follows that if proper opportunity is given for the action of convection, there will result a rapid and complete separation of the heat-producing gases and the inactive and non-heat-producing gases.

To secure this separation by convection would clearly necessitate increasing the height of the chamber in relation to the horizontal sectional area so as to allow relative zones of reaction and inaction therein, the incoming gas being admitted to the zone of reaction, and the outgoing gas being drawn from the zone of inaction.

The horizontal sectional area of the chamber would have to be proportionate to the volume of gas admitted and the height proportionate to the time necessary for the quantitative reaction of such volume, and the gas travel would be vertically downward, subject to convection currents, instead of horizontally forward.

Bearing these considerations in mind, it becomes clear what may be the effect of increasing the length of the *vertical* axis of the chambers or, in other words, the height in relation to other dimensions.

The gas mixture, admitted to the chamber, immediately sets up a strong exothermic reaction. If the chamber is high, convection currents will be induced and the reacting gas will immediately tend to rise and to remain at the top so long as the reaction goes on, only the inert, non-reacting, non-heat-producing, and therefore cooler gases tending to return to the bottom of the chamber. If the relationship of the height to the area and the volume of gas admitted is correct, the reaction taking place in the chamber will be quantitative and the gas drawn from the chamber at the bottom will consist entirely of nitrous compounds and inert nitrogen with some oxygen. In other words, the process will be completed in one chamber and the necessity of further chambers (which cost just as much as the first active chamber) for more and more sluggish processes (or decreased efficiency in work done) will be obviated and a great economy in cost of chamber space will be secured.

ALGOMA STEEL CORPORATION, LIMITED—COKE PLANT DEPT., SAULT STE. MARIE, ONT.

Daily Report of Sulfuric Acid Plant, for 24 hours ending 7 A.M., Jan. 19, 1913

Hour ending	Furnace gas Per cent. SO_2 Station 1	Niter ($NaNO_3$) Pounds	Glover Bottom		Petersen regulator Percent. N_2O_3	Gay-Lussacs Percentage N_2O_3		Chamber						
								Temps. at stations, ° F.			Difference, ° F.			Drips, ° B.
			° F.	° B.		No. 1	No. 2	1	2	3	1–2	2–3	1–3	
8 A.M.	7.5	8	241	59	4.3	2.90	0.61	180	161	79	19	84	101	47
9 A.M.		8		59				176	156	78	20	78	98	47
10 A.M.	7.5	7	240	59	4.3	2.80	0.58	172	154	78	18	76	94	46½
11 A.M.		5		59				174	154	77	20	77	97	46½
12 Noon	7.5	4	240	59	4.3	2.33	0.57	174	154	77	20	77	97	46½
1 P.M.		4		59				176	157	76	19	81	100	47
2 P.M.	7.5	4	240	59	4.3	2.33	0.56	178	158	76	20	82	102	48
3 P.M.		4		59				178	159	76	19	83	102	48
4 P.M.	7.5	4	240	59	4.3	2.33	0.50	178	159	76	19	83	102	48
5 P.M.		4		59				178	159	75	19	84	103	48
6 P.M.	7.5	4	240	59	4.3	2.33	0.46	178	159	75	19	84	103	48
7 P.M.		4		59				178	158	75	20	83	103	48
8 P.M.	7.5	4	240	59	4.3	2.33	0.50	178	158	75	20	83	103	48
9 P.M.		4		59				176	155	73	21	82	103	47
10 P.M.	7.5	4	240	59	4.3	2.33	0.46	176	155	73	21	82	103	47
11 P.M.		4		59				176	156	73	20	83	103	47
12 Midnight	7.5	4	240	59	4.6	2.33	0.46	176	157	73	19	84	103	47½
1 A.M.		4		59				176	157	73	19	84	103	47½
2 A.M.	7.5	4	238	59	4.6	2.54	0.56	178	158	73	20	85	105	47½
3 A.M.		4		59				178	159	73	19	86	105	47½
4 A.M.	7.5	4	238	59	4.6	2.54	0.62	178	159	74	19	85	104	48
5 A.M.		4		59				172	153	77	19	76	95	48
6 A.M.	7.5	4	240	59	4.6	2.54	0.72	177	158	78	19	80	99	46
7 A.M.		4		59				178	160	78	18	82	100	45½
Total		108												
Average, 24 hours	7.5			59	4.4	2.47	0.55	177	157	76	20	81	101	47¼

RAW MATERIAL USED

Name of ore—"Helen Mine."	Per cent. S—40.34
Pounds charged in 24 hours—21.000	Per cent. S available—40.00
Per cent. $NaNO_3$ on S—burnt, 1.29	Per cent. S in cinder—0.32 A.M., 0.42 P.M.

DIARY

Chamber man increased soda from 4 to 6 lbs. during night. This was not necessary. Took it off first thing this morning.

The 172° top temperature at 5 A.M. was due to pump breakdown. Had no acid upstairs to run over Glover tower.

W. E. T.

THE RESULTS OF THE HIGH CHAMBER IN PRACTICE

The considerations which have been mentioned appeared to have such force, that in March, 1905, these views as to this high chamber construction were placed before the United States Steel Corporation, who were about to erect a sulfuric acid plant. They decided to adopt this construction and a plant was ultimately erected at Vandergrift, Pa., consisting of one chamber 50 feet square by 70 feet high, together with one Glover, two Gay-Lussacs and a small unpacked cooling tower 9.5 feet square by 50 feet high located between the chamber and the Gay-Lussac towers. This plant was put into operation on January 1, 1907, and the results amply justified expectations, and proved the following facts:

(1) A large production of sulfuric acid with a minimum of chamber space.

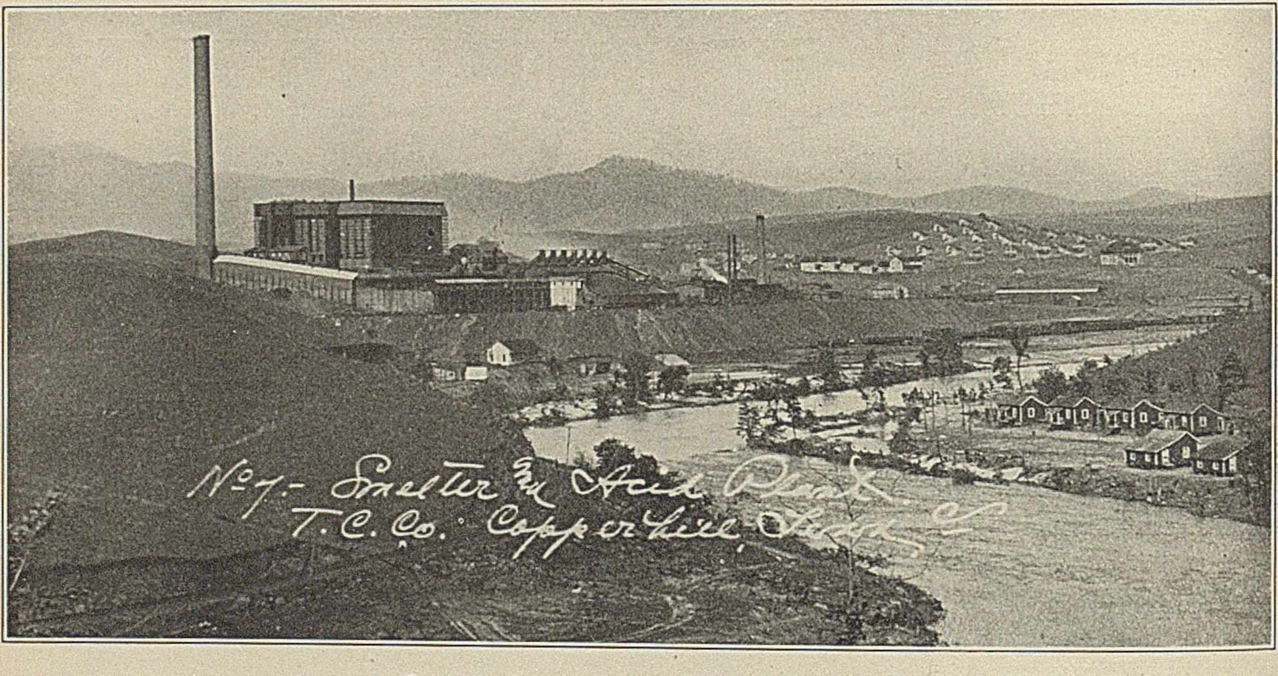
(2) A quantitative reaction in the single chamber, the SO_2 being completely converted in the chamber itself as proved by tests at the chamber exit.

(3) An insufficiency of cooling capacity—the gases on leaving the cooling tower being too hot and too moist to permit the best work in the Gay-Lussac towers.

(4) That in spite of this insufficiency of cooling capacity a reasonable consumption of niter could be attained—*i. e.*, an average between 2 and 3 per cent. of $NaNO_3$ based on the sulfur burned, while frequently for days together the consumption of $NaNO_3$ did not exceed 1.5 per cent. The insufficiency of

cooling capacity clearly interfered with a maximum output of product and a minimum consumption of $NaNO_3$.

Before this plant had been completed, the Tennessee Copper Company, who were being pressed by the Government to abate the nuisance caused by the sulfur dioxide fumes produced in their copper smelting operations, decided to erect a plant at Copperhill, Tennessee, consisting of twelve chambers of the same dimensions as those at Vandergrift. This plant now consists of thirty-six of these chambers. The work, from a technical standpoint, is not perfect, as it entails a considerable consumption of $NaNO_3$. However, the Company claims that their net profits from the production of sulfuric acid are equal to their profits from copper and other sources. This irregularity of working is due to the character of the smelter gases which contain from 4 to 6 per cent. of CO_2, and in which the percentage of SO_2 fluctuates greatly.



Tennessee Copper Co. Smelter and Acid Plant

With the advantage of the data obtained at Vandergrift and Copperhill three plants have been subsequently erected as follows:

(1) For the Cleveland Cliffs Company, at its Pioneer Iron Works plant at Marquette, Michigan, one chamber 50 feet square by 70 feet high; specified capacity in 24 hours, thirty to thirty-five tons of concentrated 66° B. sulfuric acid. This plant includes one Glover tower 10 feet in diameter by 26 feet high, two Gay-Lussacs 10 feet in diameter by 50 feet high, and two packed cooling towers 10 feet in diameter by 50 feet high, placed between the chamber exit and the Gay-Lussacs. The pyrite used at this plant is a remarkably pure concentrate, practically free from dust and arsenic; and the arrangement of the towers is such that the hot Glover tower acid is run directly by gravity from the Glover tower at a temperature of 310° to 320° F., by means of fused silica conduits into a concentrating tower. No additional labor is involved in the concentration, and the fuel cost is represented by a consumption of 100 pounds per hour of a mixture of coke breeze and slack coal fed by means of a special smokeless stoker, or a consumption of less than 70 pounds of fuel per ton of 66° B. acid.

(2) For the Dominion Iron and Steel Company, Limited, at Sydney, Cape Breton, Nova Scotia, a chamber 50 feet square by 70 feet high; specified capacity in 24 hours, forty to forty-five tons of 60° B. sulfuric acid. This plant includes one Glover tower 10 feet in diameter by 26 feet high, two Gay-Lussacs 9.5 feet square by 50 feet high and two packed cooling towers of the same dimensions as the Gay-Lussacs, placed between the chamber and the Gay-Lussacs.

(3) For the Algoma Steel Corporation, Limited, at Sault Ste. Marie, Canada, a chamber 35 feet square by 70 feet high; specified capacity in 24 hours, twenty to twenty-five tons 60° B. sulfuric acid. This plant is equipped with one Glover tower 8 feet in diameter by 25 feet high, two Gay-Lussacs 8 feet in diameter by 50 feet high, one packed cooling tower following the chamber exit and one Petersen regulator tower between the cooling tower and the Gay-Lussac, both these towers being of the same dimensions as the Gay-Lussacs.

Not one of the above installations has been made for a concern previously engaged in, or familiar with, the manufacture of sulfuric acid. In every instance the operatives are drawn from the ordinary labor available in the locality. In at least two cases the trained chemists in charge of the process had never previously seen a sulfuric acid plant. In view of these conditions, it is submitted, that the results of actual operation are entirely satisfactory. In each case (with the exception of the Tennessee Copper Company's plant, where for obvious reasons no regularity could be assured) the consumption of niter of not over an average of three per cent. was stipulated, and also a minimum output as stated in the preceding paragraph. This output in terms of chamber space was as follows:

(1) One pound of sulfur to 7.8 cubic feet of chamber space;

(2) One pound of sulfur to 7.2 cubic feet of chamber space;

Analysis of Recent Typical Daily Reports of the Vandergrift and Algoma Steel Corporation Sulfuric Acid Plants

Averages—Daily reports, etc.	First Plant erected U. S. Steel Corporation Vandergrift(*a*) Plant brimstone	Last Plant erected Lake Superior Corporation Ltd. Algoma Steel(*b*) Corp'n Ltd. Plant pyrites—"fines" (Dec. 29, 1912)
Pyrites "fines" charged in 24 hours (pounds)..	......	20,700
Pyrites contain sulfur per cent.............	....	40.34
Cinders contain sulfur per cent.............	....	0.38
Sulfur charged (pounds).	15,345	8,350
Sulfur available or burned (pounds)............	......	8,280
Per cent. of SO_2 in gas...	10.4	7.5
Nitrate of soda—per cent. on sulfur charged.....	1.56	1.15
Nitrate of soda—per cent. on sulfur burned......	......	1.16
Per cent. N_2O_3 in Petersen regulator.........	......	4.85
Per cent. N_2O_3 in nitrous vitriol..............	2.60	2.02
Per cent. N_2O_3 in final Gay-Lussac tower.....	0.17	0.60
Temp. of gas—8 ft. from top of chamber(*c*).....	205° F.	179° F.
Temp. of gas—5 ft. from bottom of chamber....	190° F.	160° F.
Temperature of gas at entrance to Gay-Lussacs.	127° F.	72° F.
Difference of temperature between top and bottom of chamber.......	15° F.	19° F.
Difference of temperature between bottom of chamber and Gay-Lussacs................	63° F.	88° F.
Drip—Degrees U. S. B...	46.5° B.	48° B.
Average strength of acid in chamber pan (U. S. B.)..................	52 to 53° B.	53 to 54° B.
Average strength of finished acid...........	62° B.	58 to 59° B.
Dimensions of chamber (feet)...............	50′ × 50′ × 70′ high	35′ × 35′ × 70′ high
Outside dimensions of towers—except Glover (feet)...............	9′6″ × 9′6″ × 50′ high	8′ diam. × 50′ high
Outside dimensions of Glover tower (feet)....	9′6″ × 9′6″ × 24′ high	8′ diam. × 24′ high

(*a*) The Vandergrift Plant has been in operation some years and was the first of this character of plant built.

(*b*) The Algoma Plant has been operated less than three months.

(*c*) Temperatures taken on side of chamber with usual bent thermometer.

(3) One pound of sulfur to 6.4 cubic feet of chamber space, or a maximum output of one pound of sulfur to 6 cubic feet of chamber space, or a minimum output of one pound of sulfur to 8 cubic feet of chamber space, or an average output of one pound of sulfur to 7 cubic feet of chamber space. In all cases these specifications have been carried out and easily exceeded. The plants have been taken over and paid for and are operating to the satisfaction of the owners.

TEMPERATURE OF REACTION IN THE HIGH CHAMBER

In the ordinary operation, readings are taken from the usual bent thermometers placed, the one about 12 feet from the top of the chamber in the side lead, and the other about 4 feet from the bottom of the pan, in the side lead. In practice the temperatures are read only on one side of the chamber. At first thermometers were placed on all four sides of the chambers, and not only near the middle of the sides, but also near the corners, until it was found that the variation in these thermometers at equal height in the chamber was practically nil.

When the chamber was first used, thermometers were placed also vertically 5 feet apart on all four sides of a chamber, and an extended series of readings taken, all of which showed a gradual diminution of temperature from the top to the bottom. This decrease in temperature, of course, was more rapid in the lower part of the chamber, but was distinctly marked after the first fifteen feet from the top, or the first two readings.

The difference between the upper thermometer (10 feet from the top) and the bottom thermometer will range from 22° F. to 7° F., according to the intensity with which the system is being driven, *i. e.*, the proportion of the amount of sulfur burned to the cubic feet of chamber space.

When the system is in normal operation, repeated tests show no more than the usual traces of SO_2 in the gases leaving the chamber. The packed cooling tower which follows the chamber serves to dry the gases leaving the chamber (by condensing the mist of sulfuric acid) before admission to the Gay-Lussac towers, and a further cooling of from 75° to 100° F. takes place in this apparatus, bringing the final temperature of the gases entering the Gay-Lussac towers to 60° to 80° F.

DRIPS

One drip reading has been found, by elimination, to be sufficient. This has been placed, by reason of convenience, near the lower thermometer about four feet from the bottom at the middle of the chamber side. If this drip is carried at 47°–48° B., the pan acid will be found invariably between 52° and 54° B. At one plant a table drip has been employed, and the readings respectively of the table drip, the side drip and the chamber acid as drawn to the storage tank, were 55° B., 47° B., and 53.6° B.

CONSUMPTION OF NITER

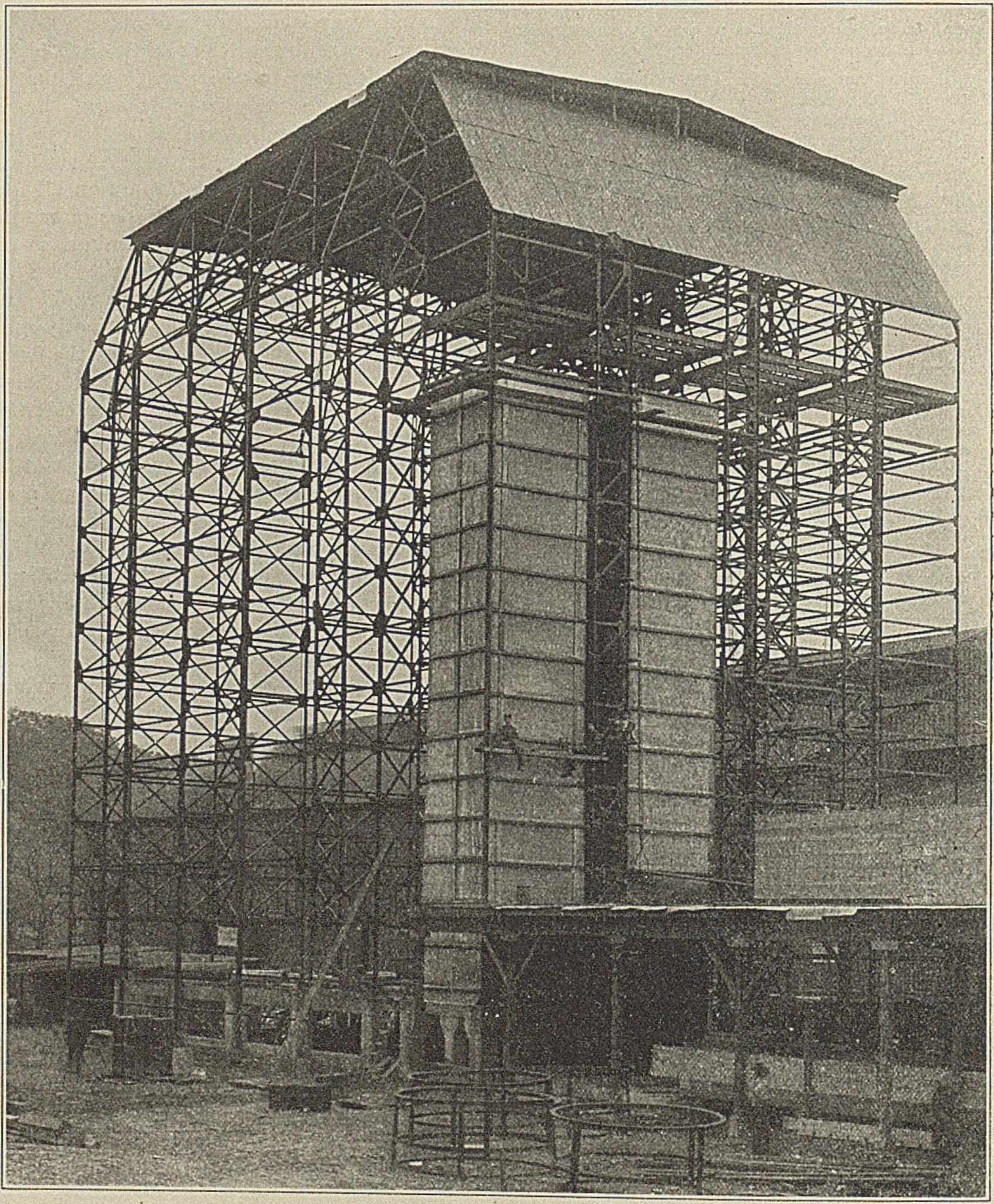
Some of these plants are of course not doing as regular work as they would do if operated by skilled labor. The consumption of nitrate of soda is frequently for considerable periods as low as 1 to $1^{1}/_{2}$ per cent., a record which undoubtedly could be uniformly maintained by competent operatives (see tables).

THE ADVANTAGES OF THE HIGH CHAMBER

The advantages of this chamber are noteworthy:

I. The Saving of Lead.—For purposes of comparison, take a chamber fifty feet square and seventy feet high, representing a capacity of 175,000 cubic feet (4952.5 cubic meters). This comparison has no reference whatsoever to the actual efficiency of such chamber space. The largest chambers which Meyer has built are probably those of the Mountain Copper Company, which he states are 12 meters in diameter and 13 meters high. These would therefore have a capacity of 1470 cubic meters each. Three chambers of this size would be required, together with a small chamber 9 meters diameter and 6.5 meters high to give the same capacity as one high chamber 50 feet square by 70 feet high. In actual construction the weights of lead used in this chamber are as follows:

The top and bottom, seven-pound lead; the sides, six-pound lead, rolled to eight-pound for two feet at the top, and five feet at the bottom. The straps for the sides six-pound, and for the top seven-pound. (These straps consist of a five-inch strip of lead burned onto the chamber sides, and extending horizontally continuously around the chamber at vertical intervals of about 5 ft. The straps are then bolted by means of clamp bars to horizontal angles; in such a way that the side lead does not touch the supporting angles or posts at any place.) The total weight of lead, including everything in connection with the chamber, is 141,000 pounds. A corresponding plant, according to the Meyer system, would require at least 220,000 pounds of lead, while a plant of three chambers of equal cubic capacity, built according to the old rectangular, horizontal system, would require from 240,000 to 300,000 pounds of lead. The saving



Vandergrift Plant in Course of Erection—Two Towers Nearly Completed in lead, therefore, is from 35 per cent. to more than 50 per cent. for equal cubic capacity, even supposing that the high chamber space is not more efficient.

II. Greater Efficiency of the High Chamber Space.—An efficiency of chamber space representing from 6 to 7 cubic feet of chamber space to one pound of sulfur burned, without the assistance of any auxiliary apparatus such as surface condensers, needs no further comment.

III. Great Economy of Ground Area and Foundations.—The fifty-foot chamber, allowing seven feet to the outside of the building for the width of the trusses, occupies only 4,096 square feet, whereas in the case of the old horizontal chambers the same chamber space would occupy about three times this area, and the Meyer Tangential System would require from two to three times this amount, depending on the size of the chambers.

IV. Fire-proof and Storm-proof Construction.—The method of construction, being entirely of steel and reenforced concrete, is fire-proof and storm-proof. The cost of this construction is about the same or rather less for equal chamber capacity than what is considered a good type of construction of the old chamber plants, where wooden frames and buildings are used.

V. Economy of Labor.—The compact arrangement of this Plant reduces the amount of labor required to the minimum. For any Plant up to 100 tons of 66° B. sulfuric acid in 24 hours the labor is as follows (12-hour shift):

When using brimstone, one chamber man who is also day foreman, one chamber man who is also night foreman, one day burner man, one night burner man—four men in 24 hours.

When using "fines" pyrite, one or two laborers will be required to handle the ore and cinder, making a total of 5 to 6 men in 24 hours.

When using lump pyrite, additional men will be needed, depending on the construction of the lump burners.

The operation of the high chamber (towers, pumping, concentration, etc.) requires only one man per shift of 12 hours.

The scope of this article could scarcely be considered as including a detailed description of the methods of steel construction and lead work.[1] It is sufficient to say that the difficulties connected with the same have been solved in a most satisfactory manner, both with reference to protection against fire and storms, and the durability of the lead work. The plants which have been longest in operation have demonstrated that the item of repairs has been considerably reduced, being approximately only about 20 per cent. of the amount usually attending the horizontal chamber construction.

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New York City

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[1] Ingenieur Awe in an article on the construction of lead-chamber walls (*Z. angew. Chem.*, **25**, 2523-2524 (1912)) describes a method of construction which was *first* used on the "Falding High Chamber" several years ago and has been continuously used since in all of the Falding installations.

TECHNICAL ACCOUNTING AND CHEMICAL CONTROL IN SUGAR MANUFACTURE[1]

By David L. Davoll, Jr.

INTRODUCTION

For the technical supervision of the manufacture of sugar, whether from beet or cane or whether the purpose be to make raw sugar or refined granulated, the activities of the chemist may well be directed along three lines of endeavor.

First, The sampling and analysis of all raw material such as coal, coke and limestone or lime. Almost without exception in the beet industry and in exceptional cases with the cane, this includes the daily systematic valuation of beets and sugar-cane deliveries for purposes of purchase. Further, it will include the analysis of field samples to determine maturity of crop in general and the effect of certain influencing factors in particular districts.

Second, The frequent and rapid testing of initial material, intermediate products at the several "stations" of manufacture and the final products, together constituting what is known as "chemical control."

Third, The keeping of the sugar account and the daily calculation of the efficiency of the various pieces of machinery and of the several intermediate processes of manufacture.

Chemical control is essentially diagnostic in character and takes advantage of the knowledge we have of how the sugar-bearing material should behave at the "stations" as now interpreted through chemical tests and of what constitutes recognized, unavoidable losses, to put in the hands of the foremen in charge the proper data.

Successful sugar accounting calls for good organization first and conscientious, well-trained chemists to carry it out. Success or failure depends upon the presence or absence of suitable conveniences, accurate factory weights and measures, calibration of utensils and the exercise of great care in the sampling, subsampling, compositing and preserving.

In the beet industry the polarization is practically the true sucrose (with raffinose absent) while in the cane industry the polarization is never the true sucrose but is the algebraic sum of the several optical activities, of all the participating bodies, not removable by lead acetate. Therefore, in this article the term polarization or "sugar" signifies the polariscopic reading when reduced to terms of 26 grams of material in 100 metric cubic centimeters. Sucrose refers to the Clerget figure.

A few terms, peculiar to the cane industry, may well be defined here.

Normal Juice.—Strictly speaking this is the whole juice of the cane as it exists in the tissues, or the combined juice of all the mill units when milling without the application of water of Saturation. It still has considerable significance in cane work but none in the beet. It usually runs from 0.1 to 0.7 degree Brix lower than the so-called First Mill Juice; it is also lower in purity. Owing to its variability under changing conditions it should be determined by actual run, without water, at stated intervals.

[1] Paper presented at the Fifth Annual Meeting of the American Institute of Chemical Engineers, Detroit, December, 1912.

It does not appear that a proper figure can be obtained in less than 1/2 hour's run. Its Brix, taken in conjunction with that of the mill raw juice, is used in calculating the extent of the dilution due to the water of saturation.

Mill Extraction.—The percentage of the sugar in the cane that has been removed by the milling process.

Retention.—The amount of sugar in the form of commercial sugar, expressed in terms of percentage of the sugar obtained in the milling process.

Total Efficiency.—The total sugar in the form of commercial sugar expresses in terms of percentage of the sugar in the cane. It is the product obtained by multiplying the extraction by the retention.

Blanc.—A product of vacuum-pan boiling upon very low products variously termed "filete" and "string-proof." It is not boiled to grain, but is made very concentrated and the density judged by pulling a small quantity out to form a "string" or rolling a small ball in cold water and noting the hardness.

It is the final boiling in all houses not provided with crystallizers, the product being set aside in storage for slow cooling and crystallization, for from two to three weeks or more.

A very brief account of the processes of sugar manufacture may assist in making clear the purpose of chemical control and scheme of sugar accounting. United States beet and Cuban raw sugars only are considered.

BEET SUGAR

The factories of the United States make standard white granulated sugar in one operation from the sugar beet, which has a sugar content of from 14 per cent. to 23 per cent. average for the entire crop or "campaign," according to territory, season and seed pedigree.

1. DIFFUSION JUICE.—The beets are washed and then sliced into strips 3¹/₂ to 7 mm. wide and 2–4 mm. thick, with cross-section V-shaped and the sugar extracted by a highly developed process of diffusion, at definite temperatures, leaving behind the "pulp," of sugar content 0.25–0.30 per cent. There results a very clean, but dark colored liquor, warm to hot, of density 0.8–0.9 that of the original juice of the cossettes (sliced beets), which goes to duplicate tanks for measurement, after which it is forced by centrifugal pumps to tall, closed tanks.

2. LIMING AND CARBONITATION.—Lime to the extent of 2¹/₂ per cent. to 3 per cent. of the beets or lime-saccharate as produced, both in the form of "milk" of 17° B., is added, while simultaneously the mass is brought to a definite, high temperature by injected steam and carbonic acid from the lime kilns is pumped in. At the precise moment of granulation of the precipitate of $CaCO_3$, etc., judged wholly by the eye and corresponding to 0.1–0.14 per cent. CaO alkalinity, the mass is released to the pumps which serve the set No. 1 filter presses.

3. THE FILTRATIONS.—After issuing from the first set of presses under 2¹/₂ atmospheres of pressure in form of a brilliant, pale straw-colored liquor, it goes to a second set of tanks for further liming and carbonitation, where the alkalinity is brought to 0.015–0.025 per cent. CaO. It is then passed through a second set of filter presses and then "sulfured." The presses are clothed with duck or jute or both. The press cakes are washed with hot water (condensed) to a definite sugar content, set No. 1 to 1 per cent., set No. 2 to 0.5 per cent., judged by applying a suitable hydrometer to the issuing "sweet waters."

4. "SULFUR STATION" NO. 1.—Bleaching by means of SO_2 gas takes place here, reducing the reaction to faint alkalinity, neutrality or faint acidity as occasion demands, but always to a definite figure. The process is perhaps invariably a continuous one.

5. GRAVITY FILTERS NO. 1.—Passing through these is preparation for evaporation under multiple-effect system.

6. CONCENTRATION.—By means of quadruple or quintuple effect the density is raised to 60° Brix, some ammonia is given off, lowering of alkalinity in the absolute but rising as result of concentration; some precipitate forms. "Thick juice" results.

The work is now in the "boiling" house.

7. "SULFUR" STATION NO. 2, OR "BLOW-UPS."—The reaction is here brought to the desired point, either faintly alkaline or acid and by careful test, in accordance with a figure that has been found to give the best results at that factory and particular district and often influenced by the season: 0.02 per cent. CaO (basis) is seldom exceeded either way. Acidity may be aided by phosphoric acid; alkalinity induced by lime or soda ash. The thick juice, like all products passing this station, is here "blown up" with injected steam and reduced to uniform Brix, quite generally 60°, to dissolve any grain, aid filtration and promote good boiling in the pan.

8. GRAVITY FILTERS NO. 2.—These serve all products in the intricate but systematic work of the "boiling" house such as thick juice, high and low wash and greens and melted sugar, removing all suspended matter in preparation for the pan-service tanks.

9. THE MASSECUITE.—This is formed in a vacuum pan working under 26–28 inches of vacuum and in masses of many tons.

The boiling to grain is carried out purely as an art by men who make it a profession. Of course, it is based entirely upon well-known scientific principles. It is essentially crystallization-in-motion, during which the product, constantly augmented in quantity by fresh injections of liquor, is resolved into grains of sugar and a more or less de-sugared mother liquor.

The final result of the process is judged upon the so-called purity test. The initial product from the beets generally carries a purity of 88; as the sugar crystallizes and is removed by the centrifugals the purity ratio necessarily lowers, thus becoming a measure of efficiency and of paramount importance.

The first boiling, followed by hot turbinating, gives, upon washing or "covering" with diluted liquors, white refined sugar, a "wash" of 90–92 purity and a mother liquor of 78–80 purity. A second boiling to grain, massecuite having 78–80 purity, discharging while hot to the crystallizers, cooling there with the aid of a helix and water jacket for about three days and then centrifugating, gives a yellow sugar and an exhausted molasses, purity 58–60. The yellow sugar is redissolved and boiled, variously combined, to white refined sugar.

If it is the intention to recover still more sugar from the molasses so reduced in purity that it will yield no more sugar by crystallization, the sugar may be precipitated by a large excess of one of the oxids of the alkaline earths.

In the United States lime oxid is used, in absolutely anhydrous and impalpable powder. Tricalcium saccharate results, later soluble in the saccharine juice to monocalcium saccharate.

10. THE COOLERS.—Under agitation the powdered lime is slowly sifted into the molasses previously reduced to 12–14° Brix, kept cool at a definite temperature meanwhile, until the density of a filtered sample indicates 6–7° Brix. It is then filter-pressed as quickly as possible.

11. SACCHARATE PRESSES.—The ordinary Kroog type of press produces 40 cakes of 1 inch thickness; the saccharate presses produce a much thicker cake, usually $2^{1}/_{2}$ inches thick; they fill and wash readily. The product from the coolers is here separated into the saccharate of 86–98 purity and a mother liquor known as "press waste water" of 6–7° Brix and 10–20° purity, all from a 58–62° purity molasses. The washing with cold water alone or combined with its own higher "sweet waters" is continued until a liquor having a Brix of $2^{1}/_{2}$ to 3° results and the purity of the last runnings mounts to 15–30. The total product of the washing is known as "wash water."

12. SACCHARATE AND SACCHARATE MILK—The saccharate press cake is transported to the liming and carbonating station of the main factory process, in the form of milk, being discharged directly into tanks provided with stirrers, where it is incorporated with liquors brought thither from various parts of the factory and bearing from known small amounts of sugar to mere traces.

A separate sugar account is required for the saccharate process and the yield should be about 67 per cent. of the sugar charged to it, in the form of refined white.

CANE SUGAR

Raw sugar factories dealing with cane aim to produce a sugar that will keep during storage and transportation, of a sugar content that will bring the highest price for the total sugar output and to get as high a yield as can be proved to be economical in the final summing up of all the conditions.

It is generally conceded that an even 96° Ventzke polarization at the port of entry brings the highest profit. It is very probable that all other grades will soon be suppressed.

1. THE MILLING.—Heavy iron mills replace the diffusion battery of the beet-sugar process. The installation generally consists of three units (individual mills) placed in tandem and composed of three rolls each; there are intervening conveyors and the whole train is preceded by a crusher of two rolls.

The crusher serves to break the outer rind and the nodes, liberating at the same time considerable juice which flows to the bed plate of the first unit. The rolls are ponderous; 7 feet long by three feet in diameter may be taken as a type.

The cane gets two compressions in passing through each unit, being sustained by the "turn bar" as it issues from between the cane roll and the top roll and passed on to be caught by the bagasse roll and the top roll for the still closer compression.

The cane, not being laden with molasses-forming salts to the great extent that the beet is, the rupture of the cells is not attended with disastrous consequences. Naturally the ratio between the fiber of the cane entering any given unit of the tandem and that of the bagasse issuing therefrom, is a measure of the efficiency with respect to the amount of liquid expressed. Where water of saturation is applied, hot or cold, it is generally sprayed forcibly upon the bagasse as it issues from the unit next the last, at the point of immediate release from pressure; the imbibition of the thin juices is the further development of the water treatment and is simple and effective. As high as five units and a crusher or in all, seventeen rolls have been employed in one tandem.

The juices from the first and second units only enter the process of manufacture, *i. e.*, under a system of combined saturation and imbibition, and, united, constitute the mill raw juice.

2. MILL RAW JUICE.—Sometimes called diluted juice.

With respect to its purity it is considered to represent the original juice of the tissues of the cane.

It is either at factory temperature or a trifle above it, depending upon the saturation water temperature; it is charged with air, turbid from suspended albuminous matter, wax, insoluble salts, clay, and fiber—this even after being strained through copper or brass sieves of 15–19 perforations per linear inch.

It is pumped to tanks for measurements or weighing and is then limed. Where heavy liming with carbonitation is not practiced (and I know of no factory in Cuba doing this) the juice is only neutralized.

3. LIMING OR "TEMPERING" THE JUICE.—This is generally accomplished in sets of three large tanks per tandem, one filling, one under treatment and one discharging. Repeated trials upon different sized tanks have shown a size corresponding to three hectoliters per ton of cane per hour to be advantageous.

Chemists carry the reaction of the juice generally about neutral to litmus paper. The subject can be said to have been scarcely attacked from the quantitative standpoint.

Continuous liming is practiced in some factories.

Lime and heat form clear juice and cachaza or "scum."

After liming, the juice is passed through heaters where the product may be even superheated if desired, depending upon whether it is desired to eliminate all the air by a "flashing" operation before complete settling. If the air is not eliminated a thick scum rises to the top at 95° C. called "blanket," a small portion sinks to the bottom, while the separation is being effected in so-called defecators varying in size from 35–100 hectoliters, net; the time for making one complete round of the defecators will be 60 minutes but capacity should be had for 90 minutes, to allow for irregular liming; 15 per cent. of the time will be consumed in filling, emptying and cleaning.

Continuous settling is effected in the Deming process and in the Hatton defecators.

The slightly opalescent, straw-colored juice is generally run, without filtration, merely decantation continuous or interrupted, directly to the multiple effect.

It should be passed through fiber or gravity filters if for nothing more than to catch much cachaza that slips into the process intermittently. The decanted cachaza is washed by decantation in small tanks and then sent to frame presses for compression and sometimes washing. Evaporation is carried to 55° Brix in a cane house, to facilitate (1) settling and (2) avoidance of false grain.

The boiling of the meladura to grained massecuite is similar in principle to that carried out in a beet factory.

Cane products grain with great facility, while beet products sometimes present great difficulty, conditions brought about by the variance in the character of the non-sugars, purity remaining the same.

Generally three grades of grained massecuites are boiled where crystallizers have been installed, all upon a nucleus of original meladura which ranges in purity from 80–92, according to district and time of season. *First massecuite,* purity 80–84, yielding a sugar polarizing 97–98° Ventzke and a corresponding green syrup or molasses of purity 60–64. *Second massecuite,* purity 70–74; corresponding molasses, centrifugated hot, 48–54; centrifugated after limited cooling in motion, purity 40–46; resulting sugar, washed by water or liquors to 96° V. *Third massecuite,* purity 58–63; corresponding molasses (final product), purity 30–35; resulting sugar polarization depending upon treatment. This last massecuite, when at 35–40° C. and 4–5 days old in crystallizers, is generally centrifugated and the untreated sugar discharged into a mixer where high-grade molasses is incorporated with it and it is again centrifugated and washed to the degree desired, generally 96. This process is styled "mingling."

In factories not provided with crystallizers (which keep the grain in motion) the exhaustion of the product when it reaches the purity 48–54 must be accomplished "at rest," which is brought about by discharging the final boiling, boiled "blanc" to a Brix of from 88–91, according to conditions, into small iron wagons or into large tanks where it is allowed to cool quietly and crystallize spontaneously for from 12–21 days or longer.

(*To be concluded in the April issue.*)

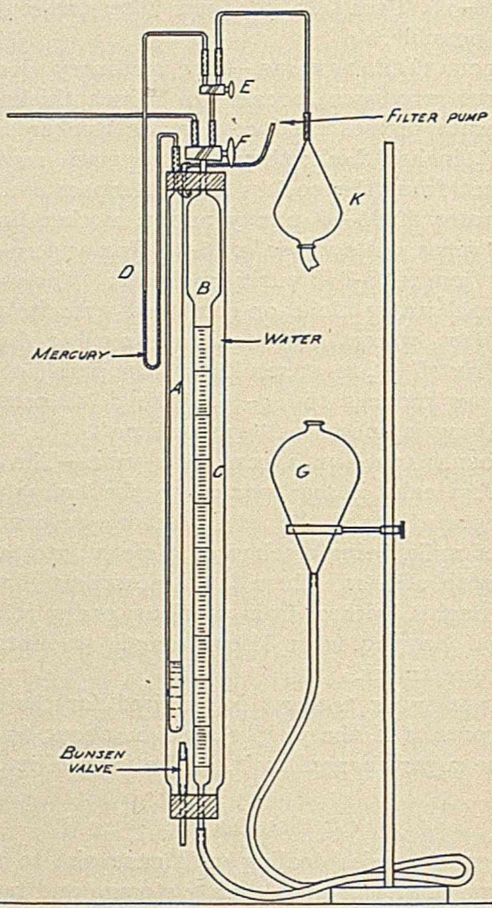
A MODIFICATION OF THE PARR TOTAL CARBON APPARATUS

By C. E. Millar

Received December 13, 1912

The following modification, while used by the writer in connection with the Parr total carbon apparatus, is equally applicable to any gas burette. It has not only been found valuable as a time saver but a great convenience since rapid changes of temperature and pressure are compensated for by the apparatus. When the amount of gas is sufficient to fill the pipette several times, as often happens in the determination of total carbon in soils, variation of temperature and pressure during the determination cause no trouble. In addition the long and tedious calculations necessary under former conditions in determining total carbon in soils and coals are reduced to multiplication of the pipette reading by a constant.

The diagram shows the modified portion of the Parr apparatus. When the sealed tube A is put in place and connected to B by means of D the heights of the mercury columns in the arms of D are carefully marked. The barometric reading and the temperature of the bath C, which should be at room temperature, are also taken. It is obvious that B and G should be empty during the operation or else the core of one of the cocks E or F should be removed in order that the gas in A be under atmospheric pressure. The sealed tube A is filled to a depth of three or four inches with a portion of the same solution used in the pipette B. Since the two tubes are in the same bath the vapor pressure is the same when the height of mercury in the arms of D is the same.



When a volume of gas is collected in B it is allowed to stand until of the same temperature as C and hence of A. The stopcocks E and F are now opened so as to connect B and A. The leveling bulb G is adjusted until the level of the mercury in D is the same as when the apparatus was set up, in other words at the mark.

The volume of gas is now read and after absorption in the bulb K and drawing back into B, the process of leveling and reading is repeated in the same manner. These readings give the volume of the gas absorbed at the same molecular concentration as the gas in A when the apparatus was set up. The reading, therefore, need only be multiplied by the factor found to apply under those conditions.

In the bottom of the bath C there is a Bunsen valve which, by turning on the filter pump at the top, allows a current of air to pass through the bath C thus keeping the liquid well stirred. The tube D should be of small bore and of sufficient length to allow for the variations of temperature from season to season.

The above modification may be fitted to the ordinary Parr apparatus or gas pipette by inserting the stopcock E.

The writer wishes to thank Professor George H. Failyer for a suggestion that led to the above modification.

Chemical Laboratory Kansas Experiment Station
Kansas State Agricultural College
Manhattan

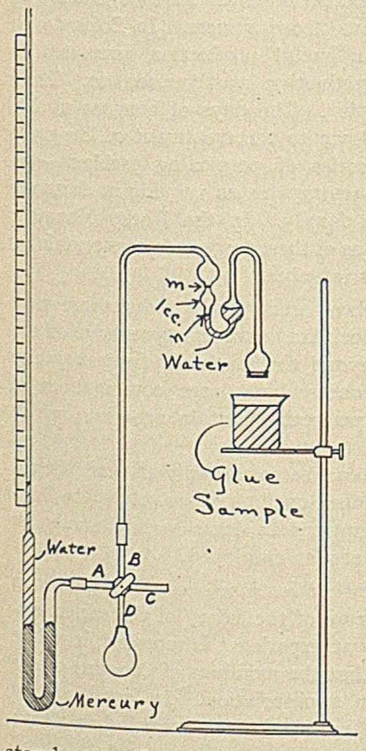
IMPROVED APPARATUS FOR TESTING THE JELLY-STRENGTH OF GLUES

By E. C. Hulbert

Received November 1, 1912

The apparatus consists of a thistle tube, the stem of which is twice bent and contains three bulbs. The two larger are about 2 cm. diameter and serve as safety traps; the smaller is accurately graduated to contain 1 cc.

Over the mouth of the thistle tube is stretched a diaphragm of thin rubber. The far end of this tube connects through a 4-way stopcock with a gage consisting of a mercury filled U tube having its far arm extended upward and of smaller bore, this arm containing water or other liquid above the mercury. A rubber bulb is attached to one arm of the stopcock, the remaining arm being left open to the air.



The sample of glue-jelly is raised by means of the adjustable stand until the liquid in the bulb tube reaches the upper graduation m, and the stand secured in this position, the stopcock meanwhile being set to connect arms A,B,C (see diagram). The cock is now turned to connect arms A,B,D and enough air forced into the system by means of the rubber bulb, to force the liquid in the bulb-tube down to the lower graduation n, the cock turned to an intermediate position and the gage read.

The principal advantages of this apparatus over others are: The figure obtained is not influenced by the condition of the surface of the sample, such as formation of a tough "skin," rapid change of temperature during the test, etc. The error due to supporting a column of water resting directly upon the glue surface, as in another somewhat similar apparatus, is avoided; this is of especial importance in case of glues of low grade.

The reading is quickly made and may be repeated three or four times on the same sample, if desired.

No error is introduced either by the apparatus itself or its manipulation; duplicate samples, tested under like conditions of temperature, etc., give identical readings. The apparatus is simple and inexpensive and may be constructed by anyone having moderate skill in glass working.

Glues may be tested either under constant conditions of temperature and strength of solution, or compared with a standard glue.

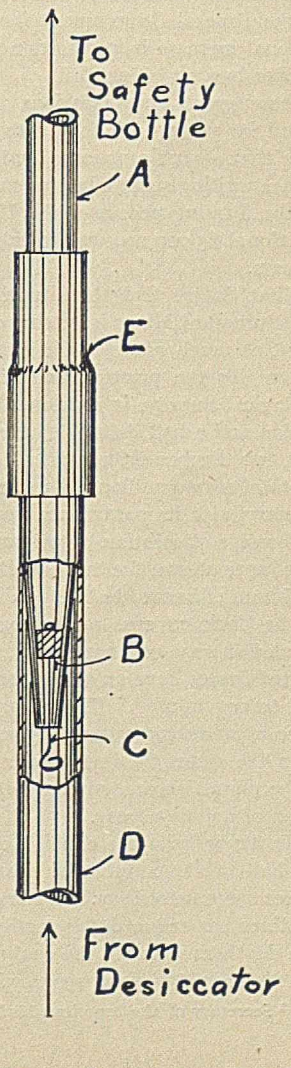
The gage scale may be graduated in any convenient way, as by comparison with another type of glue testing apparatus, or with standard glues.

Sulzberger & Sons Co.
Kansas City, Mo.

A USEFUL VALVE

By Nathan Smith

Received November 15, 1912



This simple "home-made" valve has proved very efficient in preventing the "suckback" which usually results in vacuating desiccators by water suction, when the pressure is suddenly lowered. The valve may also be advantageously employed in the Kjeldahl nitrogen determination.

A small evenly drawn tube, A (the tip of an old pipette serves the purpose), is fitted with a piece of rubber, B, which acts as a valve. A piece of wire, C, is drawn through the center of the valve and bent as shown. Another piece of glass tubing, D, is fitted over the valve tube by means of rubber connection, E.

Laboratory Schieffelin & Co.,
New York City

ADDRESSES

THE NEEDS OF THE MINING INDUSTRY

By J. F. Callbreath[1]

Received February 6, 1913

The two foundation industries of the United States are agriculture and mining. Agriculture is perhaps twice or three times as important as mining in the number of men engaged and in the value of its product.

Irrespective of their relative importance, one thing stands out preëminently and that is there can be no great agricultural prosperity without the prosperity of the mines. The population that depends upon the mines and the manufactured products of the mines supports agriculture.

Just the other day the Senate completed a piece of legislation which will give to the various State agricultural colleges the sum of three million dollars yearly for the extension of their work among the farmers. The bill was passed through both houses with comparative ease, as every one seems to have a thorough knowledge of the needs of agriculture. A report which accompanied this bill shows that the National Government has spent on the agricultural experiment colleges and experiment stations in round figures $70,000,000; that it now spends $3,940,000 in cash annually upon them; that Congress has appropriated each year $15,000,000 for carrying on the exclusively agricultural work of the Department of Agriculture.

With the additional $3,000,000 each year just granted, the total Federal aid given agriculture annually will be at least $22,000,000. The result of this generous expenditure of money by the Federal Government for agriculture is seen in the phenomenal increase in production from year to year and the final bumper harvest of last fall.

These great appropriations for agriculture have been continued for years until to-day the officers of this department declare that a vast amount of valuable information is now in existence waiting some effective means of getting it into operation by the farming people of the United States. The plea for the additional $3,000,000 was to take this information direct to the farmers.

What do we find the situation to be in regard to mining? The Bureau of Mines is getting one-half a million dollars a year, about as much money as is granted to the Department of Agriculture for the printing and binding of its publications. The Geological Survey, in existence many years, is now getting one million and a half dollars a year, a considerable part of which is used for the benefit of agriculture and other industries. This is a total of two million dollars of Federal aid for the great mining industry with its paramount unsolved problems—problems that are outside of solution by private parties.

In agriculture the men work in the open sunlight with a minimum of hazard to life. In mining nearly 700,000 men work in the dark caverns underground with a hazard that is not equaled in any other industry. The mines and quarries of the United States have an annual death roll of more than 4,000 men and 60,000 injured. This calamity of death leaves in its trail 2,500 or more widows and 15,000 fatherless children. It places upon the states the burden of caring for many of these widows and orphans, an amount that must be many hundreds of thousands of dollars a year.

We know from the experience of foreign countries that fully one-half of the number of deaths in the mines of the United States are unnecessary. We know that the work of the Bureau of Mines has reduced this terrible death-rate in the coal mines. The deaths in 1912 in the coal mines will be approximately 800 less than in the year 1907, when this work was first begun. Practically nothing has been done by the Bureau in regard to the deaths in metal mines and the rate is heavier than in the coal mines. Neither has anything been accomplished in reducing the number of deaths in the quarries which have a still higher rate than the metal mines. This work can not be taken up until Congress and the people of the United States begin to have some adequate idea of the importance of the mining industry.

So difficult has it been in the past to obtain appropriations for this humane work, that those of us who are interested have learned to be extremely modest in the appropriations we have asked for. Only slight increases are asked for the coming fiscal year, when in fact the Bureau, on the basis of what it has already accomplished, can well spend two or three million dollars each year to the benefit of the entire country, rather than the one-half million which it now has.

Taking out of consideration the unusual hazard of mining and the fact that we are killing from three to five miners in this country each year where European countries are killing one and two, there is confronting Congress the question whether it will allow Western mining to languish and grow less with a diminishing number of men employed in each state, or give to the mining industry that aid which is similarly given to agriculture to rehabilitate this industry and increase the prosperity of the country. When I say that the Western mining industry is languishing, I have statistics to bear me out in the statement.

In 1906, in the states of Colorado, Montana and Idaho, the value of gold, silver, copper and lead produced reached a value of $298,174,000. In 1910, the value of the production of the same metals in the same states was $251,000,000, a loss of near $50,000,000. In 1900 Colorado employed 40,000 miners; in 1910 that state employed 19,000 miners. In 1906 Montana employed 15,000; in 1910, 14,000 miners. In 1906, Idaho employed 7,000 miners; in 1910, 6,000 miners. In Colorado, in the year 1900 the precious metal production amounted to $50,000,000; in 1910 the production was $32,000,000. There is a definite reason for all this. The days of bonanza mining are gone and we are now down to the treatment of low-grade ores. This has led to a series of perplexing problems—the treatment of low-grade refractory ores as varying in character as the faces of the human family. In the Rocky Mountain States we have millions of tons of these low-grade ores containing practically billions of dollars in value.

The processes by which these ores may be reduced are more complex than any of the specialized problems you have on the farms. Their solution is beyond the reach of private capacity except in the rarest instances where large combinations of capital have succeeded in perfecting certain processes, which they keep to themselves.

These problems generally can be solved only through the aid of the Federal Government, and until they are solved the Western mining industry will continue to decrease in the value of production and the number of men employed.

The Bureau of Mines has asked for $250,000 to begin work on such problems and to take care of the safety of the miners employed in these mines. If past experience is repeated, it will be with the greatest difficulty that the members of Congress can be shown the need for such an appropriation. In fact it will be more difficult to obtain the $250,000 for this great industry than to obtain millions for agriculture.

I do not object to agriculture obtaining the increased yearly appropriation of $3,000,000 for the extension of the work of the agricultural colleges in the states, but I am anxious to know why a bill introduced by Representative Foster, Chairman of the

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[1] Secretary of the American Mining Congress.

House Committee of Mines and Mining, and which has the approval of the entire committee, calling for an expenditure of a maximum of $1,2000,000 yearly for mining schools in the various states, does not get an opportunity to come before the House for passage. I want to know why agriculture which has been so adequately cared for during many years can, with such ease, obtain $3,000,000 additional funds for its work, while the bill for mining schools remains on the calendar, with the fear among its friends that it may not obtain sufficient support for its passage. I want to know how Congress is to look at the proposed appropriation for one-half million dollars for buildings for the Bureau of Mines at Pittsburgh, Pa. I may say that there is no department or bureau of the Federal Government so badly and so inadequately housed as the Bureau of Mines Experiment Station in Pittsburgh. The work is being conducted under the greatest difficulties in buildings belonging to the War Department, some of them nearly one hundred years old. Further, the War Department is anxious to regain possession of its property and has been continually notifying the Bureau of Mines that it must get out. Here is an emergency as far as the mining industry is concerned. What will Congress do?

I am heartily in favor of the increased appropriation for agriculture, but mining also has similar rights and greater needs. There is no industry which adds so much per capita wealth to the country as the mining industry. There is no industry that, man for man, produces so much value to the country as the mining industry. It is an industry that must go hand in hand with agriculture. The prosperity of the country depends upon the success of both. Agriculture can not succeed without mining.

I am at a loss to understand why mining is so little appreciated in the United States when it attains such high commanding places in every other nation of the world.

THE CHEMICAL ENGINEER AND INDUSTRIAL EFFICIENCY[1]

By W. M. Booth

The chemical engineer can take his legitimate place in industrial affairs only when he begins to concern himself with values and returns and can transpose the signs and symbols of the chemist to the dollars, cents and percentages of the business world. The profession must necessarily include men with diversified mental attributes, experiences and education. Natural inventors, builders, executives, analysts and economists are examples of the men who will choose this branch of engineering as a life work.

The first and highest type of endeavor concerns itself with the invention of new processes and the perfection of these, later establishing useful industries. Relatively few men have the ability, courage and means to embark in totally new enterprises, and the larger percentage of those who do so, fail, because these three important elements are not properly balanced.

Not less important, but demanding a different type of mind, perhaps more strongly analytical in its nature, is the large and ever-increasing field open to those who are able to improve or effect economies in the processes of firms already established. Closely related to this is the examination of new enterprises that seem to have merit and which need scientific assistance and capital for development. No greater damage has ever been wrought on American investors by any class of men than by the "new chemical," "private," or "Secret process" promoters who have used the subtleties of the science as a basis for fraud.

Irrespective of the particular division of endeavor undertaken, the chemical engineer must fortify himself against all classes of misrepresentation, and must concern himself with questions of process, cost, market, location and actual capital needed in any new industrial enterprise. No considerable investment should ever be made without a complete report from conservative men, who are familiar with the industry involved.

While the basic operations of activity include agriculture, mining, transportation and manufacturing, the latter is chosen as best exhibiting the use of the chemical engineer along the lines previously pointed out.

Those who have made a careful study of the splendid papers of Dr. Munroe[1] and Dr. McKenna[2] to be found in the proceedings of this Institute have been impressed with the fact that nearly all of the manufacturing of the United States is carried on east of the Mississippi River, and that more than one-third of this is confined to New York, New Jersey and Pennsylvania. The following census report shows the increase in capital and people employed from 1850 to, and including, 1909:

	Capital invested in manufacturing	Employees
1850	$ 533,245,000	957,059
1860	1,009,856,000	1,311,246
1870	1,694,567,000	2,053,996
1880	2,790,273,000	2,732,595
1890	6,525,051,000	4,251,535
1900	9,813,834,000	5,306,143
1910	18,428,270,000	6,615,046

It will be seen that the investment at the latter date amounts to eighteen billion dollars and the number of employees to over six millions. To maintain the almost perpendicular increase in our manufacturing activities is the duty of the commonwealth, for no more useful type of industrial activity can be found, especially when our products can be placed in foreign markets at a profit.

The observations covered by this paper are necessarily personal and have accumulated during twenty years, ten of which have been devoted mainly to the questions now to be discussed. The observations cover fifty-three "going" plants, representing thirty-seven industries, and some others that have never gone further than a prospectus distributed by an ignorant or a dishonest promoter. Obviously, in a paper of this kind, a general outline only can be given.

In looking back over the years covered, it would seem as though the period has been one of unexpected crises—rising wages, unsettled market conditions, and acute competition. Among the fifty-three concerns above mentioned, there have been eight failures, two have withdrawn from business and one, only, has burned. The remaining industries are owned as follows:

Private	11
Corporations	19
Trusts	12

The failures were attributed to the following causes:

Two to incorrect conceptions of the costs and profits of a business.
Two to dishonest employees.
Two to incompetent supervision.
One to insufficient working capital.
One to manipulation of the stock in New York City in 1907.

The superintendents of twenty plants have been forced out or discharged for reasons such as inattention to duty, incompetency, lack of knowledge of the business, trust management, the introduction of new methods, inability to pay dividends on inflated stock issue, dissention among officers of the company or inability to handle help.

As I understand the matter, a general efficiency survey of any business must recognize the following conditions:

EXTERNAL

Policy of the government toward an industry. Capital upon which dividends must be earned.

Location.

Cost of plant and equipment.

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[1] Paper presented at the Fifth Annual Meeting of the American Institute of Chemical Engineers, Detroit, December, 1912.

[1] "The Chemical Industries of America," Chas. E. Munroe, *Trans. Am. Inst. Chem. Eng.*, **2**, 84.

[2] "The Centering of Great Industries in the New York Metropolitan District," Chas. F. McKenna, *Trans. Am. Inst. Chem. Eng.*, **2**, 65.

INTERNAL

Unit cost of finished product, subdivided as follows:

- Raw materials
- Labor
- Power
- Lighting
- Heating
- Cartage, freight and express
- Supplies
- Office expenses
- Repairs
- Sales
- Advertising
- Charity
- Fixed charges
- Interest
- Depreciation
- Insurance
- Taxes

Considering the capital invested, and the importance of the manufacturing business to all the people, laborers, tradesmen and stockholders, the Government is bound to respect, foster and protect these interests at all times. It would manifestly be suicidal to introduce any highly protected industry into the United States pending our tariff changes.

The passing of the pioneers who built and operated our first mills has thrown the responsibility of ownership upon the shoulders of many men totally unfitted by experience or temperament to carry on the business. To add to this misfortune of inheritance, all basic industries have expanded enormously, requiring more capital than one family or group of men could furnish. This led to the general adoption, between 1880 and 1900 of the corporate idea. While of the greatest value as an industry builder, no more pernicious influence has entered the manufacturing business than this conception and use of an artificial individual without responsibility.

Abundance of money in the banks, the abuse of personal credit, and easy bankruptcy laws have made it possible to squander the public's money without scruple. In several instances in my experience, good operating managers have attempted to maintain their profits on an inflated and unwarranted stock issue, and have failed. Good men, improved machinery and low operating costs cannot offset unnatural overhead expense. "Good will" has no place on the balance sheet of a well regulated and solvent business. Any venture in the elaboration of raw materials ought, when brought to a paying basis, to be able to return to the stockholders a net profit of at least 10%. Some old established lines of business net from 20 to 60% annually, and two good years have often paid for plant and equipment complete. Government statistics of manufactures included in the Thirteenth Census reports show the average net returns, for the year 1909, on thirty-nine of the principal manufactures in this country (see preceding table).

	Per cent.
Printing and publishing	20.33
Agricultural implements	11.09
Automobiles, including bodies, etc.	22.03
Boots and shoes	15.47
Brass and bronze products	12.25
Bread and other bakery products	25.86
Butter, cheese and condensed milk	23:23
Canning and preserving	13.09
Carriages and wagon and material	12.14
Chemicals	15.45
Clothing, men's, including shirts	20.29
Clothing, women's	33.03
Confectionery	23.12
Copper, tin and sheet iron	10.72
Cotton goods, etc.,	8.66
Flour mill and grist mill products	16.32
Foundry and machine shop products	10.23
Furniture and refrigerators	11.66
Hosiery and knit goods	15.42
Iron and steel, blast furnaces	5.90
Iron, steel, steelworks, etc.	9.38
Leather goods	16.73
Leather, tanned, curried and finished	6.75
Liquors, distilled	12.83
Liquors, malt	10.68
Lumber, and timber products	13.82
Marble and stone works	15.87
Oil, cotton seed and cake	12.04
Paint and varnish	13.40
Patent medicines, etc.	28.28
Petroleum, refining	8.24
Silk, etc.	13.18
Slaughtering and meat packing	15.02
Smelting and refining copper	23.25
Smelting and refining lead	5.29
Sugar and molasses (not beet sugar)	7.15
Tobacco manufactures	20.71
Woolen, worsted and felt goods, etc.	10.98

Considering now the matter of location, the manufacturer generally follows his market. As the growth of the population of the United States is westward, it has been found expedient to move whole industries from Massachusetts and New York to Michigan, Illinois and Missouri. Relocation of furniture and wagon plants has been quite general, because it has been found cheaper to ship the finished product to the markets than to bring the raw material east. Changes in market conditions have compelled the agricultural implement manufacturers to locate in the middle west. Formerly the manufacture of shoes was a Massachusetts industry; now, whole towns in New York and Missouri are devoted to this business.

No better illustration of economic change can be cited than the conditions at my own birthplace, a valley south of Utica, New York. On a stream furnishing from 60 to 120 H. P. at each plant, sixteen mills were in operation from 1873 to 1885. These employed about 2000 people. Forks and hoes were made in two plants, wagons in one, woolen cloth in two, cotton cloth in five, knit goods in one, silk in one, paper in two, and sewing machines and mowing machines in one. The movement of freight and finished products kept the railroad and scores of teams busy. Furnishing provisions for the employees in the mills afforded a living for many tradesmen.

One agricultural implement plant was burned; one was bought by a trust and closed; the paper mills have been idle for years; three cotton mills have gone out of business; one woolen mill was burned and the other was closed; the knit goods plant and the machinery plant burned; and the silk industry was moved to Philadelphia. To-day, there are only three really good companies operating in the valley. Originally humming with the whirr of spindles and clicking with the throw of the shuttles in the looms, the valley is now quiet, and agriculture is the main business. Those of us who lived there did not know why so many fires took our industries away. It was observed that no factories were rebuilt. I now know that it no longer paid to make woolen and cotton cloth, to spin silk, and manufacture paper in that locality. The peculiar elements which we term economic conditions took our prosperity away, with incalculable loss to all concerned, stockholders, employees and tradesmen alike. The moss-grown walls to be seen on many swiftly moving streams are monuments that mark industrial change.

Capital is sometimes invested in new projects, the aim of which is the use of raw materials that are supposed to be abundant, but that are later found to be insufficient in quality or quantity. Cement plants along the Erie Canal in New York State represent this class and also illustrate the rapid changes that may enter the elaboration of a product. In this instance the use of shale rock has taken the place of marl and clay in the manufacture of cement.

Beet sugar factories were started at Lyons, Binghampton, and at Rome, N. Y. The cost of the plant in each instance was very large, people cheerfully investing their money and expecting unusual returns. After several years of failure and loss the entire project has been given up; the empty buildings now remain after an expenditure of not less than $2,000,000. Colorado and California produce beet sugar at a profit. The location of the industry in New York state was a mistake, but the stockholders had to learn this. In the meantime, canning factories have sprung up throughout central and western New York and the well managed ones are in a flourishing condition. Cheap raw materials, plenty of help, excellent transportation facilities and a ready market are contributary causes to its success.

No science or art can determine with accuracy whether economic conditions are correct for the location of an industry. Accident or good business judgment may accomplish what statisticians and scientists cannot.

COST OF PLANT AND EQUIPMENT

With plenty of money at his disposal, the optimistic manufacturer is apt to spend too great a portion on buildings and equipment. Ample working capital should always be held in reserve. The amount thus employed will necessarily vary with the business, but from 25 to 50% is commonly set aside for this purpose in smaller industries, the capitalization of which is from $50,000 to $200,000.

It is much better to build a plant in a modest way, anticipating growth, than it is to find capital for running a concern in the midst of business expansion; especially so, if stockholders have had to wait from three to five years without dividends.

Again, a very expensive plant may be erected with consequent large overhead expense, where the income does not warrant the outlay. I found it impossible to recommend the erection of a water-gas plant in a town of 3500 people. A canvass of all prospective users showed that not more than two or three per cent. income could be expected. A similar plant in a town of about this number of people can positively pay no more than two per cent. on the investment after all avenues for gas consumption have been thoroughly exploited.

LOCATION

To aid those who wish to study the problems concerning location, I have made a list of the elements that seem important. These are as follows:

Accessibility of raw materials
Market
Transportation
Labor
Power
Water
Supplies
Climate
Hygienic conditions
Taxes
Insurance
Banking facilities
Heating
Lighting

Obviously, all of these details cannot be discussed in a general paper. The importance of one item, water, is taken up in another paper to show how carefully capital should investigate a new location, when the growth of a business warrants a change or when misfortune requires it.

With a favorable location, a demand for the product, an honest stock or bond issue, a modest initial outlay for buildings and equipment, and sufficient capital for doing business, an executive should earn a fair dividend on the investment.

INTERNAL UNIT COST

The ideal is the basis of our best effort everywhere. In manufacturing, this deal is approached when the largest quantity of good goods is turned out at the lowest possible cost. To determine how to attain the above conditions is the duty of every executive. But the task is not a simple one, for bookkeepers are not statisticians and although all of the data necessary in connection with the proportioning of expense in the manufacture of an article may be derived, it takes a large amount of study to draw correct conclusions. No simpler expedient in my estimation has been devised in this connection than unit cost. What does it cost to produce a pound, a horse power, a barrel, a yard, or a machine, any single article, many of which are made each month or year? These items may be apportioned as percentages. The separate items under fixed and operating expense become factors of the total. As an example, we will say that the production of a machine has entailed an outlay of $25.00 and that this is divided as follows:

	Per cent.		Per cent.
Raw materials..........	20	Repairs..................	1.3
Labor..................	40	Sales....................	10
Power..................	7	Advertising..............	5
Lighting and heating.....	1	Charity..................	0.2
Transportation..........	3	Interest, depreciation, taxes and insurance....	10
Office expenses..........	2.5		

This outline shows at a glance that labor is a large item in the cost of this article and that any economy that can be effected in this department will make an appreciable saving in the total outlay.

Through the kindness of Mr. E. Durand, of the Bureau of Census, I am able to present a comprehensive table illustrating the method above outlined:

STATISTICS OF MANUFACTURE. THIRTEENTH CENSUS, 1910, PAGE 30

	PER CENT. OF TOTAL EXPENSES REPORTED			
INDUSTRY	Salaries	Wages	Materials	Miscellaneous expenses
All industries..........................	5.1	18.6	65.8	10.5
Agricultural implements..................	8.6	24.3	51.1	16.0
Automobiles, including bodies and parts...	4.5	23.1	62.5	9.9
Boots and shoes, including cut stock and findings..........................	3.9	20.6	69.6	5.9
Brass and bronze products................	4.1	17.3	72.6	6.0
Bread and other bakery products..........	4.0	17.4	69.9	8.6
Butter, cheese and condensed milk........	1.4	4.3	91.0	3.3
Canning and preserving...................	5.6	13.5	72.0	9.0
Carriages, wagons and materials..........	5.7	27.0	58.9	8.4
Cars, general shop construction and repairs by steam R. R. companies............	4.3	44.7	49.2	1.8
Cars, steam railroad, not including operations of R. R. companies...............	4.3	23.0	66.7	6.0
Chemicals................................	6.5	15.0	68.2	10.3
Clothing, men's, including shirts........	5.2	20.7	57.9	16.2
Clothing, women's........................	6.0	23.0	61.1	9.9
Confectionery............................	7.6	13.1	67.9	11.4
Copper, tin and sheet iron production.....	5.8	22.4	63.7	8.1
Cotton goods, including cotton small wares	2.6	24.0	66.9	6.5
Elect. mach., apparatus and supplies......	10.0	24.5	53.8	11.7
Flour mill and grist mill products........	1.5	2.6	92.8	3.1
Foundry and machine shop production....	8.7	29.8	50.1	11.4
Furniture and refrigerators..............	7.3	30.8	51.0	10.9
Gas, illuminating and heating............	10.9	18.4	46.2	24.5
Hosiery and knit goods...................	4.4	25.5	62.7	7.4
Iron and steel, blast furnaces...........	1.8	6.8	88.4	3.0
Iron and steel, steel works and rolling mills	2.9	18.3	73.9	4.8
Leather goods............................	7.2	19.3	64.6	8.9
Leather, tanned, curried and finished......	2.2	10.5	81.2	6.1
Liquors, distilled.......................	1.0	1.6	18.4	79.0
Liquors, malt............................	7.6	13.7	32.2	46.5
Lumber and timber products...............	4.8	32.0	51.0	12.2
Marble and stone work....................	6.7	44.8	39.4	9.1
Oil, cottonseed and cake.................	3.1	4.3	87.7	4.9
Paint and varnish........................	9.3	7.4	71.1	12.2
Paper and wood pulp......................	4.0	17.2	69.7	9.1
Patent medicines, compounds and druggists' preparations......................	14.9	8.7	44.1	32.4
Petroleum, refining......................	1.8	4.4	89.6	4.2
Printing and publishing..................	16.7	26.6	32.6	24.1
Silk and silk goods......................	4.2	21.8	60.8	13.2
Slaughtering and packing.................	1.5	3.9	91.3	3.3
Smelting and refining, copper............	0.7	3.8	94.4	1.1
Smelting and refining, lead..............	0.9	3.4	94.8	0.9
Sugar and molasses, not including beet sugar..................................	0.9	2.8	92.6	3.7
Tobacco manufactures.....................	4.6	19.0	48.4	28.0
Woolen, worsted and felt goods and wool hats..................................	2.6	18.7	72.9	5.8
All other industries.....................	6.4	21.1	62.1	10.5

This is a valuable guide in any efficiency study of production costs, although too many items are grouped under expense, which may be used by any executive to conceal exorbitant bills of any nature.

The actual cost of a case of tinned goods of the season of 1912 is divided as follows:

	Per cent.		Per cent.
Raw materials.........	30.40	Interest................	1.90
Labor.................	14.40	Depreciation...........	1.80
Fuel..................	0.17	Taxes and insurance.....	1.60
Freight and express.....	0.04	Boxes and labels.........	7.30
Maintenance...........	4.80	Loss on seed............	5.80
Sales.................	3.90	Discount and brokerage..	3.10
Advertising...........	0.90	Expense................	8.70

The remaining items are not considered useful in this paper. The tin container itself includes a cost of 85% for materials and 15% for labor.

A woolen mill owner and operator furnishes the following table:

MATERIALS	LABOR	EXPENSE
Raw stock	Office	Insurance
Soap	Factory	Taxes
Dyes	Overseers	Charity
Wool		Executive salaries
Coal		Depreciation
Wool oil		Repairs
Cotton and shoddy		

Percentages were not given but the total annual expense, including the three subdivisions above shown, is divided by the number of yards of cloth turned out to determine the cost price per yard.

I am familiar with the actual cost of reducing a ton of garbage by the naphtha process. This, on a percentage basis, is as follows:

	Per cent.		Per cent.
Labor	47.43	Superintendent	3.97
Coal	20.81	Taxes	0.45
Pressing	12.37	Interest	0.28
Filter cloth	2.89	Office	0.24
Gasoline, oil and light	8.00	Commission and analyses	0.74
Freight	1.77		
			99.94

Labor is shown to cost an excessive amount. The gasoline item needs investigation.

The following elements compose the cost of a small copper instrument:

	Per cent.		Per cent.
Labor	61.62	Paint	0.22
Raw materials	27.41	Screws	0.22
Burner	6.03	Lacquer	0.55
Hanger	1.31	Bolts	0.33
Polishing	1.64	Solder	0.11
Support	0.54		

Obviously the labor cost of the instrument is excessive.

The official having charge of the cost department should ascertain at fixed intervals, by inventory and from records, the exact unit cost of any or all articles produced. This information should be made a part of a blue-print chart, carrying at the left a list of the items composing the record and a continuous line, showing the fluctuations in the cost of each, from month to month. If any one of these lines arise from causes beyond the control of the management, others must fall if the price of the finished article is to be maintained. Heating and lighting will fluctuate with seasonal changes; interest and depreciation, charity and office will remain practically horizontal. Unless the industry controls its own raw materials these usually increase in price; labor of all kinds increases; power is more expensive because coal is higher in price and water power has, in many plants, been replaced by steam. There is a very gradual rise in transportation costs and in supplies; taxes and insurance are constantly increasing everywhere.

With such records available, the operating manager is able to determine where efficiency methods could be applied to advantage.

In a former paper before the Institute, I have discussed "Power" from a percentage basis. Since that time, the use of electricity has become quite general and the H. P. year cost has been considerably lowered in a large number of plants.

STOCK AND SUPPLIES

The raw materials needed in operations of the manufacturer are usually closely bought and economically handled. I have found this particularly true in woolen, cotton and paper mills.

Every successful enterprise requiring raw stock must include an expert buyer of large experience, whose business it is to make a study of market conditions and fluctuations. Ten years ago, much more practical experience was required to fill such a position than at present. This has been brought about by the general introduction and use of specifications. One by one, natural products have been standardized; coal, wool, cotton, ores, oils, paper stock, iron and steel and natural earths; these are a few of the hundreds of raw materials used by manufacturers. Physical and chemical standards have been set to replace the guess and estimation methods that came from experience and that are often wide of the truth. A relatively inexpensive man who has a testing laboratory at his disposal can determine the actual value of raw stock much more accurately than some high-priced man can guess at it. Two items on our cost sheet can be turned downward and kept there at relatively small expense.

Unless the specification idea is carried throughout the mill to include the finished product, the work is incomplete, for the adoption of such a system invariably improves the quality of the goods turned out. For example, every piece of wood, of composition and metal that goes into an automobile or locomotive should be of the best quality, proved to be so by actual experiment. This rule holds for manufactured products generally.

One of the weakest points in the personnel of the mill organization to-day is the purchasing department. I refer more particularly to those in charge of the purchase of the supplies. This important branch of the business is often left to incompetent clerks, who antagonize salesmen generally and who buy from men from whom they get the greatest possible return—gifts, dinners, an occasional trip, or even money. Honest traveling men avoid such purchasing agents, to the permanent loss of the business.

A shrewd salesman may spend from six weeks to six months in placing an order for expensive equipment that a concern never needed and should not buy. In my experience, men totally remote from the ordinary purchase of supplies should be employed in connection with new and valuable equipment. Consulting engineers can act to good advantage, turning in reports that show the general market conditions, kinds of apparatus or material available, with the experience obtained from the use of these in other plants.

SUPPLIES

Every manufacturer uses mixtures and compounds, the exact nature of which he has no knowledge: oils, dyes, fillers, adhesives, cleaning agents, waxes and polishes, powders and salts. In some instances, such materials have a total cost of $2000 per month. Many simple substances are sold in large quantities, at inflated prices. For one concern, I was able to lower the cost of a special substance bought for $50 a ton by substituting the same material from another source at $15 per ton. Good business requires a knowledge of supplies and their component parts, for the purpose of keeping the cost down, for the protection of workmen, and to guard against fire.

No feature of factory economy should be as closely watched as the storeroom. A storekeeper should be in charge every moment of the working day, and should be held responsible for all stock handled, distributing this over a counter only, and never allowing workmen to come behind this. All orders should be signed by proper authority and a carbon copy of each transaction kept. Tools, in particular, and all stock that can be used about home, barn or garden will mysteriously leave the plant, a few cents worth at a time, if a way is found to handle the matter with an easy conscience.

SUMMARY

In the foregoing paper, I have attempted to point out to you certain methods that can be adopted in the conduct of any manufacturing business. The days of large profits, cheap raw materials and labor have gone for good. With increasing prices and competition, all executives must be economists as well. Efficiency, in its broadest sense, should include promotion, capitalization, location, organization, equipment and operation. Great stress has been laid on the efficiency of labor to the exclusion of matters of equal or greater moment. An exact knowledge of unit costs will reveal the weak points in any plant. The so-called efficiency engineer who spends three or four days in a concern and antagonizes everyone from the bosses to the office boy, can accomplish little good. Weeks and months of study are required to get at the details of the business; to make improvements is a still greater task. The good-will of the employees must be gained and kept to make any progress in economy studies. Discussion and argument naturally follow any change in policy. The man who makes the change should be on the ground to defend himself and drive home the facts as he sees them.

Syracuse, N. Y.

PERKIN MEDAL AWARD

The Perkin Medal was conferred on James Gayley for his distinguished services in the field of chemical engineering and metallurgy at the regular meeting of the Society of Chemical Industry, Chemists' Club, New York, January 24, 1913.

Mr. Gayley's study of the blast-furnace reactions covering a period of many years and his subsequent development, on the basis of these observations, of the dry-blast processes, as well as numerous other inventions in this field are fully set forth in the presentation address by Professor Charles F. Chandler and the address of acceptance by Mr. Gayley. Professor Henry M. Howe spoke on "The Value of Expert Opinion," particularly as applied to Mr. Gayley's work and Prof. Edward Hart gave a most interesting and intimate review of the career of the medalist. The program was completed by an address on "The New Age" by Dr. Rossiter W. Raymond.

At the conclusion of the program, the Society and its guests were entertained at an informal reception given by the Chemists' Club.

The addresses presented at this meeting are printed in full in the following pages. [Editor].

PRESENTATION ADDRESS

By C. F. Chandler

Mr. Chairman, Brother Chemists and Mr. Gayley:

It is my very pleasant privilege, as the senior Past-President of the Society of Chemical Industry, residing in this country, to present to Mr. James Gayley, the Perkin Gold Medal, for important achievements in Industrial Chemistry.

This medal has been awarded to Mr. Gayley by the Perkin Medal Committee of the Associated Chemical and Electrochemical Societies of America in recognition of his most valuable work in Chemical Metallurgy.

James Gayley is the maternal grand nephew of Sir Henry Bell, who established steam navigation on the Clyde, where he launched "*The Comet*" in 1812. He was born at Lock Haven, Pa., October 11, 1855, the son of Samuel A. and Agnes (Malcolm) Gayley.

He was educated at West Nottingham Academy, Md., and graduated from Lafayette College in 1876 with the degree of Mining Engineer. In 1906 he received from the University of Pennsylvania the Honorary degree of Doctor of Science, and in 1912 the same degree from Lehigh University. In 1908 he received the Elliot Cresson Gold Medal for the Promotion of the Mechanic arts from the Franklin Institute.

Mr. Gayley began his professional life as chemist for the Crane Iron Company, Catasauqua, Pa., 1877–'80. He was next superintendent of the Missouri Furnace Company, St. Louis, and later was the manager of Blast Furnaces, E. & G. Brooks Co., Birdsboro, Pa., 1880–'85. In 1885 he became manager of the Blast Furnaces at the Edgar Thomson Works, and he was subsequently promoted to the position of manager of the Edgar Thomson Works: he later became a managing director of the Carnegie Steel Company. In 1901, he was made first vice-president of the U. S. Steel Corporation, remaining in this position until 1909. He was the inventor of furnace improvements, bronze cooling plates; stand for ladle in pouring Bessemer heats and the dry air blast.

Were it not that we have with us to-night Mr. Gayley himself, it would be my duty and pleasure to give you a history of his various most valuable contributions to metallurgical chemistry. But as you would certainly prefer to learn this from his own lips, I will content myself with saying that I can not recall a more far-reaching invention than that of the dry air blast for the manufacture of iron, for which he has received, between October 23, 1894 and September 5, 1911, no less than fifteen successive patents in this country. I am told that his results in this direction alone mean a reduction of from one-half to one dollar per ton in the cost of producing pig iron, besides making it possible for the iron master to produce, in all weathers, a product of uniform quality. When one remembers that there were produced in the United States, during the past year, twenty-nine millions of tons of pig iron, it will be seen that this, Mr. Gayley's invention of the dry air blast, means a saving to the American people of from $15,000,000 to $29,000,000 per annum.

I have presented to you very briefly the great achievements of Mr. Gayley in the field of Applied Chemistry, but quite fully enough to satisfy you that your Committee is fully justified in placing Mr. Gayley by the side of Sir William Perkin and the previous recipients of the Perkin Medal, as one of our greatest Industrial Chemists and Chemical Engineers.

To Mr. Gayley:

It gives me great pleasure, as the representative of the Society of Chemical Industry, and the affiliated Chemical and Electrochemical Societies, to place in your hands this beautiful token of the appreciation and affection of your fellow chemists.

ADDRESS OF ACCEPTANCE

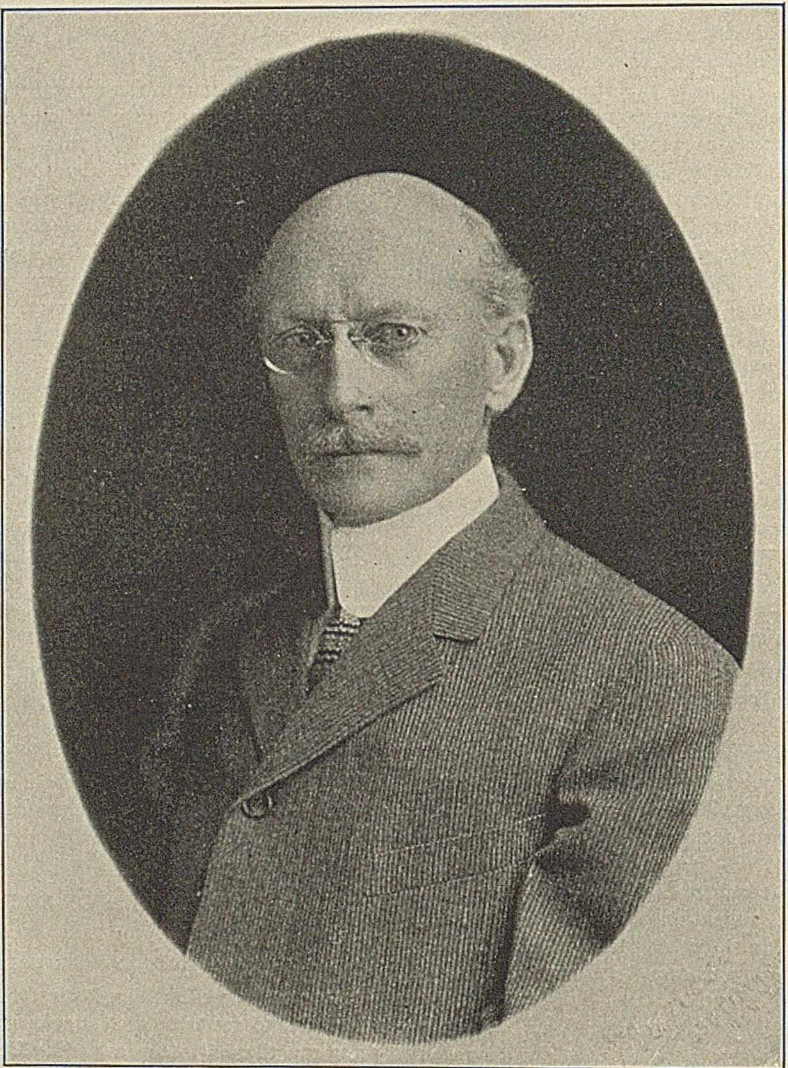
By James Gayley

Mr. Chairman, Ladies and Gentlemen:

I wish to thank the speaker for this medal which he has presented to me with such gracious words. I wish also to express my thanks to the awarding Committee and the societies which they represent in conferring this great honor upon me. I appreciate it still more because the Committee have stepped aside from what is purely the chemical industry to another great industry, that of metallurgy, which, nevertheless, is one in which the application of chemistry is the controlling factor, and this recognition of the broader field of chemistry brings with it a keen sense of appreciation of this rare honor and distinction conferred upon me. Again I thank you.

The iron blast furnace is but the chemist's crucible on a gigantic scale—operated on chemical principles and the mechanical appliances are instruments of precision, constructed and adjusted to carrying out in efficient manner the chemical reactions involved.

The "rule of thumb" practice, which ended thirty-five to forty years ago, is well-nigh forgotten—that period in which chemistry was not applied, and a ton of ore was a ton of ore all the world over, irrespective of its reducibility or chemical composition; when the iron master was more interested in the output of 1.x iron than in the fuel consumption, and would tell you that "lime made heat," and David Thomas, that grand old pioneer of the anthracite iron industry, summed up the requisites for successful furnace practice as "wind and heat." The application of chemistry to iron smelting has effected a wonderful transformation in that industry, so that to-day the chemist occupies the foreground in directing its operations.



JAMES GAYLEY, PERKIN MEDALIST, 1913

Moisture in the atmosphere has long been recognized as a variable and influential element in the manufacture of iron. Truran, in his "Manufacture of Iron," written in 1862, refers at length to the influence of moisture on the quality and make of iron, and the increased consumption of fuel in the summer season, but does not touch on the problem of its regulation or removal.

Later, in England it was proposed to extract the moisture by passing the air over lumps of chloride of calcium, but Sir Lowthian Bell showed the impracticability of that scheme on account of the rapidly diminishing power of absorption possessed by the calcium chloride.

Charles Cochrane, an eminent iron manufacturer of Great Britain, proposed and obtained a patent on extracting moisture from the air by bringing it into intimate contact with liquid calcium chloride flowing over chains suspended in a chamber; but later he declared as to the benefits to be derived from extracting moisture that "the game was not worth the candle." Nevertheless, the moisture in the atmosphere was recognized as a disturbing element. Some of the earlier makers of anthracite iron in the Lehigh Valley stated that they frequently knew in the morning what kind of iron their furnaces were making by observing the weather conditions; yet, prior to the first dry air installation in 1904, this variable element of moisture was accepted in a spirit of resignation, as something to be endured, like storm and sunshine, a condition beyond control.

It was my privilege to be invited in 1885 to take charge of the Edgar Thomson Blast Furnaces of the Carnegie Steel Company, a plant which represented the most advanced construction in blast-furnace equipment, and was supplied with the best fuel and ores. In the operation of these large furnaces, the influence of the atmosphere was forcibly impressed, as the steel works were being supplied with hot metal direct from the furnaces, for the Jones metal mixer had not come into use. It was a difficult matter to supply metal approximating uniformity even under the best conditions, and quite impossible under conditions of varying atmospheric humidity.

For several years observations were made twice a day of the moisture in the atmosphere, and a direct relation was established between the quantity of moisture and the grade of metal, with particular reference to the silicon which is the heat-producing element in the acid Bessemer converter. In the summer season, the air blown into the converter was heavily charged with moisture, and in consequence, a higher percentage of silicon in the metal was normally required, while at the same time these atmospheric conditions operated on the furnaces to produce a lower silicon metal; thus the operating of furnaces in conjunction with Bessemer converters on direct metal may well be presumed to have been a difficult and trying proposition.

Under these conditions of manufacturing, the removal of the moisture from the atmosphere, or rather its reduction to a low degree, and one of practical uniformity, appeared to hold out the only means of effecting regularity, and at the same time to promise a considerable saving. It was a matter of constant observation that a sudden change in the weather, making a quick drop in the temperature and content of moisture, would cause the furnaces to work excessively hot, but this period of dryness could not be taken advantage of, for before any changes could be made effective in the furnace charge to meet current atmospheric conditions, the moisture might have increased to its normal seasonable amount.

Observations of the humidity in the atmosphere were made daily from 1886–1890, when the first experiments for moisture removal were begun. These observations were made, at the beginning, in the morning and evening, and later were made every hour. Observations were also made of rain storms, to determine the moisture before, during and after the rain, and it was noted that the moisture usually dropped quickly as the rain began to fall. Throughout this period of moisture tests, the working of the furnace was observed in its relation to the amount of moisture in the atmosphere.

A modern furnace consumes about 40,000 cu. ft. of air per minute, and for each grain of moisture per cubic foot, there enters the furnace one gallon of water per hour for each 1000 cu. ft., that is, if the moisture was 1 g. per cu. ft., 40 gallons of water would enter the furnace per hour.

Table I—Variation of Moisture in the Pittsburgh District

	Average temperature ° F.	Weight of water per cubic foot of air Grains	Water per hr. entering a furnace using 40,000 cu. ft. of air per min. Gallons
January	37.0	2.18	87.2
February	31.7	1.83	73.2
March	47.0	3.40	136.0
April	51.0	3.00	120.0
May	61.6	4.80	192.0
June	71.6	5.94	237.6
July	76.2	5.60	224.0
August	73.6	5.16	206.4
September	70.4	5.68	227.2
October	56.4	4.00	160.0
November	40.4	2.35	94.0
December	36.6	2.25	90.0

While the moisture in January averaged 2.18 grains, yet the total variation for the month was from 0.56 g. to 2.55 g., and while June shows an average of 5.94 gr., yet the extreme variation was from 4.8 g. to 8.50 g., or in other words the moisture entering the furnace varied from 192 to 340 gallons per hour. During the midwinter period, the moisture may vary as much as 150 per cent. in the same day, but in midsummer it rarely varies more than 25 per cent.

Having secured extensive data on the varying humidity of the atmosphere over a period of five years, the problem of making those conditions uniform next presented itself.

The experience of English engineers was against the absorption process with calcium chloride, and my predecessor at the Edgar Thomson Works, Mr. J. H. Cremer, had constructed an apparatus to desiccate in the same way, but be obtained very unsatisfactory results. As our observations showed a reduction of moisture through a reduction of atmospheric temperature, the reduction of temperature through mechanical refrigeration presented itself as the obvious method of removing the moisture, and on this basis the first experiment was undertaken in 1890 in a crude way.

We began by passing air through two chambers in succession; in the first chamber the air came in contact with pipes cooled by water, and in the second chamber anhydrous ammonia was expanded through the pipes. As the air came from the blowing engine it was quite warm, but after passing through the chambers it was very cold; each chamber was only six feet long and the volume of air treated was small indeed.

Various forms of apparatus were constructed from the period 1890–1895, and tests were made as opportunity offered. During this period my duties had been changed from that of superintendent of the furnaces to general superintendent of the whole works, thus adding to my duties that of the steel works and mills. There was in consequence not much time free to make experiments, for the demands of a large steel works on its manager's time were persistent and exacting, and experiments and improvements along other well defined lines represented more immediate profit and demanded thought and attention. But the general plan for a final test on a large scale had been worked out, which was to be tried later. In 1896, I left the active management of the works to enter upon the general business of the Carnegie Steel Company in connection with the ore department and the assembling of all raw materials. If the opportunities for experimenting were rare when stationed at the works, they became more so, when located in the general office.

I therefore secured the services of a young engineer, Mr. Walter, from a refrigerating company, and when they learned the nature of my experiments they kindly loaned me a small ammonia compressor to carry on the work, and we used two air drying chambers—each four feet square and eight feet long, and filled with pipe coils, and with the necessary refrigerating appliances. A blast engine with an air cylinder three feet in diameter and capable of supplying 3500 cu. ft. of air per minute was installed, and from this engine and its small air supply the data were worked out for treating 40,000 cu. ft. of air, which was the quantity required to supply a modern blast furnace.

I mention that we had to work out the necessary data for a practical demonstration, and the suggestion might arise, why did we not avail ourselves of the experience of refrigerating firms? Indeed we tried to—the proposition was put up to several important makers of refrigerating machinery, but without any substantial benefit. To refrigerate the air for cold storage rooms was a specification they readily understood, but the treating of a hurricane of air was an entirely different problem to them. We were therefore compelled to work out the data ourselves and make out our own card of estimate.

It is unnecessary to give in detail the many and varied experiments made in treating the air, nor to describe how certain obstacles, which seemed to block our progress, were successively overcome. In our list of experiments we tried rarefaction with refrigeration but we did not obtain satisfactory results.

About the year 1900, we had plans worked out in detail for a plant to treat 40,000 cu. ft. of air per minute, and I then applied to my company for an appropriation of $100,000.00 to make this installation; here my most difficult work began—to persuade my associates that air really did carry moisture and that it could appreciably, even in a small degree, affect the working of a blast furnace.

It could hardly be expected that men who came up through the ranks of the steel works or office could appreciate the influence of atmospheric changes on a blast furnace that to all external appearances was crude and rough, but which in its interior adjustment was really on a most delicate balance. Money was readily appropriated for the mechanical appliances of the furnaces, or for securing uniformity in the physical and chemical composition of the ores, coke and limestone, and a variation therein of 10 per cent. to 20 per cent. was promptly corrected, but the atmosphere might vary from 20 per cent. to 150 per cent. even in a single day, without giving any concern, for few realized that the weight of air consumed per ton of iron was 50 per cent. greater than all the other raw materials combined. In other words, it requires about four tons of ore, coke and limestone to make a ton of pig-iron, while six tons of air are required.

The most difficult problem I had to encounter was to convince others of the merit of the process after its details of construction and operation had been worked out, and it was not until 1903 that I secured an appropriation to construct the first plant, although I had offered the year before to assume personally half the expense.

Early in June, 1904, the dry air plant, which was erected at the Isabella Furnaces at Pittsburgh, was in proper working order and ready to be put on the furnace. It was necessary to apply the dry air gradually so as not to disturb the furnace adjustment, and as there were three blast engines we arranged to first supply dry air from one engine, making the blast one-third dry air. We decided to apply this dry air at 9.00 A.M., and I had the weight of ore charge increased by 5 per cent., and this increase was timed to reach the combustion zone of the furnace at 11.00 A.M. The furnace manager regarded with dismay the addition of 5 per cent., protesting that a 2 per cent. increase was about the limit, and if the dry air did not prove efficient, then he would have a very cold furnace on his hands. Notwithstanding, the change was made. When the dry as was turned on at 9.00 A.M., the furnace soon showed the effect of it, the gas became grayer, a sure sign of the furnace working hotter, and when the 5 per cent. increase in ore had reached the hearth, it was not discernible that the furnace had been given an increased duty.

The operations of the furnace were conducted on a conservative basis, and the long time spent in working out and developing the air drying plant was rewarded, for the plant started and continued to perform its work without a hitch. By the sixth day the furnace was being supplied with two-thirds dry air and one-third natural air, and 9 per cent. increase in ore charge had come to work, the furnace continuing splendidly. I might add here that the 9 per cent. in weight of ore charge represented the same percentage increase in output, and a like reduction in the amount of fuel used per ton of iron.

On the afternoon of that sixth day in the use of dry air, I left the works, well satisfied with the progress made, and confident of the future by reason of the many little indications easily recognized by practical furnacemen, that a still heavier duty would be responded to.

I had not gone far when the sound of an explosion reached me, and, on turning, I saw great clouds of steam rising from the works. I hastened back, and feeling my way through steam and dust, I was dismayed to find that the refrigerating chamber had collapsed. There it lay a tangled mass of concrete and distorted pipe coils.

During a part of the time employed in erecting the dry air plant there was a bricklayers' strike, and the works manager, not wishing to delay construction, built the refrigerating chamber of concrete. The dry air blast conduit leading from the top of the chamber to the blast engines was constantly vibrating from the impulse of air and the vibrations of the engines. These were in turn communicated to the dome of the building, weakening the bond of the concrete; this caused the walls to collapse; in their fall they broke the steam pipe leading to the compressors. The débris was cleared away, and in less than two months the plant was again started. We began as before, and in two weeks' time had secured a fuel economy of 15 per cent., and then by small increments we gradually worked up to a point of 20 per cent. efficiency; that is, we effected a fuel saving of 20 per cent. and increased the output to the same extent. This practice was continued for about six months, but it was operating too close to the danger line to take care of irregularities in the raw materials, and the fuel economy was adjusted to 15 per cent. Even with dry air blast there is a disinclination to work up to the high limit of efficiency, for the experience of every furnace manager with natural air has shown the necessity of keeping a good margin of safety.

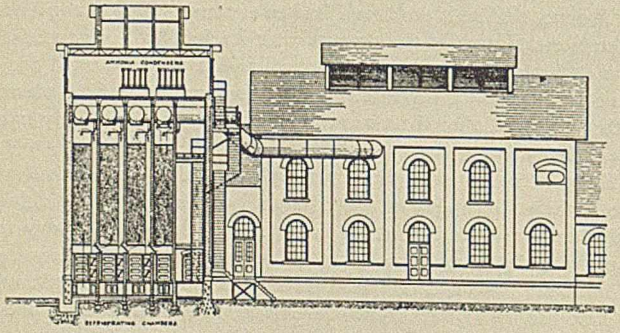


FIG. 1—SECTION OF REFRIGERATING CHAMBERS AND ELEVATION OF BLOWING ENGINE HOUSE

The apparatus for drying air is shown in the two models presented. In the first model, which represents our first installation, there is a chamber filled with pipes, and the air is cooled by indirect contact with cooling fluid, which is brine. The temperature of the brine is such that the air entering the chamber at 80° is discharged at 23°.

The second model represents a two-stage process, in which the air is brought into direct contact with refrigerated water in the first chamber, and is reduced to about 35°; then passes to the second chamber filled with pipe coils through which brine circulates and the final temperature is obtained. The two-stage method is more cheaply operated, and costs a third less to build.

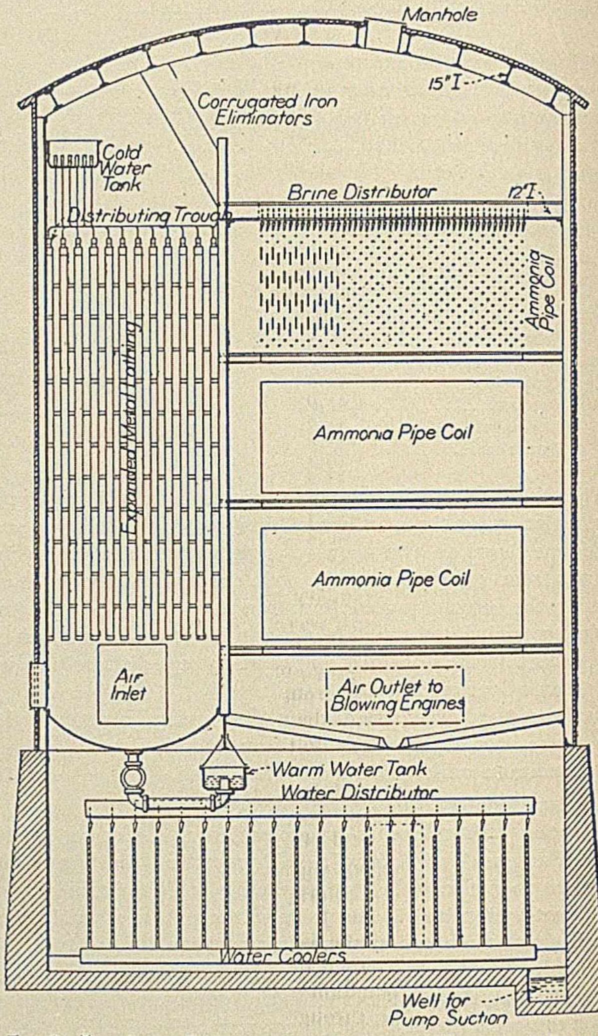


FIG. 2—TWO-STAGE REFRIGERATING CHAMBER. COOLING BY DIRECT AND INDIRECT CONTACT

It was not our purpose throughout our experiments to work out the most economical method of drying air for the first installation. What we aimed at was to construct a plant that

TABLE II.— GALLONS OF WATER DELIVERED TO FURNACE IN 24 HOURS

July, 1910	Natural air	Dry blast	July, 1910	Natural air	Dry blast
1	5,349	834	17	4,116	809
2	5,517	849	18	3,588	809
3	5,438	829	19	3,410	809
4	4,432	814	20	3,915	819
5	5,488	844	21	6,119	809
6	7,797	849	22	4,678	814
7	5,428	844	23	5,876	814
8	5,873	799	24	6,327	834
9	6,633	829	25	5,152	819
10	5,981	819	26	6,450	829
11	4,511	785	27	5,764	809
12	6,203	804	28	5,453	809
13	4,002	779	29	6,184	819
14	4,713	785	30	4,525	799
15	5,828	809	31	4,288	782
16	5,522	819			
Total, July				164,560	25,254
Equivalent in barrels of 42 gal. each				3,918	601

would efficiently and continuously produce dry air, in order to determine its economical value in the furnace.

Fig. 3 shows the variableness of moisture in natural air, and the regularity of dry air in the month of July, 1910. Table II shows the meaning of the curves in Fig. 3 when translated into terms of gallons and barrels of water.

The quantity of water extracted in July, if placed in barrels, would make a row of barrels about one and one-half miles long.

The records obtained at several works from the use of dry air, and which in each case cover a considerable period, are as follows:

Works	A	B	C	D
Output, percentage increase......	23.0	15.3	11.8	16.0
Coke used, percentage decrease...	10.5	6.0	9.8	10.5

The results obtained at different plants are modified by the raw materials used and the personal equation of the management enters into it. The results given above are taken from works in three different countries. *The claim I personally make for the process is that with an increase in output of 10 per cent., the saving in fuel per ton of iron will be reduced 10 per cent., and I consider this conservative.*

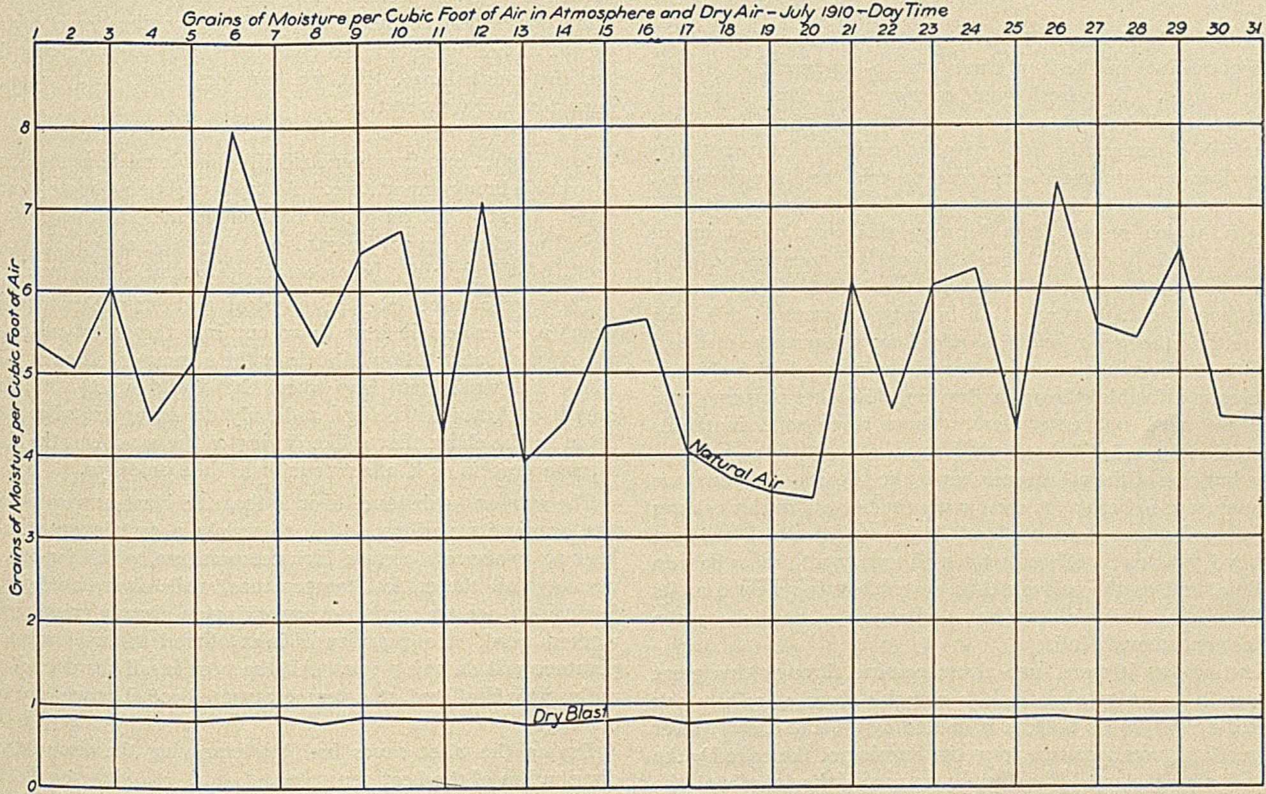


Fig. 3—Results with the Gayley Dry Blast. Graphic Chart of the Performance of a Western Dry-Blast Plant

One of the advantages obtained through dry air is the elasticity it permits in furnace operations to meet business conditions. If trade is dull the furnace can be operated to secure the maximum of fuel economy with a small increase in output, but when trade is booming and pig iron is in demand, an increased output is more desirable than a saving in fuel, and the furnace can readily be made to respond.

The manager of the Dowlais Works, at Cardiff, Wales, experimented with one furnace to determine what results could be obtained under each condition of operating.

For a period, the furnace was operated principally to secure increased output and obtained an increase of 26 per cent. with a fuel saving of 12.3 per cent.

In the next period they operated principally to obtain decreased fuel consumption and obtained an increase in output of 12 per cent. with a decrease in fuel of 17.4 per cent.

In addition to the advantages already mentioned, it is found that the furnace works with greater regularity and, in consequence, a more uniform metal is produced, and a required grade of metal can be made with precision.

In low-silicon irons the sulfur does not increase as when natural air is used.

The saving in coke effects a corresponding saving in the limestone necessary to flux the ash of the coke, and there is less phosphorus entering the metal.

As the furnace charges settle more regularly, there is less fine ore carried over with the gases.

A manager of blast furnaces using dry air recently said that they found it to be an "educator" in their practice, as it had taught them the advantage of securing regularity in many other directions.

I shall not attempt herein to set forth any of the theories advanced to account for the fuel saving, which has been found to be in excess of what is necessary to dissociate the moisture. That feature has been ably discussed by Dr. Henry M. Howe, Dr. R. W. Raymond, Prof. J. W. Richards, Mr. J. E. Johnson, Jr., and many others in this country, and also by metallurgists in England, Germany, and France; their full discussions can be found in the *Transactions of the American Institute of Mining Engineers*, the *Journal of the British Iron and Steel Institute*, and metallurgical journals.

Towards a full consideration of the dry air blast, a few points may be briefly referred to.

It did not come into its work as did the Neilson hot blast in 1828, when the art was crude, and the appliances of the furnace were poorly adapted to the work. But to the Scotchman, Neilson, all credit is due for effecting a fuel saving of thirty per cent.

Dry air instead was tried out when the equipment of the furnace was as perfect as human skill could make it. The nature of furnace operations was well understood, and its management was on a skilful and scientific basis.

Nor was it possible for dry air blast to have an experimental stage. Nearly every device or process can be tested in an experimental way, and on a comparatively inexpensive scale.

Bessemer could blow air through molten metal in a pot and demonstrate the value of the pneumatic process, but it would have been of no value to build a small plant to refrigerate a part of the air; nothing conclusive would have been gained. Nor would it have been of any value to treat the whole air supply of a diminutive or toy furnace. To efficiently demonstrate its value, it had to be applied to a furnace that was equipped and operated according to the most advanced state of the art, and not only was it essential that the whole air supply should be treated, but that the method and means of treatment should measure up in capacity and efficiency, and operate as continuously as any of the other accessories of a modern blast furnace.

This, in brief, is the story of the dry air blast.

THE VALUE OF EXPERT OPINIONS[1]

By Henry M. Howe

Interesting as are the other aspects of the Gayley process its greatest interest lies, I think, in the light which it throws on the nature of expert evidence and on the value of expert opinion.

What is this process? It is simply drying the air used for burning the fuel in the iron blast furnace—apparently a rather simple matter. To refresh your memory, the blast furnace is a huge vertical firebrick cylinder, roughly speaking. In it iron ore, which is essentially iron oxide, is converted into cast iron or pig iron by prolonged exposure to coke or its equivalent, in an atmosphere of mixed carbonic oxide, carbonic acid, and nitrogen, which results from burning this coke by means of atmospheric air forced in through appropriate openings near the bottom of the furnace, and called "the blast." The furnace is full from top to bottom of an intimate mixture of this coke and ore, together with limestone added for the purpose of forming with the barren mineral matter of the ore and with the ash of the coke a fusible silicate or slag, and for other purposes into which we need not enter here. These three solid materials collectively are called "the stock."

The burning of the coke generates at the bottom of the furnace a temperature so high as to melt away the bottom of this column of stock, or mixed coke ore and limestone, of which the last two have, by this time, been converted locally into metallic iron and lime, and as the column thus descends it is renewed at its top by adding more of this same mixture so as to keep the furnace continuously full.

There are, as it were, two rivers passing through this great cylinder in opposite directions, a sluggish river of solid stock which descends as its bottom is melted away, and a swift river of ascending gases resulting from the burning of the coke by the injected air or blast. The former traverses the length of the furnace in from 12 to 15 hours, the latter in a very few seconds. These rivers interact as they interpenetrate and flow past each other, the rising gaseous column progressively giving up its heat to the solid column and taking from that solid column the oxygen of its iron oxide, so that the gaseous stream as it emerges from the top of the furnace has taken from the descending ore all the oxygen that it is capable of removing, and has delivered over to that ore and its accompanying coke and limestone all the heat that they are capable of taking from it.

So much for the blast-furnace process, which, like every mundane process that seems simple, is in fact of a complexity so overwhelming that the human mind is inherently and incurably impotent to grasp it. We rub our eyes and, seeing as far as the ends of our noses, assume that we see to the end of the universe.

As a heat engine the blast furnace was known to be extremely efficient, as human heat engines go. Nevertheless, by the extremely simple step of drying the blast Mr. Gayley made a further important saving of fuel amounting in some cases to 20 per cent., and according to our present evidences to about 10 per cent. on an average, for usual American conditions. To those who had taken up the logical stick by the wrong end, such a saving by thus drying the blast seemed simply preposterous, as preposterous as talking a thousand miles through an iron wire, or waving a message to Europe without any wire, or as any invention is till you understand it.

Its preposterousness was promptly and convincingly exposed by the public-spirited experts whose geographical misfortune prevented their knowing Mr. Gayley's character, though those of us more favored geographically followed "Br'er Fox" till our question "Why and how?" could be answered.

The heat required for heating and dissociating the moisture of the blast is a small fraction of the total heat requirement of the blast-furnace process; how then dare we say that the removal of this heat requirement may save 10 or even 20 per cent. of the fuel?

Not a few of us had grown so used to calculating the heat needed for a given chemical and physical work like that of the blast furnace, and the heat evolved by the combustion of a given weight of fuel under known conditions, and to calculating thus the thermal balance, that we had lost sight of the supreme importance of temperature.

The atmospheric moisture, calorimetrically considered, is indeed slight, but thermometrically considered it is sometimes of overwhelming importance. As to its effect on climate Tyndall says: "To say that on a day of average humidity in England, the atmospheric vapor exerts 100 times the action of the air itself, would certainly be an under-statement of the fact."

Those who have felt the physical and mental exhilaration which a ten-mile trip from Cairo out into the desert creates by the sudden substitution of a drier for a moister climate; those whose legs would fain leap when the "wild west wind, thou breath of Autumn's being" suddenly dries our air, might well listen to the claim that a like drying of the air affects the blast-furnace process as it affects our own vital processes.

The sum and substance of it is that the blast-furnace process has, among its various duties, to supply a certain quantity of heat at or above a certain high temperature, which for brevity we may call the critical temperature, while shamefacedly confessing that we are further overworking an already grossly overworked word. No quantity of heat offered at any lower temperature will do this work. All the heat in all the suns of the milky way if offered at a temperature of 50° C., could not boil one egg.

Drying the blast saves fuel by improving the temperature-distribution of the heat generated.

Perhaps this is more readily understood if we consider an imaginary case. Suppose that matters were so ordained that he who wished to build an iron building was obliged to take, along with his iron work, a certain fixed proportion of accessories, windows, flooring, tiling, etc., the proportion fitted for a ten-story building. That would be all well enough for those who build nine- ten- or eleven-story buildings; but for the builder of a forty-story building it would be most uneconomical, because whereas the quantity of accessories, windows, flooring, tiling, etc., increases directly as the height of the building, the iron work needed increases in a higher ratio; so that, in order to get enough iron work for his forty-story building he might have to get enough accessories for a sixty-story building, and the excess would be left on his hands. This is because of bad proportioning of his supplies. A mere change in the proportion between iron work and accessories would cause a great saving.

It is somewhat so with a thermal process like that of the iron blast furnace. When combustion has raised the temperature above the critical point there is available first a certain

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[1] Address delivered at the Perkin Medal Meeting, Society of Chemical Industry, Chemists' Club, New York, January 24, 1913.

quantity of heat above that temperature, the hyper-critical heat, and second another and larger quantity of heat below that temperature, the hypo-critical heat, the heat remaining when the temperature has sunk to the critical point. The hyper-critical heat is needed for certain work which can be done only at hyper-critical temperatures, the hypo-critical heat is available for work which can be done at hypo-critical temperatures; so that, just as we have hyper- and hypo-critical heat, so we have also hyper- and hypo-critical work.

FOR HEAT ECONOMY THE HYPER-CRITICAL HEAT SHOULD BE TO THE HYPO-CRITICAL AT LEAST AS THE HYPER-CRITICAL WORK IS TO THE HYPO-CRITICAL WORK. The proportion between the two evidently depends on the temperature reached by combustion itself. The higher this temperature, the higher proportion does the hyper-critical heat bear to the hypo-critical heat.

If the temperature developed by combustion is so low that the ratio of hyper- to hypo-critical heat is too low, is below the ratio of hyper- to hypo-critical work, then in generating enough hyper-critical heat to do the hyper-critical work we are forced to generate an excess of hypo-critical heat over and above the hypo-critical needs of the process, and this excess of hypo-critical heat is used to poor advantage or is wasted. If my ratio of iron work to accessories is too low, then in providing my building with enough iron work for forty stories I am forced to provide enough of these accessories for sixty stories, and the excess is left on my hands. And as a mere change in the ratio between iron work and accessories can enrich or ruin a builder, so a mere change in the ratio between hyper- and hypo-critical heat, induced by a change in combustion temperature, can lead to a wholly disproportionate change in the economy of our own vital processes or of the process of the blast furnace.

Thus it was with Neilson's introduction of the hot blast. Formerly blast furnaces were fed with cold air, with the result that, because of the low temperature of combustion, the proportion of hyper-critical to hypo-critical heat was far below the proportion of hyper- to hypo-critical work needing to be done, with the result that the burning of enough fuel to provide enough hyper-critical heat yielded a quantity of hypo-critical heat far in excess of the hypo-critical work to be done, and this excess was used to poor advantage or wasted. Hence raising the temperature of combustion by heating the blast led to a saving of fuel which, to those unable to think, was miraculous.

The degree of economy caused by blast-drying should vary from case to case with the initial lack of hyper-criticalness in the combustion temperature; and if there is no such lack initially, as may happen conceivably, then blast-drying should cause no economy.

Other means of adjusting the ratio of hyper- to hypo-critical heat suggest themselves, such as raising the temperature of combustion by further preheating the blast, by enriching it in oxygen, by removing part of the atmospheric nitrogen, or by electric induction at the very focus where the hyper-critical work goes on; and lowering the critical temperature by changes in the conduct of the process. We have not reached the end of knowledge in general, or of the improvement of the blast furnace process in particular. But the fact that blast-drying, in removing the effects of the fluctuations in the atmospheric moisture removes a serious cause of irregularity in the working of the furnace and in the quality of its product, gives it an administrative and commercial advantage over other means of raising the combustion temperature which may well be decisive.

The explanation which I have given you is not my own, and it may not be the only one or even the chief one. It is the only reasonable one which I have heard of the accomplished fact which it aims to explain, and it seems to me adequate.

The total quantity of pig iron made in the world in one year is some sixty-six million tons, consuming somewhere about sixty-six million tons of coke and its equivalents, and this in turn represents about ninety-five million tons of coal. A 10 per cent. saving of fuel if applied to all the blast furnaces in the world would represent a saving of some nine and a half million tons of coal a year, or more than 40 per cent. of the coal production of so important a coal country as Belgium.

The scale is a large one. In how many of these furnaces such a saving can be made remains to be proved, but the results already reached lead us to expect that the total saving will be of very great importance.

The deliberation and caution, indeed the tardiness with which the iron trade has proceeded in adopting dry blast is referable to various reasons, such as hesitation to incur the certainly great expense of its installation, the competition of other devices for increasing earnings in other directions, the wish of each to get the benefit of the experience gained by its earlier adopters, uncertainty as to whether the saving found by John Doe will apply to the different conditions of Richard Roe's furnace, and the like.

But after all is said and done, that which interests us most is not the invention itself, important as that is, nor the great saving of fuel. The striking thing is the contrast between the mental attitude of the certainly very learned men of science who immediately stamped Mr. Gayley's claims as preposterous, and the attitude of this great captain of industry, who not only saw the saving to be effected but saw it so clearly that he was able to bring to pass the very costly experiments needed to prove his faith. Let us learn the lesson of humility. Natural human caution is likely to prevent the cautious from saying "I know that so and so can be done" unless they do know it; but such cases as this show that it does not prevent even the well qualified, the expert, and the prudent, from saying "So and so can not be done," though in fact it ought to prevent them, in view of the almost infinite excess of our ignorance over our knowledge.

DR. GAYLEY'S INTEREST IN EDUCATION[1]

By Edward Hart

This is the second time I have participated in rejoicings in Dr. Gayley's honor. The first festival was held early in 1902, when we dedicated Gayley Hall to chemistry and metallurgy at Lafayette College. Then, too, Dr. Howe spoke, and many of those I see here to-night were present. Dr. Thomas M. Drown, Dr. Gayley's preceptor and mine, greatly beloved, now gone to the far-away country, spoke, and Dr. Ira Remsen. It was a great celebration. One of the college boys told me that never before had so many high hats been seen on that campus.

We dedicated then a new library as well—The Henry W. Oliver Chemical and Metallurgical Library of Lafayette College, endowed by Mr. Henry W. Oliver, one of Dr. Gayley's associates, long engaged in the iron and steel business. This gift was made at Mr. Gayley's suggestion.

Dr. Gayley is a working trustee of Lafayette College. He has given time and money to the College, not once, but many times. One of his recent tasks has been the erection of a new building for the Department of Mechanical Engineering. I have often been struck with the originality and keen intuition, which he shows here as elsewhere. One of his ideas is compulsory athletics for all students.

I went to Lafayette College in 1874 with Dr. Drown, as his assistant. I was nineteen years old and had never seen the inside of a college building. Gayley was then a junior and probably knew more about chemistry than I did. In his senior year I was in charge, during Dr. Drown's absences, of the quantitative laboratory where Gayley was working. I was never, therefore, in any proper sense Dr. Gayley's teacher.

Gayley is the son of a Presbyterian minister, not blessed with great wealth; like many poor boys he received help from

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[1] Address delivered at the Perkin Medal Meeting, Society of Chemical Industry, Chemists' Club, New York, January 24, 1913.

the college funds. He was not much of a talker then, but had a keen sense of humor. At that time F. H. Daniels, of Worcester, Mass., was also an assistant in the laboratory. The boys called him Asenic, because that was the way he pronounced it. Daniels and I undertook to listen to the different preachers, and one Sunday evening visited the Presbyterian Church in Phillipsburg. Gayley and his friend Peacock were there too, and next morning we were accused of slipping over to P-burg on the sly to see the girls. What they were doing did not appear.

When he left college, Gayley became chemist for the Crane Iron Co. at Catasauqua. At that time a chemist was considered an ornament rather than a necessity, and his value was greatly increased if he were of economical habits. Mr. Frank Horn, of Catasauqua, thinks his salary was $35 a month at the start and there is a tradition at Catasauqua that the Directors of the Crane Iron Co., upon one occasion, gravely discussed Mr. Gayley's petition that his compensation be increased from $40 to $45 and finally concluded that the additional expense was not warranted. The books of the company show that he was paid at the rate of $500 a year in 1879. They also show that Mr. H. J. Seaman, who succeeded him, at one time received as much as $100 a month. Perhaps this was the belated result of Gayley's persuasive work with the Directors.

Everyone who knew Jim Gayley at Catasauqua remembers him pleasantly. He likes to go back there where everyone calls him "Jim." While there he lectured on chemistry in the Presbyterian church and afterwards appeared as one of the "babes in the wood" in a church entertainment. I venture to predict that if Dr. Gayley ever has a return engagement in either of these rôles, no building in the Lehigh Valley will contain his audience.

While in Catasauqua he lived at the hotel kept by Captain Harte. He still holds in affectionate remembrance the pies Mrs. Harte used to bake. I can remember that when he came back to visit us at the college he told us that it cost less to run his laboratory than any other in the Lehigh Valley.

The major part of the education we receive is self-education. Some men have never had much beside self-education. When I remember what Mr. Gayley was then and see what he is now, I am thoroughly convinced of the fineness of his fiber. Every time I met him he showed evidence of improvement—of the working of that yeast with which only the great are infected.

After leaving college he often came back to commencement, and I saw him once or twice in Pittsburgh. I had been plugging away at chemistry in Jenks Hall, which was the chemical laboratory from about 1881 to 1902. I taught descriptive chemistry, analytical chemistry, organic chemistry, theoretical chemistry, chemical technology, assaying, blowpiping, metallurgy and scientific German. During the intervals I worked for a living. Our laboratory was poorly equipped and I was very short of books. One day Mr. Gayley came in and after some general talk suggested that I get up plans for a new laboratory. I had heard that sort of suggestion before from others and nothing had come of it. So I acquiesced, but did nothing. Some months afterward Mr. Gayley again suggested that I get up plans and said that he would pay for them. I consulted the President who told me that Gayley had made a lot of money and that he was probably in earnest. Then I consulted an architect and in a short time Gayley Hall was in full blast. Imagine my feelings. For 20 years I had labored with my college work as one almost without hope, when suddenly all my wishes were fulfilled and I found myself with a handsome, well-equipped laboratory, an endowed library and assistants enough to enable me to do really good work. I must leave some things unexpressed. I cannot possibly express my feeling for Dr. Gayley. On the return trip to New York, after the dedication of Gayley Hall, Dr. Drown, then president of Lehigh University, was one of the party, and Mr. Gayley offered him $5000 for the University, which Dr. Drown accepted. This was used by Dr. Drown as a part of the fund for the erection and equipment of a Mechanical Laboratory at Lehigh. I think this gift was prompted more by his love for Dr. Drown than for any other reason. Lafayette and Lehigh are athletic rivals and it is necessary for Lafayette men to practice a certain amount of discretion in confessing fondness for Lehigh. If Mr. Gayley is to be convicted of love for Lehigh, he must himself confess it. I know, however, that many Lehigh men are fond of him, for Dr. Drinker is my authority; the best evidence of this is the honorary degree which Lehigh gave him last June.

About this time Mr. Gayley gave $5000 to Dr. J. A. Brashear, of the Allegheny Observatory. This gift may also have been a personal one, prompted by the love which Mr. Gayley felt for him: a feeling shared by all Brashear's acquaintances.

Sometimes Gayley was willing to educate the older generation. There is a story going the rounds, unverified, that shortly after he assumed charge at Birdsboro, the furnace began to give trouble. Of course, Mr. Brooke was greatly interested and asked many questions. Finally Gayley could endure this no longer. He told Brooke not to bother any more, the furnace would come around all right and when it did he would send for him.

My friend, Dr. J. W. Richards, says that he once asked Gayley what his calculations showed in calories with dry air as compared with natural air. Gayley replied that he didn't know nor give a whoop about the calories; what he was interested in was the pounds of coke saved per ton of iron.

When Gayley Hall was about completed he came over from New York to look at it. I found him standing out in the quadrangle looking up. "That's a fine building," he said. Then we went in and stopped a moment in the hallway. "My! that's a fine building," said Gayley. When we went into a laboratory, plain, unadorned, modeled after the laboratory of a steel works; "By God, that's a fine laboratory," said Gayley.

I have had the good fortune to know a good many men whom I consider really great men—and by the way, have you ever noticed how prolific our country is of such men? Most of them are, of course, chemists. Without being invidious, I may mention such shining examples as Dr. Drown, Dr. Raymond, Dr. Howe, Dr. Chandler, Dr. Nichols, Dr. Morley, Dr. Acheson, Dr. Remsen, Dr. Wiley, Dr. Smith of the University of Pennsylvania—one must particularize when speaking of Smiths—and Dr. Loeb, just departed, and I have noticed one characteristic common to them all—very prominent in all of them. I mean their great consideration for other folks' feelings. Mr. Gayley belongs in this class. When pleased he expresses himself fully, when not pleased—silence. Consideration for others is so natural to Jim Gayley that I don't believe he half realizes what a good fellow he is.

In essentials he seems to me very like my dear friend and preceptor Tommy Drown, whom, as his assistant for many years, I knew well. Surely, I may praise the dead whom I loved and whose memory is dear; sympathetic in all that was good and true, aspiring, just—no, generous, always fair, carefully concealing a heart of pure gold. It is a privilege to have associated with such men.

Mr. Harold Chidsey, who is one of the instructors at the Tome School for Boys at Port Deposit, Maryland, speaks in the warmest terms of Mr. Gayley's work there. In Mr. Chidsey's words, "He is a live trustee." I am quite sure this is true, for Dr. Gayley has often spoken to me about the school; about their high ideals and the wonderful work they were doing. He was particularly anxious that at Lafayette our work should be of the same high order.

What may we not hope to accomplish at Lafayette College with such leadership? I confess to the belief in a great future.

Away from the hurricanes which rage in the cañons of lower Manhattan, the effete civilization of Wall street, and the lures of Broadway, we must endeavor to build up a sacred city dedicated to the purity of truth.

THE NEW AGE[1]

By Rossiter W. Raymond

There is no possibility of denying that we live in a new age. I know that this has been said, and I suspect that it has been true, of every age since Lamech handed the administration over to Tubal Cain; but that does not prevent its being true to-day—more truly true than anything previously declared, because it is true for us. People do not agree as to the exact particulars of this novelty, or their exact value, or the spirit in which they are to be received. Enthusiastic voices of youth, shouting the war-cry "Whatever is, is wrong!" rejoice in every change as a step of victorious progress. Deprecatory voices of age declare that they recognize, in many of these changes, old errors, which, once exposed by experience and killed after long struggle, are not entitled to a resurrection; and that progress backwards is not real progress at all. Yet, after all, for good or evil or (as usual) both, it is a new age. Let us see whether we cannot point out some features in it, concerning which all parties can agree. Before engaging in this perilous attempt, permit me to remind you of the universal proposition that science is quantitative. No enumeration of facts, causes and factors can constitute a science until it begins, at least, to weigh and measure them, and to determine their relative effect and importance. It is for this reason that political science, as a body of laws deduced from established facts, and universally accepted by competent investigators, does not yet exist. It presents, in lieu of well established facts, ardent assertions, supported by statistics, more or less carefully compiled, and more or less intelligently interpreted; and the laws of a science cannot be derived by induction from such inexact and disputable data, to which, even if they be unassailably true, no relative value has been quantitatively assigned.

In the absence of the means of such scientific induction, there is sometimes at command a short method, which roughly checks the conflicting claims of theorists and reformers. The Egyptian King, when his engineers differed violently as to the grade of an irrigating ditch—each party producing the notes of its survey to support its assertion—turned water into the ditch, and decided the question-at-issue by observing which way the water ran. In like manner, when we are shown conclusively that the conditions of labor in this country are worse than in other lands, we look at the statistics of immigration, and finding a mighty current, running only one way, we form our general working hypothesis accordingly.

It is by this method that I ask you to consider our new age, as indicated not in this country alone, but throughout the civilized world, and to recognize certain world-wide facts.

1. In the first place, the old world, after many centuries, during which it barely succeeded in feeding and clothing itself, is at last growing rich. Up to, say, fifty years ago, how beggarly was the surplus—a few hundred palaces, a few thousand pictures and statues, a few millions of accumulated gold, silver and jewels. In these respects, fifty years of to-day are more than a cycle of yesterday.

2. What has been the chief cause of this change? Not government, not commerce, not war, not social reform, except so far as these contribute to the fundamental condition. For the phenomenon is the ability of the world to feed and clothe and shelter its population, and still have labor to spare for industries not directly productive; and the cause must be the greater productivity of the individual man.

3. This has been achieved, first, by increasing the equipment of the man by means of knowledge, and then by his use of that knowledge in summoning and controlling the powers of nature, to reinforce his own. In short, applied science, under and before all other causes, has transformed human life.

In connection with the first point—the increase of individual knowledge, permit me to repeat a statement, uttered by me ten years ago, in an address on the Dynamics and Ethics of Engineering. It is so delightful to be able to reiterate without change an opinion ten years old!

"What is Engineering? The control of nature by man. Its motto is the primal one—'Replenish the earth and subdue it'—not merely depend upon it, like a stationary plant or roaming beast, or hunting savage, taking what it gives—but change it by creative work, and use it with sovereign skill. Is there a barren desert—irrigate it; is there a mountain barrier—pierce it; is there a rushing torrent—harness it. Bridge the rivers; sail the seas; apply the force by which all things fall, so that it shall *lift* things. Recover the solar energy long stored in black darkness to reinforce the sunshine of to-day with heat and light and power. Nay, be 'more than conqueror,' as he is more who does not merely slay or capture, but makes loyal allies of those whom he has overcome! Appropriate, annex, absorb, the powers of physical nature into human nature!

"In the execution of this primal command, the use of machinery has been the factor and measure of progress. It is a truism of to-day that the most civilized nation is the one which uses the most coal. Probably that will not always remain as accurate a test as now. Regions which have no coal may come to utilize water power, and its transmission by means of the electric current, so as to realize something of the benefits of modern engineering. So I will not say that it will always be the country that uses the most coal, but I think it will always be true that the nation which is foremost in the utilization of some natural power will be also foremost in every branch of human progress.

"For what is the species which we seek to perpetuate? Not an animal form merely, which can eat, fight or hide, or run away, nurse children, and die. No! the species is *progressive* man, linked in his origin with the grass and the beast, but in his destiny with the universe and its Creator.

"For we have to consider now not merely the descent, but also the ascent of man—a nobler evolution, which I have attempted to describe in modest verse.

THE ASCENT OF MAN

He stood upon the earth, and turned
  To gaze on sky and land and sea,
While in his ear the whisper burned,
  'Behold, these all belong to thee!'

O wondrous call to conquests new!
  O thrill of blood! O joy of soul!
O peaks with ever-widening view!
  O race, with still-receding goal!

He heard; he followed, evermore
  Stumbling and falling, wandering far,
Yet still advancing, while before
  His footsteps shone the guiding star.

He cleft the seas; the torrent loud
  He harnessed to his need or whim;
He bade the lightning of the cloud
  Run with his words, and toil for him.

He pierced the rock; he scaled the steep;
  Destroyed; created; brought to light
The secrets of the deepest deep,
  The glories of the highest height!

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[1] Address delivered at the Perkin Medal Meeting, Society of Chemical Industry, Chemists' Club, New York, January 24, 1913.

The Future and the Past he scanned;
  With sense refined and vision keen,
Explored, beyond this lower land,
  The treasures of a realm unseen.

Until he stood with regal brow,—
  No more, as on the primal sod,
A creature yet ungrown, but now
  Lord of two worlds, and friend of God!

"This is the species with which we have to deal. It is sometimes said that evolution, as shown in lower spheres, stops with the appearance of man. But the most astonishing product of evolution—its last and strangest chapter—is man himself! man plus an infinite capacity of spirit; man plus machinery, which is but the expression of that capacity; man, whose eyes survey planet and animalcule alike; whose ear catches whispers from all the world; who hears what the past has said; who speaks to the future; whose feet speed over continents and oceans; whose hands tear open mountains, turn rivers from their channels; fill the air with the din of mighty industries; shake the ground with the thunder of battle. Do you say it is the printing press, the steam engine, the dynamo, the explosive mixture, that do these things? I tell you it is man; and these are parts of him!"

But man has always been learning, and always applying knowledge. Why is it that this element of progress has become so much more potent in this present age than ever before? The crude off-hand answer is the true one—there has been a marvelous increase of knowledge, and a corresponding increase in the application of it.

Now this is a cumulative effect, like the annual rise of the Nile. In the high mountains of Abyssinia, ten springs make one brook, ten brooks come together to form one creek, ten creeks form a river, tributary to the Atbara or White Nile, and the Atbara flows to its confluence with the Blue Nile, carrying the muddy contents of all its tributaries. In the dry season, the Atbara itself is but a slender stream, or even a series of water pools only, connected by underground seepage in its sandy bed. But when the heavy rains descend upon its innumerable sources, there are simultaneous floods in all the brooks uniting to larger floods in all the creeks, and so on, until the Atbara is traversed by a mighty wave, advancing like a wall at terrible speed—and the overflow of the Nile has begun, to be felt as a mysterious blessing for 1500 miles. Such is the effect of ten thousand separate springs of human knowledge, when they flow together, to exert their power at once.

Yet this figure is not complete, as a description of the New Age. For the number of the separate sources has been multiplied, through education, in an enormous increase of individual investigators and individual discoveries. I know that in these days it is unpopular to insist upon the rights of the individual. Liberty, property, competition and personal ambition, are all to be sacrificed to collectivism and solidarity. What the result of this wave of socialism may be, no man can yet predict, except so far as history records the rise and fall of similar tides of passion and fashion. But one thing abides sure: the men who discover and apply new knowledge are going to be, as they have always been, the real leaders of the race. No democracy can dethrone them; no juggling with phrases of legislation or declamation can disarm them. They pursue their quiet, irresistible way, while the crowd of partisans, quarreling fiercely among themselves, unconsciously follow these unrecognized yet invincible guides. And there are more of them, a hundredfold, than there ever were before!

And these men are drawn more closely together than ever before. The natural sciences are swiftly becoming one science; and those who utilize by means of science the powers of nature are beginning to be thrilled with a new sense of brotherhood. They are all *Engineers*, for the mighty enginery of Civilization is in their hands.

The old distinction between abstract and applied science is well-nigh obliterated. The practitioner sometimes follows, sometimes leads, the theorist; it would be more correct, perhaps, to say that they go forward side by side.

Many years ago, Charles Sumner, in the Senate of the United States, proposed an appropriation by Congress, in aid of experiments to test the possibility of telegraphing without a connecting wire. The appropriation was not made; and I remember with what a smile of conscious superiority many regarded that fresh instance of the audacity with which amateurs rush in, where experts know there is no footing. Later, when we studied the lectures of Prof. Hertz and other leaders in abstruse electrical science, how little did we realize that such refinements of theoretical inquiry, swiftly reinforced by practical ingenuity of application, would presently fill with human speech the atmosphere of the world.

Again, the free exchange of knowledge has made its energy well-nigh omnipotent. For in thought, as in mechanics, the measure of energy is not mv, but mv^2—not knowledge alone, but its product when multiplied by the square of the rapidity with which it is communicated.

The old practice of secrecy in applied science has largely passed away. We have found out that when we light another's candle from our own, we have more light ourselves than we had before.

Even business interests, I think, do not, in the long run, favor secrecy. Sir Henry Bessemer's family derived, for several generations, considerable wealth from the manufacture of a bronze powder, the secret of which was jealously kept. But the publicity given to the Bessemer process brought him a much greater reward, and to mankind a greater benefit. You tell me that this was an artificial reward, conferred by the patent law. But the law of patents is but a wise device to secure, through publicity, the double benefit of the immediate use and rapid improvement of an invention, while recognizing the rights and rewarding the labors of the inventor. It is a just bargain as regards the interest of the public; and it is really generous to the inventor. As Sir William Siemens well said, an important invention, given at once freely to the world, might lie in the street for years unnoticed. Some one must be induced by the prospect of a special reward, to take it up, perfect it in practice and push it into the competition of industry.

But I am not discussing the patent law; and I mention it mainly for the purpose of saying that I do not sympathize with the opinion which I have heard in some quarters, that the patentee of a new invention ought not to be permitted to contribute a description of it as a technical paper to a technical society. Under proper restrictions, excluding "puffs," unsupported assertions, and prophecies without experimental proofs and practice, such contributions are among the most valuable that any society can admit into its Transactions.

On the other hand, the maintenance of technical secrets, instead of their free communication, does not, in my judgment, based on half a century of observation, justify itself in the long run. The proprietor of such a secret unconsciously regards himself as beyond the need of improvement, and wakes too late, to discover that he has been surpassed in his art, and that his hoarded advantage has decayed on his hands. Three separate managers in a certain industry once communicated to me, under the seal of confidence, each his own secret process for perfecting a certain product. They were all doing the same thing; and if they had compared notes, they would have found out much sooner than they did, that there was a better thing which they could have adopted.

Apart from all these considerations of immediate business policy, the advantages of communication and coöperation, whenever practicable, are conspicuous and unquestionable; and their tremendous universal effect is manifested to us in this new age through the activity of technical schools, technical journals, and above all, technical societies.

Perhaps the greatest metallurgical novelty of the 20th century has been the development of electric smelting through the utilization of water-power. Through this industry, Sweden and Norway, rich in minerals but poor in fuel, are coming rapidly to the front, as competitors with those nations which have hitherto been recognized as natural leaders by reason of their supplies in coal. This change would be momentous, if it concerned those two nations only; but lately I found my professional brethren in Japan, standing tiptoe, as it were, beside the mountain torrents of their Island Empire, watching eagerly, and already beginning to imitate, the metallurgical practice of the Antipodes.

In short, gentlemen, I have arrived, by way of this desultory and imperfect discussion, at the conclusion which I might have stated with eloquent confidence at the beginning: namely, that your Society and similar organizations for the interchange of knowledge constitute the most potent agency and the brightest glory of this New Age.

Whatever you may think of the demonstration, I humbly submit that you ought to be pleased with the proposition!

CURRENT INDUSTRIAL NEWS

By W. A. HAMOR

THE PRODUCTION OF CALCIUM CARBIDE AND CYANAMIDE IN NORWAY

The *Chemiker-Zeitung* (**37**, No. 6, 65) reports that the plants of the Alby United Carbide Factories, Ltd., of London, in Odda, Norway, will, after the completion of the extension to its works in June, have an annual production of 80,000 tons of calcium carbide, of which, according to a thirty-year contract, 57,000 tons are to be delivered each year to The Nitrogen Fertilizer Co., the rest remaining for the production of acetylene. The Nitrogen Fertilizer Co. has taken over all the shares of the Alby factories, and, in return, has given sufficient of its own stock to be absolutely dominated by the Alby works. The Nitrogen Fertilizer Co. has also acquired the plant of the Northwestern Cyanamide Co., near Odda. The annual capacity now amounts to 18,000 tons of calcium cyanamide. The production is sold out for months to come, for the demand is not only for cyanamide for fertilizer purposes, but also for use in the production of ammonia according to Ostwald's process. There is a reason, therefore, why the Nitrogen Fertilizer Co. has interested itself financially in the Nitrate Products, Ltd., which concern owns the patents on Ostwald's catalytic process for the principal countries.

POTASSIUM SALTS FROM SEAWEED

The American Fertilizer, **38**, 49, states that the British Consul-General at San Francisco reports that two attempts are now being made to extract potassium salts from seaweed on a commercial scale, but that the industry has so far not passed the experimental stage. One company treats 40 tons of kelp daily, obtaining about 20–30 per cent. of potash from it. The kelp is gathered from the beach, and so contains a good deal of sand. It is dried on the beach by being turned over by hand, and is then burned in an open hearth. The residue is a chloride of potash, which has to be further treated, as only sulfate of potash is used in California. This process requires no fuel, and, though it is crude, yet the results afford good reason for thinking it may be made profitable in time. The other method consists of gathering the kelp in the water by a special cutting device carried on a barge, which ensures that no sand is present, the potash produced being therefore much cleaner. The kelp is treated in closed retorts heated by petroleum. One ton of dried seaweed is estimated to produce about 550 pounds of chloride of potash, 200 pounds of sulfate of potash, 5 pounds of iodine, 200 pounds of fertilizer and 45 pounds of gum, creosote and waste. It is intended to equip the factory at which the kelp is treated with an apparatus for the production of cyanide and chlorate of potash.

THE COBALT OXIDE MARKET

The Engineering and Mining Journal, **95**, 214, states that the trade in cobalt oxide is closely controlled, and the price during 1912 was held at $0.80 per pound without change; for 1913, an advance has been made, and $0.90 has been announced as the figure for this year's contracts. Except for the imports by the International Nickel Co.'s subsidiaries, there is but little cobalt oxide imported. There is a duty of 25 per cent. on the commercial article, but a few customers adhere to the established European brands and are willing to pay the premium their use entails.

There is some recovery from the Cobalt-district ores, but by no means so great as would be possible with a more widely extended use of the metal. There is no production of the metal as a by-product of copper refining. It is reported that there are over 3,000,000 pounds of cobalt-nickel residues at the Canadian and American refineries treating Cobalt-district ores, but statistics of production are not available.

The Canadian Government is conducting experiments to test the application of cobalt as an alloying material for steel. The results are reported to be encouraging, but it is likely that the final outcome will be only a market on the basis of the prices of nickel, that is, from $0.30 to $0.40 per pound for the metal; this conclusion is based upon the chemical similarity, practically speaking, of cobalt and nickel. An outlet for some cobalt products may be looked for in the direction of paint driers and special cutlery, etc., metals; but the principal use now for cobalt is as a coloring for blue glass and for pottery enamel.

THE MARKETS FOR CAUSTIC SODA AND SODA ASH

The following information is taken from *The Chemical Trade Journal*, **52**, 3:

A certain amount of caustic soda is imported into France, but large quantities are produced in that country and the exports are almost 15 times as great as the imports. The exports of soda ash are said to be about 20 times as great as the imports. The French caustic soda and soda ash come principally from the northeast in the Department of Meurthe-et-Moselle, a region rich in salt mines. The principal soap-making centers of France are Marseilles (kitchen soaps) and Paris (toilet soaps).

In Germany, soda ash and alkali seem to be controlled very completely by syndicate arrangements, outside of which they can not be had at present. A large number of German concerns produce these articles, the most important being, perhaps, the Deutsche Gold- und Silber-Scheideanstalt, of Frankfort, which controls the Electro-Chemische Fabrik Natrium and the Chemische Fabrik Residua, both of Frankfort. All German houses have given over their export business to Brunner, Mond and Co. and the United Alkali Co., of Liverpool, and these firms seem to be the instruments through which the export trade is distributed.

The imports of caustic soda into Italy average about $800,000 per year, with a probability of increase as Italian glass, soap, paper and cotton manufacture are further developed. Italy itself produces only a limited quantity of this material, and it is therefore dependent largely upon foreign markets for its supply. Imports of soda ash amount to over $1,000,000 annually. The imports of crude caustic soda come principally from Great Britain and France, and the same countries lead in exports of soda ash to Italy.

The caustic soda and soda ash used in the Durban district (Natal) is at present purchased from Great Britain and Germany. The consumption of caustic soda in Natal is from 100–150 tons per month, and of soda ash from 40–50 tons per month. There are two soap factories in Durban, and these are the only concerns which use caustic soda or soda ash to any extent.

The imports of caustic soda and soda ash into Japan are practically all from Great Britain. About 10,000,000 pounds of caustic soda and 22,000,000 pounds of soda ash are imported annually for consumption in soap, glass and paper factories.

THE PRESERVATION OF TIMBER

Several papers of interest to the chemical engineer were presented at the annual meeting of the American Wood Preservers' Association in Chicago, January 21–23, 1913.

H. von Schrenk discussed the requirements for successful timber treatment. He pointed out that the amount of material actually treated each year is ten times that of seven or eight years ago. In order to obtain good results from treated timber, he said that the following points must be observed:

(1) Only perfectly sound timber should be treated.
(2) Properly seasoned material should be used.
(3) A good preservative is essential.
(4) Proper injection as to quantity and penetration is necessary.
(5) Proper subsequent handling of the timber is essential.

Investigations of premature failures of so-called treated timber have shown that they were attributable, almost without exception, to the non-observance of one or more of these principles. In the early days of wood preservation much timber was treated which was sap rotten. However, the consumer frequently makes demands which cannot be fulfilled, and if they are they are bound to result in ultimate failure. The inspection of material before treatment should be made with great care, and every treating company should be empowered to refuse to treat material which it knows to be defective. The same holds true for improperly seasoned material. In treating green-red oak ties with 2 gallons of creosote oil by the full-cell process, the penetration is insignificant and internal sap rot is bound to occur in a comparatively short period of time.

H. F. Weiss reported the results of an investigation wherein he compared zinc chloride with coal-tar creosote for treating ties. In 1911, there were 9,445,961 ties treated with zinc chloride and 16,510,721 ties with coal-tar creosote in the United States. Zinc chloride and coal-tar creosote, when used under normal conditions, are both effective preservatives of cross-ties, and there is little choice between them so far as annual charges are concerned. Creosoted ties generally cost initially more than Burnettized ties, the cost of treatment being two or three times as great. Creosoted ties last, on the average, longer in the track than Burnettized ties, hence require less frequent renewals and changes in the roadbed. If creosote advances appreciably in price, it will very probably result in stimulating the number of ties treated with zinc chloride.

TAR AS A FUEL FOR OPEN-HEARTH FURNACES

The Indiana Steel Company, of Gary, Ind., has been investigating the use of tar as a fuel for its open-hearth furnaces in the place of producer gas. It is reported that the results of these experiments have been very successful, and at present two furnaces in open-hearth unit No. 1 are being run exclusively with tar as it comes from the by-product coke oven plant. The tar is handled very much like fuel oil, being atomized with steam in burners similar to those used for oil.

A NOVELTY IN GAS ENGINES

The Gas Age, 31, 77, states that a recent novelty brought out in England was a gas engine fed with powdered coal through a hopper at the ignition end. The coal passes through tubes (2 inches in diameter in a 100 H. P. engine) into the combustion and exhaust chambers, where the heat drives off the gas, which then becomes mixed with the air drawn through the tubes. In the "Low" coal gas engine the inventor claims to be able to produce 1 H. P. with one-half pound of coal and 90 pounds' pressure. If the claims are correct, this "gas producer engine" may interest those who are obliged to dispose of large quantities of coal dust.

THE BRITISH METAL TRADE IN 1912

	Imports Long tons	Exports Long tons
Copper	130,581	57,441
Tin(*a*)	60,348	43,663
Lead	205,375	58,685
Spelter	157,604	10,709
Minor metals (nickel, aluminum, minor metals and alloys)	7,889	28,549
Mercury	1,582	1,079
Pyrites	907,157	None

(*a*) Tin ore and concentrates imported were 28,652 tons, principally from Bolivia and South Africa.

IRON AND STEEL STATISTICS FOR 1912

Iron Ore

Production and Consumption

	Long tons
Lake Superior	48,410,477
Southern States	7,590,000
Other States	3,485,000
Production in the United States	59,485,477
Imports	2,200,000
Total supplies	61,685,477
Deduct exports	1,307,000
Increase in stocks	200,000
Consumption	60,178,477
The consumption in 1911 amounted to	43,571,536

Sources of Iron Ore Imports

	Long tons, 11 mo. ending November 30, 1912
Cuba	1,259,813
Sweden	315,796
Newfoundland and Labrador	124,585
Canada	95,714
Spain	83,631
Total imports amounted to	1,904,594

Production of Pig Iron

	Tons
Foundry and forge	6,096,254
Low phosphorus	282,359
Bessemer	11,385,297
Basic	11,394,477
Charcoal	347,025
Spiegeleisen	96,346
Ferromanganese	125,379
Total production	29,727,137
Imports	125,000
Exports	268,000
Consumption	30,100,000
Consumption per capita	703 pounds

THE STEEL INDUSTRY

The total production of steel for 1912 was about 29,745,000 long tons, of which about 62.5 per cent. was basic and 37.5 per cent. acid. The United States Steel Corporation supplied about 45 per cent. of the steel production of the United States; it earned approximately $108,178,307, an increase of $3,822,744 over 1911, although the tonnage of shipments increased about 35 per cent. and the prices rose about $6.00 per ton.

The iron and steel trades are now in a strong position, and this prosperity seems likely to continue for some time. There was an increase of 20.4 per cent. in exports of iron and steel, including machinery, over 1911, while there was a decrease of 0.04 per cent. in the imports; and the foreign trade for 1913 should be strong.

THE PRODUCTION OF AMMONIUM SULFATE IN 1912

The world's output of ammonium sulfate during 1912 was nearly 1,290,000 tons, more than half as much as the total output of nitrate of soda. This production was thus distributed:

	Tons(a)
Germany	465,000
United Kingdom	379,000
United States	155,000
France	68,500
Belgium	49,500
Austria-Hungary and remainder of Europe	170,000
Total output	1,287,000

(a) The figures given throughout refer to ammonia production calculated into its sulfate equivalent, as customary.

It will thus be seen that Germany is now the largest producer, which is partly ascribable to the development of the by-product coke-oven and producer plants, and partly to the increase in German consumption; in 1912, there was a decrease, instead of an increase, in the German exports.

The production in the United Kingdom was as follows:

	Tons
Gas works	166,000
Coke, carbonizing and producer gas plants	132,000
Shale oil industry	61,000
Iron works	20,000
Total British output	379,000

In 1912, the United Kingdom exported 286,864 tons of ammonium sulfate, 39,333 tons of which went to the United States, 86,659 tons to Japan, and 66,731 tons to Spain and the Canaries. Unless Germany enters into the export trade more prominently during 1913, the producers of the United Kingdom look for an increased demand from the United States and Spain. Prominent British traders still regard the United States position as the crux of the market.

About 215,000 tons of ammonium sulfate were consumed in the United States in 1912, of which amount 60,000 tons were imported. Of the home production, 155,000 tons, about 75 per cent. was produced by the by-product coke plants, and the balance by the bone-black and coal gas industries. The increase in production over 1911 was 28,000 tons, due to the larger operations of the by-product coke industry. At the beginning of 1912, there were 4,624 by-product coke ovens in operation and 698 in course of construction. Additional by-product plants are announced for 1913, and it is expected that the domestic recovery of ammonia will be carried out on a larger scale hereafter. At all events, American manufacturers are not being intimidated, apparently, by either synthetic ammonia or fixed-nitrogen.

THE PETROLEUM INDUSTRY IN THE UNITED STATES IN 1912

Day (*Am. Gaslight J.*, **98**, 46; *The Gas Age*, **31**, 85) estimates the crude petroleum production for 1912 in comparison with that of 1911, as follows:

	Barrels of 42 Gallons	
State	1911	1912
California	81,134,391	87,000,000
Oklahoma	56,069,637	52,000,000
Illinois	31,317,038	28,000,000
Louisiana	10,720,420	10,000,000
West Virginia	9,795,464	11,800,000
Texas	9,526,474	10,500,000
Ohio	8,817,112	8,500,000
Pennsylvania	8,248,158	8,000,000
Indiana	1,695,289	1,200,000
Kansas	1,278,819	1,300,000
New York	952,515	700,000
Kentucky	472,458	500,000
Colorado	226,926	200,000
Other States	194,690	500,000
Total production	220,449,391	220,200,000

The Engineering and Mining Journal, **95**, 152, gives a lower estimate, namely, 218,970,815 barrels.

The decline in production during 1912 exerted an influence on the price; for example, Mercer, Pa., black, quoted at $0.87 per barrel at the beginning of 1912, was quoted at $1.53 at the end of the year; North Lima, Ohio, jumped from $0.84 to $1.22, Indiana from $0.79 to $1.17, Kansas and Oklahoma from $0.50 to $0.80, and Electra and Henrietta, Texas, from $0.53 to $0.85. Judging from the dividend disbursements by the constituents of the "old" Standard Oil Company, 1912 was a year of prosperity for the Eastern oil companies. While the California oil companies made a creditable showing in dividends, the year closed badly for them, owing to the Standard's refusal to take low-grade oil after the existing contracts expired.

The exports of mineral oils from the United States for the year ended December 31, were 1,736,230,014 gallons in 1911 and 1,844,530,045 gallons in 1912. The exports in 1912 included 173,522,223 gallons of crude; 1,023,681,414 gallons of illuminating oils; 213,559,784 gallons of lubricating oils and paraffin; 175,089,771 gallons of naphthas and gasoline; and 259,276,853 gallons of residuum.

During the year the demand for gasoline greatly increased; there was a considerable extension in the use of oil-driven tractors and agricultural machinery, and the employment of oil engines to reduce power costs in remote districts became more fully appreciated.

Modern refining methods were described in brief during the year by Thomas T. Gray (*The Mineral Industry*, **20**, 577), who also indicated some technical problems requiring solution.

CONDITIONS OF THE PLATE GLASS INDUSTRY IN THE UNITED STATES[1]

The annual production of plate glass in the United States is about 60,000,000 square feet, about 47 per cent. of which is produced by the Pittsburgh Plate Glass Company and the remainder by the following eleven separate companies:

Allegheny Plate Glass Co., Glassmere, Pa.
American Plate Glass Co., Kane, Pa.
Columbia Plate Glass Co., Blairsville, Pa
Federal Plate Glass Co., Ottawa, Ill.
Ford, Edward, Plate Glass Co., Rossford, Ohio.
Heidenkamp Mirror Co., Springdale, Pa.
Kittanning Plate Glass Co., Kittanning, Pa.
Penn-American Plate Glass Co., Alexandria, Ind.
Saginaw Plate Glass Co., Saginaw, Mich.
St. Louis Plate Glass Co., Valley Park, Mo.
Standard Plate Glass Co., Butler, Pa.

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[1] Much of this information has been taken from *The Glassworker*, **31**, Nos. 16 and 17 (1913). For a review of the present status of the window glass industry in the United States, see This Journal, **5**, 80.

None of the American product is exported, with the exception of a negligibly small quantity to contiguous territory to supply pressing requirements. The capital invested in the plate glass industry in this country is about $49,000,000, the smallest concern in the industry having a capital investment of about $1,000,000. The average number of men employed directly in the industry is about 11,000, but those indirectly employed will equal more than twice this number.

The average cost of manufacturing plate glass in the United States in 1912 was 23.98 cents per square foot without considering depreciation; with depreciation added, but without allowing anything for interest on bonds or capital invested, the cost of plate glass per square foot was 28.45 cents. In 1909, the same cost figures were 28.16 cents and 33.71 cents, respectively. A comparison of the foregoing costs, before deducting depreciation or any interest on bonds or capital, with the average selling prices for the same periods, shows that all glass under five feet was sold at a large loss, while the margin of profit on glass over five feet was but 1.81 cents per square foot in 1909 and 4.66 cents in 1912. If we include depreciation, there existed on sales of plate glass over five square feet an actual loss in 1909 and but 0.19 cent profit per square foot in 1912.

The production of plate glass throughout the world is estimated at 125,000,000 square feet, a little less than one-half of which is produced and consumed in this country. The producing countries in Europe are Belgium, Germany, Austria, France, England, Italy, Spain, and Russia; but Belgium produces more glass than any of the other countries, at the lowest cost, and exports 95 per cent. of its product. The production of all but two of the European plate glass factories is curtailed and prices are fixed under a trust combination; each factory is said to have a capacity of 45 to 50 per cent. more than their present production. Since the organization of the International Convention abroad, the plate glass manufacturers of Europe have prospered and many improvements in machinery have been made, particularly in polishing machines. At present the cost of manufacturing plate glass in Belgium is $1.06 to $1.25 per square meter (10.76 square feet), and rough glass is being manufactured at a cost of less than $0.485 per square meter.

The American plate glass manufacturers are not associated with the European syndicate and consequently the latter has established a low range of prices for the American market. It can undersell competitors in any market in the world and then recoup its losses by adding them to the price of plate glass in markets that it controls, and is in an immediate position to make a strong invasion of the American market.

THE PRESENT STATUS OF THE SUGAR INDUSTRY[1]

The year 1912 was indeed a peculiar one in the sugar industry in the United States. In the beet sugar States, with one or two exceptions, the yield was the highest on record, while in Louisiana the output of cane sugar was the lowest recorded for a generation. The "campaign" is now over in the latter State and the product is put at less than 170,000 tons, as against 316,000 tons in 1911. Hundreds of acres of land in the cane parishes were inundated when the levees broke last spring and the young cane was destroyed. Then, too, a freeze occurred early in November, 1912, and another the latter part of the same month. Nevertheless, the Louisiana cane planters have laid plans for a successful campaign in 1913, although during much of the month of December persistent rains retarded field work and delayed the planting of the seed cane.

The 1912 statistics of the beet sugar industry are given in the accompanying table. The heavy beet yield all over the country has had the natural result of enlivening interest in the industry, and there is hardly a State in the beet belt from Ohio west to the Pacific but has one or more projects on foot with the view to locating new beet sugar factories. Some of the projects are sufficiently developed to insure operation in 1913 if it were not for the tariff situation; in the case of others not so well developed, work has been suspended until the question is definitely settled. It is of interest to note that Texas is rapidly coming to the front as a sugar State.

THE BEET SUGAR INDUSTRY IN THE UNITED STATES FOR 1912

STATE	Factories in operation	Acres harvested	Yield of beets in short tons	Sugar production in short tons
California	10	100,612	980,894	155,432
Colorado	17	142,369	1,683,158	223,181
Idaho	4	18,500	184,000	25,132
Michigan	16	114,012	1,026,208	119,611
Nebraska	2	19,248	213,000	25,150
Ohio	5	27,500	268,214	28,433
Utah	6	36,160	469,000	59,590
Wisconsin	4	23,400	250,000	30,000
One factory states: Ariz., Ill., Ia., Kan., Minn., Mont., Nev., Ind.	8	49,800	466,710	63,637
Total	72	531,601	5,541,184	730,166

For comparison, the following European statistics on beet sugar crops for 1912 are available:

	Tons
Russia	1,182,700
Germany	2,604,000
France	893,620

THE PRODUCTION AND CONSUMPTION OF RUBBER IN 1912

The statistics which follow are taken from *The India Rubber World*, 47, 194.

PRODUCTION

	Tons
South America (East Coast)	40,700
South America (West Coast)	2,000
Central America and Mexico	5,000
Africa	15,000
Assam, Rangoon and Borneo	2,500
Guayule and Jelutong rubber	10,000
Plantation	28,500
All other sources	1,000
Total production	104,700

CONSUMPTION

	Tons
America	48,000
Great Britain	17,250
Germany	16,000
France	10,000
Russia	7,000
Belgium	2,000
All other countries	8,000
Total consumption	108,250
Excess over production	3,550

IMPORTS INTO THE UNITED STATES

During the fiscal year ending June 30, 1912, 110,210,173 pounds of crude rubber were imported.

For the ten months ending October 31, 1912, 97,308,544 pounds of crude rubber were imported.

From January 1 to November 18, 1912, the United States received 3,572,959 pounds of Ceylon grown rubber.

THE UTILIZATION OF BLAST-FURNACE AND COKE-OVEN GASES.

Gouvy (*Eng.*, 94, No. 2446, 684) states that the French iron and steel works have failed to derive from their waste gases all the advantages possible, largely from having considered the matter from too general an aspect, in caple of studying each

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[1] Most of the above information has been taken from *American Sugar Industry*, 15, 21.

proposition individually. The course to be adopted should vary with the local conditions, such as the price of fuel, the quantity of coke passed through the blast-furnace, and with the amount of power which can be used in the works themselves and by their subsidiaries, as mines, cement works, etc., all of which afford a field for the use of gases now wasted, in addition to what can be sold to other undertakings. When coke-oven gas is available, as well as that from blast-furnaces, the possible methods of utilization are largely increased. The adoption of regenerative heating has greatly reduced the quantity of gas which it is necessary to consume in the ovens themselves, so that fully 50 per cent. of the total gases liberated are disposable for other purposes.

In the case of the existing French works, Gouvy considers that the program to be adopted must be largely governed by the character of the present steam plant, which it is not always financially possible to modernize. When, on the other hand, new works are being laid out, it is generally feasible to arrange the power plant so that it is operated by the waste gases entirely. It should, in such a case, be possible to operate by the waste gases the blowing-engines, the electric power station of the works, and the rolling mills. If coke-oven gas is also available, it should, moreover, be possible to work thus the reheating furnaces, the steel furnaces, and the other accessory heating plant.

The first essential to good results with blast-furnace gas is to clean it, so that, as used, it does not contain more than 0.5 gram of dust per cubic meter. This greatly increases the evaporative power of any boiler heated with the gas. Thus, with the uncleaned gas, about 2 cubic meters are required per kilogram of water evaporated, as compared with only 1 to 1.2 cubic meters per kilogram with the uncleaned gas. The great effect of cleaning on the evaporative efficiency is due to the fact that the dust is an excellent non-conductor, and, if present, is deposited upon the heating surfaces, greatly impeding the transmission of heat across them to the water. In a Westphalian works, where the cleaning plant reduces the dust content to less than 0.25 gram per cubic meter, the consumption of gas is only 0.8 cubic meter per kilogram of steam superheated to 250° C.

The same considerations are applicable to blast-heating stoves, on which the dust deposit has a very prejudicial effect. Its presence impedes the transfer of heat from the bricks to the gases, and *vice versa*, and at the same time, if a very hot blast is used, it acts as a flux, making the bricks waste rapidly. Cowper stoves, as usually worked, require from 50 to 60 per cent. of the whole of the furnace gases available, while if these gases are cleaned, the consumption for the same blast temperature will be about only 40 per cent. of the total supply. Since, however, the gas is cooled in the cleaning process, special burners must be provided to insure its ignition. The cost of cleaning to the limit stated should not exceed $0.00146 per 1,000 cubic feet. For engine-running the gas should be further purified, so as to bring its dust content below, as a maximum, 0.03 gram per cubic meter. To purify the gas sufficiently for use in gas-engines involves an additional outlay over and above the cost of the preliminary cleaning of $0.00124 per 1,000 cubic feet.

Where coke-oven gas is available, still further economies can be effected. The by-products alone are worth from $0.20 to $0.30 per ton of coal coked. If the ovens are fitted with regenerators, about 50 per cent. of the total volume of the gas evolved is available for use elsewhere. This volume varies with the quality of the coal from 250 cubic meters up to 330 cubic meters per ton coked, and its calorific value ranges from 3,500 calories up to 5,500 calories per cubic meter. The coke-ovens at Heinitz, in the basin of the Saar, consume 700 tons of coal daily. With the waste gases a central station of 10,500 horse-power is operated and power is furnished to the surrounding district. The price realized at the switchboard is $0.01 per kilowatt-hour, and the cost of generation, including capital charges, is below $0.004. A very suitable use for coke-oven gas is in heating reverberatory furnaces and steel furnaces; blast-furnace gas is hardly suitable for such purposes, although it is being tried in Germany.

ABUSES IN WATER FILTRATION

G. H. Pratt, at the last meeting of the New England Waterworks Association, said that the success of a water filtration plant depends, first, upon the selection of the method of purification best suited to local conditions; second, upon the proper operation of the works after installation. Slow sand filtration for a water high in algae is not to be recommended, because of the undue clogging of the beds, but by the use of aeration and prefilters conditions may be greatly improved. Plain sand filtration should not be depended upon to treat a very highly colored water; and the rates of filtration should not be changed quickly, for such a practice tends to disturb the bacterial action at the surface of the bed. A slow sand filtration plant which would handle a given water satisfactorily might, as was the case at Providence, R. I., be installed without covering the beds. In the case of rapid sand filters supervision must be particularly close, and constant chemical control must be maintained.

THE COST OF HYPOCHLORITE DISINFECTION

The following data relating to hypochlorite disinfection is taken from *The Engineering Record*, 67, No. 1, 16:

Commercial bleaching powder packed in sealed drums holding 700 to 800 pounds each, with a guaranteed strength of 36 to 38 per cent. of available chlorine, may be purchased in carload lots for about $0.0125 per pound. Assuming a disinfectant containing $33^{1}/_{3}$ per cent. of available chlorine at a cost of $0.02 per pound, the treatment of a sewage with 0.1 part per 100,000 available chlorine would require 25 pounds of disinfectant at a cost for chemicals of $0.50 per million gallons. To produce complete sterilization, the cost would be well over $19.00 per million gallons for sewages and the effluents from contact and trickling filters, and would vary from $1.50 to over $19.00 for effluents from sand filters. To produce a bacterial quality which would conform to the drinking water or 100–10–5 standard, the cost would vary from $3.75 to over $19.00 per million gallons for raw sewage and effluents from trickling filters, from $7.50 to over $19.00 per million gallons for settled sewage, from $15.00 to $19.00 per million gallons for strained sewage and contact filter effluents, would be over $19.00 for septic sewage, and would vary between $1.75 and $9.50 per million gallons for the effluents from sand filters which were not originally of that quality. To produce a bacterial quality to correspond to 1000–100–50 standard, or one which would be about equal to that of the better class of streams not seriously polluted, the cost would be from $1.75 to $5.60 per million gallons for raw sewage. These cost estimates are for chemical only, and do not include operating and sinking fund charges.

THE PAPER INDUSTRY OF JAPAN

Paper (January 1, 1913, p. 25) states that after cotton-spinning, the manufacture of paper is the most important industry of Japan.

As raw material, the so-called paper mulberry bush (Kodzu), *Broussonetia papyrifera* (mitsumata), serves. While the manufacture of domestic paper is very old, the production of "European" paper was first commenced by the Oji paper mill, working with rice-straw, rags and old paper.

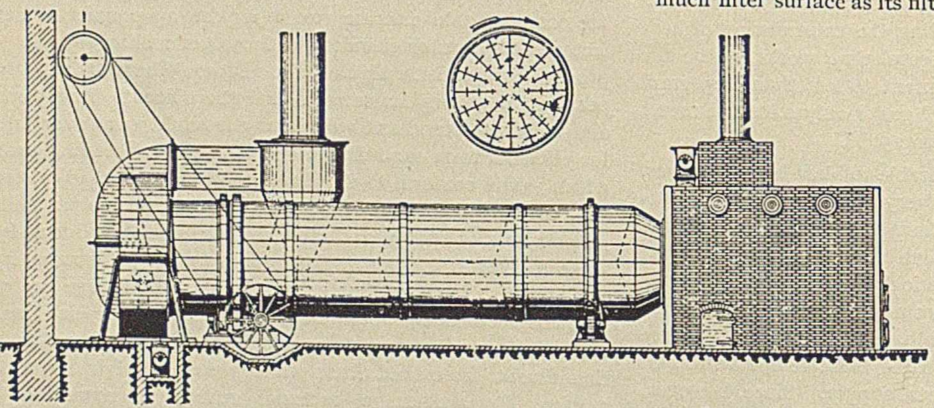
After the Saigon uprising in 1877, as a result of the establishment of numerous printing plants, the demand for European paper increased enormously. The manufacture of domestic paper, mostly conducted with hand apparatus, is carried on principally by the rural population as a secondary occupation, during the dull season. It gives occupation to about 60,000 families. Experimental stations in the most important districts test and select the best raw materials, also imported fibrous material. Guilds supply the country people with raw material and provide for the sale of the product.

Since the acquisition of the island of Saghalien, it supplies the manufacturers with woodpulp, which is cheaper than the domestic vegetable fibers; in Shiraoi, too, on the island of Hokushu, a large woodpulp plant has been set up. The manufacture of European paper is conducted chiefly in the large cities, such as Tokio, Osaka and Kobe.

In 1909, there were 27 establishments engaged in the business, with 182 machines and 6,486 employees, turning out 124,000 tons of European paper. The Mitsui Bussan Kaidha, the greatest Japanese business house, has erected at Tomokomai on Hokushu a mill that produces daily 65 tons of newsprint. The product, for the most part, goes to India, Australia and China. Altogether the value of the product of the hand mills is placed at $8,964,400; of the mills equipped with machines at $7,497,000. The exports have a value of only $1,618,400 while the imports amount to $4,498,200. According to this, Japan consumes, annually, paper to the value of $19,373,200. In the imports, Germany takes the lead, followed by Austria and England. The paper imported from the two first-named countries consists principally of imitations of Japanese paper.

THE GERLACH DRIER

In the Gerlach drier, System A, the drying is done with hot air. The cut shows the design of a tube-mill drier, provided with partitions arranged as illustrated; these partitions are claimed to effect the finest division and a continuous mixing of the drying material.



In the Gerlach drier, System B, the drying is done with steam. For handling viscous, adherent, sticky or gummy materials, the apparatus consists of a pre-drier and a regular drier.

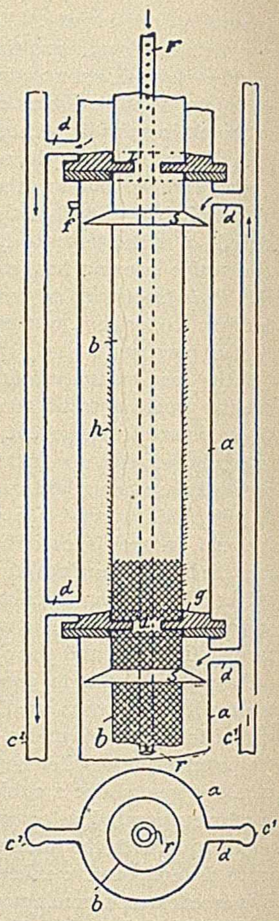
ARLEDTER'S SAVE-ALL APPARATUS

A new apparatus which is said to serve to recover from the waste waters of paper and cellulose mills and similar establishments, the fibrous matter, kaolin, and like materials, suspended in them, has been devised by Fritz Arledter, of Hamburg, Germany. According to the description of this apparatus in *Paper*, January 22, 1913, p. 19, the waste water is discharged in a clarified condition available for immediate reuse, all the fibers, clay, etc., being recovered.

One form of construction of the apparatus is shown in the accompanying illustration: *a* is a pipe of wood or metal of any desired section, in which a sieve-tube (also a felt tube), *b*, is vertically disposed, so that between the tubes *a* and *b* a space is left. For the entrance of the waste water, the side tube c^1 serves, and for the discharge of the fibrous substance, the side pipe c^2. Both pipes are connected by supporting spouts, *d*, with the pipe *a*. Under the inlet supports, on the sieve-tube *b*, deflecting distributors, *s*, are placed. The bottoms g are inclined towards the discharge spouts. By increasing to any desired extent, the number of double pipes, the filter column can be built as high as desired.

The working method of an apparatus of this character is as follows: If waste water bearing fibrous substances, loading matter, etc., is conducted through the conduit c^1 into the filter, the waste water will fall through the spout *d* into the space between the two pipes *a* and *b*, filling it from the bottom upward. The hydrostatic pressure of the rising column of water forces the water through the filter wall *b*, the fibers being deposited on the sieve pipe *b* and themselves forming a filter for the water. The umbrella or roof-shaped ring *s* serves to protect the fiber filter from being washed down from above. The clarified water flows away from inside the sieve pipe, at *b*, for further use.

The fiber filter grows automatically from the bottom upwards. The waste water uses just as much filter surface as its filtration demands and this, in the development of the tube, is always available. By this means, an automatic regulation of the filter process is created and uninterrupted working is obtained.



If the layer of fibers exceed a certain thickness, retarding the passage of the fluid, the excessively thick portion falls down of itself and its reconstruction commences anew. On the other hand, it is an easy matter to loosen the fiber felt or blow it off, a perforated pipe, *r*, leading into the sieve pipe *b*, through the holes in which steam, water under pressure or compressed air can be forced to such an extent, that the fiber coating on the inside of the pipe can either be loosened or forced completely off. The pipe *r* also serves for washing out purposes. A pipe for the supply of steam can also open into the pipe *f*, whereby the fiber felt *h* forming on the sieve pipe *b* can be loosened and kept permeable for fluids; moreover, the cleansing of the pipe conduits can also be effected, at any time, by the admission of steam.

The displaced fibrous mass drops to the bottom g and slips through the discharge spouts into the discharge pipe c^2, whence the mass can be conducted to the hollander or stirring vat. Downwards too, towards the bottom g, a layer of fibers is formed which prevents the waste water to be filtered from flowing right off through the pipe c^2. The concentrations can be regulated by throttling of valves. The waste could also be subjected to repeated filtration.

A reverse flow of the waste water to that above described can also be provided for, as regards the regulation of height as well as the flow from one pipe into the other. The separate pipe systems can also be disposed over one another, or in battery form, alongside of one another, and suitably connected so that a repeated filtration of water and fibrous liquor would be possible. To the side pipe conductors c^1 and c^2, pumps can be connected; moreover, for the purpose of cleansing the entire plant and detaching the fiber felt, water can be admitted at various points.

At certain points alum solutions can be admitted, also electricity for the purpose of loading the fibers, decomposition of chemicals, etc. The apparatus works uninterruptedly and automatically.

USES OF CAST SILICON

According to *Metallurgical and Chemical Engineering*, 11, 103, silicon is used in the form of pipes for the conveyance of acid gases at a high temperature from stills to condensers, also in the construction of the condensing batteries themselves, proving more efficient than stoneware owing to its high thermal conductivity and ability to withstand sudden changes of temperature. Silicon pipes are also used for the transportation of hot liquid sulfuric and nitric acids, but it can not be used for hydrochloric acid unless the discoloration resulting from the slight solubility is immaterial. It is used as a lining in centrifugal pumps, acid valves and pipes for the elevation of corrosive liquids by compressed air, and for the construction of plows in ore roasters it is said to have proven efficient. Cast silicon is also being employed in the form of shallow pans and pots for the concentration of zinc chloride solutions, supplanting the enameled stoneware vessels which are rapidly destroyed. Cast silicon ware is said to be produced in practically all shapes required by the chemical industries, such as, for example, pipes, evaporating vessels, receivers, tile, alembics, crucibles, and pump parts.

NOTES AND CORRESPONDENCE

NOTE ON THE INFLUENCE OF THE LIME-MAGNESIA RATIO UPON PLANT GROWTH

Editor of the Journal of Industrial and Engineering, Chemistry:

Starting from the poisonous action of potassium oxalate upon the cellular nucleus and chlorophyll bodies, further considering that the injurious action of magnesium salts on plant cells can be prevented only by calcium salts[1] and finally regarding the fact that a certain excess of calcium salts over magnesium salts retards plant growth, I had inferred that a certain ratio of these two bases must be most favorable for plants.[2] Many experiments by various authors (chiefly observing cereal and leguminous crops) have confirmed this natural conclusion. Where contradictory results were obtained it could be easily shown that too great a number of plants in the pots had prevented their normal development, since the roots spreading along the walls of the pots were hindered in their normal functions. Indeed such barley plants had reached only a weight of from 2 to 6 grams. A review of the work done in this direction will soon be published in English, to which interested readers may refer.

In the article by Gile and Ageton (THIS JOURNAL, 5, 33) it has been pointed out that since citrus, pineapple and sugarcane thrive well on soils with varying amounts of lime and poor in magnesia, the ratio of the two bases seems to have no influence. These are but other examples of apparent exceptions in regard to the lime factor. Such exceptions are the lime-loving plants, e. g., the grapevine, as the writer has pointed out repeatedly.[3] These plants are capable of precipitating as oxalate the excess of lime carried into the plant by the transpiration process. Thus an unfavorable ratio of lime to magnesia in the cells is avoided since the lime in the form of insoluble oxalate cannot further participate in the physiological processes. Now it is well known that the pineapple plant and citrus show a large amount of calcium oxalate crystals in the cells. It is not yet known whether cane grown on soils rich in lime and poor in magnesia stores crystals of calcium oxalate. Among the gramineae plants this is only occasionally observed, as with millet.

It has been stated in the article referred to that cane grows well on soils containing only traces of magnesia. In this case inquiry should have been made as to the manure applied, since tankage, cottonseed meal and stable manure contain so much magnesia and lime in an easily available form that the plants thus manured need not depend on these bases in the soil, in fact, become almost independent of the lime factor in the soils, as I have pointed out long ago.[1] When, however, fields are manured exclusively, as is often done, with inorganic manure, and only with regard to nitrogen, potash and phosphoric acid, then the ratio of lime to magnesia[2] present in the soil will certainly exert an influence on development.

The yields of cane on different soils with different ratios of lime to magnesia were compared in the article referred to and the conclusion drawn that there exists no relation between this ratio and the cane production. This conclusion was not justified since no information was procured as to the manuring and the amount of tankage applied, and the quantity of lime and magnesia thus supplied to the plants in an easily available condition was not taken into account. Also, yields on *different* soils with *different* ratios of lime and magnesia and *different* manure cannot be compared; only those on different plots of *one and the same soil* in which different ratios have been produced are comparable. Further, the opinion that only compounds contained in soil solutions are available to plants—which is certainly erroneous—led to the erroneous conclusion that all plants in the field were independent of the ratio of lime to magnesia. Voelcker in England, Bernardini in Italy, Warthiadi in Munich, as well as the writer and his co-workers, have all experimented with various soils and observed the great influence of their various ratios of lime to magnesia upon the development of cereals and other plants.[3]

It was further asserted that it is chiefly the ratio of lime to other mineral nutrients that comes into play and not that to magnesia. The literature on the subject disagrees with this assertion.[4] But in order that the lime factor can fully make its impression upon the development of plants, the amount of easily available phosphoric acid is also very important, as I have repeatedly mentioned.[5]

SUMMARY

I. Citrus and pineapple are lime-loving plants; an excess of lime absorbed by these plants can be rendered innocuous by the transformation of that excess into oxalate. Thus a properly working ratio of lime to magnesia is secured in the cells when the plants grow on soils too rich in lime. Hence the view, that

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[1] Potassium salts can only retard it. Cf. *Flora*, **102**, 110.

[2] *Bull.* **45**, Bureau of Plant Industry, Washington, 1903, p. 43, and *Landw. Jahrb.*, **35**, 539.

[3] *Bull.* **45**, 68–70.

[1] *Bull.* **45**, 70; *Landw. Jahrb.*, **35**, 532.

[2] It is supposed that the availability of both these bases is equal or nearly so.

[3] These soils were in our case surely not "alkaline" soils. Compare also the confirmation by Portheim and Samer in Vienna and of Hansteen in Christiania.

[4] *Landw. Jahrb.*, **35**, 533 (1906); *Bull. College Agr., Tokyo*, **7**, 396; *Flora*, **102**, 110.

[5] *Bull.* **1**, 16, Bureau of Plant Industry, and *Landw. Jahrb.*, **1902**, 569,

the favorable development of these plants on soils very rich in lime and poor in magnesia would be a proof against the influence of a certain ratio of lime to magnesia in soils upon other plants which do not produce oxalates, is not tenable.

II. Experiments upon the influence on cane growth of different ratios of lime to magnesia produced in one and the same soil, *manured exclusively with mineral compounds*, have thus far not yet been made. Hence the opinion that the growth of cane is independent of that ratio is not justified.

OSCAR LOEW

HYGIENIC INSTITUTE
MUNICH, GERMANY

AN IMPROVED METHOD OF CRUDE FIBER ESTIMATION

Editor of the Journal of Industrial and Engineering Chemistry:

This crude-fiber method is an improvement on the Morgan P. Sweeney modification of the official method, the improvement consisting in filtration through asbestos and sand in a Gooch crucible, treatment with hydrochloric acid, washing without transfer, and ignition in the Gooch crucible. The details are as follows:

To a 1- or 2-gram sample add 200 cc. of boiling 1.25 per cent. sulfuric acid, and boil for 30 minutes.

Neutralize with 10 per cent. sodium hydrate, using phenolphthalein as an indicator; add 200 cc. of 2.656 per cent. boiling sodium hydrate; make volume up to 425 cc. and boil for 30 minutes.

Filter through a porcelain Gooch crucible containing an asbestos pad and 10–12 grams ($^1/_2$ inch) of very fine acid-washed sand, compacted by water and suction. Treat with hydrochloric acid; wash with hot water until free from chlorides, and then with alcohol and ether; dry, weigh, ignite and weigh.

Sand satisfactory for this purpose has been purchased from the Berkshire Glass Sand Co., of Cheshire, Mass. Coarse sand was found not to be useful, the reason being that it allows the fiber to pass through and then to clog the asbestos pad.

The improved method usually gives higher figures than the official method. In general the results check, one with another, more satisfactorily, and the method is very much easier to use.

The unsatisfactory character of the official method is too well known to require comment. This new procedure eliminates all transfer of the sample and also the unstandardized cloth strainer, for which it substitutes a filter which is thoroughly efficient, and which at the same time allows of very rapid filtration of solutions such as those from linseed and cottonseed meals which are very difficult of filtration by any other method. The use of hydrochloric acid facilitates the final washing.

A microscopical examination of the crude fiber and the filtrates from the official and from our modified methods showed that the higher results obtained by the latter were due to undissolved woody and chitinous particles which passed through the linen but which were retained by the sand and asbestos. In the products examined there was no evidence of contamination of the crude fiber as determined by the improved method by gummy or protein substances.

Objection to the Sweeney method has been raised on account of difficulty with the colored extracts of some feeds in determining the neutral point. This objection is not insuperable in any case and in using this method with a wide range and considerable number of products we found that it was in rare instances only that the color of the extract interfered with the use of the indicator.

The slow filtration by the Sweeney method is entirely overcome in our modification.

Objection has also been raised to the Sweeney method on account of possible interference of the fat with the solution of the acid- and alkali-soluble constituents. This possibility, however, has not been proved to affect results appreciably.

Whatever the method under consideration, we are unable to judge of its accuracy by comparison with the official method because of the lack of definiteness as to the character of the cloth strainer and because of the incomplete retention of the crude fiber of some products by such a filter.

Below are a few figures by this method, and the official method. The sample in each case weighed 1 gram. The figures are weights in grams of crude fiber.

	Official method	Modified method
Cowpeas......	0.0544	0.0568
	0.0560	0.0564
	0.0570	0.0554
Linseed oil meal	0.0922	0.1110
	0.0908	0.1118
	0.0910	0.1112
Corn meal.....	0.0228	0.0272
	0.0212	0.0280
	0.0238	0.0272
Soy beans.....	0.0470	0.0482
	0.0472	0.0484
Distiller's grain (corn)......	0.1232	0.1239
	0.1228	0.1240
Oats, grain......	0.1215	0.1217
	0.1212	0.1215
Wheat bran.....	0.0837	0.0856
	0.0830	0.0868
Wheat, grain....	0.0324	0.0332
	0.0331	0.0332
Patent flour.....	0.0010	0.0025
	0.0012	0.0025
Gluten feed.....	0.0892	0.0886
	0.0891	0.0881
Cottonseed meal.	0.0705	0.0710
	0.0703	0.0720

E. B. FORBES AND J. E. MENSCHING

OHIO AGRICULTURAL EXPERIMENT STATION
WOOSTER, Dec. 27, 1912

AMERICAN MINE SAFETY ASSOCIATION

As the result of a conference which was held under the auspices of the United States Bureau of Mines last September, between men who are interested in the saving of the lives of miners, there has been formed a society known as the American Mine Safety Association, with headquarters at 40th and Butler Streets, Pittsburgh, Pa. This association, which is now enrolling among its members the leading coal and metal mine operators, mining engineers, and mine safety engineers of the country, has for its purpose the conservation of the life and health of the miner and a reduction in property loss due to explosions or fires in mines. It will attempt to place before the miners standard methods to be used in rescue work and in first aid to the injured.

The work of the Bureau of Mines in reducing the number of deaths in the mines has led to the adoption of many different types of rescue apparatus, such as the oxygen helmets which the rescuers wear in a gas-filled mine, and also to the use of many different methods of resuscitation and first aid to the injured. Hundreds of mines within the last three or four years have been equipped with rescue apparatus, rescue corps, and first-aid corps. Many of these men who are called upon in emergencies have developed their own ways of doing things and it is the opinion of the organizers of this association that the most efficient methods and apparatus should be found through actual use and recommended to the mining industry as a general standard.

H. M. Wilson, the engineer in charge of the Experiment Station of the Bureau of Mines at Pittsburgh, has been selected as chairman of the executive committee of the Association and has been instructed to carry on the work of organization.

There were 2,719 persons killed in the coal mines of the United States during the year 1911, and there were 9,106 seriously and 22,228 slightly injured. Nearly an equal number of men were injured in metal mines and quarries.

In the hope of reducing the number of these accidents and of shortening the period of disability of the injured, it was felt that there should be greater uniformity in methods of conducting rescue operations in mines after explosions, fires, or other disasters, and of giving first aid to the injured.

The Director of the United States Bureau of Mines therefore called in Pittsburgh, September, 23, 1912, a National Mine Rescue and First Aid Conference. About half a hundred men concerned in safety in mines attended. There were nearly an equal number of mine operators, mine surgeons and mine safety inspectors and rescue men; they represented geographically all of the principal mining regions from Pennsylvania to Colorado, and from Alabama to Michigan. They voted to form a permanent organization and to hold the first annual meeting in the fall of 1913.

THE ANALYSIS OF ORGANIC DYESTUFFS

Editor of the Journal of Industrial and Engineering Chemistry:

Sub-Commission XI of the International Commission of Analyses, appointed to study the question of the analysis of Organic Coloring Matters, has examined the possibility of unifying the analytical methods at present employed, and whether such unification would be of practical use. Reports have been received from various countries, and the Committee reporter has arrived at the conclusion that the unification of the methods employed in the analysis of organic coloring matters is not possible at the present state of the industry, and would not be of any practical use.

However, an international agreement might be advantageous in certain cases, such as that of those organic dyestuffs which are taken as standard in the levying of custom duties. Such an agreement would protect both exporter and importer and prevent disputes. It might also be of use in the dye industry in the case of certain dyestuffs. So that if the unification of the methods of analysis of all organic dyestuffs is not feasible, at least useful work might be done as regards certain special cases.

The International Commission of Analyses, presided over by Professor Lindet, after taking cognizance of the reports on this subject presented at the Eighth International Congress of Applied Chemistry held at New York in September, 1912, adopted the following resolution: "That Sub-Commission No. XI, in pursuance of its work, shall investigate special cases, in which the unification of the methods of analysis of organic dyestuffs offers some interest from the international standpoint."

I therefore beg you, Mr. Editor, to request, through the medium of your esteemed paper, all the heads of official analytical laboratories, and factory laboratories, as well as all those who are interested in this branch of chemistry, to have the kindness to direct the attention of our Sub-Commission (or the President or one of the members) to any case in which they consider that an international agreement would be desirable in the direction that we have indicated above. The members of the Sub-Commission for the United States are: Dr. B. Hesse, New York; Prof. Dr. S. P. Mulliken, Boston; and L. A. Olney, Lowell, Mass.

FRÉDÉRIC RÉVERDIN, *President*

GENEVA, SWITZERLAND

PLATINUM THIEF

Fellow Chemists: In THIS JOURNAL, 5, 82, I published a brief account of the recent theft of platinum from the laboratory of this Company and others, the capture of the man believed to be the thief, and the status of the prosecution.

In the recent county court session at Easton, Pa., Leon Cohn, defendant, was found guilty of stealing platinum from this Company. He received a sentence of two years imprisonment and a fine. The evidence admitted in the trial was restricted to that attaching directly to the circumstances attending his visits to this laboratory, and his arrest at Sparrows Point, Md.

After the jury's verdict, Cohn admitted to the court that he had stolen platinum, and declared that he would steal it again if he had the opportunity. I make this statement as a warning, and suggest, with emphasis, that those who have similar cases against him should not forget to start their prosecutions in proper time. Now that one conviction has been secured, subsequent cases should be relatively easy.

Before this case came to trial, one dealer in New York City was found who had bought part of Cohn's stolen platinum, and another who had, at least, sent him money. Neither of these parties would appear as a witness at the trial. Incidentally it may be remarked that there is little hope of any platinum stolen by Cohn ever being recovered. Most of us, doubtless, have entertained a false sense of security in the possession of platinum entrusted to our care, in the belief that it would be exceedingly difficult for a thief to dispose of it. Years ago there probably was more reason for this belief. Being in possession of the evidence secured in the course of this investigation, I am convinced that the disposal of platinum at the present time is a comparatively easy matter. The wide-spread and increased use of platinum in the dental and jewelry trades is substance for reflection in this regard.

It would seem unnecessary to remark that in the future there should be no recurrence of such a long series of platinum thefts by a single man, as have been perpetrated by Leon Cohn. A quiet warning, which was so effectively issued in the present case through the Secretary's office of the American Chemical Society, should result unfailingly in the apprehension of the criminal, or cause a cessation of his activities.

In conclusion, I wish to thank the many chemists who have endeavored to coöperate in the prosecution of the case just completed.

I have a considerable mass of correspondence relating to the matter, and any part of it will gladly be loaned if it may prove of value in the future.

Members of the State constabulary, who assisted in our case, will be glad to coöperate with officers at other points where it is desired to institute prosecutions. Any communications, which may properly be referred to them, will be forwarded as received.

R. J. WYSOR, *Chief Chemist*

BETHLEHEM STEEL COMPANY
SOUTH BETHLEHEM, PA.

MAGNESITE IN LOWER CALIFORNIA

Consul Lucien N. Sullivan, La Paz, Mexico, reports the presence of beds of high-grade magnesite in the region of Magdalena Bay. Analysis shows this product to contain 92 per cent. pure magnesium carbonate. The largest bed has an area of some 30 acres and is located on Margarita Island. Denouncements have been made on 13 deposits, covering a total area of 250 acres. There are 50 acres on Cedros Island.

Compañia de Desarrollo y Explotacion de la Baja California, with main offices at San Diego, Cal., will install one calcining plant on Cedros Island and another on Margarita Island, each to have a capacity of 200 tons per day. Experts who have examined the properties, taken measurements by sinking numerous test holes, and estimated the contents of the beds, report that there are more than a million tons in sight. The material is so hard that it must be blasted before it can be removed.

The U. S. Geological Survey states that the only deposits of magnesite in the United States which have been commercially utilized are those situated in the valley and coast counties of California. The total production reported in 1911 was 9,375 tons of crude magnesite, valued at $75,000, estimating the average value of the ore at $8 a ton. This is somewhat higher than the price fixed for the crude ore in 1910 (when the 12,443 short tons of crude magnesite produced were valued at $74,658), but is thought to be the present value of this product as mined and laid down at the point of shipment. Most of the magnesite produced in California is calcined at the mine, thereby reducing its weight approximately one-half and correspondingly increasing the value of the product as shipped.

The principal markets of the Eastern States and the Mississippi Valley are supplied by importation. Imported magnesite is quoted at \$10 to \$11 per long ton for the raw and \$30 to \$35 for the calcined and powdered product at New York. Imports for the year 1911 amounted to 122,075 short tons of calcined and 12,974 short tons of crude magnesite, not including the refined magnesia salts used for medicinal and other purposes.

Crude magnesite is used for the manufacture of carbon dioxide gas. Magnesium chloride is an excellent bleaching agent. The light carbonate of magnesia (alba levis) is used for medicinal and toilet purposes. The sulfate, known as Epsom salts, is mainly used in warp sizing or weighting in cotton mills, and lesser quantities are used for medicinal purposes. The hydrate is used in sugar manufacture.

Calcined magnesia, generally in the form of brick, is now universally recognized as the best material for lining basic open-hearth furnaces, copper-smelting furnaces, cement kilns, etc. It may be employed to advantage wherever high temperatures and chemical reactions are usually detrimental to dolomite-chromite, and silica brick. It is also used in the manufacture of boiler and steam-pipe covering, stoppers, sleeves, hot-metal ladles, tuyères, and nozzles.

Sintered magnesite tubing of assorted sizes is regularly made for chemical and electrometallurgical work; magnesite crucibles are made of various forms and different degrees of fineness. A coating of crushed magnesite is sometimes laid on hearths for heating steel stock for rolling to prevent the scale formed from attacking the fire-brick of the hearth.

As a building material calcined magnesite is most largely used for constructive fire-proof flooring and tiling, but it is also made into artificial stone and marble, hollow tile, drain-pipe, partitions, staircases, wainscoting, tanks, trays, wash-tubs, ornamental moldings, building blocks, cement, doorways, window casings, etc. Experiments are under way in California for making a protective paint for both wood and metal work to prevent corrosion or fire. It has lately come into quite extensive use as a flooring in steel railroad cars, being mixed with other substances to make it plastic so it may be laid in one continuous piece without seams. The fine waste of the crude ore mined is now being utilized in the California chicken ranches as a substitute for oyster-shells and other substances fed to fowls for hardening the egg-shells.

DEFECTIVE ARTIFICIAL SILK

The annual report just issued by the German Imperial Testing Department states that quite a number of samples of defective artificial silk were received during the past year for examination. The faults in the material took the form of chalky-looking places which were without luster and very tender. In all cases the goods had been produced by the guncotton process. The defects were traced to the presence of free sulfuric acid, due to the decomposition during storage of cellulose sulfate compounds formed in the process of manufacture. A number of apparently pure silk goods were also tested in the laboratories for artificial silk, and in several instances this was found to be present in a large proportion. The mixture is stated to have been very difficult to distinguish from pure silk by mere visual examination even by experts, the adulteration having been made during the throwing operation by doubling fibers of artificial silk along with those of the natural silk, and the pieces woven from the yarn so produced.

INCREASED WORLD'S CONSUMPTION OF NITRATE

The half-yearly report of W. Montgomery & Co. gives the increase in the world's production and consumption from year to year since 1902.

Year	Production Tons	Consumption Tons
1902	1,351,000	1,259,000
1903	1,462,000	1,412,000
1904	1,535,000	1,447,000
1095	1,728,000	1,547,000
1906	1,795,000	1,636,000
1907	1,818,000	1,658,000
1908	1,940,000	1,732,000
1909	2,077,000	1,938,000
1910	2,427,000	2,241,000
1911	2,482,000	2,355,000
1912	2,540,000	2,504,000
Total	21,155,000	19,729,000

Production and consumption have been running a close race at times, especially during the past four years, and it is quite possible that the coming season will feel the effect of a limited supply. At present it seems that there is very little margin for expansion in consumption either in Europe or America.

Imports of nitrate of soda into the United States in the fiscal year 1912 amounted to 481,786 tons, valued at \$15,427,904, as against 546,394 tons in 1911, valued at \$17,101,155.

THE EFFECT OF EXPOSURE ON BITUMENS—A CORRECTION

Editor of the Journal of Industrial and Engineering Chemistry:

My attention has been called to the fact that the article on "The Effect of Exposure on Bitumens" appearing in This Journal 5, 15, has been credited to the Institute of Industrial Research, while the work described in this paper was done in the laboratory of the U. S. Office of Public Roads. Kindly note this correction in the columns of your Journal.

Prévost Hubbard

THE DETERMINATION OF "VOLATILE" IN COAL—A CORRECTION.

In my article under the above title, This Journal, 5, 169, the first ash determination should be 5.40 per cent. instead of 4.40 per cent.

Fred C. Weld

THE DETERMINATION OF LIME IN COW FECES—A CORRECTION

In This Journal, 5, 37, second column, foot-note 2 should read Fresenius's "Quant. Anal.," Vol. II, p. 808.

R. Adams Dutcher

BOOK REVIEWS

Transactions of the American Ceramic Society. Volume XIV. H. F. Staley, Editor, Columbus, Ohio.

This well-appearing volume of 888 pages represents the work of the American Ceramic Society for the year 1912 and illustrates the important field being covered by this active organization. The object of the society is the study of the raw materials and products of the silicate industries, comprising the entire field of clay, glass and cement technology. Practically all of the important work being done in this field in the United States appears in these transactions which are indeed a credit to the modern spirit pervading our American industries. Among the papers we find reports of work done upon a great variety of subjects. The titles of the contributions are as follows:

"Influence of Silica and Alumina on Porcelain Glazes," by R. T. Stull.

"The Plasticity of Clay," by F. F. Grout and F. Poppe.

"Results of Tests on some Bricks from the Provinces of Western Canada," by H. Ries.

"Some Data on the Deformation Points of Feldspar Mixtures," by A. S. Watts.

"A Study of Glaze Composition on the Basis of 'Norms'," by R. C. Purdy.

"Downdraft Kiln Bottoms," by T. W. Garve.

"Colors Produced by Nickel Oxide in Ceramic Mixtures Containing Zinc Oxide," by F. K. Pence.

"Drying Defects in some Cretaceous Clays of the Great Plains Region of Canada," by Jos. Keele.

"The Influence of Composition of the Stain in the Production of Red from Chrome-Tin Pink Stains," by R. C. Purdy.

"A Comparison of the Rattler Test and the Sand Blast Test for Paving Bricks," by Edward Orton, Jr.

"A Thermal Study of Boric Acid-Silica Mixtures," by A. V. Bleininger and P. Teetor.

"The Behavior of Granular Limestone in Burned Clay," by C. F. Binns and M. A. Coats.

"The Production of Black Spots upon Terra Cotta Glazes," by C. W. Parmelee and R. Heidingsfeld.

"A Glaze Imitation of Granite," by R. Heidingsfeld.

"The Relation between Manufacture and Properties of Refractory Clay Wares," by W. Stout.

"Production of Vitrified Black and Chocolate Floor Tile," by R. C. Purdy.

"High Voltage Insulators and High Potential Testing," by E. T. Montgomery.

"The Effect of Temperature on the Dielectric Strength of Porcelains," by G. Weimer and C. T. Dun.

"The Relation between the Crushing Strength and the Porosity of Clay Products," by G. H. Brown.

"Studies of Flint Clays and their Associates," by S. L. Galpin.

"Examples showing the Usefulness of the Rational Analysis of Clays as a Means of Controlling Glaze Fit," by R. C. Purdy.

"Note on the Fuel Consumption of Some Whiteware Kilns," by T. Gray.

"Note on Load Tests Made on Magnesite, Chrome and Silica Brick," by G. H. Brown.

"Note on the Specific Heat of Clay," by J. M. Knote.

"Bodies for Inverted Mantle Rings," by R. L. Clare.

"Light Green Chromium Stains," by A. R. Heubach.

"Kaolin Mining in the South Appalachian Mountains," by A. S. Watts.

"An Apparatus for the Determination of the Expansion Coefficient of Solid Bodies," by P. A. Boeck.

"Recent Developments in the Refractories Industry," by F. T. Havard.

"A Comparison of Ten White Enamels for Sheet Steel," by R. D. Landrum.

"The Cause and Control of Crazing in Enamels on Cast Iron," by H. F. Staley.

"The Testing of Paper Clays," by C. S. Gwinn.

"Investigations on the Dielectric Strength of Some Porcelains," by B. S. Radcliffe.

"The Working of Some Montana Clays," by R. R. Hice.

"Notes and Data on Operation of the Denny-Renton Double Deck Tunnel Drier," by S. C. Karzen.

"The Power Consumption of Brick Machinery," by R. R. Hice.

"Design of an Economic Furnace for Tunnel Drying," by S. C. Karzen.

"A Note on the Accidental Pink Discoloration of a White Enamel for Terra Cotta," by D. F. Albery.

"Some Chemical Reactions of Interest to the Plate Glass Chemist," by F. Gelstharp.

"Behavior of an Acid Glass in a Tank Furnace," by J. M. Knote.

"Solubility of Some Salts in Glass," by F. Gelstharp.

"Matte Glazes," by R. C. Purdy.

"A Theory for the Cause of Matteness in Glazes," by F. K. Pence.

"Microscopic Examination of Twelve Matte Glazes," by H. F. Staley.

"The Dehydration of Clay," by G. H. Brown and E. T. Montgomery.

"Philippine Pottery," by C. H. Crowe.

"The Chemical Rôle of Boron in Glazes," by R. C. Purdy.

"The Replacement of Tin Oxide by Antimony Oxide in Enamels for Cast Iron," by R. E. Brown.

"The Necessity of Cobalt Oxide in Ground-Coat Enamels for Sheet Steel," by R. D. Landrum.

"Cobalt Colors Other than Blue," by R. T. Stull and G. H. Baldwin.

"Notes on the Performance of a Producer Gas-Fired Continuous Kiln," by W. D. Richardson.

"Note on the Dissociation of Calcium Hydrate," by R. K. Hursh.

"Oil as a Fuel in Burning Ceramic Wares," by J. K. Moore.

"Note on the Relation between Preheating Temperature and Volume Shrinkage," by R. K. Hursh.

"The Nomenclature of Clays," by C. F. Binns.

"Observations on the Effect of Pressure on the Vitrification of Clay," by L. Parker.

"The Effect of Acids and Alkalies upon Clay in the Plastic State," by A. V. Bleininger and C. E. Fulton.

"Note on Terra Cotta Glazes," by C. W. Parmelee and R. Heidingsfeld.

"The Melting Points of Pyrometric Cones under Various Conditions," by S. Geijsbeek.

The printing of the text is excellent and the illustrations, including three color plates, are very satisfactory. From the secretary's report it appears that the society is in a flourishing condition which, after all, is the most valuable criterion of the activity of an organization. A. V. BLEININGER

Preparation and Uses of White Zinc Paints. By P. FLEURY. Translated by DONALD GRANT. Scott, Greenwood & Son, London. D. Van Nostrand Company, New York. Price, $2.50.

The author, Mr. Fleury, is a French master painter, with a fondness for scientific study but without technical training. He frankly states that his predilections are for lead paints rather than zinc but, as the French Government has ordered the use of white lead to be discontinued after July 20, 1914, he has experimented to determine the best way of using zinc oxide, and this book gives the results.

The practical part of the book is the best, his formulas for paint and directions for its application being good. He calls particular attention to the necessity of varying the proportions of oil and turpentine to pigment and to each other, to suit the different pigments and classes of surface to be painted. His theories, however, are frequently questionable. He speaks of different grades of zinc oxide as varying in fineness, weight and durability. This is not correct, the variation in the gravity of commercially pure zinc oxide is less than the probable error in the determination. The final size of grain is the same in all cases, but the grain seen is always an aggregation. These aggregations vary considerably in size and compactness, which conveys the impression of differences in size and gravity. In speaking of the oxides commercially free from lead, he states that the lower grades are more durable. This again is a mistake, the grading is simply by color, except in the case of White Seal, and all are equally durable. With leaded zincs the case is different; they are graded both by color and lead content, which varies from 3 per cent. to 40 per cent. lead sulfate. Regarding the relative values of these as protective coatings, we have not much information, but what there is indicates that the presence of lead sulfate is not injurious. Where the paint is used for decoration the lead salts are all undesirable, although the sulfate is less so than the carbonate or hydroxide.

The chapter on the manufacture of zinc oxide is very incomplete as it does not mention the direct process by which most of the zinc oxide used is made.

He very properly calls attention to the inferiority of mixtures of zinc sulfide and barium sulfate to lithopone, and speaks well of the latter for inside work, while condemning it for outside. In this he agrees with the general opinion, although recent improvements in its manufacture seem likely to modify this conclusion.

Covering power is defined as *hiding power*, and the author says "that paint which masks the under surface best with the least thickness of paint, is the one that has the greatest covering power." An excellent method is described for a practical test, and the result of a very careful set of experiments by Lenoble of Lille is given. These show that weight for weight, or bulk for bulk, zinc oxide has a greater covering power than white lead, but, owing to its requiring more oil, it needs a greater number of coats to give an equal amount of pigment on the same surface.

There is a good chapter on grinding in which the author points out the necessity of having all materials perfectly dry, of keeping the oil at a normal and uniform temperature, and of properly regulating the speed of the mills. He states what is perfectly true, but not always recognized, that good materials may make a bad paint if improperly ground.

An excellent account is given of the experiments of the Dutch Commission and of the legislation prohibiting and regulating the uses of white lead. The author freely admits his prejudice in favor of lead, but endeavors to be, and in the main is, fair in his comments.

The last chapter is on "Qualitative Analysis," and it is unfortunate that it was written, as the author's knowledge of chemistry is rudimentary. The translator has endeavored to make up for this by numerous foot-notes to this chapter, pointing out the errors and omissions in the text, and thereby partly makes up for the curious and involved English which he frequently uses.

With the exception of the chapter on "Analysis" and some of the theories advanced, the book is good, but hardly a safe one to put in the hands of a non-technical reader. For the technologist who wishes to learn more of the painter's view of pigments, it can be recommended.

George C. Stone

Refractories and Furnaces. By F. T. Havard. McGraw-Hill Book Co., New York, 1912. Price, $4.00.

This timely work deals with a subject of prime importance to metallurgy and aims to present the principles underlying the production and use of refractories. The introduction gives a brief historical review of clay working, of which the part dealing with the development of the American fire-brick industry is the most satisfactory. In Chapter I, the usual classification of acid, basic and neutral refractories is adopted. The table of formation and melting temperatures of silicates in Chapter II might have been greatly enlarged. The description of the manufacture of silica brick, Chapter III, is concise and satisfactory. The figures given for the burning of silica brick give rise to a discrepancy; in one place it is said that the temperature rises as high as 1600° C., in another that it is 1500° or 1700°.

The treatment of the subject of refractory clays is not as satisfactory as might be wished and it is evident that the writer is on unfamiliar ground. Thus considerable attention is given to the old Bischof refractory coëfficient which has been discarded long since. The work of Richters, Seger, Cramer, Ludwig, Mellor, Rieke, Flach, on fire clays is not mentioned. The use of waste gases from kilns for steam raising is not common in this country. The Cassel kiln cannot be said to be an economical kiln; if anything it is less so than the downdraft. Chapter V, dealing with the basic and neutral refractories, is interesting and instructive. Chapters VI, VII, VIII and IX, dealing with the construction of metallurgical furnaces and the use of refractories, form a valuable summary of this subject. This applies particularly to copper furnaces. The use of magnesium oxychloride and carborundum as an acid-proof mortar for the Glover tower, etc., does not seem promising nor as satisfactory as the barium sulfate-water glass cement frequently employed.

In Chapter X instances of the use of refractory bricks in industrial furnaces are briefly described. More attention might have been given to glass furnaces where the proper use of refractories is exceedingly important. Some interesting facts have been compiled in Chapter XI, the use of refractory mortars and paints. The use of coatings for rendering furnace walls more heat-resistant as quoted on page 218 is misleading.

In Chapter XII, refractory hollow ware, the treatment of retorts is quite satisfactory; no mention is made of glass pots. Chapter XIII, on the testing of refractories, is inadequate. No mention is made of Purdy's method of classifying fire clays. The statement that "the refractory must stand a temperature of at least 50° C. above that at which it is used" needs elaborate amplification. The viscosity of refractories at furnace temperatures is an important property which requires discussion and which is not satisfactorily measured by the Cramer test. The Deville furnace is not satisfactory in this day of carbon resistance furnaces. Chapter XIV contains compilations of the work of Wologdine, Hering and others. The subject of heat measurement is considered in Chapter XV. While two diagrams are given of the electric resistance pyrometer, the subject of the thermocouple is dismissed too briefly. Mention might have been made of the Holborn-Kurlbaum optical pyrometer. The book concludes with a chapter on common bricks.

The work as a whole is interesting and while not quite satisfactory to the specialist in silicate technology, fills an important need on the part of the user of refractories. The typographical execution of the book is excellent.

A. V. Bleininger

The Science of Hygiene. By Walter C. C. Pakes. New edition, revised by A. T. Nankivell. 8vo., 164 pages. D. Van Nostrand & Co., New York. Price, $1.75.

In the preface of this book, the reviser tells us that it is intended for candidates for the degree of Doctor of Public Health in Great Britain. A careful perusal of the book shows it to be very elementary in character, going into altogether too many details in the discussion of analytical methods. In this country, the book is of such a quality as would be used by high school students, and would also be of value in public libraries where very young men go to find books of a semi-popular nature. The book has the appearance of having been hastily written, and still more hastily proof-read, as is evidenced by the following errors which were noted:

Page 7: 4.55 grams instead of "parts" per one hundred thousand.

Page 56: "Redux" instead of "reflux."

Page 77: "Filter" funnel instead of "separating" funnel.

Page 92: Fixed acidity of Sherry 270 instead of 0.270.

Page 107: 100 grams *is* taken.

The spelling throughout the book is not altogether uniform. The author speaks of "grammes," and of milligrams and kilograms, also mixes metric, and common weights and measures, and refers the specific gravity to a basis of 1000 instead of to 1.000 as is now customary. A number of antiquated expressions, such as "Baryta water" are used.

The following errors in chemistry are especially glaring:

Page 27: In mentioning the white precipitate of lead sulfate formed on the addition of H_2SO_4 to a water containing lead, no mention is made of the fact that the presence of barium would also give a white precipitate.

Page 53: The Kjeldahl method as described would undoubtedly lead to a loss of ammonia before the distillation.

Page 84: The ashing of flour in a platinum dish is recommended. The author apparently forgets the effect on platinum of the phosphorus present in cereals.

Page 93: In testing for arsenic, the author recommends the Reinsch test, but makes no mention of the Gutzeit test and its modifications which latter methods are quicker and more easily carried out.

The book is divided into the following divisions: Analysis of water, milk, butter, flour, bread, coffee, spirits, wines, beer, vinegar, air, soils, disinfectants, microscopy, meat inspection. There is also a chapter on reagents and standard solutions in the middle of the book, while additional directions for reagents are given in each chapter, oftentimes without mention of the solvent to be used. It would considerably improve the book if all the directions for the preparation of reagents and standard solutions were in one place, preferably in the back of the book in an appendix.

The best part of the book consists of the collection of typical water analyses with notices on their interpretation. Thirty such analyses are given and well explained.

D. D. Berolzheimer

NEW PUBLICATIONS

By D. D. Berolzheimer, Librarian, Chemists' Club, New York

Cellulose, Chemistry of, with Special Reference to the Textile and Fibre Industries. By C. G. Schwalbe. Vol. II. L. 8vo., pp. 660. Price, $7.00. Gebrueder Borntraeger, Berlin. (German.)

Chemical Industries of Belgium, Netherlands, Norway and Sweden. By Thomas H. Norton. 8vo., pp. 85. Dept. of Commerce and Labor, Special Agents Series No. 65.

Civil Engineers' Pocketbook. By Albert I. Frye. 8vo., pp. 1658. Price, $5.00. D. Van Nostrand Co., New York.

Dairy Technology: Dairy Products and their Uses. By C. Larsen. Sm. 8vo., pp. 298. Price, $1.50. John Wiley & Sons, New York.

Electrical Photometry and Illumination. By H. Bohle. 8vo., pp. 234. Price, $2.75. Chas. Griffin & Co., London.

Food Inspection and Analysis. By Albert E. Leach and Andrew L. Winton. 3rd Edition. L. 8vo., pp. 1001. Price, $7.50. John Wiley & Sons, New York.

History of Chemistry. By James Campbell Brown. Vol. II. 8vo., pp. 543. Price, $2.75. J. & A. Churchill, London.

Hydrocellulose, Acetylcellulose and the Determination of Acetic Acid in Acetylcelluloses. By W. Beck. 8vo., pp. 58. Thesis, University of Leipzig, Leipzig. (German.)

Inorganic Chemistry, Gmelin and Kraut's Handbook of. Vol. III, Part 1. By C. Friedheim and F. Peters. 7th edition. L. 8vo., pp. 1568. Price, $16.50. Carl Winter, Heidelberg. (German.)

Iron and Steel, An Outline of the Metallurgy of. By A. H. Sexton and J. S. G. Primrose. 2nd edition. 8vo., pp. 586. Price, $3.25. Scientific Publishing Co., London.

Lead Poisoning and Lead Absorption. By Thomas M. Legge and Kenneth W. Goadby. 8vo., pp. 308. Price, $3.50. Longmans, Green & Co., New York.

Leather Dyeing and Preparing. By M. C. Lamb. 2nd edition. L. 8vo., pp. 427. Price, $5.25. Julius Springer, Berlin. (German. Translation.)

Metals, The Casting of. By E. A. Schott. Lex. 8vo., pp. 285. Price, $3.50. B. F. Voigt, Leipzig. (German.)

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Metal Working, Handbook of Autogenous. By Theo Kautny. 8vo., pp. 712. Price, $4.50. Carl Marhold, Halle. (German.)

Metals, the Rarer, The Mineralogy of. By E. Cohen and W. O. Wooton. 12mo., pp. 240. Price, $1.50. Chas. Griffin & Co., London.

Metals, Mixed, or Metallic Alloys. By Arthur H. Hiorns. 3rd edition. 8vo. Price, $1.50. Macmillan Co., New York.

Metallurgy, Treatise on. By L. de Launay. Vol. III. 8vo. Price, $18.00. Ch. Beranger, Paris. (French.)

Nitrocellulose Produced from Sulfite Nitrocellulose and Strawfiber, Use of. By K. A. Nitzelnadel. 8vo. Price, $0.25. Dissertation, University of Dresden, Dresden. (German.)

Organic Compounds, The Preparation of. By E. de B. Barnett. 8vo., pp. 326. Price, $2.25. A. & J. Churchill, London.

Paper: The Testing of Wood Pulp. By R. W. Sindall and W. N. Bacon. 8vo. Price, $2.50. D. Van Nostrand Co., New York.

Peat as a Source of Power. L. 8vo., pp. 164. Price, $1.50. Julius Springer, Berlin. (German.)

Petroleum: Laboratory Book of Mineral Oil Testing. By Jas. A. Hicks. 8vo., pp. 74. Price, $1.50. Chas. Griffin & Co., London.

Petroleum, the Motive Power of the Future. By W. S. Tower and J. Roberts. 8vo., pp. 254. Price, $1.50. Hodder & Stoughton, London.

Photochemistry, Textbook of. By A. Benrath. 8vo. Price, $1.50. C. Winter, Heidelberg. (German.)

Public Health Chemistry and Bacteriology. By David McKail. 8vo., pp. 409. Price, $2.50. William Wood & Co., New York.

Qualitative Analyses. By Wilhelm Biltz. 8vo., pp. 129. Price, $1.50. Akademische Verlagsgesellschaft, Leipzig. (German.)

Rubber. By Rudolf Ditmar. 8vo., pp. 140. Price, $1.75. Julius Springer, Berlin. (German.)

Soap-Makers' Handbook. By William T. Brannt. 2nd ed. 8vo., pp. 535. Price, $6.00. Henry Carey Baird & Co., Philadelphia.

Sugar Industry in the Philippine Islands, Handbook on the. By G. E. Nesom, Herbert S. Walker, *et al.* L. 8vo. Price, $2.00. Bureau of Science, Manila.

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Water from Mains, Detection and Colorimetric Determination of Lead Copper and Zinc in. By L. W. Winkler. *Zeitschrift fuer angewandte Chemie*, Vol. 26, 1913, No. 3, pp. 38–44.

White Lead, The Toxicity of. By Charles A. Line. *Drugs, Oils and Paints*, Vol. 28, 1913, No. 8, pp. 295 and 301–302.

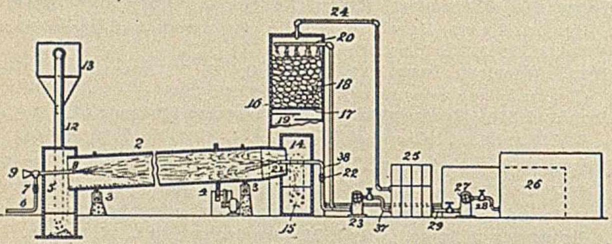
Wood Preservation. By H. v. Schrenk, F. M. Bond and H. F. Weiss. *Engineering Record*, Vol. 67, 1913, No. 4, pp. 108–110.

RECENT INVENTIONS

By C. L. Parker, Solicitor of Chemical Patents, McGill Building, Washington, D. C.

Suspensible Sulfur. H. W. Hillyer, Dec. 24, 1912. U. S. Pat. 1,048,161. The patentee claims the invention of a new form of sulfur which he terms suspensible sulfur, the material being suspensible in water and which, when so suspended, is not coagulable by sulfuric acid. The product is to be used as a fungicide.

Recovery of Sulfuric Acid and Iron from Ferrous-Sulfate Sludge. C. A. Weeks, Dec. 24, 1912. U. S. Pat. 1,048,247. In the patentee's preferred process, concentrated ferrous sulfate solution is sprayed into the upper end of a heated, slightly inclined, rotating, tubular furnace, heated by a jet of gas admitted to the lower end of the furnace. The ferrous sulfate is gradually heated within the furnace until the greatest temperature zone is reached, at which the calcination of the ferrous sulfate crystals takes place with the result that ferric oxid is formed and the whole content of sulfur gases with other vapors are released. The ferric oxid is discharged from the lower end of the furnace, while the sulfur gases and other vapors pass from the higher end of the furnace where the temperature zone is cool and are delivered into the condensing tower where it is subjected to treatment of a spray of weak solution of sulfuric acid. This condenses and absorbs the contained sulfuric acid gas, the other gas passing on. The absorbed sulfuric acid gas is withdrawn from the condensing tower and stored or used over again; the ferric oxid is collected from the lower end of the furnace, cooled and recovered as ferric oxid free from sulfur.



Formic Acid. W. H. Walker, Dec. 31, 1912. U. S. Pat. 1,048,609. In making formic acid by the reaction of sulfuric acid on a formate, the patentee uses a formate substantially free from water whereby the reaction proceeds without injurious rise of temperature or substantial loss of formic acid.

Waterproofing Concrete. A. C. Horn, Dec. 31, 1912. U. S. Pat. 1,048,695. The composition comprises Chinese wood oil, calcium stearate and casein, and a preservative compound.

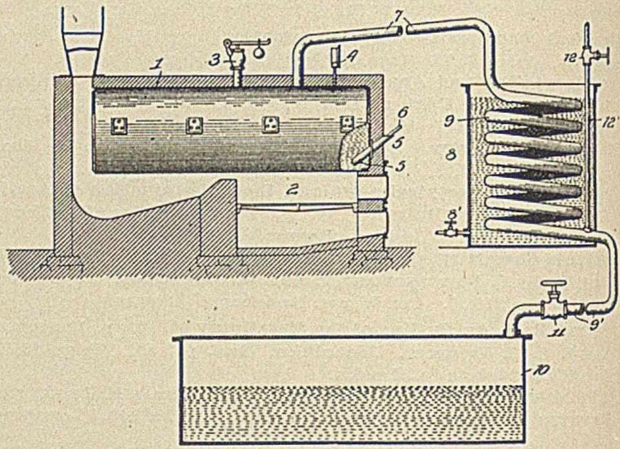
Electric-Furnace Lining. Frederick M. Becket, Jan. 7, 1913. U. S. Pat. 1,049,801. The lining consists of a refractory material having an adherent facing of ferrotungsten reduced from the refractory material.

Luminous or Flaming Arc Electrode. Joseph L. R. Hayden, Jan. 7, 1913. U. S. Pat. 1,049,854. The electrode contains titanium carbid and cerium titano-fluorid.

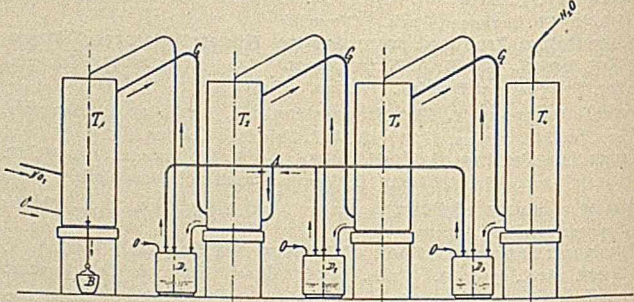
Tar-Paint Manufacture. Friedrich Raschig, Jan. 7, 1913. U. S. Pat. 1,049,916. The paint consists of an emulsion of tar, clay and water, produced by mixing clay with water and then gradually stirring tar and water into the mixture until the tar is uniformly distributed.

Purifying Asbestos. W. C. Arsem, Jan. 7, 1913. U. S. Pat. 1,049,972. Difficultly removable iron oxid is removed from asbestos by soaking the crude material in a 50% solution of orthophosphoric acid for from 24 to 72 hours and washing the fibers to remove the adherent acid.

Gasolene Manufacture. W. M. Burton, Jan. 7, 1913. U. S. Pat. 1,049,667. On distilling at from 650°–850° F. the volatile constituents of the liquid portions of the paraffin series of petroleum distillations having a boiling point upward of 500° F., low-boiling products of the same series are obtained, by maintaining a pressure of 4 to 6 atmospheres on the vapors throughout their course from the still through the condenser and while undergoing condensation. From time to time the accumulations of gas from the products of condensation are released.



Nitric Acid. Moest and von Berneck, Jan. 7, 1913. U. S. Pat. 1,049,754. In the production of concentrated nitric oxid gases it has been found that a more concentrated nitric acid is obtained if oxygen is used instead of atmospheric air for converting these gases into nitric acid in the presence of water.

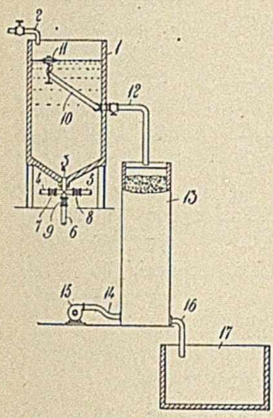


The patentee charges with oxygen the apparatus serving for the conveyance of the liquid in the absorption system and reintroduces the oxygen leaving the apparatus simultaneously with the entrained vapors containing the nitric acid.

Non-Inflammable Cellulose Compound. W. G. Lindsay, Jan. 7, 1913. U. S. Pat. 1,050,065. The composition contains 100 parts of acetyl cellulose, from 10 to 20 parts of triphenylphosphate and from 1 to 2 parts of urea in admixture with acetylenetetrachlorid and acetone.

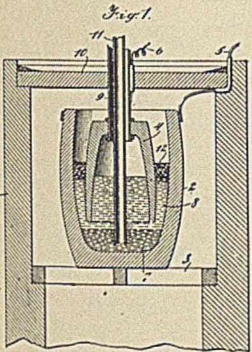
Concentrated Nitric Acid. Moest and von Berneck, Jan. 14, 1913. U. S. Pat. 1,050,160. The patentees produce concentrated nitric acid in a direct way by treating dilute nitric acid with a mixture of nitrogen peroxid and oxygen. The mixture is subjected to the action of oxygen to expel the non-oxidized nitric oxid gases from the nitric acid.

Separating Copper from Other Metals. T. A. Edison, Jan. 14, 1913. U. S. Pat. 1,050,629 and 30. In separating metallic copper from other metals the associated metals are treated with a solution of ammonium sulfate or copper sulfate containing a reducible haloid of a metal of variable valence and supplying an oxygen-containing gas to the mixture.



Alcohol from Sulfite Liquor. P. B. Ekström, Jan. 14, 1913. U. S. Pat. 1,050,723. In the process of manufacturing alcohol by fermenting the saccharine matters of sulfite liquor from sulfite pulp, it is difficult to maintain a quantity of free oxygen sufficient for the development of the yeast, due to the fact that the liquor contains reducing compounds, or compounds which are easily oxidized during the fermentation, that rapidly consume the free oxygen that may be supplied by usual aëration.

The patentee's process consists in adding to the liquor before fermenting the same, a catalyzer for oxidation and then aërating the liquor. The catalyzer effects a rapid oxidation of those compounds which later on during the fermentation process would be capable of consuming the free oxygen in the liquor.

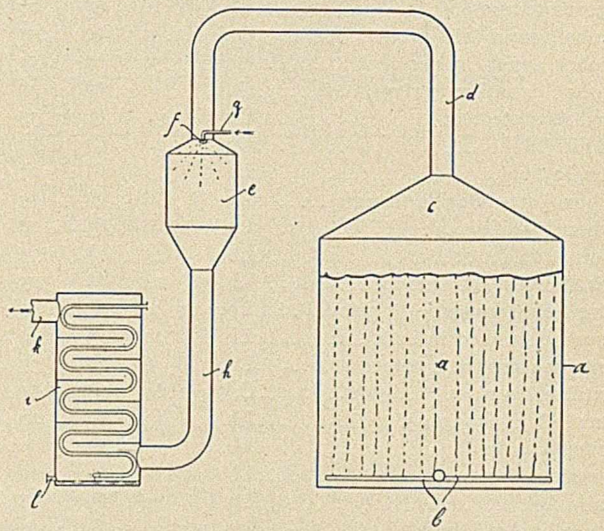


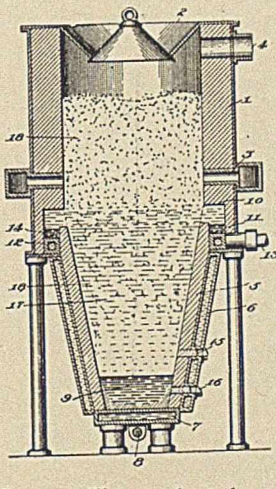
Economic Method of Obtaining Gases. C. E. Acker, Jan. 21, 1913. U. S. Pat. 1,050,902. This is a process of continuously separating gases from gaseous mixtures or from liquids or solids containing them and is adapted for the production of nitrogen from atmospheric air as well as the production of hydrogen through the decomposition of steam or water.

In the production of nitrogen from atmospheric air, such air is brought into contact with a heated metal capable of being oxidized, such as lead. A metal oxid is formed and the nitrogenous residue separated therefrom. The metal oxid is dissolved in a menstruum preferably consisting of a mixture of fused alkali metal salts, and the metal oxid is reacted upon by a reducing agent which is held in solution in such menstruum. The process may be continuously carried out in the apparatus illustrated.

Potash, Iodine and Chlorine from Seaweeds. Cameron and Moore, Feb. 4, 1913. U. S. Pat. 1,051,984. The filtered and partially evaporated solution obtained by lixiviating the ash of seaweeds is electrolyzed in a cell having its anode and cathode chambers separated by a diaphragm, thus oxidizing the liberated iodin to iodic acid. By treating the anode solution with ferrous sulfate (or iron) and sulfuric acid, the iodin is separated from the iodic acid. Potassium hydroxide is found in the cathode solution.

Regaining Alcohol from Air Passed through Fermenting Vats. A. Zeckendorf, Jan. 21, 1913. U. S. Pat. 1,051,051. The more recent fermentation processes in which a large quantity of air is caused to pass through the fermenting mash it has heretofore not been possible in all cases to economically separate the alcohol absorbed. The patentee adds to the mixture of alcohol and air a vapor, such as steam; this is quickly taken up by the mixture of alcohol and air and has every opportunity to absorb the alcohol. Since the water can be more easily removed from air than alcohol, the absorbed alcohol—with the exception of but small traces—can now be separated from the air by simply condensing the vapor. The alcohol is therefore nearly entirely regained.





Ferrosilicon by Reduction. E. A. Byrnes, Jan. 21, 1913. U. S. Pat. 1,051,194. According to this process, refractory metal-bearing ores and compounds and specifically a charge consisting of a silicon compound, a source of iron and coal for the production of ferrosilicon, are reduced by submerging the lower portion of the charge in a vertical molten resistor-column, preheating the unsubmerged upper portion by passing air and hot products of combustion upward through it, and heating the submerged lower portion by passing through the molten resistor an electric current of energy-density increasing downward toward the zone of reduction. The electric terminals are preferably of molten ferrosilicon, one being a ring surrounding the upper end of the resistor-column and the other a body of the metallic product beneath the lower end of this column. As reduction proceeds the excess slag and metal are tapped out and additions are made to the charge.

Acetone and Higher Alcohols by Fermentation of Starch Sugars and Other Carbohydrate Materials. A. Fernbach and E. H. Strange, Nov. 12, 1912. U. S. Pat. 1,044,368. In this process carbohydrate matter is mixed with water, and a suitably degraded yeast added. The mixture is then sterilized, a ferment of the type of the butylic bacillus of Fitz added and the mixture fermented in the absence of air.

MARKET REPORT

AVERAGE WHOLESALE PRICES OF STANDARD CHEMICALS, ETC., FOR THE MONTH OF FEBRUARY, 1913

ORGANIC CHEMICALS

Acetanilid	Lb.	21	@	23
Acetic Acid (28 per cent.)	C.	2.00	@	2.15
Acetone (drums)	Lb.	16½	@	17½
Alcohol, grain (188 proof)	Gal.	2.55	@	2.57
Alcohol, wood (95 per cent.)	Gal.	50	@	52
Alcohol, denatured (180 proof)	Gal.	40	@	42
Amyl Acetate	Gal.	2.35	@	2.55
Aniline Oil	Lb.	10⅛	@	10⅝
Benzoic Acid	Lb.	23	@	27
Carbon Tetrachloride (drums)	Lb.	8½	@	9
Carbon Bisulfide	Lb.	6½	@	8
Chloroform	Lb.	25	@	35
Carbolic Acid (drums)	Lb.	14	@	15
Citric Acid (domestic), crystals	Lb.	40	@	40½
Camphor (refined in bulk)	Lb.	42½	@	44
Dextrine (imported potato)	Lb.	6	@	7
Dextrine (corn)	C.	2.60	@	2.81
Ether (U. S. P., 1900)	Lb.	14	@	20
Formaldehyde	Lb.	8½	@	9½
Glycerine (dynamite)	Lb.	18½	@	19
Oxalic Acid	Lb.	7⅞	@	8⅛
Pyrogallic Acid (bulk)	Lb.	1.20	@	1.40
Salicylic Acid	Lb.	29	@	31
Starch (corn)	C.	1.82	@	2.27
Starch (potato)	Lb.	5¼	@	5¾
Tannic Acid (commercial)	Lb.	35	@	35½
Tartaric Acid, crystals	Lb.	30¼	@	31

INORGANIC CHEMICALS

Acetate of Lime (gray)	C.	2.50	@	2.60
Acetate of Lead (brown, broken)	Lb.	7¾	@	8
Alum (lump)	C.	1.75	@	2.00
Aluminum Sulfate	C.	90	@	1.75
Ammonium Carbonate, domestic	Lb.	8	@	8½
Ammonium Chloride, gray	Lb.	6⅛	@	6½
Aqua Ammonia (drums) 16°	Lb.	2¼	@	2½
Arsenic, white	Lb.	4½	@	5
Brimstone (crude, domestic)	Ton	22.00	@	22.50
Barium Chloride	C.	1.70	@	1.80
Barium Nitrate	Lb.	5	@	5¼
Borax, crystals (bags)	Lb.	3¾	@	4¼
Boric Acid, crystals (powd.)	Lb.	7	@	7½
Bromine, bulk	Lb.	30	@	35
Bleaching Powder (35 per cent.)	C.	1⅜	@	1½
Barytes (prime white, foreign)	Ton	18.50	@	22.50
Blue Vitriol	Lb.	5¼	@	5½
Calcium Chloride	C.	65	@	90
Chalk (light precipitated)	Lb.	4	@	5
China Clay (imported)	Ton	11.50	@	18.00
Feldspar	Ton	7.00	@	9.00
Fuller's Earth, powdered	C.	80	@	85
Green Vitriol (bulk)	C.	55	@	60
Hydrochloric Acid (18°)	C.	1.15	@	1.55
Iodine (resublimed)	Lb.	3.05	@	3.10
Lead Nitrate	Lb.	8⅛	@	8¼
Lithium Carbonate	Lb.	65	@	70
Magnesite (raw)	Ton	7.50	@	8.50
Nitric Acid, 36°	Lb.	3⅞	@	4¼
Phosphorus	Lb.	35	@	90
Phosphoric Acid, sp. gr. 1.75	Lb.	21½	@	25½
Plaster of Paris	Bbl.	1.50	@	1.70
Potassium Bromide	Lb.	39	@	40
Potassium Permanganate (bulk)	Lb.	9¾	@	11
Potassium Cyanide (bulk) 98–99%	Lb.	19	@	24
Potassium Iodide (bulk)	Lb.	2.60	@	2.65
Potassium Chlorate, crystals	Lb.	9	@	10½
Potassium Nitrate (crude)	Lb.	4¾	@	5
Potassium Bichromate, 50°	Lb.	6⅞	@	7
Quicksilver, Flask		40.00	@	—
Salt Cake (glass-makers')	C.	55	@	65
Silver Nitrate	Oz.	39⅛	@	40
Soapstone in bags	Ton	10.00	@	12.00
Sodium Acetate	Lb.	4	@	4½
Sodium Chlorate	Lb.	8¼	@	9½
Sodium Bicarbonate (English)	Lb.	2¾	@	3
Sodium Bichromate	Lb.	5	@	5½
Sodium Hydroxide, 60 per cent.	C.	1.60	@	1.65
Sodium Hyposulfite	C.	1.30	@	1.60
Sodium Nitrate, 95 per cent., spot	C.	2.62½	@	—
Sodium Silicate (liquid)	C.	65	@	1.50
Strontium Nitrate	Lb.	6⅞	@	7⅝
Sulfur, Roll	C.	1.85	@	2.15
Sulfur, Flowers (sublimed)	C.	2.20	@	2.60
Sulfuric Acid, 60° B.	C.	85	@	1.00
Talc (American)	Ton	15.00	@	20.00
Terra Alba (American), No. 1	C.	75	@	80
Tin Bichloride (50°)	Lb.	14¼	@	14½
Tin Oxide	Lb.	52	@	54
Zinc Chloride (granulated)	Lb.	4½	@	5
Zinc Sulfate	Lb.	2¼	@	2½

OILS, WAXES, ETC.

Beeswax (pure white)	Lb.	42	@	45
Black Mineral Oil, 29 gravity	Gal.	13½	@	14
Castor Oil (No. 3)	Lb.	9½	@	10½
Ceresin (yellow)	Lb.	12	@	22
Corn Oil	C.	5.80	@	5.85
Cottonseed Oil (crude), f. o. b. mill	Gal.	39	@	39½
Cylinder Oil (light, filtered)	Gal.	21½	@	32
Japan Wax	Lb.	9¾	@	10
Lard Oil (prime winter)	Gal.	88	@	92
Linseed Oil (double-boiled)	Gal.	50	@	52
Paraffine Oil (high viscosity)	Gal.	26	@	28
Paraffine (crude 120 & 122 m. p.)	Lb.	3½	@	3¾
Rosin Oil (first run)	Gal.	34	@	36
Spindle Oil, No. 200	Gal.	18	@	19
Sperm Oil (bleached winter), 38°	Gal.	72	@	74
Stearic Acid (double-pressed)	Lb.	9⅜	@	10
Tallow (acidless)	Gal.	64	@	67
Tar Oil (distilled)	Gal.	30	@	31

METALS

Aluminum (No. 1 ingots)	Lb.	25½	@	26
Antimony (Hallet's)	Lb.	9	@	9½
Bismuth (New York)	Lb.	2.10	@	2.15
Copper (electrolytic)	Lb.	14½	@	15
Copper (lake)	Lb.	14⅞	@	15
Lead, N. Y.	Lb.	4.30	@	4.40
Nickel	Lb.	50	@	55
Platinum (refined)	Oz.	46.00	@	—
Silver	Oz.	62	@	—
Tin	Lb.	49	@	50
Zinc	Lb.	6.35	@	6½

FERTILIZER MATERIALS

Ammonium Sulfate	C.	3.25	@	—
Fish Scrap, domestic, dried	Unit	nominal		
Blood, dried	Unit	2.80	@	—
Tankage, high-grade	Unit	2.70	&	10
Bone, 4½ and 50, ground, raw	Ton	28.00	@	—
Potassium, "muriate," basis 80%	Ton	38.55	@	—
Phosphate, acid, 16 per cent.	Ton	7.00	@	8.00
Phosphate rock; f. o. b. mine:				
Florida land pebble, 68 per cent.	Ton	3.00	@	3.25
Tennessee, 70–80 per cent.	Ton	5.00	@	5.50
Pyrites, furnace size, imported	Unit	0.13½		
Castor meal	Unit	3.10		
Mowrah meal	Ton	9.00	@	—