THE JOURNAL OF INDUSTRIAL AND ENGINEERING CHEMISTRY

VOL. I. MARCH, 1909. No. 3

THE JOURNAL OF INDUSTRIAL AND ENGINEERING CHEMISTRY
PUBLISHED BY
THE AMERICAN CHEMICAL SOCIETY.

Published monthly. Subscription price to non-members of the American Chemical Society $6.00 yearly.

Vol. I. MARCH, 1909. No. 3

EDITORIALS.

EDUCATION.

THERE have been a large number of papers published within the past year which dealt with the preparation of engineers in general, or of chemists and chemical engineers in particular. Most of them were written by practical engineers. If the teachers of the land have read them all, they are still justified in following their own concepts to a considerable degree, because of the disparity on the one side and impracticability on the other. If the teachers have read but part of them and have been deeply impressed, they may be changing good plans for poorer ones. They may be making the future graduate of their courses still more of a hand-book of ready data and of evanescent processes, of quick conclusions and of decapitated originality. They may be turning out a machine to fit the present requirements of a certain kind of mill, which mill will pay a royalty of sixty dollars a month for the use of the machine, but is this the best attainable? Should not such requirements be considered merely as a fortunate demand for by-products? Are not the "seconds" of the chemist-plant good enough for the mill which wants a cheap machine? The broken saggers of a porcelain factory might be a useful by-product, but should we forget the making of the porcelain?

In chemical industries and engineering, the one thing most needful is character; which means integrity, individuality, energy, push, etc. These must be greater than the average to warrant greater than average success. The memory of the stored facts, the familiarity with the prices and names of common chemicals, the details of their manufacture, etc., need be only an average-grade-accessory. If a man cannot think independently, cannot talk or write decently, cannot listen internally, and finally does not do so frequently and better than the average, then the retention of stored knowledge will be a useless task to him.

It may be maintained that such assets as character, interest, judgment, etc., cannot be poured through a funnel, that they cannot be driven in with a hammer, that they are hereditary in the man or absent forever, but is it true? Are not these desiderata largely the result of intimate contact with such personal qualities as distinct from physical contact with other things? They are not openly demanded of the teacher by the student as he demands chemical knowledge, nor are they likely to be demanded as long as every man feels that he does not need them. But may they be acquired? In practice it is the difference in these traits which is of greater influence than accumulated knowledge, though the latter be ever so necessary. A well-trained engineer in any science will very quickly look and act and become like a well trained chemist if he finds himself called upon to go deeply into chemistry. A poorly trained chemist may have trouble keeping up with the office boy on routine steel analysis if he lets himself latibulize. The deciding difference between the two is in the personality or character, and not in the stock of knowledge. It seems as though the automatic power of centering and maintaining interest in things was one of the most desired traits to impart to a very young chemist.

W. R. Whitney.

THE NEXT STEP IN PUBLICATION FOR THE AMERICAN CHEMICAL SOCIETY.

Several years ago Professor A. A. Noyes proposed a classification of the articles in the *Journal of the American Chemical Society* with a separate paging for each class. A little more than two years ago the writer presented to the Council of the Society a scheme for the publication of a set of separate journals representing the more important subdivisions of chemistry. The plan proposed was imperfect in some of its details and it involved a coördination of conflicting interests which is probably too Utopian for complete realization. The American Chemical Society has, however, accepted the most vital part of the proposal and has established a separate journal devoted to the interests of industrial and engineering chemistry. Every one will, I am sure, agree that the development of publication has been so rapid recently that we should wait till we can see more clearly the effect of present policies before taking another step forward. It is, however, wise for us to begin to consider the direction which future development should take.

The purposes which have led to the establishment of the Journal of Industrial and Engineering Chemistry are: First, to care better for the interests of a class of chemists and manufacturers who form a very large group of our membership. Second, to furnish a more suitable medium for the publication of articles in this field in the hope that we may bring a large portion of the best industrial articles together in a single journal where they will be easily accessible to the chemists of the country. Third, to gain new members with the support which they will give. A fourth reason which might apply in other cases would be the desire to combine the articles from some fields of chemistry which is represented in the *Journal of the American Chemical Society* with those published in some existing journal, with the hope of securing for this journal the publication of practically all American work in the field.

All of the reasons given lead many physical chemists in America to desire the publication by the American Chemical Society of a journal of physical chemistry. The difficulty of separating general and physical from inorganic articles, as indicated by German experience, makes it seem desirable to combine the three classes of articles in one journal. Such a journal, if established, would take about two-thirds of the articles which will probably be published in the *Journal of the American Chemical Society* during the present year. Evidently what would be left could no longer be called, appropriately, the *Journal of the American Chemical Society*, and the establishment or adoption by the society of a journal of physical chemistry means also the establishment or adoption of a journal of organic and biological chemistry. This has not, perhaps, been sufficiently considered by the physical chemists.

If we assume that the publication of a journal of physical and inorganic chemistry is desirable, the next questions are those of practical detail. In the first place it is to be hoped that some arrangement can be made, satisfactory to the Editor of the *Journal of Physical Chemistry*, by which the articles published In the *Journal of the American Chemical Society* can be united with those published in the former.

The financial side of the question is best considered in connection with the different plans which may be proposed:

(1) The journal might be published on a subscription basis. It is extremely doubtful if sufficient financial support could be secured in this way, but, even if it could it would mean that we should take away from one class of members of the American Chemical Society those articles in which they are especially interested, compelling them to do without or pay extra for them. It would, in effect, raise the dues for those interested in physical chemistry. I am sure this is not desirable.

If two journals were published in place of the *Journal of the American Chemical Society*, with an increase of 800 pages of material, and both journals were to go to all our members the additional cost of printing would be about $2500. If we add $700 for the salary and expenses of the editor, we have $3200 a year as the sum needed for the establishment of this journal. This expenditure might be met by one of the following plans:

(2) On payment of the present dues of $10 members might be permitted to select any three of the four journals which we would then publish.

Any one wishing all four would pay $3 additional. If we assume that 500 would do this (a liberal estimate) this would give $1500, while about $2800 would be saved by the decrease of 3500 copies in the total circulation of the three journals. Such a plan would, in effect, add $3 to the dues of all who wished, as now, to have access to original papers in all fields and would deprive all the rest of the papers in some one field. The increase would be especially felt by students and young men just beginning their professional career. The injury which would result from giving to the majority of our members a one-sided literature appears to me very serious. We ought not to think chiefly of the members of the council of the society and of chemists of similar character and position but rather of members with limited means and of the many who are isolated so that the journals published by the society are the only ones they can see. The decreased circulation would also make the journals less attractive as a means of publication and might also interfere seriously with advertisements.

(3) An increase of $1 in the dues would somewhat more than meet the increased expense. It seems to be generally agreed that such a plan is wholly inadvisable and that the dues must not be increased again for any purpose.

(4) Add 640 new members. Even with the four journals it would cost only about $5 to print an extra copy of each, pay the postage, and pay the additional expenses for the secretary, local sections, etc. Hence each new member adds $5 to the funds available for publication.

(5) Secure an endowment for publication. Every one will admit, I think, that the maintenance of our publications is of very great importance for the future of chemistry in America and it does not seem too much to hope that some of the leaders of industries which have profited so largely by our science may yet contribute liberally to their support. What has been accomplished during the past two years is certainly a sufficient basis for an earnest appeal.

It seems to be clear from what has been said that we ought to wait for the publication of a journal of physical chemistry till it is possible, financially, to do this and send the journal to all of our members. For the first time in the history of chemistry the American Chemical Society has succeeded in uniting all classes of chemists in a large organization which cares adequately for the interests of both pure and applied science. Those of us who attend the general meetings of the society see very clearly the advantages which come from the association and acquaintance of chemists with radically different habits of work and points of view. But we must remember that, after all, these meetings reach only a small fraction of our membership, while the journals now go to all of our members. We have recently provided, by the establishment of this Journal, a more suitable form of publication for articles in industrial and engineering chemistry. I am sure that industrial chemists would be the last to say that we ought not to continue to provide, as in the past, for the suitable publication of researches which have been undertaken without reference to any industrial application.

W. A. Noyes.

SAMPLING.

Mr. Bailey has done a service to analytical chemistry by his careful physical and mathematical investigation of sampling, the results of which are published in this number. While the work concerns itself with the sampling of coal, the results are of wide application. In the past, and at the present time, in thousands of cases the analysis has to bear the criticism which should fall upon improper sampling. The analyst, who is not his own sampler, can analyze only the sample which is delivered to him. Almost any analyst can relate how samples representing carload lots of coal were delivered to him in pint milk bottles and lubricating greases in candy sacks. Members of the engineering profession are not altogether blameless in encouraging faulty sampling, and occasionally the chemist himself is deserving of censure in this regard. The simple truth that a correct analysis is dependent upon a correct sample, would appear to be sufficiently axiomatic to require no demonstration.

W. D. Richardson.

ORIGINAL PAPERS.

THE SYNTHESIS, CONSTITUTION, AND USES OF BAKELITE.[1]

By L. H. Baekeland, Sc.D.

Received February 8, 1909.

Since many years it is known that formaldehyde may react upon phenolic bodies. That this re-

---

[1] Read before the N. Y. Section of the American Chemical Society on February 5, 1909

action is not so very simple is shown by the fact, that according to conditions of operating or to modified quantities of reacting materials, very different results may be obtained; so that bodies very unlike in chemical and physical properties may be produced by starting from the same raw materials. Some of these so-called condensation products are soluble in water, other ones are crystalline, while some others are amorphous and resinlike. Then again, among the latter resinous products some are easily fusible and soluble in alcohol or similar solvents while other ones are totally insoluble in all solvents and infusible. This paper will deal with a product of the latter class.

The complexity of my subject compels me to make a brief historical outline which will allow us to form a clearer idea of the scope of my work and differentiate it from prior or contemporary attempts in subjects somewhat similar.

That phenols and aldehydes react upon each other was shown as far back as 1872 by Ad. Bayer and others.[1]

The substances obtained by these investigators were merely of theoretical interests and no attempt was made to utilize them commercially; furthermore their method of preparation was too expensive and too uncertain and the properties of some of their resinous products were too undecided to suggest the possibility of utilizing them for technical purposes.

Until 1891 attempts at synthesis with formaldehyde were generally limited to the use of its chemical representatives, either methylal, methylen acetate, or methylen-haloid-compounds.

With the advent of cheap commercial formaldehyde, Kleeberg[2] took up again this subject using formaldehyde solution in conjunction with phenol and in presence of strong HCl. Under spontaneous heating he obtained a sticky paste which soon becomes a hard irregular mass. The latter is infusible and insoluble in all solvents and resists most chemical agents; boiling with alkalies, acids or solvents will merely extract small amounts of apparent impurities.

As Kleeberg could not crystallize this mass, nor purify it to constant composition, nor in fact do anything with it after it was once produced, he described his product in a few lines, dismissed the subject and made himself happy with the study of nicely crystalline substances as are obtained by the action of formaldehyde and polyphenols, gallic acid, etc.

The mass obtained after Kleeberg's method, is a hard and irregular porous substance containing free acid which can only be removed with difficulty after grinding and boiling with water or alkaline solutions. The porosity of the mass is due, as we shall see later, to the evolution of gaseous products during the process of heating.

In 1899 Smith,[1] realizing probably that Kleeberg's method does not lend itself to molding homogeneous articles, tried to moderate the violent reaction by using a solvent like methyl-alcohol or amyl-alcohol in which he dissolves the reacting bodies, as well as the condensing agent, muriatic acid. Even then the reaction is too violent if formaldehyde be used, so he does not use formaldehyde, but instead he takes expensive acetaldehyde and paraldehyde, or expensive polymers of formaldehyde. After the reaction, he slowly evaporates the mixtures and drives off the solvent at 100° C. He thus obtains, by and by, a hardened mass in sheets or slabs which can be sawed, cut or polished. In his German patent specification[2] he insists on the fact that in his process the methyl- or amyl-alcohol not only act as solvents but participate in the reaction and he states that this is clearly shown by the color of the final product, which is dependent on the nature of the solvent he employs. He mentions that his drying requires from 12–30 hours; my own experience is that it takes several days to expel enough of the solvent; and even after several months, there is still a very decided smell of slowly liberated solvent. During the act of drying I observed in every instance warping and irregular shrinking of the mass which thereby becomes deformed and makes this method unfit for accurate molding.

In 1902 Luft,[3] tried to overcome these difficulties in a somewhat similar way. Like Kleeberg he uses a mixture of formaldehyde, phenol and an acid; but recognizing the imperfections of the product and desiring to make of it a plastic that can be molded, he mixes the mass before hardening, with suitable solvents such as glycerine, alcohol or camphor. He virtually does the same thing as Smith with the difference, however, that he adds his solvents *after* the main reaction is partially over and uses his acid condensing agent in aqueous solution. His aim, as clearly expressed in his

---

[1] *Ber.*, **5**, 1095; **19**, 3004 and 3009; **25**, 3477; **27**, 2411.

[2] Annalen, **263**, 283 (1891).

[1] Engl. Pat., Arthur Smith, 16247, August 9, 1899.

[2] D. R. P. A. Smith, 112685, October 10, 1899.

[3] D. R. P. Adolf Luft, 140552, April 29, 1902; U. S. P. 735278.

patent specifications, is to obtain a mass which remains "transparent and more or less plastic." After pouring his mixture in a suitable mold he dries at a temperature of about 50° C. He too insists on the advantages of using solvents and in his German patent (page 1, line 44) he states that from 2 to 10 per cent. glycerine must remain in the mass; moreover he arranges matters so as to retain in his mixture all the expensive camphor. The whole process of Luft looks clearly like an attempt to make a plastic similar to celluloid and to prepare it and to use it as the latter. The similarity becomes greater by the use of camphor and the same solvents as in the celluloid process.

I have prepared Luft's product; it is relatively brittle, very much less tough and flexible than celluloid; it does not melt if heated although it softens decidedly; acetone swells it and suitable solvents can extract free camphor and glycerine from it.

And now we come to an attempt of another kind, namely the formation of soluble synthetic resins, better known as shellac substitutes.

Blumer[1] boils a mixture of formaldehyde, phenols and an oxyacid, preferably tartaric acid and obtains a fusible, alcohol-soluble, resinous material, which he proposes as a shellac substitute. This substance is soluble in caustic soda lye; it can be melted repeatedly, and behaves like any soluble fusible natural resin. Blumer in his original English patent application puts great stress on the use of an oxyacid and seems to think that the latter participates prominently in the reaction; he uses it in the proportion of one molecule of acid for two molecules of phenol and two molecules of formaldehyde.

Nathaniel Thurlow, working in my laboratory on the same subject, has conclusively shown several years ago that the identical material can be obtained by the use of minute amounts of inorganic acids; he has shown furthermore that equimolecular proportions are not necessary; in fact they are wrong and harmful if the reaction be carried on in such a way that no formaldehyde be lost; he showed also that in order to obtain a fusible soluble resin, an excess of phenol over equimolecular proportions must be used, unless some formaldehyde be lost in the reaction.

So as to avoid confusion, I ought to mention here that Blumer and Thurlow's resin is relatively very brittle, more so than shellac and that no amount of heating alone changes it into an insoluble, infusible product.

As to the real chemical constitution of this interesting product which I have tried to establish by indirect synthesis, I shall read a paper on this subject at one of the next meetings of this society.

About a year later, Fayolle[1] tries to make gutta-percha substitutes by modifying Luft's method: he adds large amounts of glycerine to the sulphuric acid used as condensing agent, and obtains a mass that remains plastic and can be softened and kneaded whenever heat is applied. On trial, this method gave me a brittle unsatisfactory substance of which it is difficult, if not impossible, to wash away the free acid without removing at the same time much of the glycerine. In this relation, Luft's way of adding the glycerine after eliminating the acid, seems more logical.[2]

Later,[3] the same inventor modified his method by adding a considerable amount of pitch ("brai") and oil thus trying to make another gutta-percha substitute which also softens when heated and remains plastic.

In 1905 Story[4] modifies all above methods in the following way: He discontinues the use of condensing agents and of added solvents; but he takes a decided excess of phenol, namely 3 parts of 40 per cent. formaldehyde and 5 parts of 95 per cent. cresol or carbolic acid; by this fact the latter is present in excess of equimolecular proportions. He boils this mixture for 8–10 hours, then concentrates in an open vessel which drives off water and some formaldehyde, and which increases still more the excess of phenol; after the mixture has become viscous he pours it into suitable molds, cools down and afterwards hardens by slow *drying below 100° C.*, or as stated in his patent, at about 80° C. His product is infusible and insoluble. But this method has some very serious drawbacks which I shall describe summarily and which Story himself recognized later.[5]

His process is necessarily slow. Leaving out of consideration his long preliminary boiling, the hardening process at temperatures *below 100° C.* is really a *drying* process where the excess of phenol that provisionally has acted as a solvent is slowly expelled. This assertion I have been able to verify beyond doubt by my direct experiments

---

[1] Engl. Pat., Louis Blumer, June 5, 1902, No. 12880.

[1] French Pat., E. H. Fayolle, 335584, September 26, 1903.

[2] See also addition patents to original French Pat. add. Pat. 2414, February 8, 1904 and 2485, February 18, 1904. Fayolle.

[3] French Pat., E. H. Fayolle, 341013, March 7, 1904.

[4] Engl. Pat., Henry Story, 8875, 1905.

[5] See his addition Patent, Belgium 210965, September 30, 1908.

where hardening was conducted in closed vessels at below 100° C. and where I succeeded in collecting phenol with the eliminated water. The evaporation or drying process may proceed acceptably fast for thin layers, or thin plates, but for masses of a somewhat larger volume, it requires weeks and months; even then the maximum possible hardness or strength is not reached at such low temperatures. All this not merely involves much loss of time, but the long use of expensive molds, a very considerable item in manufacturing methods; furthermore, during the act of drying, the evaporation occurs quickest from the exposed surface, thus causing irregular contraction and intense stresses, the final result being misshapen molded objects, rents or cracks.

Story states that if pure phenol be used the reaction proceeds very slowly; I should add that in that case the reaction does not take place, except very imperfectly, even after several days of continuous boiling. Even then in some of my own experiments made with pure commercial crystallized phenol and with commercial 40 per cent. formaldehyde, I obtained products not of the insoluble type, but similar to the soluble fusible products of Blumer and Thurlow.

Taken in a broad sense, Story's process is very similar to Luft's with this difference however, that he foregoes the use of an acid condensing agent and instead of using a solvent like alcohol, glycerine or camphor, he uses a better and cheaper one, namely an excess of phenol. In further similarity with Luft and Smith's his method is, as he expresses himself in his patent text, a *drying* process.

Like Smith and Luft he is very careful to specify temperatures *not* exceeding 100° C. for drying off his solvent.

Shortly after Story filed his patent, DeLaire[1] obtained a French patent for making soluble and fusible resins either by condensing phenols and formaldehyde in presence of acids, in about the same way as Blumer or Thurlow and then melting this product; or by dissolving phenol in caustic alkalies *used in molecular proportions*, then precipitating the aqueous solution with an acid and afterwards resinifying the reprecipitated product by heating it until it melts. I should remind you that the French patent laws allow patents without any examination whatever as to novelty. And I should state also that DeLaire simply uses here the old and well-known processes of Lederer[1] and Manasse[2] which consists in making a phenol-alcohol by the action of formaldehyde on an aqueous solution of a phenolate and subsequent treatment with an acid.

It is a well-known fact that these phenol-alcohols, for instance saligenin, if heated alone or with an acid, will give partial anhydrides such as saliretin and homosaliretin,[3] $C_{14}H_{14}O_3$ or $C_6H_4(OH)CH_2OC_6H_4CH_2OH$,[4] fusible and soluble in alcohol, or caustic soda, and precipitable from the latter by the addition of chloride of sodium.

Trisaligenosaligenin, $C_{28}H_{26}O_5$ or $4C_7H_8O_2—3H_2O$,[5] and heptasaligenosaligenin, $C_{56}H_{50}O_9$ or $8C_7H_8O_2—7H_2O$,[6] are both higher anhydrides of similar resinous character, the first one obtained by the action of sulphuric acid on saligenin, the latter by the action of acetic anhydride.

The direct homolog of saliretin, which is methylsaliretin or homosaliretin, has properties similar to saliretin, melts at 200° or 205° C. and is less soluble.[7]

No wonder then if the English[8] and the German patents[9] of DeLaire vary considerably in text and claims from the French patent; the claims are reduced merely to a method consisting in resinifying phenol-alcohols by heating them under reduced pressure or vacuum. The resins of DeLaire are fusible, soluble products, having all the general properties of saliretin and homosaliretin.

In order to complete my enumeration of methods where alkalies are used, I ought to mention two processes which aim at products very different from those which we have in view. Speyer[10] produces an antiseptic which easily gives off CH_2O. For this purpose he uses naphthol or polyphenols like resorcin or pyrogallol and adds an excess of ammonia and of formaldehyde. This gives him an insoluble powder which easily liberates CH_2O and NH_3. It is a well-known fact that ammonia reacts on formaldehyde and produces hexamethylentetra-

---

[1] French Pat., DeLaire 361539, June 8, 1905.

[1] *Journal Praktische Chemie* [2], vol. 50, page 224.

[2] *Ber.*, 1894, 2409–2411; D. R. P. Bayer, 85588; U. S. P., Manasse, 526786, 1894.

[3] Beilstein, *Organ. Chemie*, Vol. 2, 1896, page 1109.

[4] R. Piria, *Ann. Chem.*, **48**, 75; **56**, 37; **81**, 245; **96**, 357. Moitessier, Jahresbericht, 1886, page 676.

[5] K. Kraut, *Ann. Chem.*, **156**, 123; Gerhardt, *Ann. Chim. Phys.* [3], 7, page 215.

[6] F. Beilstein and F. Seelheim, *Ann. Chem.*, 117, page 83.

[7] C. Schotten, *Berichte*, 1878, page 784.

[8] Engl. Pat., DeLaire, 15517, 1905.

[9] D. R. P., DeLaire, 189262, July 19, 1905.

[10] D. R. P., Arthur Speyer, 99570, November 2, 1897.

min, $C_6H_{12}N_4$,[1] which easily acts upon acids and forms again CH_2O, NH_3 and methylamin.[2]

Two recent patents relate directly to the manufacture of soluble fusible resins. One of Farbenfabriken Fried. Bayer & Co.[3] uses orthocresol so as to obtain an odorless shellac substitute.

The other issued to Grognot[4] also for a shellac substitute, adds glycerine first, then after the reaction is over distils the solvent off.

After I had filed my own patent claims in the U. S. which gave me International Convention privileges, Helm[5] described, after me, amines or ammonium salts as condensing agents for the manufacture of synthetic resins with phenols and formaldehyde. He does not clearly indicate the chemical or physical properties of his resins. He furthermore makes the rather ambiguous statement that ammonium nitrate can be used as well as aniline. I have shown (see below) that in the case of ammonium nitrate the end-product may be a fusible soluble resin, while in the case where aniline is used I obtain finally an insoluble infusible resin.

It is true that Helm uses large amounts of aniline and nitrate of ammonium; his indicated proportions are very close to molecular proportions and this undoubtedly has, as I will show later, a great influence on the nature of the resulting products.

Knoll,[6] who also applied for patents after the filing date of my U. S. patents, uses sodium sulphite or *neutral*, or *acid* or *alkaline* salts as condensing agents; disregarding again the fact established by me, that according to whether an acid, a base, an acid salt, or an alkaline salt be used, the resinous products may be totally different.

This will close my review of the work done by others and I shall begin the description of my own work by outlining certain facts, most of which seem to be unknown to others, or if they were known their importance seems to have escaped attention. Of these facts I have made the foundation of my technical processes.

As stated before, the condensation of phenols with formaldehyde can be made to give, according to conditions and proportions, two entirely different classes of resinous products. *The first class* includes the products of the type of Blumer, DeLaire, Thurlow, etc. These products are soluble in alcohol acetone or similar solvents, and in alkaline hydroxides. Heating, simply melts them and they resolidify after cooling. Melting and cooling can be repeated indefinitely but further heating will not transform them into products of the second class. They are generally called "*shellac substitutes*," because they have some of the general physical properties of shellac.

The second class includes the products of Kleeberg, Smith, Luft, Story, Knoll as well as my own product, in so far only as their general properties are concerned; but each one of them may be characterized by very distinct specific properties which have a considerable bearing on any technical applications. Broadly speaking, this second class can be described as infusible resinous substances, derived from phenols with aldehydes; some of them are more or less attacked by acetone, by caustic alkalies or undergo softening by application of heat. *At least one of them* is unattacked by acetone and does not soften even if heated at relatively high temperatures. None of them can be re-transformed into products of the first class even if heated with phenol.

These insoluble infusible substances can be produced directly in one operation by the action of formaldehyde on phenols under suitable conditions, for instance the process of Kleeberg (see above). Or they may be produced in two phases (see Luft and Story above), the first phase consisting of an incomplete reaction giving a viscous product that is soluble in alcohols, glycerine, camphor or phenol, and which on further heating or after driving off the solvent may gradually change into an infusible product.

In order to be able to stop at the first phase, the condensing agents may either be omitted (see Story above) or they may be used moderately (see Luft above) or they may be diluted with suitable solvents, for instance methyl- and amyl-alcohol (see Smith above) or with glycerine (see Fayolle above).

In all these processes there is a further treatment by which the solvent is driven off during a *drying* process. For example, in the process of Smith or Luft, alcohol or glycerine is thus expelled partially; and in Story's process the excess of phenol is driven off in the same way by slow

[1] Wohl, *Ber.*, **19**, 1892; Tollens, *Ber.*, **17**, 653.

[2] See also Moschatos and Tollens, *Ann. der Chemie.*, **272**, 280.

[3] D. R. P., Farbenfabriken vorm. Friedr., Bayer & Co., 201,261, April 16, 1907.

[4] L. Grognot, U. S. P. 391436, 1908.

[5] Engl. Pat., Louis Helm, 25216, November 13, 1907.

[6] Eng. Pat., Knoll & Co., 28009, December 19, 1907; Swiss Pat., Ditto, 40994, December 7, 1907.

drying under 100° C. In all these drying processes, some of the solvent is left, either purposely, so as to insure flexibility or plasticity, or it is left involuntarily, because at temperatures of 100° C. or below it is impossible to expel the totality of these solvents, part of which are stubbornly retained by the mass.

If I except the processes of Blumer, Thurlow or DeLaire, and generally those which have in view fusible and soluble resins, in all above-mentioned patent specifications temperatures *of 100° C. or much below* are insisted upon.

And yet I have convinced myself by often repeated experiments that temperatures *above 100° C.* and *considerably above 100° C.* are best suited or indispensable for the complete and rapid transformation into a final insoluble, infusible product of exceptionally desirable qualities. If this be so why have my predecessors not used temperatures above 100° C.?

Why do some of them recommend temperatures as low as 80° C. (Story) and even 50° C. (Luft)? Why do they prefer to make this final hardening a long and slow operation which does not give the best, the hardest, the most resisting product? (See confirmation of my statement by Story in Belgian addition patent 210965, September 30, 1908.)

For the simple reason that if their initial mass be heated at too high a temperature it gives off gaseous products, mainly composed of formaldehyde; this produces bubbles in the mass, makes it spongy, porous, and unfit for commercial use.

More direct experiments have proved to me that during the first stages of the process, we have to deal with a phenomenon that has all the characteristics of chemical dissociation with liberation of CH_2O.

If the initial mass be heated at temperatures above 100° C. the tension of this gas becomes very pronounced. At 100° C. the tension may become as high as 1 kilogram per square centimeter (above atmospheric pressure), but this tension subsides as soon as the final product is formed.

I shall explain later how I have utilized this knowledge to good advantage, and how I counteract this dissociation simply by exercising a compensating external pressure.

In the historical part of my paper, reference has repeatedly been made to the use of condensing agents. We have seen how Kleeberg, Smith, Luft, Fayolle, Blumer and Thurlow use acid condensing agents. Others like Speyer, Hentsche, Lederer, Manasse, DeLaire, use alkalies, but every time in relatively large proportions: practically one molecule or over; but the products thus obtained are of a nature very different from the substance I am about to describe.

Story, on the other hand, adds no condensing agents whatever. True, he is able, with commercial impure carbolic acid, to obtain a reaction after about 8–10 hours' boiling, and this heating process has then to be supplemented by much longer drying. But if his process be carried out with pure or crystallized phenol, it takes many days of continuous boiling before a reaction sets in; even then the product obtained is of a dubious character hovering between a resin of class one (fusible and soluble) and a resin of class two (infusible and insoluble). It is more likely to be a fusible and soluble resin if for some reason or another the process has been carried out with an excess of phenol or if, some way or another, too much CH_2O has escaped in the after concentration. For instance by following his description and boiling for 5 days in a return condenser a mixture of 50 gr. pure crystalline phenol and 30 gr. 40 per cent. commercial formaldehyde, then concentrating in an open dish, I obtained the fusible soluble resin of Blumer or Thurlow which on further heating remains fusible and does not change into the insoluble infusible product as described by Story. I obtain the same result if the boiling be carried on in presence of a small amount of any acid, any acid salt, or any salt which on hydrolyzing may split, so as to give a preponderant acid reaction. This effect is shown by salts of mineral acids and heavy metals; it is shown even with ammonium chloride, and on acting upon formaldehyde liberates free hydrochloric acid.[1]

On the other hand, if I use an alkaline salt or a salt, which on hydrolyzing splits into a weak acid and a strong base, as for instance sodium acetate, I obtain under the same circumstances a resin of the insoluble, infusible variety even if to some extent, a slight excess of phenol has been used, showing conclusively that within certain limits the amount of phenol does not change the general character of the reaction. All what may happen in that case is that the final product is rendered impure by some excess of phenol which can be

[1] See Cambier, Brochet, *Compt. rend.*, **120**, 557.

driven off afterwards by a drying process similar to that of Story.

I have obtained similar results with many other alkaline salts as for instance ammonium carbonate, sodium carbonate, potassium carbonate, sodium bicarbonate, potassium bicarbonate, trisodium phosphate, borax, potassium cyanide, sodium silicate, soap, etc.

In the same way I have used sodium sulphite, which on acting on CH_2O liberates sodium hydroxide according to following reaction:

$$CH_2O + SO_3Na_2 + H_2O = CH_2(OH)SO_3Na + NaOH.$$

I might say that a similar effect is obtained from all substances which can act directly or indirectly as bases.

In other terms: *the quality as well as the quantity* of condensing agent has an enormous influence on the nature of the final products.

For the manufacture of insoluble, infusible, condensation products of formaldehyde and phenol, bases used in moderate amounts have very decided advantages. They accelerate the reaction without degenerating same into a violent and irregular process. The relatively small amount of base which may remain present in the finished product, either in combined or uncombined form, does not involve the same objectionable features for its technical uses as the presence of free acid.

Furthermore, for some reason or another acid condensing agents seem to favor the formation of soluble and fusible resins, while for some other reason, bases seem to favor the formation of insoluble, infusible resins.

Moreover, by the use of small amounts of bases, I have succeeded in preparing a *solid* initial condensation product, the properties of which simplify enormously all molding operations as we shall see later.

I have tried all organic or inorganic bases which I could obtain readily. I have tried the hydroxides and carbonates of the alkali metals, the hydrates of alkaline earths, ammonia and its alkaline salts, hydroxylamin, organic amines, pyridine, carbamide and other amides of weak acids; and the effect, with slight variations, is always about the same; it is quite natural, that for reasons of economy or expediency, I should prefer the commercially more available bases.

I wish it distinctly understood that in order to obtain my technical effect I use the bases in relatively small quantities, say less than one-fifth of the amount which would be required to transform the phenol into phenolate.

If larger amounts of base be used, the results are technically much inferior; in fact the process changes gradually into such as give phenol-alcohols or compound condensation products of ammonia or amines with formaldehyde, all products very different from those I desire to make.

I have good reason to believe that in my process the bases only act as catalyzers and intervene only temporarily in the reaction. They seem to be expelled in free condition during the last stage of the process. For instance if I use ammonia, I find this ammonia back in the free state in the final hard condensation product.

A careful study of the condensation process of phenols and formaldehyde, made me discover that this reaction instead of occurring in two stages can be carried out in three distinct phases. This fact is much more important than it appears at first sight. Indeed it has allowed me to prepare a so-called *intermediate condensation product*, the properties of which simplify still further my methods of molding and enlarge very much the scope of useful applications of my process.

The three phases of reaction can be described as follows:

First phase. The formation of a so-called *initial condensation product* which I designate as *A*.

Second phase. The formation of a so-called *intermediate condensation product*, which I designate as *B*.

Third phase. The formation of a *final condensation product*, which I designate as *C*.

As to the properties of each of these condensation products I can define them in a few words:

A, at ordinary temperatures, may be liquid, or viscous, or pasty, or solid. Is soluble in alcohol, acetone, phenol, glycerine and similar solvents; is soluble in NaOH. *Solid A* is very brittle and melts if heated. All varieties of *A* heated long enough under suitable conditions will change first into *B* then finally into *C*.

B is solid at all temperatures. Brittle but slightly harder than solid *A* at ordinary temperatures: insoluble in all solvents but may swell in acetone, phenol or terpineol without entering into complete solution. If heated, does not melt but softens decidedly and becomes elastic and somewhat rubber-like, but on cooling becomes again hard and brittle. Further heating under suitable conditions changes it into *C*. Although *B* is infusible it can be molded under pressure in a hot mold to a homogeneous, coherent mass, and the latter can be further changed into *C* by the proper application of heat.

C is infusible, insoluble in all solvents; unattacked by acetone, indifferent to ordinary acids, or alkaline solutions; is destroyed by boiling concentrated sulphuric acid, but stands boiling with diluted sulphuric acid; does not soften to any serious extent if heated, stands temperatures of 300° C.; at much higher temperatures begins to be destroyed and chars without entering into fusion. It is a bad conductor of heat and electricity.

The preparation of these condensation products *A* and *B* and their ultimate transformation in *C* for technical purposes constitute the so-called Bakelite process. This can be described easily:

I take about equal amounts of phenol and formaldehyde and I add a small amount of an alkaline condensing agent to it. If necessary I heat. The mixture separates in two layers, a supernatant aqueous solution and a lower liquid which is the initial condensation product. I obtain thus at will, either a thin liquid called *Thin A* or a more viscous mass, *Viscous A* or a *Pasty A*, or even if the reaction be carried far enough, a *Solid A*.

Either one of these four substances are my starting materials and I will show you now how they can be used for my purposes.

If I pour some of this *A* into a receptacle and simply heat it above 100° C., without any precaution, I obtain a porous spongy mass of *C*. But bearing in mind what I said previously about dissociation, I learned to avoid this, simply by opposing an external pressure so as to counteract the tension of dissociation. With this purpose in view, I carry out my heating under suitably raised pressure, and the result is totally different.

This may be accomplished in several ways but is done ordinarily in an apparatus called a Bakelizer. Such an apparatus consists mainly of an interior chamber in which air can be pumped so as to bring its pressure to 50 or better 100 lbs. per square inch. This chamber can be heated externally or internally by means of a steam jacket or steam coils to temperatures as high as 160° C. or considerably higher, so that the heated object during the process of Bakelizing may remain steadily under suitable pressure which will avoid porosity or blistering of the mass.

For instance if I pour liquid *A* into a test tube and if I heat in a Bakelizer at say 160–180° C., the liquid will change rapidly into a solid mass of *C* that will take exactly the shape of its container; under special conditions it may affect the form of a transparent hard stick of Bakelite. It is perfectly insoluble, infusible, and unaffected by almost all chemicals, an excellent insulator for heat and electricity and has a specific gravity of about 1.25.

It is very hard, cannot be scratched with the finger nail; in this respect it is far superior to shellac and even to hard rubber. It misses one great quality of hard rubber and celluloid, it is not so elastic nor flexible. Lack of flexibility is the most serious drawback of Bakelite. As an insulator, and for any purposes where it has to resist heat, friction, dampness, steam or chemicals it is far superior to hard rubber, casein, celluloid, shellac and in fact all plastics. In price also it can splendidly compete with all these.

Instead of pouring liquid *A* into a glass tube or mold I may simply dip an object into it or coat it by means of a brush. If I take a piece of wood, and afterwards put it into a Bakelizer for an hour or so, I am able to provide it rapidly with a hard brilliant coat of Bakelite, superior to any varnish and even better than the most expensive Japanese lacquer. A piece of wood thus treated can be boiled in water for hours without impairing its gloss in the slightest way. I can dip it in alcohol or other solvents, or in chemical solutions and yet not mar the beautiful brilliant finish of its surface. But I can do better, I may prepare an *A*, much more liquid than this one, and which has great penetrating power, and I may soak cheap, porous soft wood in it, until the fibres have absorbed as much liquid as possible, then transfer the impregnated wood to the Bakelizer and let the synthesis take place in and around the fibres of the wood. The result is a very hard wood, as hard as mahogany or ebony of which the tensile- and more specially the crushing strength, has been considerably increased and which can stand dilute acids or water or steam; henceforth it is proof against dry rot. I might go further and spend a full evening on this subject alone and tell you how we are now bringing about some unexpected possibilities in the manufacture of furniture and the wood-working industry in general. But I intend to devote a special evening to this subject and show you then how with cheap soft wood we are able to accomplish results which never have been obtained even with the most expensive hard wood.

In the same way I have succeeded in impregnating cheap ordinary cardboard or pulp board and changing it into a hard resisting polished material that can be carved, turned and brought into many shapes. I might take up much more of your time by simply enumerating to you the applications of this impregnation method, with wood, paper, pulp, asbestos, and other fibrous and cellular materials; how it can be applied for fastening the bristles of shaving brushes, paint brushes, tooth brushes, how it can be used to coat metallic surfaces with a hard resisting protecting material; how it may ultimately supplant tin in canning processes; but I have no doubt that your imagination will easily supply you a list of possible technical uses even if I defer this subject for some other occasion.

As to Bakelite itself, you will readily understand that it makes a substance far superior to amber for pipe stems and similar articles. It is not so flexible as celluloid, but it is more durable, stands heat, does not smell, does not catch fire and at the same time is less expensive.

It makes excellent billiard balls of which the elasticity is very close to that of ivory, in short it can be used for similar purposes like knobs, buttons, knife handles, for which plastics are generally used. But its use for such fancy articles has not much appealed to my efforts as long as there are so many more important applications for engineering purposes.

Bakelite also acts as an excellent binder for all inert filling materials. This makes, that it can be compounded with sawdust, wood pulp, asbestos, coloring materials, in fact with almost anything the use of which is warranted for special purposes. I cannot better illustrate this than by telling you that here you have before you a grindstone made of Bakelite and on the other hand a self-lubricating bearing which has been run dry for nine hours at 1800 rev. per minute without objectionable heating and without injuring the quickly revolving shaft.

If I mix Bakelite with fine sand or slate dust I can make a paste of it which can be applied like a dough to the inside of metallic pipes or containers, or pumps, and after Bakelizing, this gives an acid proof lining very useful in chemical engineering.

Valve seats, which are unaffected by steam, steam-packing that resists steam and chemicals, have been produced in a similar way.

Phonograph records have been made with it, and the fact that Bakelite is harder than rubber, shellac, or kindred substances indicates advantageous possibilities in that direction.

For the electrical industry, Bakelite has already begun to do some useful work. There too its possible applications are numerous. Armatures or fields of dynamos and motors, instead of being varnished with ordinary resinous varnishes, can simply be impregnated with *A*, then put into a Bakelizer and everything transformed into a solid infusible insulating mass; ultimately this may enable us to increase the overload in motors and dynamos by eliminating the possibility of the melting or softening of such insulating varnishes as have been used until now. But the subject of dynamos and motor construction is only at its very modest beginnings and I prefer to mention to you what has been already achieved in the line of molded insulators of which you will find here several very interesting samples.

This brings me to the subject of molding Bakelite.

For all plastics like rubber, celluloid, resins, etc., the molding problem is a very important one. Several substances which otherwise might be very valuable are useless now because they cannot economically be molded. The great success of celluloid has mainly been due to the fact that it can easily be molded. Nitrated cellulose alone, is far superior in chemical qualities to celluloid, but until Hyatts' discovery, it could only be given a shape by an evaporation process and its applications were very limited. The addition of camphor and a small amount of solvent to cellulose nitrate was a master-stroke, because it allowed quick and economic molding.

In the same way white sand or silica would be an ideal substance for a good many purposes, could it be easily compressed or molded into shape and into a homogeneous mass. But it *cannot*; and therefore remains worthless. And that is the main difference between a *plastic* and a *non-plastic*. It so happens that Bakelite in *C* condition does not mold; it does not weld together under pressure even if heated; only with much effort is it possible to shape some kind of an object out of it, but someway or another the particles do not stick well together; in other terms it is not a true plastic. Therefore the molding problem has to be solved in the anterior stages of the process. We have seen how

Smith, Luft, and Story tried to solve a similar problem by the admixture of solvents and subsequent evaporation, but we know now that these very solvents imply most serious drawbacks.

I have already shown you how I am able to mold and harden quickly by pouring liquid *A* into a mold and heating it in a Bakelizer. But even that method is much too slow for most purposes. Furthermore, molds cost money; any rubber or celluloid manufacturer will tell you that the item of molds represents a big portion of the cost of his plant. If an order for 10,000 pieces has to be delivered and it takes an hour for molding, it will require between three and four years to fill this order with one mold and if the mold costs $100 it will require $5000 for molds alone if the order has to be finished within 20 days. For that very reason I have devised my molding methods so as to use the molds only during the very minimum of time. I have succeeded in doing so in several ways. One of the simplest ways is the following:

As stated before, the use of bases permits me to make a variety of *A* that is solid although still fusible. The latter is as brittle as ordinary rosin and can be pulverized and mixed with suitable filling materials. A mixture of the kind is introduced in a mold and put in the hydraulic press, the mold being heated at temperatures preferably about or above 160–200° C. The *A* melts and mixes with the filler, impregnating everything; at the same time it is rapidly transformed into *B*. But I have told you that *B* does not melt, so the molded object can be expelled out of the mold after a very short time and the mold can again be refilled. All the molded articles are now in *B* condition; relatively brittle but infusible. At the end of the day's work or at any other convenient time all the molded articles are put in the Bakelizer and this of course without the use of any molds; in this way they are finally transformed in "*C*" Bakelite of maximum strength and hardness and resisting power.

The process can still be further simplified. Instead of using *A*, we can use *B* and mold it in the hot press where it welds and shapes itself. After a very short time, the *B* begins to transform into *C* and can now be expelled from the mold. If the transformation in *C* is not complete, a short after-treatment in the Bakelizer will finish everything. I have succeeded thus in reducing the molding to less than two minutes for small objects.

The valuable properties of *B* may be used in many other ways; for instance *A* may be poured into a large container and be heated slowly at 70° C. until it sets to a rubber-like mass and shows that it is transformed into *B*. This block of *B* if warm has very much the consistency of printers' roller-composition, but is brittle when cold. The warm flexible mass can now be removed from its container or, divided, cut, or sawed to any desired shape and the so-shaped articles can be simply placed in a Bakelizer; no melting nor deformation can occur, so we need no mold while maximum heat is applied to bring everything in condition *C*.

I could multiply these examples by numerous other modifications of my process but I believe that what I have said will be enough to convince you of its many uses; we are studying now applications of Bakelite in more than forty different industries on some of which I shall report on some future occasion.

The chemical constitution of Bakelite and the nature of the reactions which occur in the Bakelite process are problems which I have endeavored to solve. This subject is not by any means an easy one. Indeed, we have to deal here with a product that cannot be purified by crystallization nor other ordinary methods, which is insoluble, does not melt nor volatilize; in other terms, it is not a product which is amenable to our usual methods of molecular weight determination. Its chemical inertness makes it unfit for studying possible chemical transformations and unless my friends, the physico-chemists, will come to my aid, discover some way for establishing some optical properties or other physical constants, we are very much at a loss to establish the molecular size of my product.

But I have been so fortunate as to be able to obtain some insight into its chemical constitution by a rather round-about way: Indeed, I have succeeded in making Bakelite by indirect synthesis.

As stated previously, oxybenzylalcohol if heated at 150° C., or in presence of acids, gives various partial anhydrides, called saliretin , which may resinify further if heated at higher temperatures. Saliretin products are more or less soluble in alcohol and acetone and in NaOH solution, from which they may be reprecipitated by means of NaCl.

We have already seen that DeLaire in heating phenol alcohols in vacuum obtains soluble resins. But I have heated saligenin in sealed tubes under pressure at 180° C. for 8 hours, with and without the addition of small amounts of ammonia. In both cases I obtain a substance which is hard when cold, but which softens when heated, but does not melt. It swells in acetone and in NaOH and dissolves partially. This substance is not my intermediate condensation product *B*, because no amount of heating can transform it in *C*.

If, however, I heat oxybenzylalcohol in presence of enough CH_2O or its polymers in a sealed tube at 180° C. for 8 hours, I obtain a substance entirely similar to Bakelite in properties and in chemical composition.

By varying the proportions and repeating the experiment a great number of times I succeeded in establishing that, unless I use at least 1 molecule of CH_2O for 6 molecules of oxybenzylalcohol I do not obtain Bakelite but a product containing saliretin compounds.

The same result occurs by heating 6 molecules of phenol and 7 molecules of CH_O in presence of a small amount of a base.

If I use somewhat less formaldehyde or if for some reason or another all the formaldehyde does not enter into reaction, I obtain a substance which may still be attacked by acetone, probably because it contains uncombined phenol or saliretins after the reaction is over.

But I have found that all these substances, whether they are obtained by heating 6 molecules of phenol alcohol with at least one molecule of CH_2O, or whether they are obtained by the action of phenol on formaldehyde under heat and pressure in presence of small amount of bases, can be purified and brought to about constant composition as follows:

The substance is pulverized, washed with 5 per cent. KOH solution, with dilute HCl, with alcohol, with acetone and finally dried to constant weight in vacuo.

The powder so obtained still contains traces of potassium, which I did not succeed in eliminating. The amount of same is very small, about 0.09 per cent. of ash, but it seems to cling tenaciously to the product and makes it somewhat hygroscopic making weighing for analytical purposes very difficult, and accounts for some variations in the results.

The organic combustion of all these products gave the following results:

1 mole CH_2O 6 mole saligenin	C = 77.48 H = 5.96 O = 16.56	77.88 5.97 16.15
1 mole CH_2O 4 mole saligenin	C = 76.47 H = 5.44 O = 18.09	76.35 5.40 18.25
13 mole CH_2O 12 mole saligenin	C = 76.59 H = 5.97 O = 17.44	76.57 5.97 17.46
1 mole phenol 1 mole CH_2O and 1 per cent. NH_3	C = 77.48 H = 5.60 O = 16.92	76.61 5.80 17.59
10 cc. phenol 10 cc. 40 per cent. formaldehyde and ½ per cent. NH_3	C = 77.92 H = 5.71 O = 16.37	75.62 5.78 18.60[1]

If we take into consideration the great difficulties encountered in purifying methods, these results seem to indicate that we have to deal here with a definite organic substance of constant composition, which according to its methods of preparation may exist with impurities mixed in various proportions. These impurities are probably free phenol, or free CH_2O or saliretin products.

From the indirect synthesis of Bakelite by means of oxybenzylalcohol and CH_2O, I am led to consider Bakelite in its simplest form as a polymerized oxybenzyl — methylen - glycol — anhydride which, in case of ordinary phenol, might be represented by the following formula:

$$n(C_{43}H_{38}O_7).$$

The reaction being represented by:

$$n(6C_7H_8O_2 + CH_2(OH)_2) = n(C_{43}H_{38}O_7) + n(7H_2O.)$$

This formula corresponds acceptably to the analytical results if we take in consideration the difficulties of purification.

	C.	H.	O.
Calculated	= 77.44	5.75	16.81
Found (average)	= 77.68	5.96	16.36

(with product of 6 mole saligenin + 1 mole CH_2O).

I consider Bakelite *C* as a direct polymer of another anhydride which is represented by my intermediate condensation product or Bakelite *B*. Bakelite *B* is a more complete anhydride than Bakelite *A*. As to Bakelite *A*, I am unable to arrive at a constant composition, for the reason that it easily gives off water, changing gradually its composition until heating converts it slowly into *B*, after passing through various mixtures of *A* and *B*.

My supposition has a strong appearance of probability by the following experiment:

If I put a mixture of phenol and formaldehyde in proper proportions and with some small amount of a base in a sealed glass tube and heat just long enough to produce *A* the formerly homogeneous liquid mixture separates into two layers. The initial condensation product *A* forms a lower stratum, and a supernatant layer of liquid indicates the elimination of water. The same thing occurs if

---

[1] Beginning oxidation during drying.

anhydrous phenol is heated with paraform in presence of a small amount of base.

If this *A*, first properly freed from any physically retained water, be introduced into another sealed tube and heated further, I may succeed, with some precaution as to the duration of heating, in stopping just in time so as to transform everything into *B*, the intermediate condensation product. I then see that a new amount of water is set free which will assemble on top, giving evidence of further dehydration. At the same time we notice that the mass *B* has not contracted in volume to any important extent.

If now this *B*, properly freed from water, be heated in another sealed tube, it will be transformed finally into *C*. But this time we see no further elimination of water. On the other hand there is a decided contraction of volume.

This contraction of volume, together with the remarkable increase in physical and chemical inertness, points out towards the probability that *C* is simply the polymer of *B*.

I am fully supported in this belief by the fact that analysis has shown me that *B* and *C* contain the same percentage of carbon, hydrogen and oxygen.

With homologs of phenol we obtain the direct homologs of these anhydrides; for instance with orthocresol we get the polymer of ortho-methyl-oxybenzyl-methylen-glycol-anhydride.

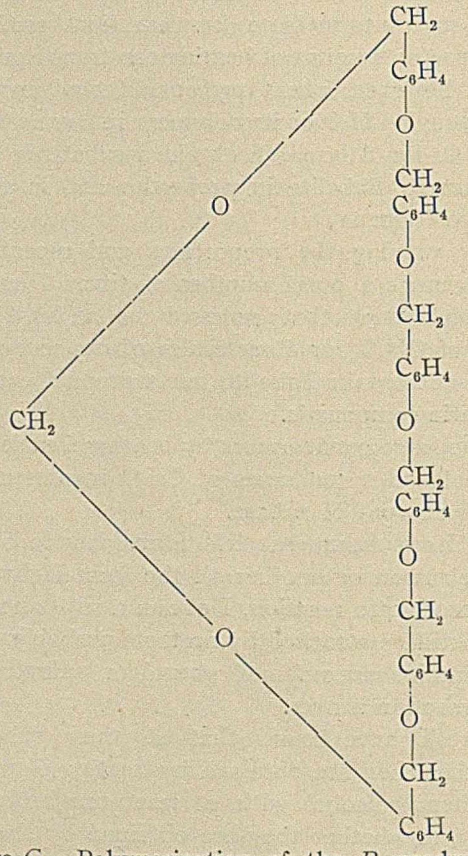
It may be of interest to remind you that many years ago, Oscar Low[1] called our attention to the great importance of formaldehyde as a starting point of synthesis in plant life. By the photochemical action of sunlight on CO_2 in presence of water in chlorophyl, oxygen is liberated and produces CH_2O. This is the beginning of a process of further synthesis building up more complicated bodies.

If we accept these premises then the theory of the Bakelite process is easy to explain:

Stage A.—Formation of a partial anhydride of a phenol alcohol and methylen glycol containing, hydroxyl groups, which can fix NaOH.

Stage B.—Formation of a higher anhydride by further elimination of water. This higher anhydride seems no longer to possess hydroxyl groups, but by addition of NaOH may still form alkaline compounds. This induces me to believe that in some way or another we shall suceed in obtaining alkali compounds of the kind which on being treated with dilute acids will regenerate *A*.

Until we have anything better, I shall propose the formula:



Stage *C*.—Polymerization of the *B* product resulting in greater chemical inertness and disappearance of active corners of the molecule.

I shall also call your attention to the fact that the willow tree produces in its cells, salicin, which is the glucoside of saligenin; this same saligenin or oxybenzyl alcohol in presence of more CH_2O has given me Bakelite.

On the other hand Bertrand,[1] and Vlchirch and Stevan[2] and more recently R. Majima and S. Cho[3] have called our attention to the phenolic nature of resinous substances, specially Japanese lacquer. The latter substance has some analogy with Bakelite and exudes from the *Rhus vernicifera Dc.* which is a plant that is somewhat related to our American "poison ivy."

---

[1] *Ber.*, **22**, 475 and **23**, 388 and 480.

[1] *Ann. Chim. Phys.* [6]. **12**, 115 (1898); *Bull. Soc. Chim.* [3]. **11**, 614 and 717 (1894).

[2] Tschirch und Stevan, *Arch. d. Pharm.*, **243**, 504 (1905).

[3] *Ber.*, **15**, November, 1907, page 4390.

So after all, the synthesis accomplished in my laboratory seems to have a decided similarity to some intricate biological processes that take place in the cells of certain plants.

In order not to increase too much the length of this paper, I have merely given you the brief outlines of years of arduous but fascinating work, in which I have been ably helped by Mr. Nathaniel Thurlow and more recently also by Dr. A. H. Gotthelf, who attended to my analytical work.

The opened field is so vast that I look forward with the pleasure of anticipation to many more years of work in the same direction.

I have preferred to forego secrecy about my work relying solely on the strength of my patents as a protection.

It will be a great pleasure to me if in doing so, I may stimulate further interest in this subject among my fellow chemists and if this may lead them to succeed in perfecting my methods or increase still further the number of useful applications of this interesting compound.

(CONTRIBUTION FROM THE ARTHUR D. LITTLE LABORATORY OF ENGINEERING CHEMISTRY.)

ACCURACY IN SAMPLING COAL.[1]

BY E. G. BAILEY.

In 1899 the American Chemical Society was presented with a "Report of the Committee on Coal Analysis;" and so far as the chemical analysis is concerned the method recommended by this committee has been very closely followed by the majority of laboratories. In many specifications for the purchase of coal on the analysis and heat unit basis, it is stated that the sample shall be analyzed in accordance with the method adopted by the American Chemical Society, while the greater number of these specifications merely mention the sampling of the coal delivered and do not state definitely how the sample shall be taken and quartered down.

One specification covering the delivery of about 200,000 tons of coal per year states: "Sampling will be done by a representative of the company and contractor may have a representative present. Sample will be taken from each delivery and kept in hermetically sealed jars." Another specification on which almost twice this amount of coal is purchased describes the sampling as follows: "A small quantity of coal will be taken from each weighing hopper just before the hopper is dumped, while the lighter is being unloaded. These quantities will be thrown into a receptacle provided for the purpose, and when the lighter is empty the contents of the receptacle will be thoroughly mixed, and a sample of this mixture will be taken for chemical analysis."

The specifications on which the United States Government purchase state: "Samples of the coal delivered will be taken by a representative of the Government.... The sample taken will in no case be less than the total of 100 pounds to be selected proportionally from the lumps and fine coal, in order that it will in every respect truly represent the quantity of coal under consideration.

"In order to minimize the loss in the original moisture content, the gross sample will be pulverized as rapidly as possible, until none of the fragments exceed one-half inch in diameter. The fine coal will then be mixed thoroughly and divided into four equal parts. Opposite quarters will be thrown out and the remaining portions thoroughly mixed and again quartered, throwing out opposite quarters as before. This process will be continued as rapidly as possible until the final sample is reduced to such amount that all of the final sample thus obtained will be contained in the shipping can or jar and sealed air-tight."

In one instance, the Government found 46.23 per cent. ash in a sample of coal delivered to a public building. The contract was based on 4.78 per cent. ash, and they obtained samples as low as 6.70 per cent., while the average for 10 samples was 11.58 per cent. ash. Referring to this instance, Mr. Woodwell, in a paper before the American Society for Testing Materials, says: "Moreover, the coal passed the visual inspection of officers charged with the duty of checking the weights and of noting the presence of slate, dirt, etc."

The United States Geological Survey has analyzed and tested some "bone coal," and their analyses show only 45.43 per cent. ash in West Virginia 11-B and 28.08 per cent. ash in West Virginia No. 24.

The "bands" or streaks of "bone coal" found in the Pittsburg seam as mined in West Virginia contain only 25 to 30 per cent. ash. A sample of "bone coal" from Pennsylvania was found to contain 30.56 per cent. ash. It is very difficult to conceive that a coal higher in ash than "bone coal" would be delivered by any coal company, and still more improbable that any inspector would not

---

[1] Read before the Industrial Division of Amer. Chem. Soc., Baltimore, December 31, 1908.

detect the nature of such material from the most casual inspection. It seems more likely that such results were due to a non-representative sample.

Coal which undoubtedly did not vary more than 2 per cent. in actual quality was delivered under one of the other specifications, and the analyses show a variation of over 15 per cent. ash in individual samples.

In another instance, coal was delivered from one cargo to a state institution during two consecutive months, and one sample was accumulated throughout each month. The second sample showed over 20 per cent. more ash than did the first. This resulted in a penalty of over $1.50 per ton on all coal delivered for the month. The coal was all burned, so there was no chance to obtain a check sample, and the coal contractor had nothing to do but accept the reduced price, thus losing more than all the profit on the entire year's contract because of this one sample.

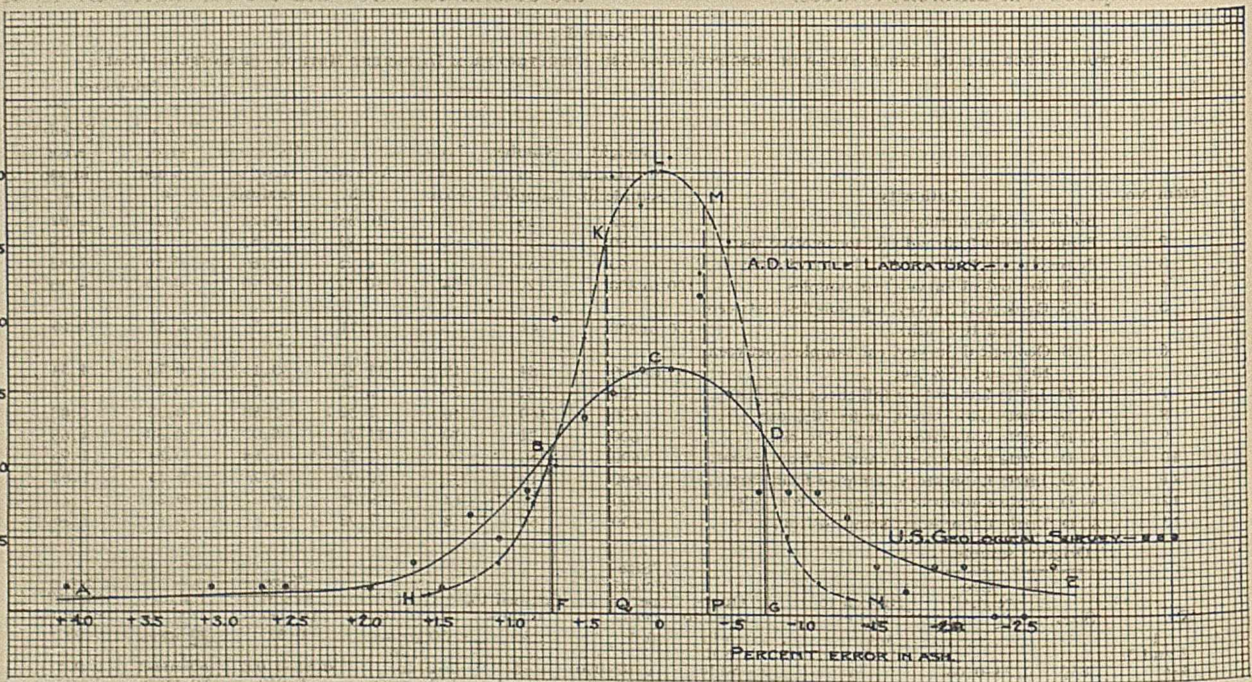


Fig. 1.

It is not strange that the coal companies are reticent about taking contracts on the analysis and heat unit basis, when they are at the mercy of non-representative samples. If there were only a corresponding error of sampling in the opposite direction, the results might average up very well in the end, providing a sufficient number of samples had been taken, but with a specified ash of 8 per cent., it is impossible to go 20 or 30 per cent. below the standard, while samples have shown this much above.

The impression seems to exist among many chemists, coal buyers and even coal companies, that the quality of coal from any one mine or region may vary a great deal more than is actually the case. The chemist may notice that certain results seem discordant and check the analysis, from his pulverized laboratory sample, and find that his analysis is correct. How many times does he check the taking and quartering down of the original sample? Here is where the discrepancies and inconsistencies are usually found; not in the quality of the coal itself or in the chemical analysis.

It is not intended to convey the impression that the quality of coal itself does not change, for it certainly is affected by the care of preparation, as well as local impurities which vary to some extent even in the same mine. It is because of these variations and the different quality and character of coal coming from different mines and regions that the coal is analyzed, but until the necessity of taking representative samples is realized by every one connected with the testing, buying and selling of coal, the errors in sampling will overshadow the variations in quality.

CAUSE OF ERRORS IN SAMPLING.

The word error as used here does not apply to that class of analyses which have been so often used for advertising purposes and have been made from selected samples. It applies only to cases where two or more samples are separately taken from some one lot of coal of which each sample is supposed to be representative. The error of an individual ash determination is the difference between its percentage of ash and the average of all samples taken from the one lot of coal.

It has been found that these errors follow the probability curve so closely that the probable error of sampling by a certain method may be calculated by the method of least squares, just as the surveyor or physicist calculates the probable error of a number of observations or determines the limit of accuracy of a certain instrument.

Table I gives the errors on a large number of check samples from different sources.

TABLE I.

ACTUAL ERROR AND PROBABLE ERROR IN PERCENTAGE OF ASH RESULTING FROM DIFFERENT METHODS OF SAMPLING COAL.

							Calculated.	
Item No.	Authority.	Weight of original sample lb.	Number of samples.	Lots of coal.	Average ash.	Maximum error by analysis.	Probable error in ash r.	Possible error in ash u.
1	Boiler tests by author	125	16	1	10.68	3.24	0.71	4.02
2	Pennsylvania Railroad Co., locomotive tests	60	17	1	7.83	3.52	1.21	6.19
3	Pennsylvania Railroad Co., locomotive tests	60	80	1	6.25	2.60	0.50	2.83
4	U. S. Geological Survey, car samples	100 to 800	120	34	10.17	4.10	0.73	4.16
5	U. S. Geological Survey, car samples, between 5% and 10% ash	100 to 800	48	13	6.71	2.06	0.56	3.19
6	U. S. Geological Survey, car samples, between 10% and 15% ash	100 to 800	64	19	11.88	3.11	0.76	4.34
7	A. D. Little Laboratory (all check samples)	600	181	56	8.28	1.58	0.33	1.86
8	A. D. Little Laboratory, under 10% ash	600	160	46	7.52	1.58	0.33	1.88
9	A. D. Little Laboratory, over 10% ash	600	21	10	11.78	0.93	0.30	1.68
10	A. D. Little Laboratory, sampled from vessel	600	136	37	7.42	1.58	0.34	1.96
11	A. D. Little Laboratory, sampled from cars	600	18	7	10.02	0.93	0.33	1.88
12	A. D. Little Laboratory, sampled from piles	600	27	12	9.95	0.57	0.22	1.27
13	A. D. Little Laboratory, sampled from cars	250	62	8	7.98	1.61	0.41	2.36

	Original sample.									
No.	Size.	"Size-weight" per cent.	Broken to. inches	Quartered to.	"Size-weight" per cent.	Crushed to, inches.	Quartered to.	"Size-weight" per cent.	Pulverized to.	"Size-weight" per cent.
1	...	....	3/4	5 lb.	0.5	¼	50 grams	0.22	80-mesh	0.002[1]
2 and 3	...	....	"Almond"	5 lb.	...	....	½ lb.	....	....	....
4-6	1¼	0.23-0.03	½	25-50 lb.	0.02-0.01	¼	2-10 lb.	0.01-0.002	60-mesh	0.005[1]
7-12	...	About 0.1	2-mesh	4 lb.	0.05	8-mesh	100 grams	0.012	80-mesh	0.002[1]
13	...	About 0.2	2-mesh	2 lb.	0.10	8-mesh	100 grams	0.012	80-mesh	0.002[1]

The results of items Nos. 4 and 7 have been plotted in Fig. 1. These curves involve no mathematics whatever, but represent the per cent. of analyses in which the errors fall between 0 and 0.2, 0.2 and 0.4, etc., both above and below the average. The significance of two probability curves plotted to the same scale, is that their areas are equal, and curve with the highest apex corresponds to the largest number of small errors. Whatever area B K L M D C one curve one may have above another near the zero axis must be equaled by the areas A B H and E D N in the regions of greater error. The probable error in the two instances are represented by the distances O Q (0.33) and O F (0.73). This means that the area Q K L M P is equal to the sum of the areas H B K Q and P M D N lying outside of the probable error, or in other words, there are as many errors greater than 0.33 as there are less than this amount. The possible error or u, is the error that is likely to occur only once in 10,000 analyses, and would be represented on the curve as the point where the vertical ordinate is 0.01%. As this data follows the probability curve very closely, the formula for probable and possible error can be safely applied in determining the reliability of different methods of sampling.

In this table the last three columns, denoting the determined error and the calculated errors, are given in percentages based on the coal as 100 per cent., and not on percentages of the amount of ash present in the coal. In each of these cases the average of all samples made on the same lot of coal was determined, and the error of each individual sample from this average was taken. The sum of the squares of each of these errors was substituted in the following formula:

$$(1)\quad r = 0.6745 \sqrt{\frac{\Sigma v^2}{n-1}}$$

---

[1] Based on one gram sample taken from bottle for analysis.

"r" is the probable error.

"v" is the individual error.

"Σv^2" is the sum of the squares of the individual errors.

"n" is the number of samples analyzed to obtain the averages from which the errors were determined.

The formula used for calculating the possible error is:

$$u = 3.84\sqrt{\frac{\Sigma v^2}{n-1}}$$

"u" represents the possible maximum error likely to be encountered once in 10,000 different samples.

The other symbols correspond to Formula 1, the only difference being the change in the constant.

The "size-weight" percentage referred to in the lower part of Table I is the percentage of the largest pieces of slate to the total weight of the sample previous to its last division or quartering. This ratio or percentage appears to be the most important factor to be considered in the sampling of coal.

Item No. 1.—Table I represents 16 samples taken from one car of Pennsylvania coal after it had been carted from the car and shoveled into a shed. During one boiler test when about three tons (including the samples), were taken from the pile, one shovelful was put into each of 16 barrels after every filling of the wheelbarrow. In this manner sixteen separate samples were accumulated, each consisting of one shovelful for each wheelbarrow-load taken from the pile, and any one sample should represent at least the three tons of coal taken from the bin, if this method of sampling is to be relied upon, and 125 pounds original sample is sufficient. This coal probably contained some pieces of slate weighing as much as one pound. These sixteen analyses are given complete in *American Society of Mechanical Engineers Transactions*, Vol. 27, page 636.

The data represented by Items 2 and 3, Table I, are taken from "Locomotive Tests and Exhibits" by the Pennsylvania Railroad System, Louisiana Purchase Exposition, 1904. This report states: "The coal finally selected came from the Scalp Level mines (near Johnstown, Pa.), owned by the Berwind-White Coal Mining Co." Some of this coal was also used by the U. S. Geological Survey at St. Louis and according to their reports the coal came from Eureka No. 31 mine.

The method of handling and sampling the coal used at the Pennsylvania Railroad testing plant, is given in their report as follows: "All coal was brought to the plant in low side gondola cars, and was shoveled over the sides of the car into boxes of a capacity of about 1000 pounds each. After dumping and before sprinkling, a small sample was taken and placed in a covered box...... About 60 pounds of coal were taken as a sample; this was crushed to the size of an almond and reduced by quartering to about five pounds. It was then ground fine and reduced to half a pound by further quartering, after which it was pulverized and a regular chemists' sample taken."

Each analysis given in this report is supposed to represent the coal used on a test, which varied from 1300 to nearly 15,000 pounds, averaging 6739 pounds. While the quality of coal used on any one test may not have been exactly the same, yet there can be no doubt but that 7,000 pounds taken from any part of the cars shipped from this mine would contain a more uniform ash than is shown by the analyses of the individual 60-pound samples. A careful study of these results shows that there is no apparent relation between the variation in ash and time, as the results of consecutive tests show great variations, for instance, 7.25, 11.35, 7.63 per cent. ash. Averaging the different series of tests the ash is 7.83, 6.30, 6.10, 6.23, 6.31, 6.27, 6.25, and 6.30, indicating that after the first series of tests the quality of coal itself ran remarkably uniform. It appears that the coal received subsequent to the first series of tests had been better prepared, as the ash averages about 1.5 per cent. less. The errors are greatly reduced, indicating that the amount of slate and impurities has a great deal to do with the accuracy of sampling in addition to the "size-weight" per cent.

The data for Item No. 4 was taken from Professional Paper No. 48 of the United States Geological Survey and includes all analyses made of the different car samples taken at St. Louis during the period covered by this report. These results have been tabulated on pages Nos. 281 to 284. The errors have been determined from the average of all plant samples as a basis, which is probably not absolutely correct, considering the fact that the car samples were taken by a different method than were the samples from the steam, coke and gas plants—the principal difference being in the size of the initial sample. The car samples consisted of from 200 to 800 pounds original sample, while the samples taken from the gas plant were said to weigh about 100 pounds. This report also states that the samples from the steam plant were collected in galvanized iron cans which were about 18 inches in diameter and 30 inches high. On this basis, the capacity of these cans would be a little over 200 pounds; but it is not stated whether or not these cans were completely filled during each test. Apparently no mention has been made of the quantity of coal taken from the original sample in the coke plant.

From these various plants, the samples were quartered down by the conical pile method to 50 or 25 pounds and were then turned over to the chemical laboratory, where they were further reduced in size and quantity, as indicated in the lower part of Table I. All of this coal had been crushed to about 1¼ inches previous to any of the samples being taken.

Mention is made in several of the recent publications of the United States Geological Survey that there is a decided tendency for the heavier pieces, such as slate and pyrites, to settle to the bottom of the car. If this is true, it is probable that some of the errors here indicated may be somewhat greater than was actually due to the sampling. However, the same tendency holds true in the case of the other sets of samples presented in this table, and the comparison is undoubtedly justified.

Items Nos. 5 and 6 include the same data as is represented by Item No. 4, except that the results have been classified according to the average ash content in the various coals. It is noted from this comparison that the probable error in the coals containing between 5 and 10 per cent. ash is 0.56 per cent., while it is 0.76 per cent. ash in the coals containing over 10 and less than 15 per cent. ash.

Item No. 7 includes all of the check samples which have been made in a commercial laboratory during the past year. One hundred and eighty-one samples from 56 different lots of coal are represented. In these 56 different lots, there were only 14 varieties of coal included. The different lots represent either individual vessel cargoes, carloads or piles. It is noted that the maximum error found in these 181 samples is 1.58 per cent., while the probable error of each individual sample is 0.33 per cent.

Items Nos. 8 and 9 give the same analyses classified as to percentage of ash, and it is noted that there is practically no difference.

Items Nos. 10, 11 and 12 give the same analyses classified as to vessel, car and pile samples, and it appears that the probable error is less in the latter case than in the vessel and car samples, which are practically the same.

Item No. 13 includes 62 samples which have not been included in any of the above items. This coal was discharged at tide-water and loaded into railroad cars. A wheelbarrowful was taken from each car of coal as it was received at the plant, from which a sample was worked up and analyzed. The eight different lots indicate the number of cargoes received, while in fact there were only three kinds of coal and 42 of these analyses were made on one kind only. The averages of the five or more samples from each different cargo of this one coal varied only 0.8 per cent.

The important point brought out by Table I is the decreasing error with the larger original samples. For instance, the comparable cases where the writer was personally familiar are Items 1, 11 and 13.

Item.	Wt. original sample, lbs.	Actual error.	Calculated possible error.
1	125	3.24	4.02
13	250	1.61	2.36
11	600	0.93	1.88

Likewise, the results from such reliable laboratories as the Pennsylvania Railroad and United States Geological Survey show similar error when only 60 and 100 pounds have been taken as original samples. It should be understood that the car samples taken by the Government have probably been as well or even better taken than those from Little's laboratory; but the wide range of error is undoubtedly due to the samples from the steam, gas and coke plants, where only 100- to 200-pound original samples were taken.

EXPERIMENTAL SAMPLING.

After realizing the importance of keeping the ratio of the largest pieces to the weight of the sample, or the "size-weight" per cent. low, a series of experiments was made in order to determine how the variation in percentage of ash was affected by this ratio. The coal selected was a Pennsylvania semi-bituminous coal which was known to contain about 5 per cent. of ash in the form of slate and impurities in addition to the intrinsic ash of the coal itself. This coal contained very little pyrites or sulphur, as indicated by the following analysis:

Moisture	0.56%
Volatile	15.86%
Fixed carbon	72.08%
Ash	11.50%
Sulphur	0.93%
B. T. U.	13791

About 29 pounds of this coal were taken originally, and all of it was put through a 2-mesh sieve. It was then "quartered" or divided by means of a riffle sampler; this was repeated until about 144 grams remained to be divided. This remaining amount was divided and one-half (72 grams) of it was pulverized to 80-mesh and put into a bottle; the other half of this, instead of being returned with the large part of the original sample, was divided, making two 36-gram samples, one of which was pulverized to 80-mesh and bottled, while the remaining 36 grams were divided again, and one of the 18-gram samples pulverized, while the other was returned to the original sample. The original sample was again divided in the same manner and the complete operation repeated five times, thus giving five 72-gram, five 36-gram and five 18-gram samples. The remaining original sample was then screened through a 4-mesh sieve, and all that was too large was broken in a mortar until all of it passed through 4-mesh. Fifteen more samples were obtained from this coal in the same manner employed to get the fifteen 2-mesh samples. All of the remaining original sample was then put through an 8-mesh sieve and the entire operation repeated and carried one step further, making twenty 8-mesh samples, five each weighing about 72, 36, 18 and 9 grams.

A second and third series of samples were also obtained from the remaining original 8-mesh sample, but the second series was carried down to a 1-gram sample, and the third series was divided down to a 0.3-gram sample. In the third series, the samples of 2 grams and smaller were divided by quartering on glazed paper with a spatula; all other divisions were made with the riffle sampler. The samples of 2 grams and less were not pulverized, but the entire amount of coal contained in them was weighed up and burned in platinum crucibles.

The fourth series, consisting of ten 144-gram samples, was later made from the original sample, and from each of these 80-mesh bottle samples ten grams were taken and a composite sample mixed. Ten analyses were made from this one composite sample, after which it was further pulverized in an agate mortar until it all passed through a 200-mesh sieve. Eleven more analyses were then made from this sample. In all there were 193 samples made from this one original 29 pounds of coal.

The phase of sampling which is particularly considered in this paper is the percentage of ash, consequently the ash only was determined from each of these samples, excepting a proximate and B. T. U.

TABLE II.

ASH DETERMINATION WITH DIFFERENT "SIZE-WEIGHT PERCENTAGES."

Weight of sample gram	Composite.	Composite.	144	72.1	35.2	17.15	8.85	73.0	4.52
All through sieve before dividing	...	...	8-M	8-M	8-M	8-M	8-M	4-M	8-M
"Size-weight" per cent	0.0002[1]	0.0014[1]	0.00835	0.0167	0.0341	0.070	0.136	0.150	0.266
Pulverized to	200-M.	80-M.	80-M.	80-M.	80-M.	80-M.	80-M.	80-M.	80-M.
Ash per cent	...	...	...	...	...	...	...	...	10.21
	...	...	...	...	...	...	...	...	10.60
	...	...	...	11.08	12.06	11.96	11.58	...	10.76
	...	...	...	11.58	12.10	11.06	11.04	...	10.48
	...	...	...	11.78	11.92	12.30	11.28	...	11.64
	...	...	...	11.72	12.16	12.14	11.88	...	11.74
	11.50	...	...	11.82	12.04	12.60	11.10	...	11.22
	11.50	11.34	11.38	11.24	11.20	11.30	12.06	...	11.33
	11.48	11.48	11.28	11.68	12.02	12.04	11.46	...	11.40
	11.54	11.62	11.78	11.20	12.06	12.70	11.04	...	11.33
	11.48	11.50	11.46	11.52	11.08	11.90	12.44	...	11.20
	11.46	11.48	11.38	11.30	11.88	11.66	11.54	...	10.27
	11.54	11.40	11.30	11.64	11.30	11.18	11.26	11.98	12.32
	11.48	11.36	11.16	11.66	11.26	11.26	11.52	12.32	11.28
	11.50	11.60	11.00	11.10	11.36	10.66	11.58	11.95	11.40
	11.54	11.40	11.16	11.48	10.90	12.06	11.44	12.30	11.32
	11.48	11.42	11.40	11.04	10.98	11.34	10.80	12.16	10.32
Average	11.50	11.46	11.33	11.46	11.62	11.68	11.47	12.14	10.65
Maximum error from 11.50	0.04	0.16	0.34	0.46	0.66	1.20	0.94	0.82	1.29
Error of average from 11.50	0.00	—0.04	-0.17	—0.04	+0.12	+0.18	—0.03	+0.64	—0.85
Calculated									
r or probable error	0.02	0.07	0.18	0.19	0.33	0.43	0.34		0.58
u or possible error	....	0.40	1.04	1.05	1.87	2.48	1.91		3.28

[1] Based on one gram taken from bottle for analysis.

TABLE II.—(*Continued.*)

Weight of sample gram	36.4	2.2615	18.2	1.1368	71.8	0.5867	35.5	0.3001	17.8
All through sieve before dividing	4-M.	8-M.	4-M.	8-M.	2-M.	8-M.	2-M.	8-M.	2-M.
"Size-weight" per cent	.305	0.53	0.61	1.06	1.16	2.045	2.35	4.00	4.65
Pulverized to	80-M.	Not pulverized.	80-M.	Not pulverized.	80-M.	Not pulverized.	80-M.	Not pulverized.	80-M.
Ash, per cent	...	...	...	10.47	...	...	...	...	...
	...	...	...	11.39	...	...	...	...	...
	...	...	...	9.74	...	...	...	...	...
	...	...	...	9.37	...	...	...	...	...
	...	...	...	10.45	...	...	...	...	...
	...	10.91	...	10.29	...	...	...	...	...
	...	9.57	...	12.41	...	...	...	...	...
	...	10.37	...	9.32	...	...	...	...	...
	...	10.88	...	10.06	...	...	...	...	...
	...	10.40	...	10.80	...	...	...	...	...
	...	11.22	...	10.39	...	10.73	...	10.79	...
	...	12.13	...	11.30	...	12.79	...	10.66	...
	...	11.23	...	12.79	...	13.50	...	13.48	...
	...	10.20	...	10.11	...	9.14	...	12.18	...
	...	9.61	...	11.34	...	10.77	...	15.86	...
	11.64	11.49	11.64	12.09	11.88	10.06	11.68	11.52	15.56
	12.92	10.55	11.84	12.97	10.50	9.74	14.12	8.71	9.76
	10.76	11.55	10.70	9.72	13.80	12.01	11.72	9.58	8.84
	10.82	12.20	11.74	10.30	12.10	11.21	11.20	13.32	12.78
	12.48	10.92	11.76	7.60	11.24	11.91	11.36	8.95	9.18
Average	11.72	10.88	11.54	10.65	11.90	11.19	12.01	11.50	11.22
Maximum error from 11.50	1.42	1.93	0.80	3.90	2.30	2.36	2.62	4.36	4.06
Error of average from 11.50	+0.22	—0.62	0.04	—0.85	+0.40	—0.31	+0.51	0.00	—0.28
Calculated									
r or probable error		0.60		1.01		0.90		1.65	
u or possible error		3.41		5.72		5.14		9.40	

determination that was made from the 200-mesh composite sample. In every case the coal was burned to ash in platinum crucibles placed in a gas muffle.

Each ash determination made is given in Table II and arranged in columns according to the "size-weight" per cent. The percentages of ash in each column are in order as determined. The average of the eleven 200-mesh composite ash determinations has been taken as representing the true average ash contained in the coal as it was taken from the ten 144-gram, 8-mesh samples after they had been pulverized to 80-mesh. It is noted that the maximum error of individual samples from the true average of 11.50 gradually increases with the "size-weight" per cent. The probable and possible errors have been calculated according to formulas (1) and (2), and these values also increase with the increasing percentage of the largest piece to the weight of sample. It will be noted that the probable error of taking one-gram quantities out of the 200-mesh sample is 0.02 per cent. This is probably the limit of accuracy of weighing on the balance, as a 12-milligram rider is used and it is customary to weigh no closer than 0.2 milligram, consequently no possible error was calculated in this instance.

Had the importance of obtaining a large number of results been fully realized at the start, the work would have been more concentrated, especially with the larger "size-weight" percentages, which would have resulted in a more uniform increase in the individual errors, as well as the values calculated for "*r*" and "*u*."

This work might be criticized by some because of its being carried out on a comparatively small scale, but when the results compare as favorably as they do, whether 2-mesh samples weighing 17.8 grams or 8-mesh samples weighing only 0.3 gram are used, as noted in the last two columns of Table II, there can be little doubt that it would hold true from the largest sizes of commercial coal down to a sample as small as 200-mesh.

It will be noticed from these ash determinations that even in cases where the "size-weight" per cent. is comparatively high and variations of over 5 per cent. occur in ten or more analyses, there may be cases where three or more consecutive determinations agree within 0.50 per cent. Too often have chemists supposedly determined their range of error by making only a few check determinations. Two analyses or check samples are of little value in arriving at the limit of accuracy in any case; ten checks are not conclusive, but it would be better to repeat the determination 50 or 100 times at least.

In Fig. 2 the results of Table II have been plotted in terms of "size-weight" percentage and percentage of ash. The analyses given in the first and third columns are left out because of the impossibilty of putting so many points in such a small space. Also, the results of the second column are indicated by two short horizontal lines extending to the left of the vertical axis and represent the extremes only. The lines showing the limits of the plus and minus errors have been drawn through the exterior points of the various sets of analyses, connecting the greatest errors, rather than in and out, as it would necessarily have had to be drawn in some instances where too few results were determined under one set of conditions. One exception had been made in the instance of the 7.60 per cent. ash on a sample where the "size-weight" per cent. was 1.06. This one point is evidently one in ten thousand and probably would not have been approached again even had a thousand samples of this class been analyzed.

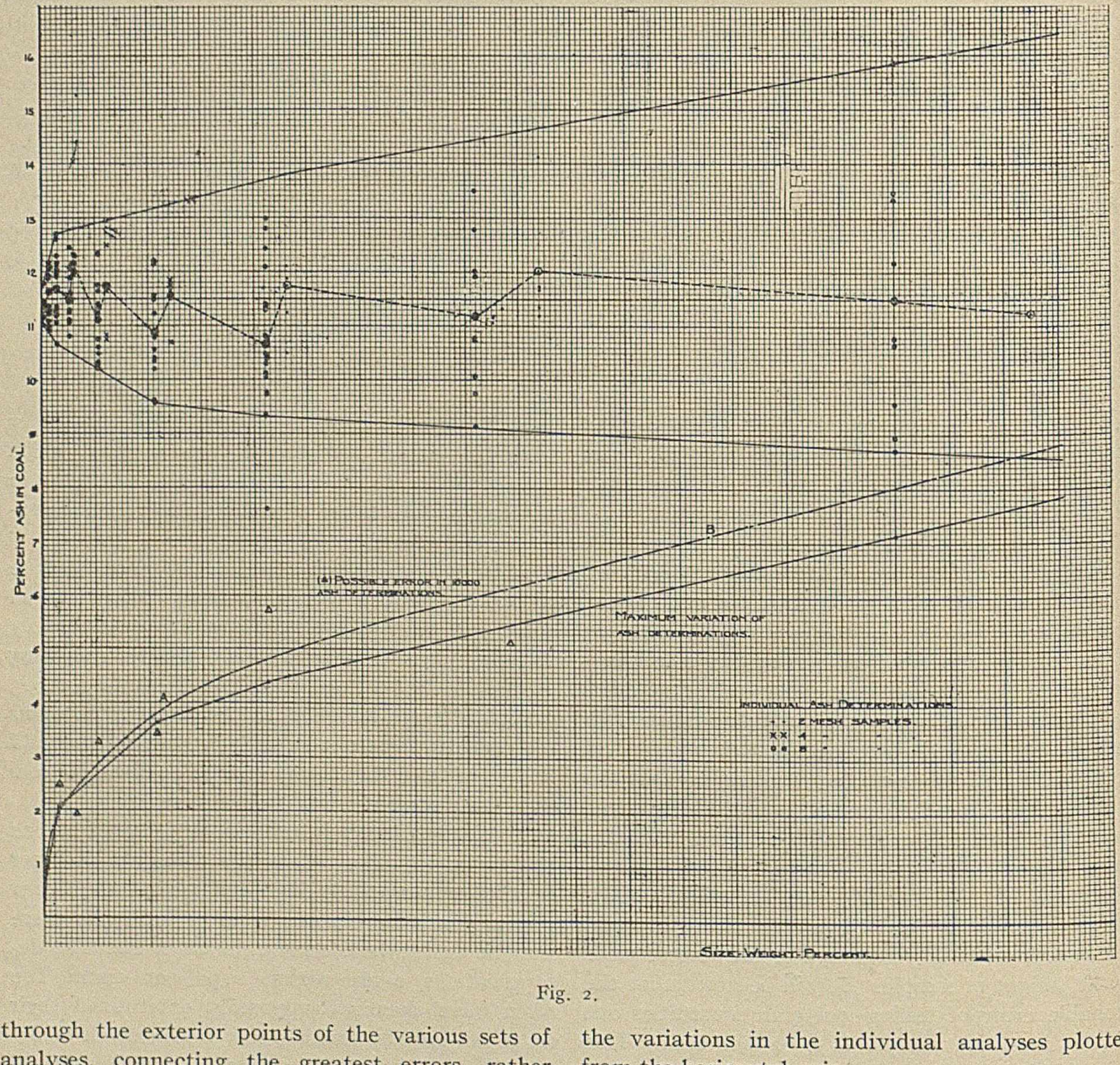


Fig. 2.

The points with large circles connected by a broken line represent the average ash of each set and are found both above and below the true average line, with some surprising variations.

The lower full line curve of Fig. 2 represents the variations in the individual analyses plotted from the horizontal axis.

The triangular points are plotted from ("*u*") the possible error in 10,000 as given at the bottom of Table II.

The curve A B has been drawn with respect to these points, giving some weight, also, to the nature of the variation curve as actually determined.

This possible error curve represents the error from the average only, while the determined variation curve is the sum of the plus and minus errors. Had more determinations been made in the several cases, the possible error or ("*u*") points would undoubtedly have fallen more consistently.

From these results, it will be at once realized that the "size-weight" per cent. should be less than 0.1 per cent., if errors of 2 per cent. are not to be exceeded or the probable error is to be less than 0.4 per cent. ash with a coal that contains about 5 per cent. ash in the form of slate and foreign impurities when quartered.

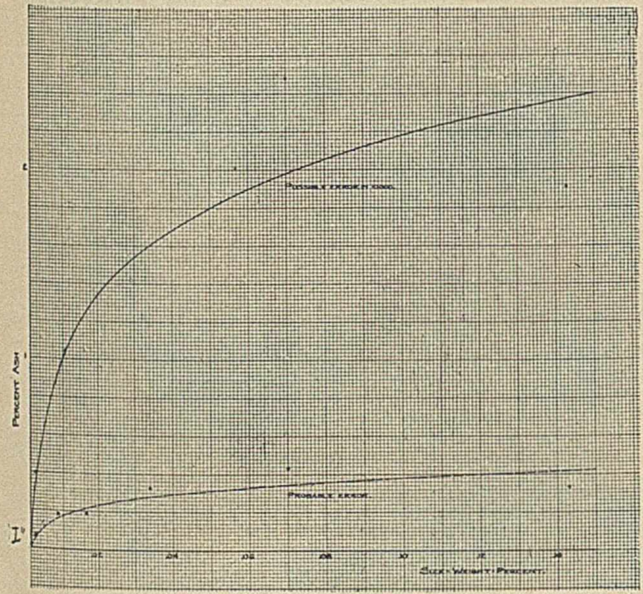


Fig. 3.

Fig. 3 gives the possible error curve from Fig. 2 drawn to a larger scale, and according to this, the "size-weight" per cent. must be as low as 0.01 per cent. if an error of 1 per cent. in ash is not to be exceeded. Even with this small ratio, the probable error is almost 0.2 per cent.

APPLICATION OF THE LAW OF PROBABILITY.

Fig. 2 shows that the error does not increase directly with the "size-weight" per cent., but it rises very rapidly from the origin, making a curve similar to a parabola. When the "size-weight" per cent. is 0.5 per cent., the determined variation in ash is 3.5 per cent., or seven times the variation due to an odd piece of slate going into one sample one time and into another the next. It, therefore, indicates that it is not a question of one piece of slate or high ash coal that causes the error, but several individual pieces which, according to the law of chance, fall more on one side than on the other.

In order to determine the extent of this variation, a series of experiments was made as follows: Sixteen small pieces of coal (4-mesh) were placed in a can containing a quantity of sand and the mixture divided in the riffle sampler. The number of pieces of coal falling on the right-hand side of the sampler were counted, and this half divided again, and the operation repeated until no coal fell on the right-hand side. Theoretically each division should have halved the number of pieces of coal, giving 8, 4, 2, 1, 1 or 0 for the successive divisions, but out of 100 times there were only two sets of divisions where these conditions were fulfilled, and from 100 sets of similar divisions with 32 pieces of coal, corresponding conditions were not fulfilled at all. Some of the results obtained were:

After 1st	2nd	3rd	4th	5th	6th	7th	8th Division.
4	3	0					
11	8	6	3	2	2	1	0
7	2	1	1	0			
9	5	3	2	0			
12	4	1	1	1	0		
8	5	5	0				

After 100 such sets of divisions had been made with both 16 and 32 pieces of coal, it was realized that the first division of each set was of the greatest importance, and that a large number of separate divisions was necessary to determine the law governing the error; consequently 1300 divisions were made, using 8, 16, 32, 64 and 128 pieces. With a fewer number of pieces—four, for instance—it took only a few divisions before the extreme possible variation was reached, *viz.*, either getting 4 or 0 pieces on the right side from the first division.

While the riffle sampler is pretty well known, it might be well to describe the one used in this work. It consists of a series of 20 troughs ½-inch wide, made of tin and soldered together so that each adjacent one slopes in the opposite direction at an angle of about 30 degrees from vertical. The sampler is put over the sides of two rectangular cans setting close together, and the sample to be divided is poured from a similar can on to the tops of the 20 troughs. The top edges of these troughs are level, so that the flat stream of coal falling on the sampler is divided into 20 streams ½-inch wide. Each alternate twentieth part of the entire sample goes into one of the collecting cans, while the other can receives the other half. In dividing down a sample of coal for analysis, the sample may be divided several times, resulting in ½, ¼, ⅛, etc., of the original until the sample is reduced to the desired quantity. The term dividing has been used in preference to "quartering," because in this case the sample is in reality divided into 20 parts instead of four.

The results of the several hundred divisions made of the different numbers of counted pieces of coal are given in Table III.

TABLE III.

RESULTS OF COUNTING MARKED PIECES OF COAL AFTER BEING DIVIDED ON A RIFFLE SAMPLER.

1 Number of pieces coal (n)	**4**	**8**	**16**	**32**	**64**	**128**
2 Times divided..........	58	100	200	300	400	300
3 Theoretical number after division............	2	4	8	16	32	64
4 Average of all divisions, pieces..............	1.86	3.92	8.04	16.61	32.33	65.88
5 Per cent. above or below item 3..............	—3.5	—1.0	+0.3	+1.9	+0.5	+1.5
6 Smallest number pieces found..............	0	1	3	9	21	47
7 Largest number pieces found	4	8	12	23	43	80
8 Maximum variation. Number pieces............	4	7	9	14	22	33
9 r of probable error—formula (1)............	....	0.91	1.17	1.67	2.61	3.79
10 u or possible error—formula (2)............	....	4.00	6.63	9.51	14.90	21.80
11 u' or possible error from empirical formula (3).	....	3.94	6.40	9.88	14.80	21.77
12 Per cent. Item (11) to Item (1)............	....	49.3	40.0	30.9	23.2	17.0

From Items 4 and 5 it will be noted that the averages of all divisions are reasonably close to the theoretical average.

The important item in this table is 8, or the maximum variation in the number of pieces of coal falling on one side of the sample at different times as compared with the total number of pieces in the sample divided. The data from these divisions make it possible to plot very good probability curves, as seen in Fig. 7.

Items 9 and 10 have been calculated by substituting the individual errors of the several hundred divisions in formulas (1) and (2), which have been previously explained and used in calculating the probable and possible errors of the commercial samples. Item 9, after being divided by two in order to change it from variation to error, has been plotted in Fig. 4 as "maximum error found." The probable error as well as the probable error in 10,000 determinations have been plotted to the same scale.

From the possible error curve as plotted in Fig. 4, it appears that this curve is similar to a parabola, conseqeuntly a formula has been derived by substituting the different values of u and n from Items 1 and 10, Table III, in the formula

$$u = a + b\sqrt{n}$$

and solving for the constants a and b by the method of least squares. This results in formula

$$(3) \qquad u = 2.10\sqrt{n} - 2.01.$$

Where u is the possible error in number of pieces above or below the average, n is the number of pieces of coal contained in the sample before it is divided.

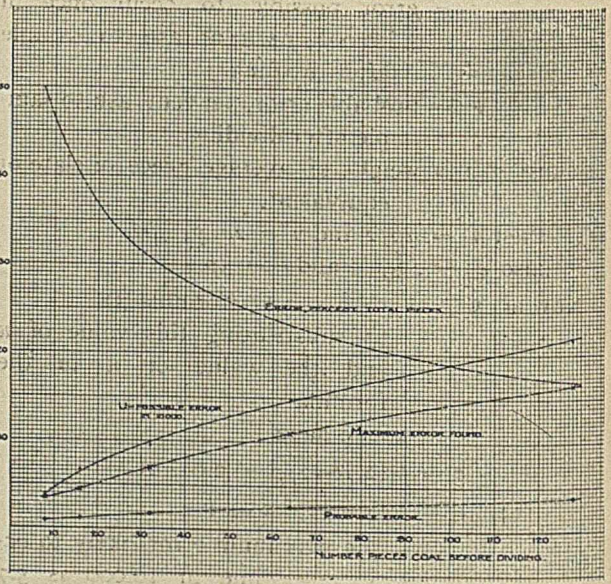


Fig. 4.

It is noted from Item 12 and the curve on Fig. 4, plotted from these results, that in spite of the increasing error in the number of pieces with the greater number of pieces divided, the percentage of error to the total number of pieces divided grows less. From this fact, together with formula (3), we readily see that in the case of sampling coal the important point lies in the number of pieces of slate, bone or coal having higher than the average percentage of ash. If a certain lot of coal contains a certain percentage of ash in the form of slate, etc., the error in sampling will be greatly diminished as the pieces of slate are broken finer and the number of them thereby increased. It seems reasonable to assume that the weight of the average piece of slate in a sample of coal would be about one-half that of the largest pieces, although this might be greater or less, depending upon how the sample was broken up.

If a = per cent. ash due to slate, free impurities, etc., in sample to be divided

p = "size-weight" per cent. or per cent. of largest pieces of slate in sample to be divided

n = number of pieces of slate, or equivalent, in sample to be divided

u = error possible to occur once in 10,000 pieces of slate after dividing

e = error possible to occur once in 10,000, per cent. ash to sample after dividing

r = probable error, per cent. ash to sample after dividing.

Assume $p/2$ = weight of average piece of slate or equivalent,

then $a = np/2$ and $n = 2a/p$

also $e = 2\,up/2$ and $e = up$.

Now as $u = 2.10\sqrt{n} - 2.01$ (formula 3) by substituting this value of u and the value of n in the formula

$e = up$ we have

$e = p\,(2.10\sqrt{2/p} - 2.01)$ or

(4) $e = 2.97\sqrt{ap} - 2.01\,p$

The maximum error likely to occur one time in 10,000 is 5.7 times greater than the probable error, therefore

(5) $r = 0.522\sqrt{ap} - 0.353\,p$

From these formulas it is apparent that the error in sampling is dependent upon the percentage of ash in the form of slate, etc., and the "size-weight" percentage, and by assuming different values for a and p the possible and probable errors have been calculated and plotted in Fig. 5.

Curve "AB" from Fig. 2 has also been plotted in Fig. 5 to the same scale, and it is noted that it follows the 5 per cent. ash calculated error curve very closely excepting where the "size-weight" percentage is below 0.2 per cent. Even here, the curves are of the same general nature and rise very abruptly from the origin, indicating that in order to keep the error within any reasonable limits, the "size-weight" percentage must be very small.

It is very reasonable to expect that the possibility of error would increase with the percentage of ash existing in the sample as slate and other such impurities. It does not follow that the error is necessarily greater in sampling a high ash coal as compared with a low ash coal, for if the coal contains a high intrinsic ash it is already more or less uniformly mixed throughout the coal. It is the difference between the total ash in any given lot of coal and the intrinsic ash, or that contained in the purer lumps of coal, which affects the error of sampling for any given size and weight of sample to be divided.

The difference in the percentage of ash in car samples and mine samples is a very good indication of the amount of ash that causes the variation in sampling.

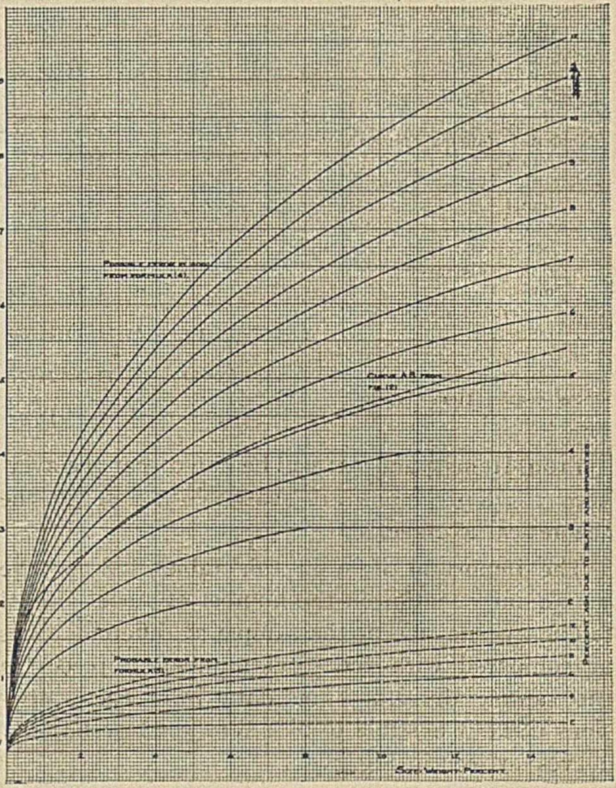


Fig. 5.

In Bulletin 316 of the United States Geological Survey, Mr. Burrows has compared the ash in mine samples with the ash in the run of mine, screened coal and slack shipped under inspection from the corresponding mines.

From 87 mines the average ash in the run of mine samples was 2.67 per cent. higher than in the corresponding mine samples. In the 67 cases of screened coal being loaded, the ash averaged 3.06 per cent. higher, and from 15 slack samples the ash was 5.97 per cent. higher than in the corresponding mine samples. In several individual instances the ash in the car samples was more than 10 per cent. higher than in the mine samples.

United States Geological Survey Bulletin 323 also gives data bearing on this point. On pages 30 to 35 the results of washing 23 different coals in a solution of 1.35 specific gravity are given, and the difference between the ash in the original sample and that part of the coal which floated in this solution was 4.75 per cent. In several instances the difference exceeded 10 per cent. ash.

While the ash due to slate and impurities may vary from less than one per cent. to over 10 per cent. in commercial coals, the average appears to be somewhere near 3 per cent.; but considering the number of instances where the difference is greater than this amount, it seems advisable to assume 5 per cent. for this value, as it cannot be determined in individual cases very readily, and in no case will it be definitely known before the sample is taken.

Of the 23 coals washed in the 1.35 specific gravity solution, only three contained over 7 per cent. ash in the washed coal, and in some cases this intrinsic ash is as low as 1.52 per cent.

In Bulletin 316, Mr. Burrows also classifies the sample of run of mine coal according to the ash in the mine samples. In the case of 35 coals, where the ash in the mine samples was less than 7 per cent. (averaging 4.80), the car sample contained 2.97 per cent. more ash, while with the 47 samples, where the ash in the mine samples was more than 7 per cent. (averaging 9.50), the car samples showed only 2.69 per cent. more ash than did the corresponding mine samples. This indicates that the total ash is no indication whatever of the amount of ash existing as slate, etc. It is largely dependent upon

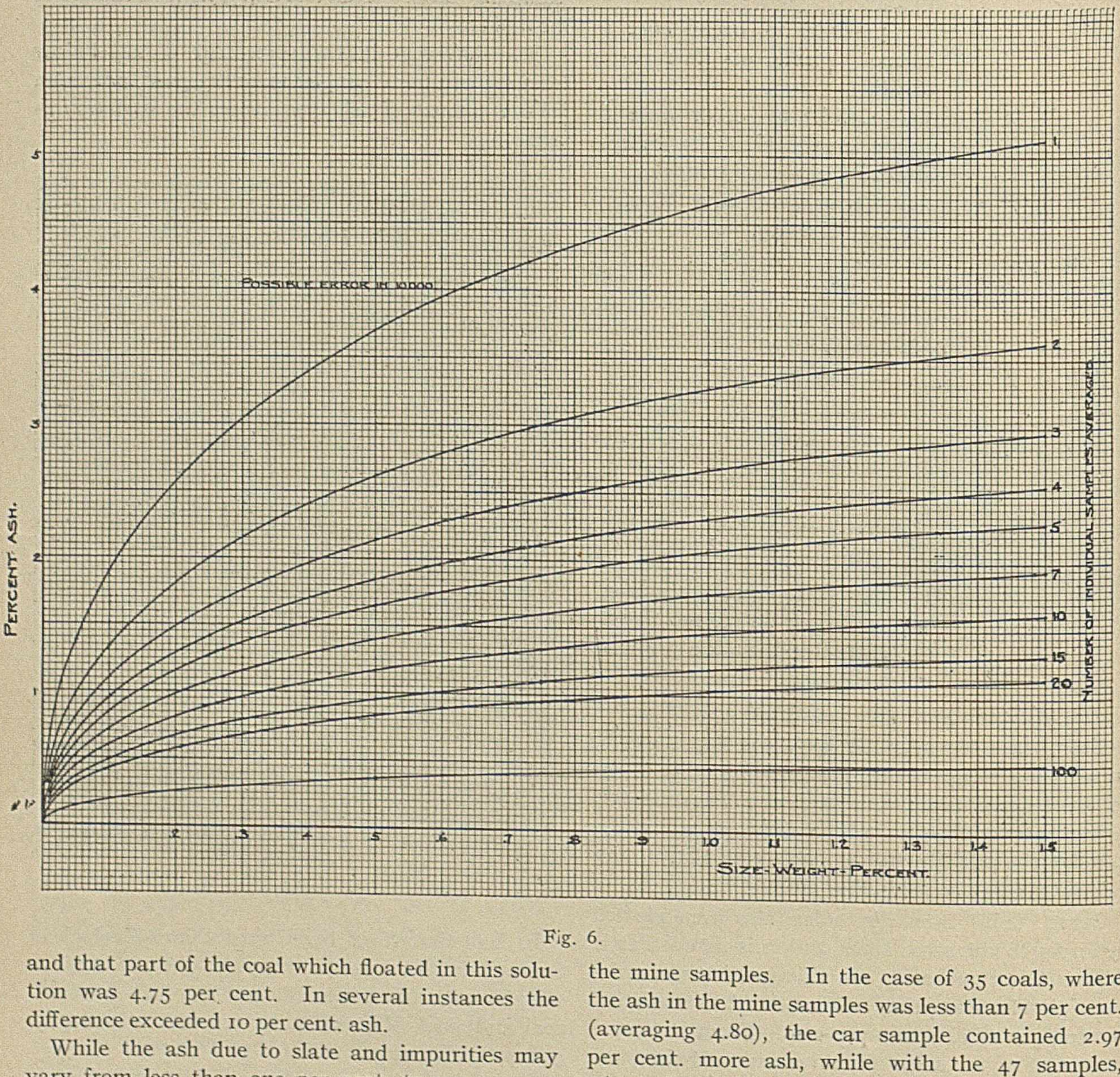


Fig. 6.

the nature of roof, floor and partings found with any seam of coal, together with the care in mining and preparation of the coal. The percentage of slate and free ash increases as the sample is broken and crushed to the smaller sizes, as the breaking frees thin streaks of slate, bone and pyrites from the lumps of coal to which they may have been attached in the original sample.

Throughout all of the experimental work presented in this paper, the dividing of the samples was done on the riffle sampler, but two samples of coal containing 16 and 32 pieces of coal, painted white, were each mixed and quartered 100 times by means of a spatula, and the number of pieces of white coal in each quarter were counted. From the variation in the sums of the number of marked pieces in the two opposite quarters, the probable error was calculated. A probability curve was drawn from the 32-piece sample and shown in Fig. 7 in comparison with the probability curve from 300 divisions of 32 pieces with the riffle sampler. It will be noted that there seems to be a little greater error by the quartering method. The pieces of marked coal in two adjacent quarters were also added, and from the results obtained the discarding of two opposite quarters seems to give little, if any better results than if the conical pile had been simply halved. However, this phase of sampling was not investigated with sufficient thoroughness for any definite conclusions to be drawn.

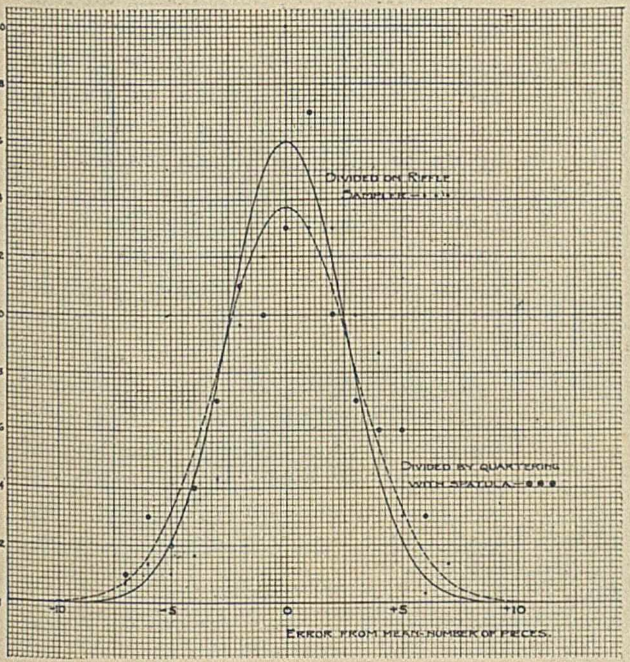


Fig. 7.

AVERAGE OF ANALYSES.

Many laboratories have realized the large errors likely to occur in individual samples and have attempted to obtain the true quality of any lot of coal by taking several duplicate samples and averaging the results of the several separate analyses.

According to the law of averages, the probable error of the average of any number of observations is expressed by $r_0 = r/\sqrt{n}$ where r is the probable error of the individual observations and n is the number of observations averaged; so the probable error of the average of four samples is only one-half the probable error of the individual samples, providing the same method has been used in taking each sample. The same is true of the possible error.

From the 1300 divisions and counting of the number of pieces of coal, the results were averaged by twos, fours, sixes, eights, tens, etc., and the probable error of the averages determined. It was found that the above formula held true to within reasonable limits, hence it was used in calculating the values which have been plotted in Fig. 6. This set of curves is the same as the curve of Fig. 5 obtained from formula (4), where $a = 5$ per cent., except the values of e for corresponding "size-weight" percentages which have been divided by the square root of the number of samples averaged.

This set of curves indicates that if the possible error is to be less than one per cent., the "size-weight" percentage must be less than 0.024 per cent. if one sample only is taken, while if 10 samples are taken the "size-weight" percentage may be 0.33 per cent. in each sample and still be assured that the possible error will be only one per cent. In order that these conditions may be fulfilled, the one sample should be 13.7 times larger when originally taken from the lot of coal than is each of the 10 samples. The result would be that 27 per cent. less coal need be taken and broken up for the original 10 samples than in the one individual sample. Similarly, 47 per cent. less coal need be taken originally, providing it is divided into 20 separate samples. As the greatest difficulty is in taking and quartering down the original sample, it might often be better to take several original samples, and to mix them after they have been quartered down to a comparatively small size, making one sample for the final pulverizing and analysis.

WEIGHT OF VARIOUS SIZES OF COAL AND SLATE.

In order to determine the weights of the largest pieces of slate that would pass through different sizes of bar screens and sieves, pieces of both slate and coal were weighed for the different sizes from 4-inch screen to 80-mesh per inch sieves. The results are given in Table IV. On account of the few pieces of slate larger than 2-inch screen, their weights are rather discordant when compared with the weights of coal of the same size, consequently, in plotting Fig. 8 it was assumed that for sizes larger than 2½ inches the weight of the slate was 2.25 times that of the coal. The specific gravity of the coal, slate and pyrites was determined as follows: Coal, 1.36; slate, 2.55; pyrites, 4.28; thus making the ratio of the specific gravities of slate to coal 1.87: 1. It is noted from Table IV that the ratio of the weights of slate to coal is greater than this in each case, where a sufficiently large number of pieces were weighed, until they were broken to 8-mesh. This is probably due to the slate usually occurring in the form of flat pieces; and in breaking the slate it tends to form long, slender pieces rather than cubical, as does the coal; while the pyrites may exist in more or less lens-shaped pieces, yet in breaking it forms more nearly cubical pieces, so that its weight was taken as 3.15 times the weight of the different sizes of coal. The ash or iron oxide resulting from the

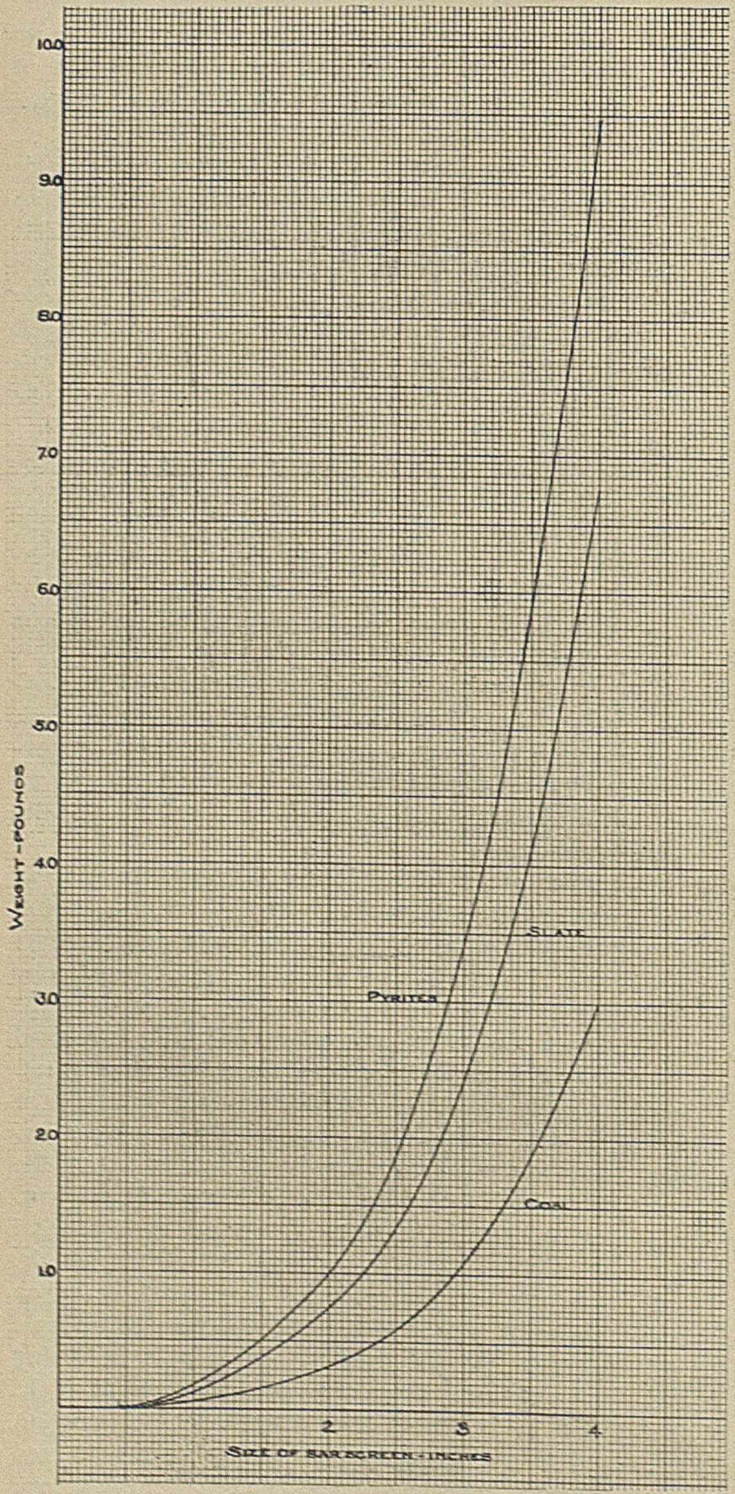


Fig. 8.

TABLE IV.

WEIGHT OF COAL AND SLATE THROUGH BAR SCREENS AND SIEVES.

		Slate		Coal.		
Through.	Over.	Number pieces weighed.	Average weight per piece. Lbs.	Number pieces weighed.	Average weight per piece. Lbs.	Ratio average weight of slate to coal.
4	3½	2	3.8	6	2.4	1.58
3½	3	2	1.97	6	1.43	1.38
3	2½	5	1.24	16	0.806	1.54
2½	2	5	0.98	23	0.435	2.25
2	1½	14	0.553	47	0.236	2.35
1½	1	51	0.240	138	0.106	2.26
1	½	152	0.0463	648	0.0195	2.37
½	2-mesh	238	0.0076	601	0.00368	2.06
Mesh.	Mesh.		Gram.		Gram.	
2	4	108	0.805	597	0.340	2.37
4	8	1057	0.0899	702	0.428	2.10
8	10	592	0.0168	643	0.00898	1.87
10	20	931	0.0036	1069	0.001925	1.87
20	40	770	0.000201	....	......	....
40	60	1000	0.0000606	....	......	....
60	80	2000	0.0000257	....	......	....

burning of pyrites is a little more than that due to a piece of slate of corresponding size.

Some of the slate used for these experiments was found to lose 11 per cent. upon being heated in the muffle. Figs. 9 and 10 give the weights of different sizes of coal and slate between 2-mesh and 80-mesh from Table IV. In Fig. 9 the results are plotted to nominal sizes of sieves, but Fig. 10 takes account of the actual size of the openings. The lengths of the sides of the larger openings were measured with a microscope in the sizes from 20 to 200-mesh.

Sieve. Mesh.	Length of side. inch.
2	0.425
4	0.25
8	0.10
10	0.076
20	0.0358
40	0.0164
60	0.0134
80	0.0089
100	0.0060
200	0.0030

The weights of the various sizes of coal, slate or pyrites can be read from the curves of Figs. 8 9 and 10, which have been drawn through the points of average weight. In several instances the largest pieces were weighed up separately and usually were a little higher than the results read from the curves for corresponding sizes.

WEIGHT AND SIZES OF SAMPLES.

From the foregoing data we are enabled to take and quarter down a sample of coal and be assured that the possible error may be within certain required limits. While accuracy of 0.25 per cent. would be desirable, it is seen from Fig. 3 that in order to be certain that errors would not exceed this amount, the "size-weight" percentage must be 0.001 per cent. In sampling run of mine coal, where pieces of slate weighing 5 pounds are often encountered, the original sample would have to consist of no less than 225,000 pounds; or if the coal had been previously crushed to 2 inches, the original sample should contain 37,000 pounds. Samples of this size are practically out of the question considering the expense necessarily involved as compared with the value of the material being sampled.

Any sample, however, should be within one per cent. of the true ash, in which case the probable error would be 0.18 per cent.; and from Fig. 3 the "size-weight" percentage should be 0.01 per cent. in order that this degree of accuracy would be assured. The "size-weight" percentage is based

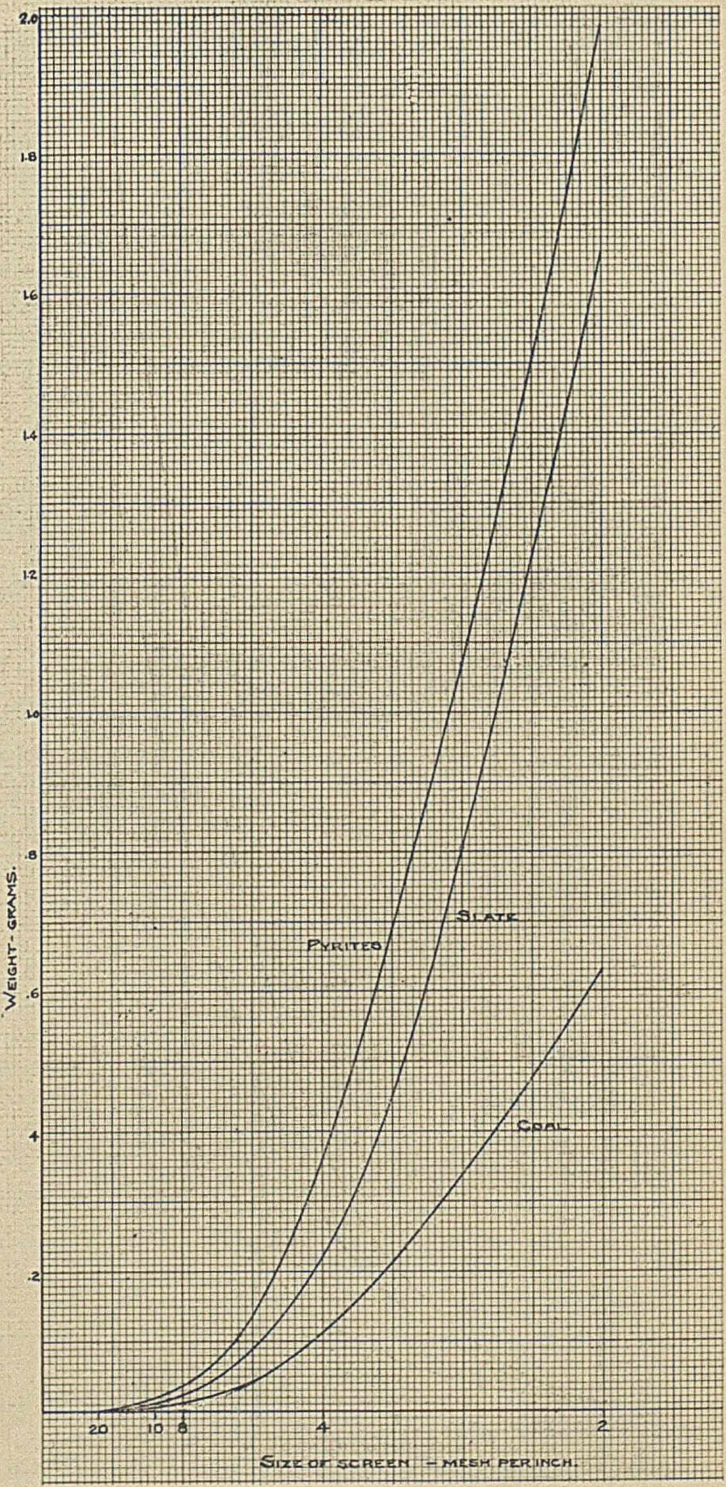


Fig. 9.

on the sample about to be divided, and the same piece would be double this percentage of one-half of the divided sample; so that in taking the original sample the largest piece need be only 0.02 per cent. of the total and still be within the required limit of error.

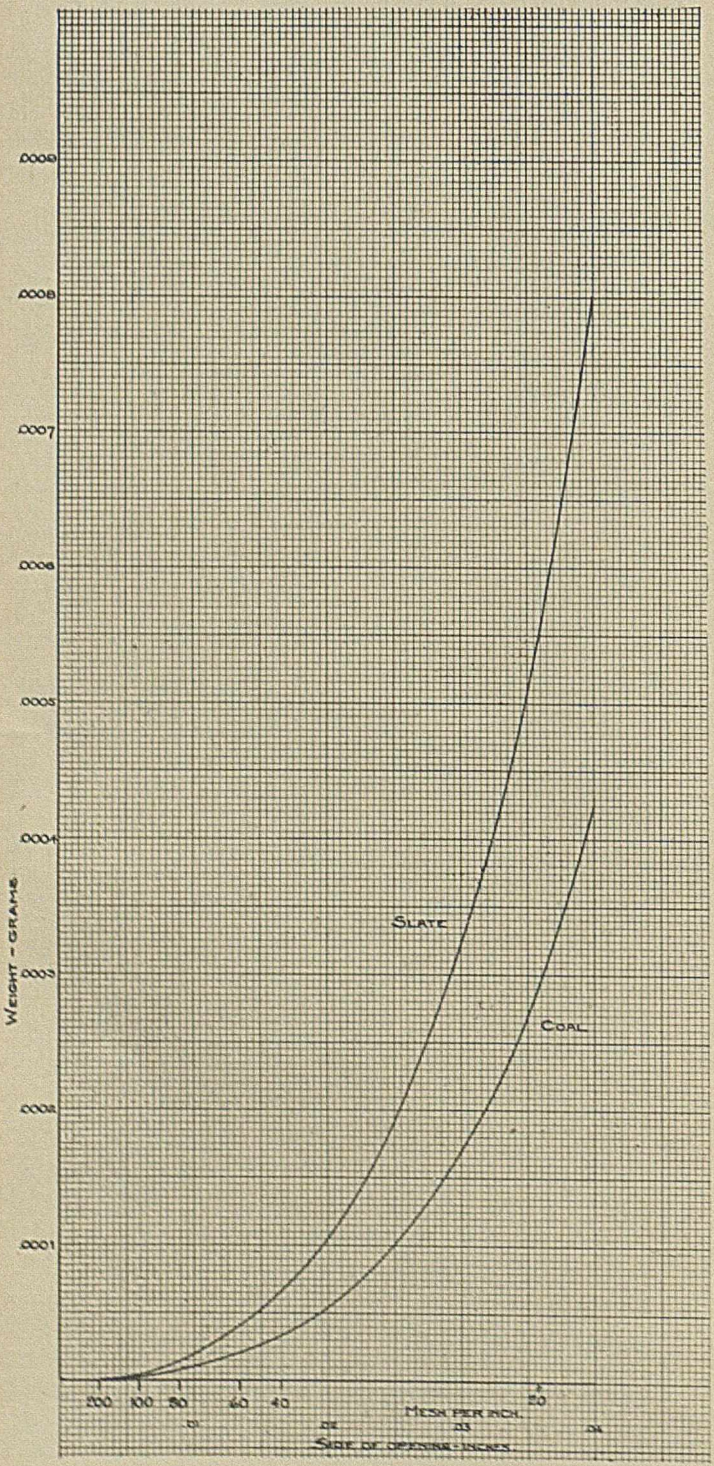


Fig. 10.

TABLE V.

(*a*) Size of slate contained in coal and amount of original sample required to insure the error of sampling being less than 1 per cent. ash.

Size of slate, inches.	Weight largest piece of slate. Lbs.	Original sample should weigh. Lbs.
4	6.7	39,000
3	2.5	12,500
2	0.75	3,800
1½	0.38	1,900
1¼	0.24	1,200
1	0.12	600
¾	0.046	230
½	0.018	90

(*b*) Size to which slate and coal should be broken before quartering or dividing samples of various weights:

Weight of sample to be divided. Lbs.	Should be broken to inches.
7500	2
3800	1½
1200	1
460	¾
180	½
40	2-mesh
5	4 "
½	8 "
¼	10 "

(*c*) Limit beyond which sample should not be divided when crushed to different sizes in laboratory:

Size of coal. Mesh.	Should not be divided to less than Gram.	
2	8300	
4	1100	
8	120	Should be pulverized to at least 60-mesh
10	55	
20	3	

Table V gives data which will be found very useful as a guide in sampling coal under various conditions. From this table, which is based on a "size-weight" percentage of 0.01 and coal containing 5 per cent. ash as slate and free impurities, the importance of taking a large initial sample will be realized. The size of the original sample is undoubtedly the weakest point in the existing methods of sampling coal, and the absurdity of a 100-pound sample is apparent. In collecting the original sample, the coal should be taken in fairly large increments. The taking of handfuls or small shovelfuls of run of mine or large-size coal makes it practically impossible to obtain a really representative sample in the beginning. Increments of 15 to 20 pounds are none too small in order that the lumps and fine may be proportionally represented. However, unless the importance of the "size-weight" percentage is realized by the chemist, a sample—no matter how well taken from the original lot of coal—may be ruined by quartering to a smaller quantity than is proportional to the size to which it has been crushed.

The methods used by the average person in taking samples to be sent to commercial laboratories is sufficient explanation for the lack of confidence they have in the analysis as made by the chemist. While the complete history of the sample is seldom available to the chemist, yet he can determine the relation between the size and weight of the sample as it comes to him. From a large number of such samples received during the past year, there have been less than 3 per cent. of them where the largest pieces were 0.02 per cent. or less of the total. Table VI gives a classification of the condition of commercial samples as received from various sources.

TABLE VI.

"Size-weight" per cent. of sample as received.	Per cent of samples received from outside parties.
Less than 0.1	12
Between 0.1 and 0.5	13
" 0.5 " 1.0	20
" 1.0 " 2.0	24
" 2.0 " 3.0	13
" 3.0 " 4.0	3
" 4.0 " 13.6	15

Oftentimes some of the above samples have been half of a sample which has been divided between two laboratories, and when the results show a wide variation, as they naturally would, it is impossible to convince the person sending the samples that the fault is more likely to be in the dividing of the sample than in the analytical work. For his own protection, the chemist should determine the exact condition of any such sample when received at his laboratory and report same on the certificate with the analysis. This relieves him of much controversy and has been found to be a very effective method of showing the public the folly of paying $5 to $25 for the analysis of a 25-cent sample.

SUMMARY.

1. The present methods of sampling coal are almost as various as is the number of persons taking such samples, with the result that errors of 3 to 5 per cent. in ash are of ordinary occurrence and extreme errors of 15 to 30 per cent. are frequently encountered.

2. The method of taking a sample must be specified in every detail in contracts between the two parties buying and selling coal on an analysis and heat unit basis before satisfaction, and justice will be given to both parties.

3. The errors in sampling evidently follow the laws of chance when any one method is used, and from the calculated probable errors a comparison of the accuracy of different methods can be made. See Table I and Fig. 1.

4. The most important factor causing errors in sampling is the size and number of pieces of slate, pyrites, bone and other impurities in proportion to the weight of the sample when originally taken or when divided or quartered. This ratio has been termed the "size-weight" percentage, and the results of 193 analyses from different samples of the same coal show that there is a decided increase in the percentage of error in ash with the larger "size-weight" percentage. See Table II and Figs. 2 and 3.

5. From over 1300 divisions of samples containing marked pieces of coal it was found that the variation in the number of pieces in each half varied according to the law of probability, and that the variation increased with the number of pieces in the sample divided. From this data an empirical formula was deduced, which showed that the error in dividing a sample is a function of the percentage of ash due to slate, etc., and the "size-weight" percentage. The error calculated by means of this formula checked remarkably close with the results of actual analyses. See Table III and Figs. 4, 5 and 7.

6. The average of any number of samples reduces the error in proportion to the square root of the number averaged, providing all samples have the same "size-weight" percentage; or, if a certain degree of accuracy is required a less aggregate weight of original sample need be taken when two or more samples are obtained and averaged, see Fig. 6.

7. The weight of various sizes of slate and coal was determined by weighing up a large number of each, passing through a screen or sieve of one size and over the one of the next smaller size. The sizes ranged from 4 inches bar screen to an 80-mesh sieve. This data is given in Table IV and Figs. 8, 9 and 10.

8. A knowledge of the relation existing between the "size-weight" percentage, amount of ash due to slate and the possible error, makes it feasible to intelligently take a sample of any coal with an assurance that the results will be accurate within certain limits. See Table V, also Figs. 2, 3, 5 and 6.

9. The deductions from the experiments and data reported in this paper explain the cause of

many samples varying so widely in quality, even though taken from the same car or lot of coal, the most common fault being that the original samples taken have been too small. The absolute necessity of taking large samples will lead to mechanical or automatic sampling in many places.

10. The chances of error in the chemical analysis are insignificant when compared with the errors in the existing methods of sampling coal. The chemist should be responsible for the sampling in every possible instance.

11. Almost every one who has given any consideration to the sampling of coal or other similar material, has said that a large sample should be taken and that it should be broken or crushed fine before the successive quarterings. How large, and how fine, have been left to the judgment of the individual taking the sample, and it is the object of this paper to answer these questions with some degree of accuracy. As this data is of a preliminary nature, it is hoped that others will not only verify these results, but add to them, in order that the sampling of coal and other material may be reduced to a more scientific basis.

THE COMMERCIAL MANUFACTURE OF AMORPHOUS CALCIUM PHOSPHIDE.

By Charles E. Munroe.

Received January 8, 1909.

In 1891, I was directed to prepare calcium phosphide on a limited commercial scale. The Howell Automobile Torpedo had then been developed to such an extent that it had been tentatively accepted as a service weapon, and officers and seamen gunners of the Navy were being trained, by target practice, to become skilled in its use. As devised, this torpedo was so constructed that, as ready for service, it would, when stationary or running at low speed, fill with water to such an extent as to sink the torpedo and drown its priming charge and detonator so as to render them harmless, for, if in action a live torpedo failed to hit the enemy at which it was aimed and became vagrant, it would prove a menace to friend as well as foe. Although, in target practice, the war heads were not used it was necessary that otherwise the torpedo should be arranged and should operate as in action. But the sinking of the torpedoes at the end of their runs was, owing to the difficulty of locating them in deep water, a serious impediment to practice, as they were expensive. Hence, it was most desirable to recover them. Various devices were invented with the object of surmounting this difficulty. The one which proved most efficient was the inserting in the nose of the torpedo of a can of calcium phosphide, with holes punctured in the top of the can, for as the torpedo took the water the reaction of the phosphide with the water entering the can gave rise to a cloud of smoke, which was visible by day, and a faintly luminous flame, which was visible by night, and these phenomena enabled one not only to easily follow the course of the torpedo during its run but also to locate it with great accuracy after it had sunk. It thus became easily possible to recover the torpedo by grappling.

As there was, previous to the erection of this furnace, no manufacture, so far as was known, of calcium phosphide in this country, it was imported from Europe at a cost of $2.25 per pound. Quite apart from the high cost a difficulty was met in importing it, owing to the custom's officers, who insisted on breaking open the sealed packages and inspecting their contents greatly to the disgust of both the inspecting officers and the importers, though for different reasons, and to the damage of the material. For these various reasons, I was directed to manufacture it and the material was then supplied by the Government to the company furnishing the torpedoes.

From the literature it was learned that Dumas[1] placed fragments of quicklime in a retort set horizontally on a grate and disposed phosphorus at the extremity of the neck, which was closed with a stopper. When the lime had been heated to redness the phosphorus was passed over it in the state of a vapor, thus producing calcium phosphide.

Thenard[2] made use of a clay crucible, of about 1 liter capacity, pierced at the bottom by a hole 1 to 2 cm. in diameter. The neck of a glass flask, of about 250 cc. capacity was passed through the hole in the bottom of the crucible and luted in place, and the whole arranged in a furnace with a double grate so that the flask hung suspended above the lower grate. The flask was filled with phosphorus and the crucible above it with quicklime. When the lime had been heated to redness, heat was applied to the glass flask and thus the vapors of the phosphorus were passed through and about the lime. The operation lasted 30 to 40 minutes and about 500 to 600 grams of the phosphide were obtained.

---

[1] *Ann. Chim. Phys.* [2], **33**, 363 (1826).

[2] *Ibid.* [4], **14**, 12 (1845).

A preferred form of crucible recommended by Thenard, was one which was divided into two parts by an earthenware grid, the lower section, about 1/3 to 1/4 the height of the crucible, being used to hold the phosphorus. Such a crucible is pictured by Pelouze et Fremy[1] but they effected the division by placing a small crucible inside the large one and placing the grid on the small one. However, they continued the use of a furnace with two grates.

Gattermann and Haussknect[2] made use of a covered Hessian crucible, resting on the grate of a crucible furnace, in which to put the lime, and when the latter had been brought to a glow, they dropped in sticks of phosphorus by means of a vertical iron tube which passed through the covers of the furnace and crucible, and reached to within 2 cm. of the bottom of the crucible, the upper end of the iron tube having a glass tube fitted into it by means of an asbestos packing.[3] They used about 500 grams of lime and added to it 350 to 400 grams of phosphorus.

This was the state of the art as I found it at the time of beginning this work. Experiments were made with glass and earthenware vessels, but they proved unsatisfactory. Tests were then made with an iron tube, butt-welded at one end, and this resisted the action of molten and vaporized phosphorus so well that crucibles were made from 5 inch wrought iron tubes with welded bottoms, their dimensions being as follows: depth inside 6 inches, outside 7 inches, diameter at top, inside 5 inches, outside 5 5/7 inches. They were provided with grooved lids, 1/4 inch thick and 6 inches outside diameter, which were fitted with asbestos gaskets. A slotted lug was welded on each side of each crucible and a wedge-shaped iron key was provided so that by driving it through the slots the cover of a crucible was easily fastened on tightly and firmly. Through one side of the cover a wrought iron pipe, 12 inches long, and 1 inch in diameter, was swedged, the lower end of the pipe being flush with the inside face of the crucible cover. This pipe was provided with a cap 4 inches long and 1 1/4 inches inside diameter. Three of these pots are shown in the photograph. They held about 1500 grams of lime each. They were heated with coal in an ordinary square crucible furnace.

The lime used was "black marble lime, wood-burned." It was of high quality and it was broken into lumps 1 to 1 1/2 inches in diameter. The crucible was charged with the lime, the cover put on and keyed, and the whole lifted by the feed pipe and embedded in the fire in the furnace. When the crucible had become heated to between a dark and cherry redness the cap was lifted from the feed pipe, sticks of dry phosphorus were dropped in and the cap replaced. So soon as fuming ceased, which with the right proportion of phosphorus was almost immediately, the crucible was lifted from the furnace and allowed to cool with the cap on so as to prevent air from reaching the phosphide in any considerable volume during the cooling process. The operation required about 1/2 hour. With 1400 grams of lime to the pot and 200 grams of phosphorus added about 1600 grams of phosphide were obtained. With a larger quantity of phosphorus the yield was no greater as the excess of phosphorus fumed off. Using four pots, an unskilled workman, after short practice, turned out 28 pounds per short day. The cost, counting labor and materials, was 20 cents per pound. The same pots were used, without repairs, for some eight years.

The phosphide produced in this way was very active, giving off spontaneously inflammable hydrogen phosphide promptly on contact with water. A perforated tin container holding a pound of this amorphous calcium phosphide, when submerged in 18 feet of sea water, gave a flame, which rose some two feet above the surface of the water, and voluminous smoke, and it continued intermittently to give off flame and smoke for over five hours. It was found to be more satisfactory in its behavior than the imported article. Portions of it made 17 years ago seem to-day to be as active as when freshly manufactured.

Since this work was accomplished, H. Moissan[1] has obtained calcium phosphide by reducing tricalcium phosphate with carbon in an electric furnace, and also by the action of phosphorus on metallic calcium. The product thus obtained was crystalline and it reacted with water to produce phosphine (H_3P), which was not spontaneously inflammable, while the product obtained by the action of phosphorus on lime, after the manner described above, is amorphous and reacts with water to produce hydraphosphine (H_4P_2), to which is attributed its property of generating a spon-

---

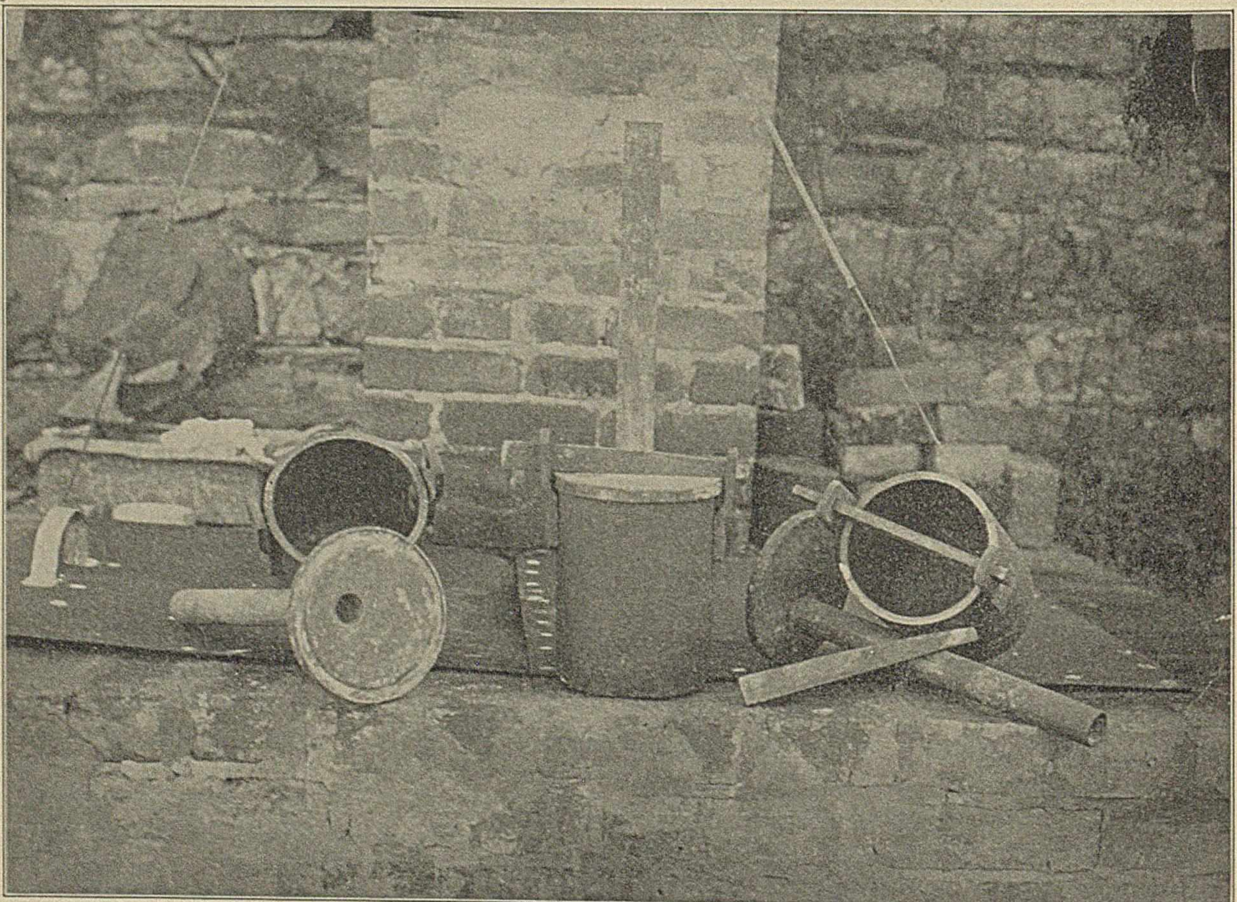
[1] *Traite de chimie*, **2**, 558 (1865).

[2] *Berichte*, page 1175 (1890).

[3] This apparatus, except for the glass extension tube, is practically identical with that of M. Silas shown in Mémoires Militaires et Scientifique, 5 [XXXII], pp. 9, 1877.

[1] *Comptes rendus*, **128**, 787 (1898).

taneously inflammable product. E. Renault[1] has also obtained the crystalline calcium phosphide in the electric furnace, carrying out the operation in both iron and carbon crucibles, but he states that under certain circumstances he obtained, from the tricalcium phosphate, a phosphide which yielded both H_3P and H_4P_2. This is quite to be expected for I recall that when operating the plant at Spray, N. C., in 1895, for the production of calcium carbide I noted the presence of calcium phosphide in the product, due to reduction of calcium phosphate in the lime, and I attributed the spontaneous inflammation of the acetylene, and other gases, produced by the reaction of the carbide with water, to the simultaneous formation of hydraphosphine.



No doubt if there were any considerable demand for amorphous calcium phosphide it could be most economically met by electric furnace methods, but as the uses for this material are limited the method I have described is sufficient and commends itself for its simplicity, low cost of installation, the wide accessibility of the apparatus and materials used, and the ease with which it can be carried out by unskilled labor.

With this manufacture in successful operation in the Navy there was naturally a desire to further utilize the product in the solution of other naval problems. It had then been recognized that the electric search lights installed in the Navy were not an unmixed blessing for while they might be used in locating the enemy they never failed also when in use to locate the vessel that carried them, and therefore they failed in enabling one to take the enemy by surprise. It was evident that this defect could be remedied by means of a self-igniting light which could be thrown, say in a shell from a gun, among the enemy. With this in view, experiments were made with the product from this furnace but though perfectly clear, yet quite dark, nights were chosen for the tests, when the vessels in the harbor were at anchor with some of their sails spread, and the seamen

---

[1] *Comptes rendus*, **128**, 883–884 (1898).

gunners who laid the test shells were clad in white duck working suits, yet the intrinsic luminosity of the phosphide flame was so slight that no useful result was obtained. Quite recently, however, according to reports, this result has been successfully accomplished and we find in calcium carbide, mixed with calcium phosphide, a material which, when thrown to a distance, will, when alighting in water, produce a self-igniting flame which has a high intrinsic illuminating power.

But the economic use to which this material has been longest put and even to-day, probably, the chief use to which it is put, is as an attachment to life-saving emergency buoys which are carried aboard passenger vessels in locations from which they may be readily thrown at the call of "Man Overboard," for though the flame given by the phosphide is dim yet it is often sufficient to enable the person struggling in the water to locate the buoy. This application is described in great detail in De l'application de phosphure de calcium a l'appareil éclairant des bouées de sauvetage,[1] and it therefore will not be enlarged upon here.

THE GEORGE WASHINGTON UNIVERSITY.

---

[1] Mémoires Militaires et Scientifiques, 1878, XXXII.

THE TEMPERATURE OF THE LEAD BUTTON IN CUPELLATION.

BY ROBERT H. BRADFORD, PH.D., Prof. of Metallurgy, State School of Mines, University of Utah.

Received December 24, 1908.

Excellent work with the assay furnace was done by early investigators. They had no means of ascertaining exact temperatures, and hence in their writings they attempt to describe the desired temperatures by the appearance of the objects to the eye, using such color terms as would most nearly apply to the degree of heat under consideration. They recognized the importance of employing proper muffle temperatures in cupellation, as is indicated by the following careful description of the same given by an early text-book on assaying:[2] "When the interior of the muffle is reddish white the matters to be cupelled may be introduced. When the cupels are filled, the furnace is closed, either by the door or by pieces of lighted fuel, so that the fused metals may become of the same temperature as the muffle. When this point has been gained, air is allowed to pass into the furnace; the metallic bath is then in the state termed 'uncovered;' that is, it presents a convex surface, very smooth, and without slag. When the air comes in contact with it, it becomes very lustrous, and is covered with luminous and iridescent patches, which move on the surface, and are thrown toward the sides. These spots are occasioned by the fused oxide of lead which is continually forming, and which, covering the bath with a very thin coating of variable thickness, presents the phenomenon of colored rings.

"As silver is sensibly volatile, it is essential, in order that the smallest possible quantity be lost, to make the cupellation at as low a temperature as may be. On the other hand, the heat ought to be sufficiently great, so that the litharge may be well fused and absorbed by the cupel.

"Experience has proved that the heat is too great when the cupels are whitish, and the metallic matter they contain can scarcely be seen, and when the fume is scarcely visible and rises rapidly to the arch of the muffle. On the contrary, the heat is not strong enough when the smoke is thick and heavy, falling in the muffle, and when the litharge can be seen not liquid enough to be absorbed, forming lumps and scales about the assay. When the degree of heat is suitable the cupel is red, and the fused metal very luminous and clear."

Modern texts are less elaborate in their descriptions, yet they lay especial stress on the importance of maintaining proper temperatures in the assay muffle.

The modern pyrometer, for accurately measuring high temperatures, has recently come to fill a long-felt want in many metallurgical operations. Since the need of employing definite temperatures in assaying is generally recognized, one would expect that the pyrometer would be employed more commonly in the muffle, and greater accuracy observed in stating degrees of temperature. It is gratifying to see some recent magazine articles and texts on assaying employing exact figures for representing temperatures required for the various operations in the furnace.

It is difficult to reconcile some explanations of cupellation phenomena, based on pyrometer readings, with certain well established facts. The lack of sufficient exact data may account for some apparent discrepancies, as the following instance will show. One very recent manual of assaying[1] in describing the process of cupellation says: "Little flakes of PbO form on the surface of the

---

[2] John Mitchell: "Manual of Practical Assaying," 2nd edition, p. 368-370 (1868).

[1] C. H. Fulton: "Manual of Fire Assaying," 1907, pages 70, 71.

molten lead and slide down the convex surface of the button, and are absorbed by the porous mass of the cupel. If the temperature of the cupellation is between 700 and 750° C. as it should be, this litharge is solid, as litharge melts at 906° C." The same author says further that the actual temperature of cupellation has never been determined, and that a determination of the temperature of the cupelling lead will, in his opinion, very much modify the present theory.

If it is necessary as stated by Mitchell to have the driving lead button constantly hot enough to keep the litharge in a molten condition, in order that the oxide shall be absorbed by the cupel, then the temperature of 700 to 750° C. as used by Fulton cannot be high enough, if applied to the button, because litharge freezes at 906° C.

Since it is necessary to have a draught of air through the muffle in order that oxygen may be supplied to the cupelling button and since the air is cool as it enters the muffle, the temperature of the air above the button is considerably lower than that of the muffle itself or of the cupel interior, and very much lower than that of the cupelling metal. In previous magazine articles and text-book descriptions the recorded temperatures are apparently those of the surrounding atmosphere rather than the lead button, and very little information is on record concerning the temperature of the driving button.

It was with the object in view of determining the minimum temperature of a cupelling button, that the series of experiments herein reported were undertaken. The instrument used in the experiments was the Le Chatelier thermo-electric pyrometer, illustrated in Fig. 1. It consists of a platinum wire and a 90% platinum 10% rhodium wire twisted or soldered together, forming a thermo-electric couple, the loose ends of the wires being attached by means of copper leads to a galvanometer of high resistance. The cool ends of the wires were kept at a known temperature by being immersed in water along with a thermometer. The twisted junction of the wires was readily placed in any position in the muffle. In order to calibrate the instrument the deflections and cold junction temperatures were taken with the twisted junction at certain known temperatures. A curve was then plotted, showing the relationship between deflection and temperature. From such a curve any deflection of the galvanometer was interpreted in degrees Centigrade.

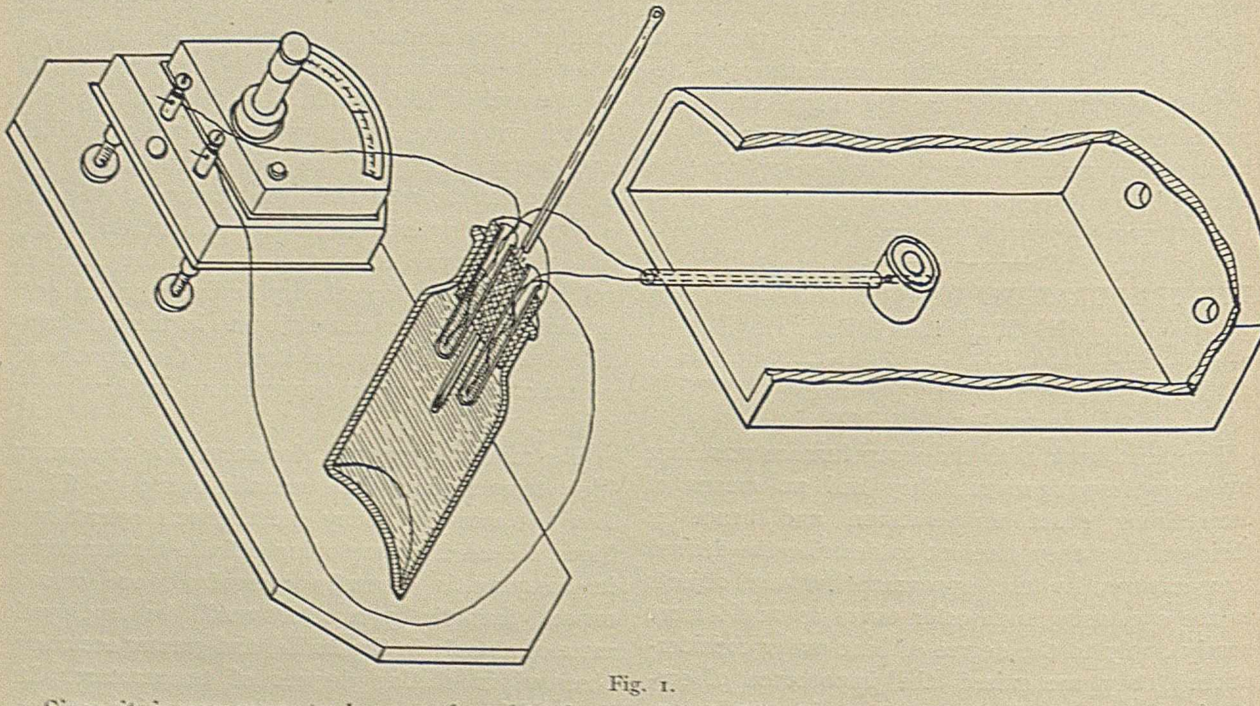


Fig. 1.

When the bare pyrometer wires were brought in direct contact with the driving lead they were immediately alloyed and fused. To prevent this destruction of the wires when employed with molten lead the couple was protected at the hot junction by a thin coating (see Fig. 2) of fire-clay. This coating was made on the spot from moistened raw material. It was made thin so that it would offer but little interference with the surface oxidation of the lead button. The temperature of the heating button was determined by placing the protected couple well within the molten lead, then reading the galvanometer and the cold junction thermometer, and interpolating on the calibration curve to find the degrees Centigrade.

The minimum temperature of the lead button itself requisite for starting cupellation was investigated by introducing buttons into cupels placed at varying distances from the front of the muffle and hence at varying temperatures. Pyrometer readings of the heating metal and the cupel interior were carefully taken in each instance.

Before driving commenced it was found that the temperature of the molten metal rose to 900° C. or above—approximately to that of melting litharge. If the heat conducted from the cupel or radiated from the muffle was not sufficient to raise the temperature to that of melting litharge the button froze and would not "uncover."

In order to confirm the temperature readings of the cupelling metal the degree of heat of the interior of the cupel was determined by boring a small hole into the dry cupel with a one-eighth inch drill bit, and inserting the junction of the pyrometer wires well within the cupel. Holes were drilled to points beyond the centers of the cupels, and were made at different levels in the different cupels, from directly underneath the bottom of the cup-shaped depression, to the bottom of the cupel directly above the supporting muffle. The method of getting the temperature of the cupel interior is illustrated in Fig. 2.

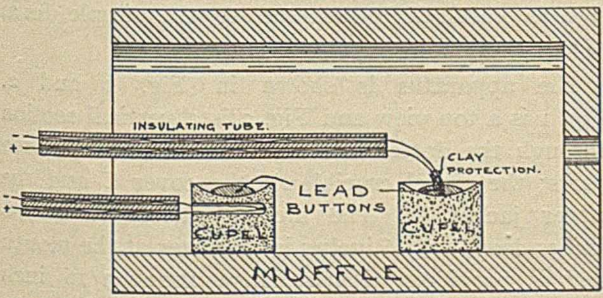


Fig. 2.

The temperature of the interior of the cupel was readily ascertained by the method previously described. In every case in which the cupel had been properly pre-heated beforehand, as is done in ordinary practice, the temperature directly under the button, although lowering as the lead melted, rose to 900° C. or above before cupellation commenced. Below this point and nearer the muffle the temperature was usually a few degrees higher when the button opened. The melting metal had taken its heat from the upper layers, cooling these more than those near the bottom.

The temperature within the cupel and near the molten lead remained at 906° C. or above so long as the button continued to drive. If the button froze the temperature of the interior rapidly lowered, showing that its heat had been, in great part, derived from the heat of combustion of the driving lead. Upon freezing, the temperature of the button rapidly declined because its heat of combustion had been cut off.

If the interior of the cupel was well below 906° C. when the button was introduced and the cupel and contents then pushed back into the muffle, the button began to drive when the heat radiated from the arched roof of the muffle gave the litharge coating of the lead a temperature of 906° C. The bare couple could be safely placed in contact with the litharge in the above instance. The button rapidly heated to a much higher temperature if left in this position as cupellation proceeded. An intensity of heat resulting from the above conditions is not safe to use, owing to the excessive losses in silver and gold. Good practice would require that the cupel and contents be drawn forward to a cooler part of the muffle.

The temperature favorable for the formation and retention of "feathers" of litharge was determined by placing the bare couple among the crystals as they formed around the button. The temperature of the forming crystals in every instance was below 906° C. as measured near the driving lead. So long as the button continued to drive, the cooler the surrounding air and cupel surface, the more favorable was the temperature for the formation of the litharge crystals. After the cupellation was finished these crystals could be heated to 850 to 890° C. for many minutes without showing any tendency to fuse as indicated by their sharp edges, and without volatilizing to any considerable extent. If their temperature was raised to the melting point of litharge they immediately fused and were absorbed by the bone ash cupel.

The results of these experiments seem to establish the following facts:

When once the lead commences to oxidize rapidly and the molten litharge escapes into the cupel, the temperature of the button immediately rises owing to the heat of combustion of the oxidizing lead, and the heat from this source is sufficient to keep the button driving, *i. e.*, to keep the litharge molten and thus in a condition to run down the sides of the button and in most part to be absorbed by the cupel—even though the cupel and contents are drawn forward to a cooler position. With a gentle draught through the muffle the temperature one-quarter inch above and near the front of the cupel may be maintained as stated by one text,[1] as low as 625–650° C. and cupellation continued, although the danger of freezing is very much lessened if the temperature is kept between 650° and 750°. So long as the heat of combustion of the driving button is able to maintain the temperature of the lead at that of melting litharge the cupellation proceeds. If the cupel is brought so near the mouth of the muffle that the transference of heat from the button is sufficiently rapid to overcome the heating effect of the combustion of the lead, the button cools; and when its temperature goes below the freezing point of molten litharge, it freezes, *i. e.*, solid litharge covers the molten lead and cupellation ceases.

If the surrounding air is kept well below the temperature of melting litharge, the surface of the cupel around the driving button becomes cooled below that temperature, and the molten lead oxide as it runs off the convex surface of the lead, solidifies, and crystals of litharge, or "feathers" form. These thin crystals volatilize slowly after forming, but do not disappear as a rule unless the temperature of the cupel surface is raised above 906° C. when they melt and are partly absorbed by the bone ash of the cupel.

UNIVERSITY OF UTAH,
SALT LAKE CITY.

[1] Richard W. Lodge: "Notes on Assaying" (1905), page 60.

A NICKEL CRUCIBLE FOR THE DETERMINATION OF CARBON IN STEEL.

BY HENRY E. K. RUPPEL.

Received November 27, 1908.

The high price of platinum crucibles for carbon determinations led the writer to seek some substitute to supplement the work of the Shimer crucible in regular use in his laboratory. Nickel naturally suggested itself for this purpose on account of its inexpensiveness, its high melting point and its resistance to oxidation. Crucibles of this material have been used for the past year for the determination of carbon in steel and the results, obtained both by direct combustion of the steel and by solution in potassium copper chloride, have been so satisfactory that it seemed advisable to publish them together with a description of the apparatus.

At the beginning, a word should be said in regard to the conditions under which such crucibles can be used to advantage. A conservative estimate of the life of these crucibles is about thirty to fifty determinations when the solution method is used. On account of the low cost of these crucibles they can be used to advantage in those analytical laboratories where the number of carbon determinations performed is limited and the high initial cost of platinum precludes its use, especially in college laboratories where a single crucible may serve the needs of an entire class. They may also find a place as supplementary crucibles in steel laboratories where carbon determinations are a matter of daily routine. Since such crucibles are used up in time, it is necessary to make them as simple as possible and to provide for a separate water jacket not directly attached to the crucible.

The principle of a water-cooled stopper for platinum crucibles to be used in the determination of carbon in steel was first published by P. W. Shimer[1] and several forms are at present on the market. Although the present paper is intended solely to show that satisfactory results can be obtained with a nickel crucible, nevertheless the writer feels justified in giving a somewhat detailed description of the cooling apparatus as the design is particularly adapted to the simple form of nickel crucible employed.

The apparatus is shown in Fig. 1 and 2. Fig. 1 is a top view and Fig. 2 is a vertical section through the dotted line $x\ y$. It consists of three parts; the nickel crucible *A*, the cover *C* and the water jacket *B*. The nickel crucible is 1 7/8 inches deep, 1 9/16 inches in diameter at the mouth and is provided with a flange about 9/16 inch wide. These crucibles are now supplied to us in one piece, although we have used ordinary nickel crucibles to which a brass rim was soldered with silver solder. The area of the bottom was made comparatively large so that all the carbon might be in contact with the bottom of the crucible, even though the asbestos pad was made slightly large as sometimes happens. When this occurs with the platinum crucible, in which the

---

[1] "Carbon Combustion in a Platinum Crucible," *J. Am. Chem. Soc.*, **21**, 7 (1899).

area of the bottom is small, the asbestos pad as well as the asbestos used to wipe out the funnel, extends up the sides of the crucible. In such cases we have sometimes encountered unburnt carbon. This difficulty is further obviated by using a deep crucible which can be heated to a considerable distance from the bottom. It might be stated against this that an asbestos pad made in an ordinary funnel is not too large to fit comfortably in the bottom of the regular platinum crucible. On the other hand, the filtration becomes slower as the area of the pad decreases. We use a special funnel which will be described later and which offers a comparatively large area for filtration.

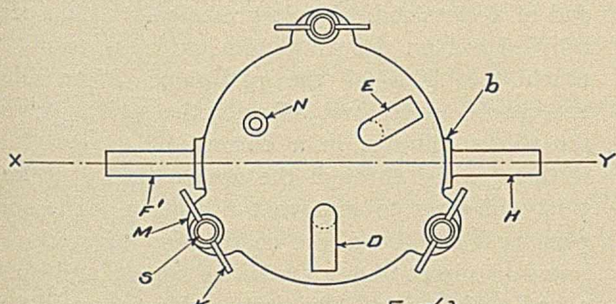


Fig. 1.

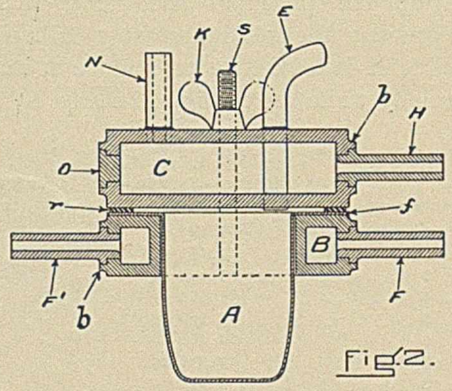


Fig. 2.

The crucible rests upon the water jacket *B*, which consists of an annular machined bronze casting 2 3/4 inches outside diameter, 1 9/16 inches inside diameter and 5/8 inch high with walls about 1/8 inch thick. This jacket is provided with two 3/8 inch holes, each of which is surrounded by a boss *b*. The holes are slightly counter-bored and the collars of the brass tubes *F* and *F'* are inserted and fastened with ordinary solder. The brass tubes are all turned from solid stock and are 1/4 inch outside diameter with 1/8 inch holes. Three brass rods *S*, 3/16 inch in diameter and threaded at each end, are permanently soldered to the ears, corresponding to *M* (Fig. 1) with which the jacket is provided, and pass through holes in the corresponding parts of the cover which is keyed down by means of the winged nuts *K*.

The cover *C* is also cast of bronze and machined. It is 2 3/4 inches in diameter, 7/8 inch high with walls about 1/8 inch thick. It is supplied with three semi-circular ears M, 3/4 inch in diameter, through which holes are drilled to admit the rods *S*. In making these castings it was necessary to provide two 3/8 inch holes through which the anchors of the core might be passed. One of these holes is closed by the brass plug *O* (Fig. 2), the other is counter-bored and the collar of the brass tube *H* inserted and soldered. The joint between the crucible and the cover is made tight by means of a rubber gasket *r* such as is used for ordinary glass preserve jars. The air enters the crucible by tube *D* (Fig. 1), which extends 1 1/4 inches below the cover, and leaves by the tube *E*. The top ends of these tubes were bent in an ordinary blast lamp after filling them with sand. When in use tubes *F* and *H* are connected by means of a rubber tube. The water, supplied by an eighth-inch tap, enters the jacket at *F'* and leaves the cover through the tube *N*.

The cost of the nickel crucible is one dollar and ten cents. The cost of the completed jacket and cover, exclusive of patterns, is about seven dollars.

The determinations given in Table I were made by dissolving the steel in double potassium copper chloride solution, filtering on ignited asbestos, and burning in a current of air. The crucible was supported through a hole in a piece of asbestos board 1/4 inch in thickness and was heated to bright redness for 3/8 to 1/2 inch from the bottom by mean of a small upright blast lamp. The time of heating was half an hour for steel and three-quarters of an hour for cast iron. Instead of using an ordinary funnel for filtering the carbon, we employed a modified form of the funnel generally used to support a Gooch crucible, in which the height of the funnel above the stem is about one inch. A copper wire is made into a close spiral at one end and passed through the stem of the funnel, loose asbestos is added, followed by a suspension of asbestos in water. After applying the suction, the asbestos is gently tamped down with the flattened end of a glass stirring rod. The finished pad is from 3/4 to one inch in diameter and about 1/4 inch thick. This pad is placed in the crucible with the carbon down and the crucible is filled loosely with ignited asbestos. The bottom is protected by means of sheet platinum shaped so as to fit snugly inside and to extend about 1/4 inch up the sides.

The combustion train consists of a small preheating furnace containing a twelve-inch porcelain tube filled with copper oxide, followed by a Liebig bulb containing potassium hydroxide solution and a small guard tube. Next in order comes the crucible, a small furnace with a twelve-inch porcelain tube containing copper oxide, a tube of beads moistened with water, a Marchand drying tube and the Geissler bulb and guard tube. The writer prefers the small porcelain tubes to the brass tubes frequently used and employs them with the platinum crucible. The above procedure is the one regularly used on carbon steels in his laboratory.

In Table I, column 1, are given some of the results obtained with the nickel crucible, column 2 contains the corresponding results obtained with the platinum crucible, except in the case of cast iron, in which the results given in column 2 are the average of the results of various chemists on standard foundry iron. The results are representative and were not selected on account of their agreement. Blanks can be easily obtained showing an increase in weight of less than 0.5 milligram and the results agree closely with those obtained by means of the platinum crucible. Many of the samples were mixtures from several rolls so that some of the variations may be due to imperfect mixing.

TABLE I.

Sample.	1. Nickel crucible. Per cent.	2. Platinum crucible. Per cent.
1	1.119	1.114
2	1.082	1.081
3	1.185	1.187
4	1.132	1.115
5	1.023	1.022
6	1.090	0.070
7	1.118	1.098
8	1.127	1.141
9	1.074	1.077
10	1.108	1.084
11	1.052	1.053
12	1.133	1.132
13	1.086	1.087
14	0.936	0.936
15	1.163	1.163
16 Cast iron A	3.63	3.67
17 Cast iron D	3.01	3.00

	Mg.
Blanks (15 minutes)	+0.3
" "	0.0
" "	+0.1
" "	+0.0
" (30 minutes)	+0.4
" "	+0.6
	+0.3

In Table II, column 1, are given the results obtained by direct combustion of steel in a current of oxygen. In column 2 are given the corresponding results found by dissolving the steel in potassium copper chloride and burning the carbon in air.

To prevent the caked iron oxide from adhering to the platinum shell, which protects the bottom of the crucible, it is filled to a depth of about 1/16 to 1/8 inch with ignited aluminum oxide, which is tamped down in the middle with the flattened end of a glass rod. Three grams of steel are carefully placed on the aluminum oxide so as not to touch the sides of the platinum. After filling the crucible with loose asbestos the steel is burned for thirty minutes in a current of oxygen. Air is then passed through the apparatus for at least thirty minutes to remove the oxygen. The samples used for the direct combustion of steel consisted of pieces about 1/16 inch square and about 0.006 inch thick. The iron oxide is readily removed at the end of the determination by withdrawing the platinum shell from the crucible. To prevent the platinum from adhering to the crucible it is advisable to remove it occasionally, especially while the crucible is new.

TABLE II.

Sample.	1. Direct combustion. Per cent.	2. Solution method. Per cent.
1	1.123	1.137
2	1.151	1.144
3	1.144	1.133
4	1.176	1.163
5	1.153	1.163
6	1.109	1.114
7	0.841	0.834
8	1.261	1.276

The following figures give some of the blanks obtained by passing oxygen through the apparatus for thirty minutes and expelling the oxygen by forcing air through for the same time:

+0.9 mg., +0.8 mg., +1.1 mg., +0.7 mg., +0.4 mg., +0.3 mg.

When the direct combustions in oxygen were first tried, the air was allowed to pass through the combustion train for only fifteen minutes to expel the oxygen, but the results obtained in this way were always too high. That this was mainly due to the presence of oxygen in the Geissler bulb is shown by the following experiments. After attaching the weighed Geissler bulb to the train, oxygen was passed through at the rate of three bubbles per second for thirty minutes, followed by air at the same rate for fifteen minutes. The Geissler bulb was then detached and weighed, after standing in the balance case for fifteen minutes. The increase in weight was 7.4 mg. in one experiment and 5.2 mg. in another. While the bulb was in the balance case, air was passed through the rest of the train in order to remove any oxygen which might have been present. The Geissler bulb was again attached and air allowed to bubble through for fifteen minutes more. After allowing the bulb to remain in the balance case for the usual time, its weight was found to be practically the same as at the beginning of the experiment. This would seem to indicate clearly that the oxygen is not completely removed by passing air through the apparatus for fifteen minutes.

Many chemists do not expel the oxygen in this way but prefer to weigh the bulb containing oxygen instead of air; this would, of course, shorten the time considerably.

In conclusion, the writer desires to express his sincerest thanks to Professor Henry Fay for his interest and to his assistant, Miss Caroline E. Shute, by whom many of the determinations were made.

BOSTON, MASS.

DETERMINATION OF IRON IN BRASSES AND BRONZES.

By I. M. Bregowsky and L. W. Spring.

Received January 15, 1909.

At various times we have had occasion to compare our analyses of brasses and bronzes with those of other chemists, on the same samples. It has practically always been noticeable that while the other chemists check us within fairly close limits on tin, lead, copper and zinc—the main constituents of brass, they nearly always either fail to mention the iron content, or report it as "a trace" or give some such value as 0.06 per cent., or possibly 0.20 per cent.—seldom more. With samples on which our determinations of iron have shown the iron content to be far above "a trace" in any sense of the term, the chemist of a well-known railroad made no mention of iron in his report, and a prominent commercial chemist reported "a little iron present" on one of the two samples, and did not mention iron in the report on the other. With another sample on which we got 0.75 per cent. iron, a metal refiner offered to wager us a snug sum that there was not over 0.006 per cent. of iron present. His chemist had reported a content of 0.003 per cent.

Now iron in the amounts present in the commercial copper alloys may or may not be considered to be a factor of importance in the usefulness or working of the alloy, yet the chemist should know what the iron content is, or at least know how to determine it when he wants it. There is little that is new in the method and many will say that such accuracy is unnecessary. However, when occasion requires some may profit by a knowledge of it.

Fresenius' "Quantitative Analysis," Cohn edition, 1904, p. 681, says that the stannic oxide obtained upon the ignition of the metastannic acid precipitate is impure, containing part of the iron, besides other impurities, and gives directions for separation. We have found, as a rule, approximately half of the iron content of the brass in the ignited stannic oxide, unless the iron in the alloy is high. The remainder will be found by precipitation with ammonia either before or after the copper is separated, if sufficient time be allowed for it to come down. Our procedure is as follows:

A gram of the magnetted borings is dissolved and run down in nitric acid (1.42), the metastannic acid filtered out as usual and ignited to stannic oxide in a clean weighed porcelain crucible. After weighing the stannic oxide, one gram of c. p. sodium carbonate and 0.25 gram of sublimed flowers of sulphur are added, the crucible covered and the mixture fused at a very low heat until the excess of sulphur is burned off. Crucible and cover, when cold, are immersed in 50 cc. of distilled water in a No. 1 beaker and warmed to disintegration of the melt. They are then rinsed and removed. If the solution is blue or green, it indicates dissolved iron which can be thrown out by addition of a little ammonium chloride, leaving the solution a light yellow. The ferrous sulphide is filtered out, rinsed with hydrogen sulphide water, dissolved from the filter, and the iron determined by titration or gravimetrically. The iron figured to ferric oxide should, of course, be deducted from the weight of the stannic oxide.

The lead and copper are removed and determined by any of the usual methods. The filtrate is brought to a boil, the iron oxidized with a few drops of nitric acid and an excess of ammonia added. The solution is kept just at the boiling point for an hour or so. A bubble going up occasionally does no harm, but hard boiling does not allow the iron hydroxide to settle and undoubtedly has led many to believe that none was present. In an hour, the bottom of the beaker will show a considerable precipitate of iron hydroxide. It is filtered out, washed well with hot water, and determined volumetrically or gravimetrically. Blanks should be run on all chemicals used and their iron content allowed for.

We often analyze magnetted drillings from brasses from many sources and seldom find one with an iron content of less than 0.40 per cent., and many run to 0.90 per cent. and above. Below are given some of our analyses showing the total iron found and the relative amounts found in the stannic oxide and the main solution. Many of these samples have been analyzed by other chemists who, in their original analyses, found little or no iron, but who now by this method check the results here given.

BRASSES AND BRONZES (FINISHED GOODS) OF VARIOUS MANUFACTURERS.

	Sample No.						
	1. Per cent.	2. Per cent.	3. Per cent.	4. Per cent.	5. Per cent.	6. Per cent.	7. Per cent.
Iron from SnO_2	0.30	0.25	0.34	0.32	0.16	0.63	0.16
Iron from main solution	0.63	0.26	0.43	0.28	0.20	0.84	0.25
Total iron	0.93	0.51	0.77	0.60	0.36	1.47	0.41

	Sample No.					
	8. Per cent.	9. Per cent.	10. Per cent.	11. Per cent.	12. Per cent.	13. Per cent.
Iron from SnO_2	0.35	0.18	0.57	0.04	0.12	0.31
Iron from main solution	0.49	0.49	0.43	0.36	0.22	1.01
Total iron	0.84	0.67	1.00	0.40	0.34	1.32

Nos. 6 and 13 were not manganese bronzes but ordinary brasses.

Brass ingots we have received from refiners gave:

	Sample No.					
	14. Per cent.	15. Per cent.	16. Per cent.	17. Per cent.	18. Per cent.	19. Per cent.
Iron from SnO_2	0.39	0.37	0.24	0.24	0.22	0.33
Iron from main solution	0.34	0.33	0.28	0.98	0.49	0.32
Total iron	0.73	0.70	0.52	1.22	0.71	0.65

Samples of brass borings received from metal dealers, after removal of the free iron by magnet, gave:

	Sample No.				
	20. Per cent.	21. Per cent.	22. Per cent.	23. Per cent.	24. Per cent.
Iron from SnO_2	0.20	0.28	0.21	0.28	0.29
Iron from main solution	0.35	0.35	0.63	0.43	0.32
Total iron	0.55	0.63	0.84	0.71	0.61

These results are sufficient to show that the ordinary copper alloy contains considerably more than "a trace" of iron. How necessary it is in a technical analysis to determine this iron is, of course, for the individual chemist or metallurgist to say. They also show that the quantity of iron carried down by the tin is, in the main, proportional to the amount of tin in the alloy.

	Sample above No.						
	11.	12.	13.	8.	2.	10.	6.
Per cent. of tin in the alloy	1.13	2.28	3.34	8.20	6.04	5.97	5.67
Per cent. of iron in SnO_2	0.04	0.12	0.31	0.35	0.25	0.57	0.63

Undoubtedly the varying gelatinous condition of metastannic acid also influences the quantity of iron which is carried down.

CRANE CO. LABORATORY,
CHICAGO, January, 1909.

(CONTRIBUTION FROM THE CHEMICAL LABORATORY OF THE NORTH CAROLINA AGRICULTURAL EXPERIMENT STATION.)

THE COLORIMETRIC DETERMINATION OF NITRATES IN SOIL SOLUTIONS CONTAINING ORGANIC MATTER.

BY W. A. SYME.

Received December 9, 1908.

The accurate determination of nitrates in soil solutions by the colorimetric method with phenoldisulphonic acid and ammonia is a matter of some difficulty on account of the large number of interfering substances which may be present in the solution. Chlorides may cause a loss of nitric acid when the phenoldisulphonic acid is added to the residue left on evaporation. Iron salts affect the color. Nitrites interfere by producing a yellow color with the reagents like that caused by nitrates. These disturbing factors can be removed to a considerable extent—the chlorides by adding silver sulphate, the iron salts with sodium carbonate, and the nitrites may be determined separately by the Griess method.

The presence of organic matter in the soil extract causes trouble in several ways. In the first place, the solution may be so intensely colored that it cannot readily be decolorized with carbon black; secondly, the organic matter may reduce the nitrates, thereby causing a loss of nitric acid; and thirdly, the strong acid acting on the organic matter produces substances of foreign color which cannot be matched by the pure nitrate standards.

The object of the work reported in this paper was to see if potassium permanganate could be used to remove organic matter from soil extracts. The extracts were prepared for some work on nitrification now in progress in this laboratory, the method of preparation being as follows: 400 grams of soil were shaken four hours with 1200 cc. of water containing a little chloroform. The solutions were filtered through a porcelain tube.[1] The filtrates were strongly colored when the soils contained much organic matter. The solution from soil 1867, a soil from a compost heap and the one most favorable for the nitrification experiments, was very rich in organic matter and could not be satisfactorily clarified by shaking with carbon black. When this solution was evaporated to dryness, the residue treated with phenoldisulphonic acid and made alkaline with ammonia, the resulting solution had a brownish yellow color quite different from the clear lemon-yellow given by a pure nitrate solution.

The method for treating this solution, and others of the same kind, was as follows: Heat 50 cc. or any convenient portion to 60–70° and add 1 cc. of dilute sulphuric acid (1 : 5). Add from a burette a dilute solution of potassium permanganate (5 to 10 g. per liter) until in excess and heat fifteen minutes on a water-bath, adding more permanganate from time to time, if necessary, to have an excess. Remove the brown precipitate by filtering into an evaporating dish, make the filtrate slightly alkaline with sodium carbonate and evaporate to dryness on the water-bath. Add water to the residue and filter into a 50 cc. flask, wash the residue on the filter, and dilute to the mark. We now have 50 cc. of a colorless soil solution nearly free from organic matter. Nitrates can now be determined by the usual colorimetric method, remembering that if nitrite were present in the original solution, it would be oxidized to nitrate.

If a soil solution, after treatment in the way described, is evaporated and the color, developed in the usual way, is compared with that given by a portion of the same solution not so treated, the difference is very marked.

The evaporation of the soil solution with sodium carbonate removes manganese and iron, but enough nitrous acid may be taken up from the air to give an appreciable color with the nitrite reagent.

Carbonaceous matter was not entirely removed by the treatment with permanganate, though the coloring matter was destroyed and the residue left on evaporating the solution to dryness on the water-bath was colorless. The quantity of carbonaceous matter left by the permanganate was not enough to cause a serious trouble when the phenoldisulphonic acid was added. The effect of the permanganate treatment can be readily seen by evaporating equal volumes of the same soil solution with and without permanganate and comparing the residues as to color, quantity of organic matter and behavior on ignition.

The method was tested by having an associate prepare a number of solutions by adding known quantities of nitrate to soil extracts containing organic matter and giving them to the writer for analysis. The results were satisfactory in every case, and the method is now used in this laboratory.

Experiments were made to find out whether nitrate is formed by the oxidation of the organic matter by the permanganate. Soil extract 1931, containing orgainc matter but no nitrate or nitrite, was treated with permanganate. No nitrate was found after the treatment. A trace of nitrite from the air was found after evaporating the alkaline solution, but none was found in the acid solution before evaporation.

A liter of a colored soil extract was heated and acidified, permanganate was added in excess to the hot solution and the brown precipitate was filtered off. The filtrate was made alkaline with sodium carbonate, divided into two parts, and evaporated. In one part, nitrogen was determined by the Kjeldahl method and in the other part by the Kjeldahl method modified to include nitrates. The same quantity of nitrogen was found in the two portions, showing that there had been no nitrate formation. This experiment was repeated with a pasture soil containing 50 per cent. volatile matter chiefly in the form of cow manure and vegetable mould. The result was the same, namely, there was no nitrate formation from the action of the oxidizing agent on the organic matter. It is therefore safe to use permanganate for the purification of soil extracts.

WEST RALEIGH, N. C.

---

[1] Bulletin 31, Bureau of Soils, p. 12.

THE DETERMINATION OF BENZOIC ACID IN TOMATO CATSUP AND OTHER FOOD PRODUCTS.

By Rodney Mott West.

Received December 20, 1908.

The determination of benzoic acid is attended by peculiar difficulties in the case of many of the food products in which it is used as a preservative. Methods have been found to be unreliable, especially as applied to tomato catsups, when acceptable in their application to other food-stuffs. The semisolid consistency of the catsup leads to the formation of emulsions during extraction with volatile immiscible solvents which can be broken only with great difficulty. A complete extraction of the preservative is thus rendered tedious and uncertain. In every instance, too, the extracted benzoic acid is so badly contaminated with more or less of a highly colored oily extract as to preclude the possibility of obtaining reliable results, either by weighing or titrating, without further purification of the acid. Numerous methods have been devised for obtaining the benzoic acid from food-stuffs in a pure state. In order to outline briefly the ground which has been covered by the various investigators their methods may be classified as follows:

I. Methods depending on the complete extraction of the benzoic acid, without previous treatment of the sample other than acidifying, and on subsequent purification of the acid in the crude extract, by
  1. Sublimation, followed by
    (a) Direct weighing or titration of the sublimate,[1] or
    (b) Collecting the sublimate in alkaline solution and subjecting to further treatment.[2]
  2. Precipitation of the benzoic acid as
    (a) Lead benzoate,[3]
    (b) Copper benzoate, or[4]
    (c) Silver benzoate.[5]

II. Methods depending on a preliminary treatment of the catsup in order to remove or render insoluble all substances which interfere with the extraction of pure benzoic acid, such as
  1. Saturating the sample with common salt and filtering,[1]
  2. Making alkaline, evaporating to dryness and extracting the dry material with ether,[2] or
  3. Distilling, either
    (a) Direct, or
    (b) With steam.[3]

None of the proposed methods falling in class I remedy the defects due to the emulsions formed during extraction. It is necessary, too, preparatory to sublimation, to dry the crude extract thoroughly at ordinary temperature, preferably in a vacuum, while the sublimation process itself requires careful attention and a large amount of time and patience for successful results. In precipitating the benzoic acid as metallic benzoates, each method presents its own difficulty, such as solubility of the benzoate in an excess of the precipitant, solubility in the alcohol used for washing, or contamination of the precipitated benzoate with foreign matter from the crude extract. By following the methods of class II, the troublesome emulsions are avoided but the method of LaWall and Bradshaw (1 under II) requires a filtration which is nearly as troublesome, which can not be accelerated by means of the suction pump on account of the gelatinous character of the suspended matter and which if carried out on the ordinary paper filter often requires several hours for its completion. Furthermore, it was found in this laboratory that the method seldom gave an extract of benzoic acid that, without purification, could be weighed with reliable results.

It is well known that benzoic acid is volatile at a temperature much below its melting point and that it distils readily with steam. Steam distillation, however, as it is ordinarily conducted, is a tedious operation and for the quantitative recovery of volatile substances a large distillate is necessary. Moreover, Hortvet[4] shows conclusively that the official method of steam distillation is entirely unreliable for a quantitative determination of the volatile acids in wine, depending to such an extent, as it does on the conditions of distillation that concordant results can not be obtained by two

---

[1] A. O. A. C.: "Official Methods for the Determination of Benzoic Acid," Bull. 107, U. S. Dep't of Agr., Bur. of Chem. Julius Hortvet and Ralph Hoagland: Report of the Chemist, State of Minn. Dairy and Food Commission, 1907.

[2] Harry S. Reed: *Jour. Amer. Chem. Soc.*, **29**, 1626.

[3] Hager's Untersuchungen, 2 Aufl., **2**, 155.

[4] F. M. Horn: *Jour. Soc. Chem. Ind.*, **7**, 225.

[5] Hillyer: *Proceedings of the A. O. A. C.*, 1908.

[1] C. H. LaWall and H. A. Bradshaw: *Amer. Jour. Pharm.*, 171-172 (1908).

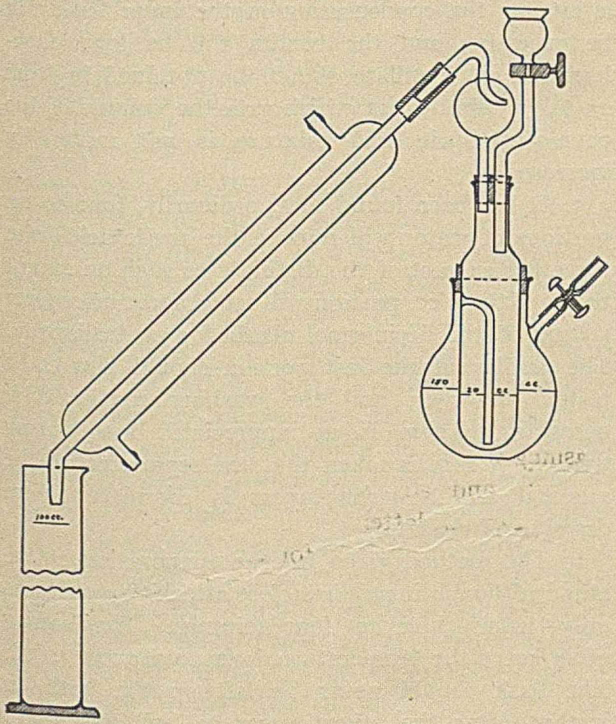
[2] A. Wynter Blyth: "Foods, Their Composition and Analysis." E. Mohler: *Bull. Soc. Chim.*, **3**, 414.

[3] Suggested by A. E. Leach, Mass. State Board of Health Report for 1905.

[4] "The Determination of Total, Fixed and Volatile Acids in Wine." This Journal, I, **1**, 31-38.

different operators or even by the same analyst using the same piece of apparatus. He shows in addition, however, that the apparatus he recommends enables a comparatively large amount of volatile acid to be carried over by a volume of distillate not so large as to interfere with subsequent operations This being true for the volatile acids in wine, it was hoped that it would prove equally true for the small amounts of benzoic acid usually found in catsup and other food products

A few slight modifications were made in Hortvet's wine apparatus (see figure). The tube leading from the outer to the inner flask was made slightly larger in diameter and was introduced half way up the side of the inner flask. This makes it possible to connect the apparatus in such a way that at the beginning of the operation the water in the outer flask will reach to the height of the contents of the inner flask. The whole apparatus was made somewhat shorter, the inner flask proportionately larger in diameter, and the outer flask was provided with a side tube closed with a rubber tube and pinch-cock by means of which the steam pressure might be relieved at any moment without danger of drawing the material under examination back into the outside flask.



Determinations with this apparatus were made on water solutions of benzoic acid of known strength in order to ascertain the per cent. of the acid recoverable, under given conditions, in 100 cc. of distillate. These results, as shown in Table I, are calculated to sodium benzoate, the form in which the acid is usually added to food products. The figures in column 2 were obtained by acidifying 10 cc. of the water solution, extracting direct with ether, evaporating the ether and determining the equivalent of the resulting benzoic acid volumetrically. The figures in column 3 were obtained similarly by extracting the 100 cc. distillate obtained from 10 cc. of the sample. In every instance the residue in the distillation flask was tested for the presence of benzoic acid with negative results. In the case of solutions 8, 9 and 10 the loss was caused by the deposition of the acid in the condenser in such large amounts that the following distillate failed to carry it all through into the receiver. The maximum weight of the acid which can be completely carried over by 100 cc. of distillate can be safely placed at 50 mg. In cases where a determination shows more than that amount the results should be checked by a second determination on half the usual quantity taken for analysis.

TABLE I.

No. of sol.	Grams of sodium benzoate in 100 cc. of sol.	Grams of sodium benzoate found in 100 cc. of sol.	Per cent. sodium benzoate recovered.	Mg. of benzoic acid obtained in 100 cc. distillate.
1	0.097	0.097	100.0	8.24
2	0.194	0.194	100.0	16.47
3	0.274	0.274	100.0	23.18
4	0.374	0.374	100.0	31.72
5	0.468	0.468	100.0	39.65
6	0.562	0.562	100.0	46.81
7	0.770	0.770	100.0	65.27
8	0.979	0.972	99.3	82.35
9	1.375	1.354	98.4	114.68
10	1.440	1.411	98.0	119.56

The subjection of a sample of catsup to a similar distillation with steam resulted in indifferent success. Although a distillate was obtained which was readily extracted with ether, the extract was badly contaminated with coloring matter and volatile oils from the tomatoes and spices. In order to destroy these volatile substances, concentrated sulphuric acid was added to the catsup, prior to the distillation, in sufficient amount to thoroughly char the vegetable tissue. The distillate obtained under these conditions was free from all coloring matter and oil and was found to contain, in addition to the benzoic acid, only acetic and such other volatile acids as are readily soluble in water. The distillate was extracted with ether without the least tendency toward the formation of an emulsion and, after the evaporation of the ether, a white crystalline residue of benzoic acid was obtained which agreed very closely both in weight and acidity with the amount of acid originally added to the catsup as benzoate of soda. Chromic acid was also tried in place of the sulphuric acid but with no apparent advantage. The method in detail for catsup follows:

About 10 grams of the sample are accurately weighed into the inner flask of the apparatus, 1.5 to 2.0 grams of paraffin added to check excessive foaming during distillation and the flask is connected with the condenser. Ten cc. of concentrated sulphuric acid are dropped in through the dropping funnel at a rate sufficient to complete the addition in from two to three minutes, the flask is gently agitated to thoroughly mix the contents and is then allowed to stand for five or ten minutes after all apparent action of the sulphuric acid has stopped. 150 cc. of distilled water are measured into the outside flask of the apparatus which is then connected as shown in the diagram, the water is slowly brought to a boil, and the boiling is continued until 100 cc. distillate have been collected. The distillate is filtered into a separatory funnel and the original receiver and filter are rinsed with two 10 cc. portions of water. Complete extraction is effected with three portions of ether using successively 50 cc., 30 cc. and 20 cc. The combined ether extracts are shaken out with water until a 25 cc. portion of wash-water requires not more than a drop of decinormal alkali for neutralization, indicating the complete removal of the water-soluble volatile acids. The ether extract is transferred to a wide-mouthed extraction flask which has been previously weighed and the ether is distilled as quickly as possible on the water-bath. At just the point where ebullition of the ether ceases the flask is removed from the water-bath, air is blown in to remove the last traces of ether, the flask with its contents is placed in a desiccator and dried over night or until constant in weight and weighed. The number of grams of benzoic acid found multiplied by the factor 1.18 will give the equivalent sodium benzoate. Considerable time may be saved, however, if the determination is made volumetrically. In such case the filtration of the distillate may be omitted as well as the weighing of the flask and drying and final weighing of the acid. The crystals of benzoic acid are dissolved in a few cc. of carefully neutralized alcohol and the solution is titrated with decinormal alkali using phenolphthalein as indicator. The number of cc. of standard alkali required multiplied by the factor 0.0144 gives the equivalent sodium benzoate in grams. The burette can be read to 0.05 cc. which is equivalent, in a decinormal solution, to 0.00072 gram of sodium benzoate. Hence, on a 10 gram sample, the results will be accurate to 0.01 per cent.

Experience with the method has shown that the following cautions should be observed:

1. The paraffin used should yield no volatile ether-soluble substance on distillation with steam under the conditions of the method described above.

2. Excessive foaming is likely to occur when the steam begins to pass into the inner flask. Careful regulation of the flame at this point is necessary. The foaming may be caused by distilling too soon after the addition of the acid, by an insufficient amount of paraffin or by an unusual amount of sugar in the catsup. A little care in manipulation will suffice in nearly every case to prevent the foam from passing over into the condenser.

3. Whenever for any reason the flame is withdrawn, the outlet tube in the outer flask must be opened or the material under distillation will be in danger of being drawn back into the water.

4. Distillation must not be carried on at too slow a rate or the condensation in the inner flask will be excessive and the results will be low. One-hundred cc. distillate should be obtained in from 25 to 30 minutes, at which rate the volume of the catsup-sulphuric acid mixture is not materially increased.

5. It has been found that, ordinarily, four 50 cc. portions of water will remove the acetic acid from the ether extract of the distillate to such an extent that a fifth 25 cc. portion will require not more than a drop of the decinormal alkali for neutralization. The acidity of the last portion should always be tested, however, and the washing continued if necessary. There is no appreciable loss during washing if care is taken that the separation of the ethereal and aqueous layers is complete before drawing off the latter.

6. The method given for the evaporation of the ether, although it requires close attention, consumes only from three to four minutes. The boiling ether keeps the temperature down to at most 40° C. The flask should never be left on the bath an instant after the ether ceases to boil or loss of benzoic acid will occur.

7. If the determination is to be made gravimetrically the ether extract should be filtered through

a dry filter into the tared flask. This will remove a large amount of the moisture with which the ether is saturated and greatly facilitate the drying in the desiccator subsequent to the evaporation of the ether.

8. The alcohol used in dissolving the benzoic acid preparatory to titration should always be neutralized just previous to its use as acidity develops on standing.

In order to test the method more thoroughly in its application to catsup, tomatoes were obtained and some catsup was made and bottled in the laboratory. One portion was kept free from a preservative; to the others were added respectively 0.10, 0.25 and 0.50 per cent. of sodium benzoate.[1] Table II shows the results obtained on these catsups. Duplicate volumetric determinations (1 and 2 in table) were made in each case. Determinations 3 and 4 were made on the same distillate: 4 is the equivalent of the benzoic acid actually weighed while 3 is the result obtained by calculation from the acidity of the weighed residue.

TABLE II.

No. of sample.	Per cent. of sodium benzoate added.	Per cent. of sodium benzoate found.				
		By calculation from acidity of residue.			By calculation from wt. of res.	4 months after bottling.
		1.	2.	3.	4.	5.
I	0.00	0.00	0.00	0.00	0.00	0.00
II	0.10	0.10	0.10	0.09	0.09	0.10
III	0.25	0.24	0.23	0.24	0.25	0.25
IV	0.50	0.47	0.46	0.47	0.47	0.47

The general belief that during the aging of a catsup the amount of recoverable benzoic acid gradually decreases led to a further determination, four months after bottling, on each of the three samples to which the preservative had been added in the laboratory. The results obtained are shown in the last column of Table II. The figures indicate that, during four months at least, no change in the benzoate occurs which renders its recovery by the proposed method incomplete. It is conceivable, however, that reactions might take place between the benzoate and the ingredients of the catsup by means of which compounds of the acid or of derivatives of the acid would be formed which would fail to yield the benzoic acid under other conditions than the destruction of the vegetable tissue and oxidation with concentrated sulphuric acid.

Some samples of wine of known purity, to which definite quantities of benzoate of soda had been added in this laboratory, were also subjected to the steam distillation method. The results, without exception, were too low. The only way in which these low results could be explained was by the presence of alcohol, which under the influence of the concentrated sulphuric acid might contribute to the formation of ethyl benzoate. Three solutions of benzoic acid were made up by dissolving known amounts in about 10 cc. of 95 per cent. alcohol and making up to 100 cc. with water. The benzoic acid was then determined in each of these solutions by distillation and by direct extraction with ether. The results are shown in Table III.

TABLE III.

No.	Benzoic acid added, gram.	Found by distillation in 100 cc.	Found by extraction of sol.
1	0.08	0.07	0.08
2	0.16	0.14	0.16
3	0.24	0.16	0.24

Direct extraction in the case of the wines was found to give too high results and consequently a method based on such an extraction could not be considered reliable. It was found necessary, therefore, to dealcoholize the wine, after rendering alkaline with a few drops of a saturated solution of sodium hydrate, before subjecting to distillation. The results obtained on the wines of known benzoate content by the various methods tried are shown in Table IV.

TABLE IV.

Kind of wine.	Source.	Grams of benzoate soda added to 100 cc.	Grams of benzoate found in 100 cc.			
			By direct extraction.	By distillation without dealcoholization.	By distillation with dilute acid.	By distillation after dealcoholization.
Claret	Garrett & Co.	0.00	...	0.00	...	0.00
Claret	Same	0.11	0.13	0.10	0.10	0.11
Claret	Same	0.20	0.22	0.16	0.16	0.20
Claret	Same	0.29	0.30	0.23	0.23	0.28
Claret	Same	0.39	0.42	0.32	0.33	0.39
Sherry	Cal. Wine Assn.	0.58	0.58	0.42	0.48	0.58
Riesling	Same	0.29	0.32	0.24	0.24	0.29
Sauterne	Irondequoit Wine Co.	0.20	0.21	0.16	0.17	0.19

In the case of substances containing alcohol, then, a convenient volume is made alkaline with sodium hydroxide, dealcoholized in the usual way on the water-bath, made up to the original volume and a 10 cc. sample taken for distillation. Where it is necessary to take the sample by weight instead of measure, dealcoholize the weighed portion as

---

[1] The benzoate used was assayed for purity and the necessary amount in addition to that calculated on the basis of 100 per cent. pure was added to the catsup to give the required per cent.

described and transfer to the distillation flask with as little distilled water as possible. Substances containing large amounts of fat, such as milk and milk products may be very successfully subjected to this distillation process for benzoic acid. With apple cider no difficulty was experienced when extracted directly with ether and the results were equally as good as by the distillation method but the latter is recommended as being safer in every case. A "hard" cider should, of course, be dealcoholized as in the case of wines. Jellies and jams having a high sugar content will be found to give the most trouble by excessive foaming. It will be found necessary to add the sulphuric acid very slowly and in beginning the distillation a great deal of patience will be required before the caramelized sugar is completely carbonized.

It is suggested that each analyst, before making a determination by the proposed method, test his apparatus with one or two solutions of known benzoate content and make sure that the time and other conditions of distillation are sufficient to give the desired results. In making up the test solutions for this purpose it must be borne in mind that very little of the sodium benzoate on the market is actually chemically pure. An assay of the benzoate should always accompany the preliminary tests of the apparatus.

In conclusion, the writer wishes to acknowledge the many helpful suggestions, contributing to the accomplishment of the above results, which have been made by Mr. Julius Hortvet, chief of this laboratory.

CHEMICAL LABORATORY,
STATE OF MINNESOTA,
DAIRY AND FOOD COMMISSION.

ADDRESSES.

TO WHAT EXTENT SHOULD COLLEGE TRAINING CONFER PRACTICAL EFFICIENCY ALONG TECHNICAL LINES?[1]

By LOUIS M. DENNIS.

For many years there has existed among employers in manufacturing and business pursuits a wide-spread scepticism concerning the value of the services of the recent college graduate. That this scepticism still exists is shown by the very wording of the topics that have been assigned to the distinguished speaker[2] who has preceded me and to myself. In the field of technical chemistry this doubt concerning the value of the college-trained chemist has been due chiefly to (1) the failure on the part of the employer to realize the value of chemistry in the conduct of his process; (2) the distrust shown by the "practical" man toward one whom he regards as a mere theorist; (3) the inadequacy of the training of many of the young men who have gone directly from the laboratory into technical practice; and (4) the belief on the part of many employers that if a young chemist lacks experience in their own or in allied processes, his services must necessarily be of but little value to them.

The first of these causes is rapidly disappearing, and the old commercial processes that were governed by guess work or inherited formula are suffering so seriously either through high cost of production due to waste and ignorance, or through inability to march with the times along the line of scientific development and improvement, that they are being forced to appeal to the chemist for aid in preserving their very existence. It is interesting to note this change of attitude on the part of many chemical manufacturers and to observe their rapidly growing respect for the well-trained chemist whom they had formerly looked upon as a sort of mysterious corpuscle existing in a malodorous medium and highly charged with negative usefulness.

The distrust shown by the "practical" man toward the theoretical chemist is still widely existent, but the rapid replacement in many branches of manufacturing of the man who has grown up in the works by the younger college graduate who brings into the process a sound theoretical training is evidence of the fact that the day of guess-work management is rapidly passing. We may therefore consider that the two chief causes for the feeling on the part of the employer that the recent graduate in chemistry is lacking in practical efficiency are the inadequate training of the chemist himself and the demand by the employer that the chemist possess practical experience.

Let us first briefly consider this question of the training of the chemist. There is no doubt that the scepticism of the employer concerning the value of the young chemist's services has frequently found full justification in the smallness of the benefit accruing to the employer from those services. The young graduate, however, has not been wholly or even largely to blame: the fault lies partly with those entrusted with his preparation for his calling and partly with his future employer. On the one hand, he was told by the enthusiastic but narrow-visioned teacher that early specialization in some particular branch of chemistry was essential to later success, while from the employer came the warning that he wished only such young men as had extensively specialized in that part of the college curriculum that related particularly to the employer's line of manufacturing.

There further existed in the minds of many educators and manufacturers the idea that the only use of a chemist in an industrial plant was as an analyst. What was the result? The college graduate went out into practice thoroughly acquainted with but a small portion of the field of chemistry and wholly ignorant of allied branches. Naturally he sought and by his teachers was recommended for a position in which this limited information could be utilized. If such an opening occurred he entered upon his work under what he and his employer regarded as most favorable auspices, and his progress was quite satisfactory until he began to meet problems outside of his specialty. He has been trained, let us say, as an analyst, and fails t

---

[1] Read before the Section on Education, American Chemical Society, Baltimore meeting, Dec. 29, 1908.

[2] Dr. Wm. H. Nichols; see February number THIS JOURNAL.

solve a works problem because of his ignorance of physical chemistry; he has specialized in physical chemistry and is unable to test a lubricating oil; he has devoted his attention to organic chemistry and cannot ascertain the reason for the perversity of a certain reaction because of his unfamiliarity with the analysis of complex gas mixtures, or with the application of the spectroscope or microscope in chemistry. He is familiar with the main branches of chemistry but cannot advise as to the construction of new apparatus because of his inability to read an engineer's drawing; he is asked concerning the installation of electric power in his laboratory or in the works and sadly shakes his head, thereby calling to the mind of his employer the remark of Rufus Choate to the jury when he had been exasperated by repeated objections from the Bench. "You may have noticed, gentlemen of the jury, that his Honor shakes his head, but there's nothing in it." Under such circumstances it is but natural that the employer should lose faith in the college-trained chemist. In business life, it is results that count, and the graduate failed to produce them. Of course I do not here refer to the genius, for he will rise superior to the limitations of his training; nor to the weak student, for his budding aspirations should early have been blasted by a rigorous faculty.

If then the employer is to be led to look with less distrust upon the college-trained man we must first of all make sure that that training is suitable and adequate. Of course, the college cannot give the student brains, but it can teach him to use his brains. As Lorimer says: "College does not make fools, it develops them; it does not make bright men, it develops them."

The question before us here to-day is: "Can proper college training in chemistry confer upon the graduate practical efficiency along technical lines?" Before entering upon the discussion of this question, let us first seek accurate definition of it. Turning to the dictionary, we find: "Efficiency"—"that which produces results. Energetic and useful activity." "Practical" is defined as "capable of applying knowledge to some useful end." The properly trained chemist should then be "capable of applying his knowledge to the production of results." Does that imply that his college training must necessarily embrace specialization in the field that he is to enter? Not at all. In the first place it should be remembered that the student in average circumstances is able to devote but four years to his college training. All of us will undoubtedly agree that two years of general culture study before the four years of special work would be of inestimable advantage to the student in fitting him for that enlightened citizenship which should be the aim of higher education, but for the majority of the young men who enter college these additional two years are wholly out of the question. The problem that concerns the educator is, then, in this limited time so to shape the work of the future chemist as to develop mental alertness and the ability to reason clearly and correctly, and to equip the student with a knowledge of the different branches of chemistry and of allied subjects broad enough to enable the graduate successfully to cope with the diverse problems of chemical practice, and to employ in their solution the most efficient line of attack. Narrowness of training such as would necessarily result from extensive specialization during the college course would so limit the student's ability correctly to interpret his observations that graduates under this system might be likened to the blind men who were asked to describe an elephant. One felt his side and said he was like a wall; another his trunk and pronounced the elephant a snake; the third happened to seize hold of his tail and declared that he was like a rope; while others, grasping an ear, a tusk, and a leg, likened him to a fan, a spear, and a tree. Doubtless each one of us could cite many instances of false conclusions drawn by the chemist because of lack of broad information. Like the illustrious Liebig when he had isolated bromine but thought it was merely an oddly behaving chloride of iodine, the chemist of narrow training "constructs a theory" that, while it suits him, leads him far astray.

Breadth of training should be the principle underlying the college curriculum in chemistry and to make clear my views upon this subject, permit me briefly to outline the course of study that experience has led me to believe to be essential to the success of the chemist in technical practice. The elements of the leading sub-divisions of chemistry itself, inorganic chemistry, analytical chemistry, organic chemistry, physical chemistry, and industrial chemistry, should be thoroughly covered, and to these should be added some acquaintance with sanitary chemistry and agricultural chemistry. Especial attention should be paid to the instruction in analytical chemistry, for if a chemist is to succeed he must be an expert analyst. But the curriculum that offers extended courses in quantitative analysis to the exclusion of other branches of analytical work will be of much less value to the student than that which broadens his training as an analyst by affording him detailed instruction in gas analysis, organic and sanitary analysis, assaying, and thorough laboratory practice in the employment for analytical purposes of the spectroscope, the polariscope, the colorimeter, and the microscope. Although this variety of instruction along analytical lines will necessarily curtail the work in advanced quantitative analysis, yet to my mind it undoubtedly yields better results than does long practice in quantitative analysis alone. The capable student who is familiar with the scientific basis of quantitative analysis and with typical determinations will readily grasp new methods and be able to carry them through with accuracy. The equipment of the chemist should not, however, be limited to chemistry alone. He must be well grounded in certain allied lines, chief among them being physics, mineralogy, and economic geology. He should also be acquainted to some extent with the principles of construction and of power transmission. I do not mean that he should be an expert engineer, but he should be qualified to pass intelligently upon questions of construction and design and to outline to the engineer in an understandable manner his wishes and needs in the mechanical development of the process in which he is interested. For these reasons his course should further include mechanical drawing, the mechanics of engineering, a full year of work in the mechanical laboratory, and instruction in the theory and use of the dynamo.

Such a curriculum will leave the student some spare time for other work during the four years, and that can best be utilized by giving him in his Senior year a small problem to investigate. The primary object of this research should be the training of the young man in methods of investigation. By that I mean the manner in which a problem is attacked, the searching of the literature for light upon the point in question, and the exercise of his varied information in the solution of the difficulties that may arise. This undergraduate research should be carefully differentiated from the more elaborate investigation of the graduate student in which discovery is the chief end in view. If time permitted it would be of almost inestimable gain to the student to carry out more ambitious and extended investigation, for I am convinced both by observation and by the almost unanimous testimony of graduates in chemistry who have gone into technical practice, that thorough drill in research far surpasses in practical value any other of the college courses. For this reason it is most desirable that not only the prospective teacher but also the young man aiming at a technical career should, whenever possible, continue his university work until he has obtained the doctor's degree. When he then enters commercial practice, the better class of positions with correspondingly larger remuneration will be open to him, and what is of chief importance to the employer, his practical efficiency will be markedly greater than that of the student who has completed only the undergraduate course.

Toward the beginning of my remarks I stated that the demand by the employer that the young chemist possess practical experience might be classed as one of the principal causes for the existence of the idea that the college-trained chemist lacks practical efficiency. The relationship between these two may seem to be remote, but the perusal of many letters from manufacturers asking for chemists shows that employers frequently confuse the efficiency that results from suitable college training with the efficiency that follows upon long acquaintance with their particular process. If we accept the definition that "practical" means "capable of applying his knowledge to some useful end" the properly trained college graduate should show this quality at the very outset of his technical career. But his practicality must not be expected to be identical in nature with that gained by another through long experience in the works. If we give him time and opportunity the college-trained chemist will, I feel confident, develop a general practical efficiency wholly satisfactory to the employer both in quality and extent, and in the meantime, he will be able to produce results of a value sufficient to justify his retention and advancement. The confusion of scientific efficiency with works efficiency reacts in undesirable manner upon the training of the chemist, because it imbues the teacher with the idea that works efficiency is obtainable through college instruction and for that reason leads many educators to incorporate in the curriculum laboratory courses in technical processes. Personally, I believe this is a serious mistake. In the first place, the introduction of such instruction forces out of the curriculum some of what I regard to be the far more profitable lines of work above-mentioned. In the second place, the attempt to familiarize the student with a technical process by having him carry out the reactions of the procedure in a beaker or a pot cannot but fail to give him an adequate idea of the process when it is performed on a manufacturing scale. If the student is well-grounded in the theories underlying the leading commercial processes and if he has listened to lectures by experts upon the apparatus and the methods that are employed in the handling of large amounts of material, he ought to be able to grasp the details of any manufacturing process in a comparatively short time. A further unfortunate result of this point of view of the employer is that it leads him to believe that because the collegian's training has been chiefly along theoretical lines, the young man can be useful to the company only in the analytical laboratory and is unfit to be sent out into the works. I realize that the answer to this will be: "But we have tried the college-trained man in the works, and he has not 'made good' ". To this I reply: "Get the right kind of a man and try again." If you are still doubtful as to his possible value adopt some such plan as that recently followed by a large manufacturing concern. They had not previously employed a chemist, but sharp competition and the desire to improve their process led them to ask us to recommend one. A young man was sent to them directly from the laboratory. The concern naturally was sceptical as to his ability to master the intricacies of their process and acting, I think, with great shrewdness, they ordered him at the outset to "go into the works and, without asking questions, steal our process if you can." He set to work and, when ready, submitted his report in writing. That he succeeded may be inferred from the fact that he soon received a hurry call on the telephone from the general manager asking him if he had kept a copy of the report. Upon replying that he always copied his letters he was ordered to tear out the copy from his letter book and destroy it at once. After relating to me his experience, the young man added: "And now I wish to make a confession. When I was a student in the laboratory I used to wonder why such a variety of work was prescribed and I often thought that a great deal of it was a sheer waste of time. I take it all back now. There was not a course in the whole curriculum that I did not draw upon in solving the details of that process."

No one realizes more fully than do I that academic theories of chemical education that are evolved in ignorance of the needs and demands of industrial practice must almost of necessity fail to efficiently equip the young chemist for a technical career. But conference and coöperation between educator and employer coupled on the one hand with willingness to shape the training to meet the requirements of the chemical industries, and on the other hand with readiness to give to the young man full opportunity to show what he can do and to remunerate him in proportion to his worth, will certainly result in distinct and far-reaching gain to our chemical manufactures and will demonstrate that the proper kind of college training does "confer practical efficiency along technical lines."

THE ATTITUDE OF TECHNICAL INSTITUTIONS TO POST-GRADUATE STUDY.[1]

By William McMurtrie.

The subject assigned to me in this discussion seems to have been somewhat mixed. I have been asked to discuss:

(1) To what extent is post-graduate training recognized or desired by employers of chemists?

(2) What should be the attitude of technical interests toward post-graduate work?

(3) What should be the attitude of technical institutions toward post-graduate study?

---

[1] Read before the Section on Education, American Chemical Society, Baltimore meeting, Dec. 29, 1908.

It matters little how the subject of our discussion is stated, it really becomes, what shall be the training or education of young men whose life work shall be the applications of chemistry or physics or both, in the industries. It is a question which the experience of a century at least has scarcely solved. The elements which enter into the answer are too varied, the results to be attained too mainfold, the conditions available, too perplexing, the personal equation too persistent. Shall the training be confined to the storage of the facts and laws of chemistry and physics in the minds and memories of the young students trusting to the exigencies which may arise, to find their application and that when the exigencies do arise the facts and laws will be brought forth and be wisely applied? Or shall we first cause storage of facts, principles and laws and afterward offer training in the methods whereby these shall be applied and employed in the solution of the problems likely to arise in the industries and to demand treatment with satisfactory results. Whatever may be said, the latter is what employers expect and demand. The questions which arise must be answered promptly and accurately. The amount of knowledge a man may have acquired and may possess is little appreciated unless it can be applied usefully and effectively. Complaint is not uncommon that young men from the technical schools are overeducated, overtrained; educated above and beyond the positions they must occupy and the work they have to do. While this is unfortunate in form of statement, it nevertheless expresses a fact. Many young men are profoundly educated in theories and laws and at the same time acquire little or no appreciation of the practical value of these theories and laws even in the advancement of the science to which they relate and certainly not in the solution of the problems of the industries and the arts of life. There is therefore, whether recognized or not, a demand for something beyond the regular course of study of the university or the technical school. Something beyond the mere cramming with facts and academic training, to the exclusion of the systematic utilization of knowledge in the promotion of knowledge. Here, then, is the problem propounded to the educator for solution: How shall young men be educated and trained to meet the demands likely to be presented to them by the chemical industries? In the chemical industries it is natural that a profound knowledge of chemical laws should be required but in addition to this there must be provided a sound knowledge of such laws of physics as may be necessary to the physical application of chemical laws. In the undergraduate school, first the laws of chemical action and the properties of matter, theoretical and descriptive chemistry, the methods of chemical analysis, qualitative and quantitative, must be taught, and it is well-known that one of the first steps in laboratory instruction in these methods is an introduction to the form of apparatus to be employed in the practice of experiment and analysis. Then comes the application of the laws of chemistry and of the properties of matter to the methods of operation to be used. Now since manufacturing or industrial chemistry is really analytical chemistry in a large way, similiar lines of instruction and training must apply in preparation for the industries. If acquaintance with the beaker, the casserole, the filter, the evaporating dish, the distilling apparatus, the balance must be provided in the analytical and research laboratory: if here must be taught the sources and mode of application of heat, the transfer of liquids, separation of vapors, liquids and solids, all these processes made in a small way in the laboratories must be made large in the works. Operations made in a large way must be studied and the means for effecting them made familiar. The operation of the chemist in the laboratory must become the operation of the engineer in the works. The industries demand that the men who shall control shall have some of that capacity known as engineering, shall know something of the materials and methods of engineering, of the larger apparatus to be employed and its management and have ability to apply the laws of physics in the larger operations of the works. Chemistry and engineering must therefore be combined in some measure at least, in the training of the men who will become most successful in meeting the demands of the "technical interests," and whether this is recognized generally or not, it is certainly desired by employers of educated chemists.

But the common complaint of the institutions and their teachers is, that the customary four years allotted to the undergraduate course is too brief for all that is demanded and required. Here, then, is the problem to be solved: what shall be provided in the undergraduate course or school and shall post-graduate study and training be provided and carried on under the direction and management of the educational institution, or shall the training which might be provided by post-graduate study be deferred and be supplied by actual practice in the works? How and by whom shall this very important question be answered? The demand of the present and of the immediate future, is for men who are able to work independently, to take care of the problems arising, and work them out to successful issue, with profitable result, directors in the industries and employers have little time, energy or freedom from detail, to devote to the training of young men in fundamental principles. Yet experience can be had only in practice and this must be paid for in the time, energy and material apparently wasted by young men in the earlier periods of their life work.

But the question still persists: Could the young man working under intelligent direction in the systematic application of the principles he has been taught save time? Will the work of one, two or three years under intelligent and patient training of competent teachers save time of the young man and his employers and relieve both of embarrassment, loss and disappointment?

The laudation of the German Chemical Industry has extended to all nations and is probably justified. In some of the most successful branches of the German Chemical Industry the practice is to take into the works only men who have served as "*privatdocent*" in the university or technical school, and to become privatdocent the candidate must generally have taken a course in post-graduate work in investigation and in the solution of problems, work leading to the doctor's degree. First a training for systematic applied work, then experience in teaching. The value of the latter in the preparation of young men for life work is, I believe, too little recognized. It is certainly true that one of the most excellent means of securing a thorough and fundamental knowledge of a subject is found in an effort to impart such knowledge to others.

I have said elsewhere that an important adjunct to the successful application of knowledge is a trained imagination. Not an imagination "like the baseless fabric of a vision" nor "such stuff as dreams are made on," but an imagination based upon knowledge which furnishes a vision of what may be accomplished and suggests means for accomplishment.

So it seems to me that the proper function of the undergraduate school is to communicate knowledge of facts and methods and that the function of the post-graduate school is to furnish training in the application of knowledge to the solution of problems, to the training of the imagination and thus to meet the demands which the industries, consciously or not, are making.

A most useful beginning in such work has been made in the laboratories for research in industrial chemistry lately established in some of the leading technical institutions. Here the subjects for research, the applications of knowledge are not of an abstract but of a concrete character, and provide training in work which may produce results immediately useful in the arts of life. Here the problems arising in the industries in every-day work are solved by students, under direction of men who have themselves been trained in the solution of such problems. By such work the imagination is stimulated and at the same time trained and directed in proper channels—habits of application established which must be fruitful later on. The designers of these laboratories and the authorities who have ordered their organization and establishment, as well as the industries which have patronized and encouraged them all deserve the highest praise. It is a step in the right direction and one which must be taken in other educational institutions if the proper and most effective training of young men for the industries is to be secured.

What then should be the attitude of the industries to post-graduate work? I answer, unhesitatingly, *favorable*. What should be the attitude of the technical institutions to post-graduate work? I answer, without hesitation, *favorable*. Post-graduate work should be earnestly encouraged from both sides, from the educational and from the industrial, and particularly from the latter. It has been fully recognized in the German institutions by providing in the technical schools courses leading to the degree of "doctor of engineering," and in the university by the establishment of similar courses and providing for the same degrees. In all educational institutions the attainment of the degree of doctor—a degree not lightly appreciated not glibly assumed in Germany—involves work of investigation leading to results, work devoted to the application of knowledge and the solution of problems. The industries in Germany are wise in choosing for their employees and directors those who have passed through the office of privatdocent and have had, therefore, experience in the training and management of men. That men may become successful without this very extended and profound training is manifest in this country and is due largely to the men themselves. But even such men would be better equipped for their work by the training provided by the undergraduate and post-graduate schools and though frequently compelled by their necessities to enter upon their life work without it they would save much labor and loss of time to have had it. Many of those who, even with limited training in the schools, have been reasonably successful in the industries and in their life work in this country have a right to speak feelingly and affirmatively upon this point.

May young men be overtrained? Surely in the laboratories and in the class-room as in the gymnasium and on the athletic field; and they may be weakened, from a practical standpoint, by their training. Yet even these are often carried by their enthusiasm to eminent success. "Fools rush in where angels fear to tread" applies equally well in the world of science and industry as elsewhere, and the struggle to get out after the rush in has produced some of the best results the world has seen, though the influence and the method may not always have been recognized or acknowledged. Each one who has had experience may furnish evidence of this fact. Effort to correct errors of one's own making often leads to splendid results. "Necessity is the mother of invention" and the needs of a man in deep trouble makes him devise means which otherwise remain dormant and without utilization. Yet errors should be avoided, and the more thorough training should lead to this.

Will the institutions meet this demand for better trained men? Will the new courses necessary to it be established? Of this there can scarcely be a doubt. The institutions are looking for the sign and will respond to it when it is plain. But what of the industries? Will the leaders make the sign prominent and clear? Will they do their share? Do they know what their share is? And do they appreciate their responsibility?

First the institutions must know what is needed and the knowledge can be acquired only by close relations with the industries. Teachers should have ready access to the industries and their work for themselves and their students. Problems should be submitted to the research laboratories and needed means and materials provided. Such coöperation must certainly lead to important progress not only in the industries, but in the related sciences and progress under such circumstances is inevitable. May the influences which control have free course and be not only justified but glorified.

THE U. S. PATENT SYSTEM: ITS USE AND ABUSE.

At the meeting of the New York Section of the American Chemical Society, held at the Chemists' Club, on January 8th, the general subject for the evening was "The U. S. Patent System: its Use and Abuse." The papers presented were as follows: F. I. Allen (late Comm'r of Patents) "Introduction: Historical and Descriptive"; W. Hastings Swenarton (of the New York Bar, late of the Examining Corps, U. S Patent Office) "Patents, Trade Secrets and Trade Names as Factors in Industrial Development—their Relative Functions;" Louis C. Raegener (of the New York Bar), "Some Defects in the Practice of our Patent System and Suggested Remedies;" B. C. Hesse, Ph.D., "Some Suggestions as to Desirable Improvements;" L. H. Baekeland, Sc.D., "The Inventor's Standpoint." All of these papers, with the exception of that of Mr. Allen, who on account of illness was unable to prepare his manuscript for publication, will be published in these columns. The subject is one of great importance and is of especial interest at this time because of the reports which are coming to this country of the operation of the new English patent law, and also because of the fact that the rules of practice of the U. S. Patent Office are at the present time undergoing revision. The following notice appeared in the December, 1908, issues of the *Official Gazette* of the U. S. Patent Office:

It is considered advisable to publish a revised edition of the Rules of Practice of the United States Patent Office, because of the changes which have been made in such rules since the publication of the last edition. In view of this fact and in order, if possible, to simplify and improve the rules governing the practice before this Bureau, I desire to obtain suggestions as to amendment of the present rules and would be glad to hear from those interested upon the subject. It is not proposed to make radical amendments to the present rules, but simply to embody such changes in or additions to the rules as will be beneficial both to the Office and to applicants.

All suggestions should be submitted prior to January 1, 1909. EDWARD B. MOORE,
Nov. 27, 1908. Commissioner.

"PATENTS, TRADE SECRETS AND TRADE NAMES AS FACTORS IN INDUSTRIAL DEVELOPMENT—THEIR RELATIVE FUNCTIONS."

By W. HASTINGS SWENARTON.

The general conception of our laws relating to patents, is that of Statutes of Monopolies. At the present time when monopolies, not comparisons, are odious, even patents, which are often described as the only legal monopolies in the United States, are fast regaining the once notorious reputation of true monopolies, which culminated in the enactment of the famous English Statute of Monopolies. That a patent is not a monopoly (and all that can be said of patents applies equally well to Trade Secrets and Trade Names), is readily appreciated when one considers the true monopoly as recognized by the common law. According to Blackstone, a "monopoly" is defined as "a license of privilege, allowed by the King, for the sole buying and selling, making, working, or using of anything whatsoever, whereby the subject in general is restrained from the liberty of manufacture or trading which he had before." "A patent," on the contrary, to quote from Robinson on Patents, "lays no burdens upon the people, except that of remaining for a while without that which they never yet enjoyed."

When one recalls the unexampled progress, directly attributable to the inventions of American citizens, which has been coincident with the growth and development of our patent system, even long prior to this era of massive aggregations of capital, he is a poor patriot indeed if he does not thrill with pride and lose all thought of the idea of monopolies, as he approaches in turn such epoch-making inventions as the steamboat of Fulton, Stephenson's improvements in the steam engine, including the tubular boiler and the smokestack-exhaust, McCormick's harvester, Bigelow's carpet loom, the telegraph of Morse, the pneumatic process first invented by the American, Kelly, for manufacturing Bessemer steel, the Hoe press, the Otis elevator and the Howe sewing machine, the Westinghouse air-brake, the open-hearth steel process of Thomas, at last a substantial rival of Kelly's Bessemer process, the Bell telephone, Edison's incandescent lamp and the multitude of others of equal or greater importance.

In addition to the direct influence of the United States patent system, through its patents, upon the commercial expansion of the United States, it is interesting to consider at this time the report of the Commissioner of Patents for the year 1848, particularly since the United States is now recognized universally as the greatest agricultural country in the world. This report which, as is evident, was a report of the Patent Office as well as the annual agricultural report, there being no separate department of agriculture at that time, stated:

"Much complaint has been made by inventors on account of a small portion of the Patent Fund employed each year for the agricultural report, and I have heretofore sympathized in such complaint. Mature reflection, however, has convinced me that no injustice is done to the interests of inventors, by such an application of the Patent Fund, but, on the contrary, the interests of the Patent Office and of the inventors themselves have been subserved by it.

"The Agricultural report of the office, by its wide dissemination throughout the country, has contributed much to increase the reputation and influence of the Patent Office, and to spread more widely among the people a knowledge of the new inventions and improvements which have been made during the year. And thus it promotes the interests of inventors, by contributing to the more rapid introduction and sale of their machines and improvements."

The United States Patent System comprehensively protects inventions which are disclosed, through its Letters Patent, and to a lesser degree, through its caveats, or what might be termed its *letters de cachet*, or sealed and undisclosed documents. Similarly its certificates of registration more adequately secure Trade Marks to their respective owners or proprietors. Trade Marks (herein considered as a species of trade names), as well as trade secrets, in addition to the protection afforded by the United States Patent System, have long been recognized under the common law as property rights.

While both trade secrets and trade marks are of ancient origin, the legal recognition and protection, under the common law, of trade marks, long preceded that of trade secrets. In fact it is virtually but a century since the courts have extended their protecting arm to cover the property rights known as trade secrets.

The trade mark is a development of the ancient shop sign. Even the Egyptians are known to have displayed inscriptions denoting their trade in conjunction with an emblem to further indicate it. Also among the ancient Greeks signs were employed to proclaim their calling. In the ruins of Pompeii and Herculaneum discoveries are reported of representations of various kinds, let into the pilasters at the side of an open shop, as, for example, a goat by a dairy; a mule driving a mill at the baker's, and at the door of the schoolmaster's, what has been termed, "the not overtempting allurement to knowledge," comprising the representation of a boy receiving a good birching. These trade emblems were doubtless introduced into England at the time of the Roman invasion.

Originally, owing to the limited number of traders, the signs were indicative of the trade, and were employed in the same capacity as we now use street numbers. Gradually, as competition increased in these particular trades, and the shops became designated by street numbers, the sign-board fell into disuse. Then it was that the trade mark came into active use, being merely in the beginning a transfer, so to speak, from the door of the shop to the article of merchandise.

Even the piracy of trade marks is of early origin, as witnessed by the following preface occurring in Alder's "Livy:"

"Lastly, I must draw attention of the student to the fact that some Florentine printers, seeing that they could not equal our diligence in correcting and printing, have resorted to their usual artifices. To Alder's 'Institutiones Grammaticae,' printed in their offices, they have affixed our well-known sign of the Dolphin wound round the Anchor! But they have so managed that any person who is in the least acquainted with the books of our production, cannot fail to serve that this is an impudent fraud, for the head of the Dolphin is turned to the left, whereas that of ours is well-known to be turned to the right."

A critical review of famous trade marks would indeed form an interesting lesson in psychology, and would doubtless supply a fund of useful information to the memory system doctrinaires. Without a detailed review of the multitude of priceless trade marks already in existence, a single reference to the influence of a recently adopted trade mark will possibly be of interest. The National Biscuit Company, since its formation in 1898, has extended its reputation chiefly through its trade marks, and if reports can be credited, the company attributes its commercial success to that unique example of a psychological trade mark "Uneeda."

The passage of the Pure Food Laws has been already a boon to the manufacturers of pure food, and with the maintenance of quality, their trade marks will ever stand foremost in the minds of the eighty million consumers of this country, who will, it is hoped, never again have occasion to buy standard commodities subject tot he doctrine of *caveat emptor* (let the buyer beware).

No consideration of the influence of invention upon industry can be complete without a review of the functions of trade secrets. A trade secret may comprise a process which produces an old product that is producible by other known methods in so nearly an identical condition as to prevent the determination of the method of production, even when the product is subjected to the most careful scrutiny. Such processes obviously would not include the Hall aluminum process, Kelly's Bessemer process, or Mushet's recarburizing process for the production of steel, and the Thomas open-hearth steel process. Even more recently than the above is the patented Knietsch contact process for the manufacture of sulphur trioxide. While all of these processes do not necessarily produce products that would invariably reveal whether or not a definite process was employed in their production, nevertheless each one at least requires a particular type of apparatus, of a substantially permanent nature, so that its indefinite concealment within a plant would be rendered difficult, or else the product is of a nature to indicate whether or not a certain patented process has been employed. The Bell process of telephony is another example of a process which, by virtue of the fact that substantially permanent apparatus is required to perform the same, is admirably adapted for the protection afforded by the United States Patent system through its Letters Patent.

A typical example of the type of process which one must perforce protect as a trade secret, or else dedicate to the public by virtue of the fact that it is not susceptible of patent protection, is the process of treating tin scrap, which was involved in the recent case of the Vulcan Detinning Company *vs.* The American Can Company, wherein the process in issue was apparently non-patentable, and yet extremely useful. It consisted of the employment of old processes in a system of apparatus which was peculiarly arranged as to the disposition of the various elements of the system, and also as regarded the relative distances of each element from certain other elements. In addition, apparently the size of the apparatus and the employment of a steam hammer, as one of the elements of the system, were important features. In spite of the fact that the title of the Vulcan Detinning Company to the process was clouded by virtue of its having acquired the process from parties who had surreptitiously obtained it from a German manufacturer, the court held that since there was an apparent breach of trust involved in the acquirement of knowledge of the process through a former officer of the Vulcan Detinning Company, that the American Can Company should not only be enjoined, but should account for all profits made thereunder.

No trade secret can be considered as coming under the protection of the common law unless it is really treated as a secret as such, and unless all parties who are cognizant of it, know that it is a secret, and are either employed in a confidential capacity, whereby they are stopped from denying their responsibility of keeping the secret, or are under contract, oral or written, and express or implied, not to divulge it. It need not, so far as the present status of the law on this subject is concerned, be either new, or sufficiently novel to be patentable.

The influence of trade secrets upon industry has been marked, from the earliest times. The guilds of the middle ages are famed for guarding inviolably their trade secrets. In fact, in many European countries, inventors were often compelled to hold as trade secrets and assume the risk of their secret being discovered, inventions which might have been patented, owing to the stringent requirements and high taxes imposed by foreign governments upon patents. In the great steel centers, at the present time, quite a different welcome awaits the rival steel manufacturer, in so far as obtaining access to these mammoth workshops is concerned, as distinguished from the reception which would have been tendered him but fifteen years ago. All specially built and peculiarly assembled machinery, used exclusively within the plant and not purchasable in the open market, as well as all secrets relating to the more economic production of steel, are now carefully guarded.

It is often a nice question for the manufacturer, to determine whether to protect his invention as a trade secret, or to procure a patent thereon. The mere fact that a second inventor may at any time enter the field, and apply for, and secure the grant of, a patent, is a matter of serious import and worthy of the most careful consideration. This has occurred often in the past, and for this reason, it is in some cases advisable to file a caveat in the confidential archives of the Patent Office, renewing it from year to year as its term expires, in order to prevent the issue of a patent to a subseqeuent applicant, and yet withal avoiding the publication of the secret until such time as the competitor appears in the field. Of course the caveat should contain as full and complete a description as any patent specification, if one would insure himself against the grant of a patent to a subsequent applicant, and since most trade secrets are carefully guarded in some secret archives, commonly of a corporation, it is obvious that no rights are sacrificed by the filing of a caveat as aforementioned.

In respect of the three classes of property rights mentioned, either the manufacturer, the merchant, or the inventor, in some cases has a misconception of, or fails to realize, the protection afforded by the United States Patent System to each property right as aforesaid. The moral effect of a patent, and of a certificate of registration of a trade mark, upon the public generally, and upon competitors in particular, is often of considerable value. This is even true in the cases of what are termed "paper patents" or "paper trade-marks," that is, patents and trade-marks which are of such a nature as to be almost certain of being ultimately upset in court. When one realizes the time and money often required under the present administration and enforcement of our Patent Laws to defeat a patent or a certificate of registration, is it any wonder that corporations and individuals who are cognizant of this fact, do not hesitate to secure every possible degree of protection for their products which the common law or the United States Patent System affords them? Similarly in the case of the trade secret, there is no question but that the moral effect of a caveat upon the employees and competitors would result in increasing the loyalty of the employe and discouraging the competitor against infringing upon the rights of his rival, who had thus endeavored to protect his trade secret.

Many there are who still believe with the Examiner who was appointed to the Patent Office in 1854, and who upon resigning shortly after declared: "I believe in a little while there will be nothing for the Patent Office to do, as everything is already patented, and I am going to get out of this and engage in some permanent business." It appears to be the same old story "that familiarity breeds contempt." On the other hand, the following effective tribute, which is probably the most unbiased and spontaneous that has ever been paid to the United States Patent System, was delivered by Mr. Karekiyo Takahashi, the special commissioner appointed by the Japanese Government to gather data regarding our Patent System as practiced in the year 1899. In response to a query as to why the people of Japan desired to have a patent system, he said:

"I will tell you. You know it is only since Commodore Perry in 1854 opened the ports of Japan to foreign commerce that the Japanese have been trying to become a great nation like the other nations of the earth, and we have looked about us to see what nations are the greatest, so that we could be like them; and we said, 'There is the United States, not much more than one hundred years old, and America was not discovered by Columbus yet four hundred years ago;' and we said, 'What is it that makes the United States such a great nation?' And we investigated and found that it was patents, and we will have patents."

SOME DEFECTS IN THE PRACTICE OF OUR PATENT SYSTEM AND SUGGESTED REMEDIES.

By Louis C. Raegener.

Mr. Chairman and Gentlemen:

In addressing you to-night I am taking a position somewhat similar to that of the illustrious Andrew Carnegie. After the tariff had given him personally all the protection he required, he came before the world and proved that protection for him at least was no longer necessary.

In my opinion the practice and procedure of trying a patent case is antiquated, time-consuming, abnormally expensive and generally unsatisfactory; final judgments are practically unobtainable, and pecuniary judgments so rare that any well-advised infringer need hardly fear them.

Once upon a time—so long ago that the memory of mankind runneth not to the contrary—our U. S. Supreme Court formulated rules of practice and procedure. If these rules are followed, the testimony in a patent suit can be ready for the court at the end of six months. Practically, however, it takes from a year to eighteen months to reach this point. The testimony of witnesses is taken at the lawyers' offices, usually on the typewriter. If taken stenographically, the stenographer benefits more by that method than the lawyers, because the lawyers and experts as a rule charge per diem, and of course the longer it takes, the more per diems.

The worst feature of this method of taking testimony is that it is practically legally impossible to stop either the witness or counsel from padding the record with testimony that would never be admissible were the trial in open court and questions could be ruled upon forthwith. The record is unconsciously padded in this way by nearly everybody. Firstly, it is in the direction of everybody's interest to do so; and, secondly, it requires less brain work to stuff the record than to concentrate one's thoughts upon what is strictly relevant and competent—thereby condensing the record.

It is not unusual for an expert witness to favor the court with his views on the witness stand for weeks and months at a time—and, by the way, eminent counsel once defined an expert to be one who swears expertly.

The legal taxable costs for a deposition of this kind to the successful party are $2.50. Of course this may have been a fair compensation a hundred years ago, but it does not compensate a day-laborer to-day for a day's work.

It is not at all unusual for a suit involving fairly important issues to cost each side at the rate of from ten to twenty thousand dollars per year.

After all the testimony has been taken it is printed, an oral argument takes place, printed briefs are submitted, and after the close of the arguments, if not before, the court goes to sleep, forgets the oral arguments, and after the lapse of considerable time renders an opinion resulting in a decree dismissing the complaint or directing an injunction and an accounting.

There is usually a stay of the accounting pending an appeal by the unsuccessful party to the Circuit Court of Appeals for that Circuit; and if the decree of the court below is affirmed, testimony for an accounting is taken before a master, and again all the elements combine to protract examinations and increase expenses. Except in extraordinarily important cases it is best to drop the accounting. The defendant is usually an irresponsible corporation or has lost or burned his books of account. The burden of proof is very heavily on the complainant, and the law very jealously protects the poor infringer against unjust attacks. If you are lucky you get a judgment which is not reversed on appeal; you must be still luckier if you collect it; and if the expenses of obtaining and collecting such a money judgment are less than its face value you are indeed a fortunate man.

I always believed with old Galileo that the earth does move, and if I succeeded in getting an injunction I usually had enough of my client's money by that time and was anxious to turn to the next job.

I have, however, heard of others occasionally collecting a judgment of some magnitude.

Now, having obtained your injunction in this United States, which is generally believed to extend from the Atlantic to the Pacific and from the Gulf to Canada, you might be foolish enough to think that your patent is valid and will henceforth be cheerfully respected. If you think that, you are wofully mistaken, for, mark you, there are nine Circuit Courts of Appeals of the United States of coördinate jurisdiction. For instance, the Second Circuit takes in New York, Connecticut and Vermont; but New Jersey and Pennsylvania are in the Third, and what is law in the Second Circuit in any particular case is not necessarily the law in the Third, as the latter tribunal will frequently—"always with a heavy heart, however"—intimate, when coming to a diametrically opposite conclusion from that reached by the Second.

So that if you get an injunction in New York City against the "Excelsior" corporation with a capital stock of five hundred dollars, the incorporators can go to Hoboken and incorporate as the Hoboken Excelsior Co., and everything, every detail that I have told you about the conduct of a lawsuit must be done all over again at similar expense of time and money.

The usual result is that the last Circuit Court of Appeals differs from the first, seldom that it agrees with it, and of course only when it cannot help itself. Otherwise it would surrender its own judgment, which those of you who are married all know is supposed to be wrong.

Now you are worse off than you were before. Before you began your first successful suit you could at least threaten or bluff with the patent and occasionally find an honest man who might give you a cent or two more for a patented article just so that he might feel that he was not a *tort feasor* every time, but now you are ridiculed and laughed at.

When I began the practice of the law over thirty years ago, appeals from the Circuit Court were as a matter of right to the U. S. Supreme Court. It did take two or three years before your case was reached, but when it was reached you got "a square deal" whether the decision was for or against you. It was the end—but now there is never an end in sight for the unfortunate patentee.

Some genius, who in my opinion ought to be crucified, invented the present system of appealing to the Circuit Court of Appeals and inserted a provision that no appeals from these Courts lie to the U. S. Supreme Court. It is true you can petition the U. S. Supreme Court to review a decision of the Circuit Court of Appeals, but as this august tribunal is exceedingly busy with many matters of the greatest importance most of such petitions are denied without an opinion, and the result is that whenever a diversity of opinions exists between two Circuit Courts of Appeals as to the validity of a patent, and a petition for certiorari is denied you can keep on litigating and guessing which of the two Courts is right, and the chance is one guess will be about as good as the other.

There have been attempts to create one Court of Appeals at Washington to which all appeals in patent cases would lie; but as the creation of such a court means the dispensation of patronage, I dare say that another decade or so will expire before such an improvement can go into effect.

I have narrated facts to you. How to cure these ills I am not prepared to say. In the earlier years of my practice I did not appreciate the great crime committed against the interests of the people, and now I am too old to make an effort to change them; but like Carnegie, to some slight extent I must admit that I have benefitted by these protracted and iniquitous laws.

You can readily see what such a condition of affairs leads to. As a young man I took for granted that all judges were honest and learned. I have never since had reason to change my mind as to the integrity of the judges, but the capacity and ability of judges in patent causes is as varied as the colors of the rainbow.

That patent lawyer is indeed inexperienced and careless if he does not carefully select his tribunal, so far as it is possible, for among the judges there are some who are so opposed to monopolies and patents that a mere look at the red seal and blue strings of Letters Patent incites them to such wrath that the patent is instantly doomed.

Please don't think for a moment that this is a joke, because nobody has ever yet been able to define what is invention or discovery and how much or how little genius must be shown, how laborious and how brilliant the work must be to dignify it with the name invention, and therefore any "learned" judge can always decree that the claims of the patent are void for lack of patentable novelty, and no tribunal can really settle that question in any particular case except a court of last resort like the U. S. Supreme Court.

Therefore it does make some difference whether you go before one court or another, even admitting that both courts decree according to their conscience; but after all, if the court of last resort finally finds in your favor, it is a poor consolation to say the judge meant well. It would have served your interest much better if he habitually meant ill and at the same time decided correctly in every instance.

While I have pointed out to you the abuses from which you as inventors and patentees suffer in the attempt to enforce your rights, I shall before closing call your attention to a ridiculous abuse from which of late the poor public suffers.

An infringer is called a *tort feasor*, and that is a bad enough name; but a contributory infringer is the worst specimen of humanity imaginable.

This theory of contributory infringement is, like alfalfa or Indian corn, the peculiar growth of our country, and I advise some of you patentees to cultivate this theory and profit thereby.

Under the law as made by the courts to-day you can sell your patented engines, your aluminum, your carbide and other patented products, upon condition that the purchaser, as long as he uses your patented device or process, shall buy his flour, eggs, oatmeal or soap, his rat traps, furniture or whatever you please, from you or your agents; and if any wicked provision or hardware dealer should be mean enough to sell any of these articles to your venders with knowledge of these restrictions he becomes a contributory infringer, and you can annoy him and sue him and make him account for your losses.

This country never does anything by halves. It gives the worst protection possible to the patentee seeking to enforce the rights plainly given him by the statutory law; on the other hand, by trick and device not found in the statutory law or in the patent laws of any other country, it enables him to hamper trade in the most beautiful manner possible.

Of course this theory of contributory infringement does much to make patent laws unpopular, and in the end the honest patentee will suffer.

You might ask me what remedies I have to suggest for all or some of these evils.

I can only say that in England all patent suits are tried in open court and more quickly disposed of in consequence. The successful party recovers, say, two-thirds of his real expenses paid to lawyers and experts—but I do not profess to be accurate on this point.

In Germany, I understand, every patent is absolutely valid after five years, and during the five years practically valid unless a proceeding is brought during this period in the Patent Office to vacate the patent.

Of course neither in Great Britain nor Germany are there nine courts of last resort and of coördinate jurisdiction. The necessity for establishing one tribunal of last resort, be it a special Court of Appeals or be it again the U. S. Supreme Court, seems to me so self-evident that no man of ordinary intelligence can doubt it.

That the public would benefit by the taking of testimony before judges with some power to rule upon the evidence goes without saying, even if this would necessitate the appointment of a score or more of additional judges.

The theory of contributory infringement will probably be exploded sooner or later or confined within reasonable limits by the U. S. Supreme Court.

The abuses to which I have called your attention will undoubtedly be remedied in time, and probably by the bar itself; but you inventors are more interested in a speedy correction of these abuses than the bar, and united action on your part would undoubtedly do more to bring about the needed changes than waiting for some one else to do the work.

141 Broadway, New York,
January 8, 1909.

THE UNITED STATES PATENT SYSTEM, ITS USE AND ABUSE.

SOME SUGGESTIONS AS TO DESIRABLE IMPROVEMENTS.

By B. C. Hesse.

The United States Patent System has been so successful in the attainment of the primary object of a patent system, namely the stimulation of invention that changes therein should not be made without full consideration.

The full, free and untrammeled exercise of the monopoly granted by the Federal Government to an inventor in return for a complete disclosure to the public of his invention should be interfered with to the smallest extent consistent with full protection of the public's rights and further, the monopoly granted to the individual should be protected to the fullest extent consistent with the public's rights.

My experience has led me to the conclusion that at some points the inventor's monopoly when fully exercised tends to inflict a damage upon the public and that, on the other hand, failure of the public to make effective the monopoly granted tends to inflict, in some instances, unnecessary penalties upon inventors.

The chemist is concerned with all the subject matter which can be the object of patent protection, namely, new and useful arts, machines, manufactures and compositions of matter; fuller protection for the chemist is hardly conceivable.

When an inventor, chemist or other, obtains a patent it is supposed to be for something new and useful and he may reasonably be expected to exploit that invention so that the public can benefit by having access to it or to the use thereof subject to such restrictions as the inventor may regard as reasonable for his own protection and remuneration.

When, however, such inventor allows his invention to lie idle and without performing any useful function and without reasonable prospect thereof either by offering it to the public for its use or by acting as a protection for related inventions which he is actively exploiting and bringing to the public, then such inventor is, in my opinion, transgressing the spirit underlying the Patent System. He is reserving for himself a field of endeavor which he has agreed to exploit and is not exploiting and by his reservation is preventing its exploitation by others. To this extent he is retarding progress.

A partial remedy for this might be a low annual tax, say, $15 or $25 or some other sum not high enough to stand in the way of any invention giving any reasonable promise of remuneration yet high enough to make it appear unprofitable to many to retain reservation in fields which they are either unwilling, unable or incompetent to develop. To this extent reservation would be curtailed and the return of reservations to what might be called the "public domain" would be affected, for exploitation by the public as occasion might arise.

It would serve no good purpose, at present, to enumerate such instances but certainly all who have read the numerous patents as they appear in the *Official Gazette* have been struck with the idea that a great many patents have little or no utility, present or prospective, and many of those coming into contact with inventors and patentees have met with many cases where the ability to develop or exploit, or the ability to get others to develop or exploit is lacking or has been wholly exhausted and have also met cases where the ideas embodied in such patents could, with profit to the public, be adapted to use by others but which have not been so adapted or developed in other arts because of such patents.

When an applicant for a patent has succeeded in overcoming the objections, if any, raised to such application by the Patent Office (which is, in effect, acting for the public) it would seem reasonable that the ground covered by the patent so issued be regarded as conceded by the public to belong to the inventor and that any one contesting the legality of such reservation should be compelled to remain out of the field until he proves the contrary.

Experience, however, has led the courts to adopt a different view and in the majority of cases a patentee has to contend for the legality of his reservation, sometimes, if not generally, expending the greatest part of his energy in a litigated case in proving his title, as it were, while curtailment of the granted monopoly is going on without restraint. The reason for this seems to be that the courts have come to the conclusion that our system of preliminary examination does not, in fact, properly safeguard the interests of the public; the reasonableness of this is clear on considering the practical impossibility of any one man, or set of men of reasonable proportion, to be so fully acquainted with the entire art involved in all inventions made and operated, as to give, in all cases, a decision which is bound to be just. The remedy, therefore, would seem to lie in the direction of improving or elaborating our system of preliminary examination because I believe an absolute abandonment of preliminary examination to be necessarily vicious.

The remedy which I beg leave to suggest is that after an application has been passed to allowance in the Patent Office the public, or so much of it as is interested, be given access to the disclosure of the invention and a reasonable time within which to oppose the final grant of the monopoly, as is done in Germany, or a patent might issue subject to the conditions that if it be successfully opposed by the public within a reasonable time from the date of issue, say, one year, the scope of the patent will be diminished to the extent the opposition succeeded or revoked as the case may be; a patent obtained under either mode to be given the right of preliminary injunction in its favor and all infringement or alleged infringement thereof to cease or to be prohibited pending final adjudication of the validity of the grant.

Of these two propositions I am inclined to favor the first because so long as the grant of a patent is withheld exploitation is not likely and enterprises based upon what may be false foundations will be less likely to be started, as might well be the case under the second suggested remedy thus causing avoidable, and therefore unnecessary, losses and disappointments.

By this means the inventor, on the one hand, would receive a patent effectively shutting off infringement and consequent interference in his granted monopoly until his opponents have properly proven the illegality of the patent. The public, on the other hand, would be spared the issue of a very considerable number of patents presumably invalid and by exercising its rights to oppose the grant of patents of doubtful validity would save to itself many opportunities of endeavor and development and the expense and annoyance of proving in court the invalidity of a granted patent together with the annoyance and interference which an invalid patent is capable of producing.

In return for this, the inventor would probably be required to have his monopoly date from the date of the disclosure of the invention to the public-at-large by the Patent Office, the time required to overcome the opposition by the public being very likely a proper charge against the patentee's term of monopoly.

It should be noted that it is not contemplated to take from the public the right to attack the legality of the grant of the patent but that that right remains fully open to the public throughout the entire life of the patent, subject to the condition that the patentee's rights must be fully respected until he is proven to be in wrongful possession.

This covers all the changes that I feel I should care to suggest.

There is another suggested change that is agitating a great many inventors, economists and the public generally, which is the so-called working of patents.

Personally, I am unfavorable to a compulsory working of any patents; personally, I believe that a patentee ought to be permitted to practise his invention where it best suits his convenience and the trade conditions he has to encounter; so long as he gives the public access to his invention, directly or indirectly, he has in my present opinion carried out the spirit underlying our Patent System. If circumstances are such that it is not profitable or possible to exploit the invention a patentee should not be compelled to work his patent in spite of such adverse conditions.

The foundation of the agitation for the working of patents seems to be a supposed loss of income or advantages to the public-at-large said to be due to inventions protected by United States Patents but imported from abroad, the corrective idea being that the country would be benefitted by having such patented articles or processes produced or practised within the United States. If that be the true reason then it seems to me to be foreign to the purpose and intent of the patent law and very probably antagonistic to a full and free exercise of its contemplated and proper functions. There may be other ways, perhaps by levying a special tariff on goods imported from abroad and patented in the United States, to accomplish this object, such levy to be reviewable by the Secretary of the Treasury and rescindable upon satisfactorily showing that the goods cannot be economically produced in this country. To attach a condition of working a patent, no matter how limited, under penalty of revocation or other restriction of the granted monopoly is, in my opinion, not the best way to accomplish the desired end and, I believe, not in harmony with the purposes and spirit of the Patent Law.

THE UNITED STATES PATENT SYSTEM, ITS USE AND ABUSE

THE INVENTOR'S STANDPOINT.

By Dr. L. H. Baekeland.

The patent laws of the United States have been conceived with an unparalleled spirit of liberality towards the inventor. The law-giver in framing these statutes had certainly in mind to offer to the inventor the maximum of protection for his intellectual property. In no other country whatever is a patent obtainable at such a small cost; nowhere is the intended protection extended for as long a period as in the United States; nowhere is the real and first inventor so much guarded against possible disclosures or unfair indiscretion of others. In most other countries the patent laws seem to have been conceived with the main idea of bringing in a source of revenue for the government. In most of them additional taxes are levied every year which make the burden of the struggling inventor a very heavy one and seem to discriminate in favor of the rich man or the rich corporation who can afford to pay the yearly tribute.

My relations with the United States patent examiners have shown me that with very few exceptions these officials are able, earnest and fair. They can compare very favorably with the best of all other countries. Their honesty of purpose and conduct has never been questioned by anybody except by people who instead of investigating the subject, merely limited themselves to sneering at them.

In late years one single case has transpired of a subordinate assistant examiner who seems to have tried to get appointed to the patent office staff with the very purpose of committing a dishonest act, hoodwinking the vigilance of the chief of his department. That such abuses have not occurred oftener is a real wonder when we take into consideration that the corps of examiners are an underpaid, ill-recognized, unrewarded set of fine men. Their devotion to their work with no hope of reward beyond a meagre salary is a fine example of good citizenship.

I know of many a time when views contrary to these were expressed by some impatient inventors who happened to believe that everybody in the world and especially patent examiners ought to be as thoroughly acquainted with their inventions as they themselves were. To such of my misguided friends, I must recall the fact that the patent examiner here in the United States or any other country is a man at whom are flung every day new ideas widely different in scope, in direction and in details. Many of these ideas are undigested or unclearly expressed. Other ones are the result of months and years of mature and deliberate specialization and it is hardly to be expected that a man, however open-minded he may be, should at the first glance be able to penetrate the subject as deeply as the intelligent, specialized inventor. The standpoint the examiner takes in a case where things are not very clear is that of a representative of the average uninitiated public whose interest he has to protect as well as that of the inventor. His attitude is best summed up in slang: "I am from Missouri," and it becomes the task of the inventor of "showing" and "showing why."

In several of my experiences with the patent examiners here and abroad, I have felt very thankful for the objections which were made to my texts and to my claims because I was shown either that I was not sufficiently explicit or concise, and by making the required amendments, I avoided much future trouble in the eventuality of an infringement. It may be a consolation to the United States patent examiners to know that their colleagues of other countries, for instance England, Germany and Austria, are criticized just as much by some dissatisfied and narrow-minded inventors, although their attitude is just as fair, unbiased and open-minded.

Summing up, I could hardly suggest an improvement in the United States Patent law without curtailing the privileges and interests of the poor inventor.

If I venture to make one suggestion, it would be to propose an act of reciprocity, which would consist in exacting compulsory working of all patents taken here by non-citizens of the United States and whose countries have patent laws with a clause for compulsory working which means great hardship for the American inventors who take out patents abroad.

On the other hand, it is very unfortunate that although the laws for filing and registering a patent in the United States are almost all that can be desired, I must lift my voice of protest when it comes to testing the rights of the inventor before the courts. Here the poor inventor is entirely at the mercy of a legalized system of piracy as carried out by infringers helped by all the tricks of lawyers, and let me say to the shame of our own profession, helped very often by experts who will back the lawyer to confuse an issue before a judge who most of the time is already incompetent on account of lack of theoretical or practical knowledge in the art. This game is so successfully played, that I know of rich companies here in the United States whose main method of procedure is to frighten, bulldoze and ruin financially the unfortunate inventor who happens to have a patent which he is not willing to concede to them on their own terms; that is to say, for next to nothing.

I could cite you several examples of prosperous companies where the money paid in salaries for the technical or scientific staff and for royalties is a mere bagatelle if compared to the fortunes paid annually to their lawyers who happen to look after their patent litigations.

Thus has it come about that an otherwise liberal patent law intended for the protection of the poor inventor has become a drastic method for building up powerful privileges in the interest of big capitalistic combinations.

The sooner we have a special and adequate patent court to which all patent litigation can be referred and which can operate without the absurd delays and abominable expenses now involved in patent suits, the sooner will cease this arrogant frustration of the generous efforts of those who framed our patent laws.

NOTES.

PHOSPHORUS FOR THE HEMPEL PIPETTE.

The method of preparing sticks of phosphorus for use in the Hempel pipette for the determination of oxygen, as usually described, does not take advantage of a little device which suggested itself to me some years ago and which I find exceedingly satisfactory.

I proceed as usual until the liquid phosphorus is expected to solidify in the glass tube. Phosphorus is very prone to undercooling and it often requires considerable patience to await its solidification. When, however, the liquid phase is brought into contact with the solid phase equilibrium is promptly established and the column of liquid phosphorus solidifies the very instant the temperature of the normal point of solidification is reached. Contact can readily be insured because the liquid phosphorus usually protrudes slightly from the end of the glass tube and if a piece of solid phosphorus is suspended by a wire in the cooling bath one can easily bring solid and liquid together while the latter is cooling.

It will be found that this little device not only simplifies and expedites the whole operation but insures greater uniformity and far greater yield of perfect sticks.

H. August Hunicke.

MITCHELL-WALKER MOISTURE TEST.

During the past year we made investigations in rapid moisture determinations in dairy products. We personally examined a number of the tests on the market and consulted the reports of other chemists on the other tests in vogue.

The beaker test, consisting of weighing into a cup a quantity of butter, heating till foaming ceases and re-weighing, we considered a good test but open to inaccuracies when used by the ordinary creamery man. Some of its disadvantages are, the necessity of weighing very accurately, thus involving a sensitive and comparatively expensive balance; uncertainty as to the right moment to stop the heating, and to re-weigh the dehydrated butter; necessity of calculating the percentage of water from the weights.

Gray's Test involves a fragile glass contrivance and gave results from 0.5 to 1.0 per cent. too low. The test is also rather expensive to operate. The "improved" Gray's Test proved to be still more fragile and awkward to clean, but gave more accurate results due to a modification in the scale.

Other tests presented undesirable features in one form or another.

We devised a test, attempting to conform to the following requisities:

1. A rapid and reasonably accurate method. 2. An inexpensive form of apparatus both to purchase and to use. 3. A durable form of apparatus and one easy to clean. 4. A method that requires no great amount of attention or care in operating, and that can thus be used by the average factory man. 5. An apparatus that can, if damaged, be easily repaired at small cost. 6. A method equally suitable for testing butter, curd and cheese.

Our apparatus consists of a thin, copper still attached by a friction joint to an upright brass condenser fitted with a block-tin inner tube. A glass receiver reading directly in percentages and with a stop-cock near its lower end, hangs from the lower end of the condenser. This apparatus is supported on an upright brass rod to which is also attached an adjustable copper spirit lamp.

In making a moisture determination in butter, ten grams of the sample are weighed into the copper still counterpoised on a small balance. Ten cc. amyl acetate are added. The still is then connected with the condenser and heat applied by the adjustable lamp till water and amyl acetate cease to drop from the lower end of the condenser. The graduated receiver is then removed and the percentage of water read off directly at the line of demarcation between the lower layer of water and the upper layer of amyl acetate. In testing curd and cheese, five grams are used instead of ten, and the percentage read off on the scale opposite the butter scale.

For the sake of economy the amyl acetate is collected and dehydrated in the still and used over again.

The average cost per test is less than half a cent. The time required for a test, including the weighing of the sample, is about ten minutes.

The test serves also for moisture determinations in such substances as oils, fats, wood pulp, bread, flour, etc.

The results obtained by this apparatus seldom vary from the gravimetric results more than two-tenths of a per cent., and usually not more than one-tenth. The apparatus is simple in all its parts, and practically indestructible, since it is made almost entirely of metal.

Descriptive bulletins may be obtained upon application to W. O. Walker, Eastern Dairy School, Kingston, Ontario, Canada.

J. W. Mitchell,
W. O. Walker.

A CONVENIENT FUNNEL FOR INTRODUCING DRY REAGENTS OF POWDERS INTO BOTTLES AND FLASKS WITHOUT LOSS OF SUBSTANCE.

In order to avoid loss of small amounts of such finely powdered materials as flour, etc., when introducing them into bottles and flasks, after weighing on the balance, it has been found very convenient to make cones or stemless funnels from celluloid film. The celluloid film required should be transparent and about the thickness of a calling card. It can be obtained from dealers in art goods. A paper cone is first made of the proper size and then the celluloid is cut, using the paper as a pattern. The lap-joint along the side can be made perfectly tight and smooth by applying the proper amount of acetone to effect a softening or partial solution of the celluloid

Charles H. Briggs.

The Howard Wheat & Flour Testing Laboratory,
Minneapolis, Minn., Jan. 7, 1909.

TILLANDSIA USNEOIDES.

Tillandsia usneoides, called Spanish moss, Florida moss, New Orleans moss, long moss and black moss, is very common in Louisiana. It is an epiphyte and grows on both dead and live trees. It can be found growing on trees that have been dead for years. On trees having a dense foliage it does not grow so well, as sunlight is one of its requirements for existence.

Many people claim that it kills the trees and they regard it as a parasite. Billings[1] says that Spanish moss does not injure the trees on which it grows and he has demonstrated that it depends wholly upon air, sunlight and rain for its life.

In the winter when the pastures are poor, farmers often chop down trees and allow the cattle to feed on the moss. In order to ascertain the feeding value and the composition of this plant the following analysis was made:

Composition of Tillandsia Usneoides (Spanish Moss).

Complete analysis.

Water	69.500
Volatile matter	28.905
Silicon dioxide (SiO_2)	0.590
Iron and alumina oxides (Fe_2O_3 and Al_2O_3)	0.286
Phosphoric acid (P_2O_5)	0.032
Calcium oxide (CaO)	0.058
Sulphur trioxide (SO_3)	0.184
Potassium oxide (K_2O)	0.313
Sodium oxide (Na_2O)	0.581
Total	100.449

Feed constituents.

	Natural state.	Water-free bases.
Protein	3.68	12.07
Ether extract	1.06	3.47
Carbohydrates	15.95	52.29
Fiber	8.24	27.02
Water	69.50	..
Ash	1.57	5.15
Total	100.00	100.00

---

[1] *Botanical Gazette*, **38**, 99–121.

Fertilizer constituents.	Natural state.	Water-free basis.
Phosphoric acid	0.032	0.105
Nitrogen	0.588	1.928
Ammonia	0.715	2.343
Potash	0.313	1.026
Water	69.500	...

J. E. HALLIGAN.

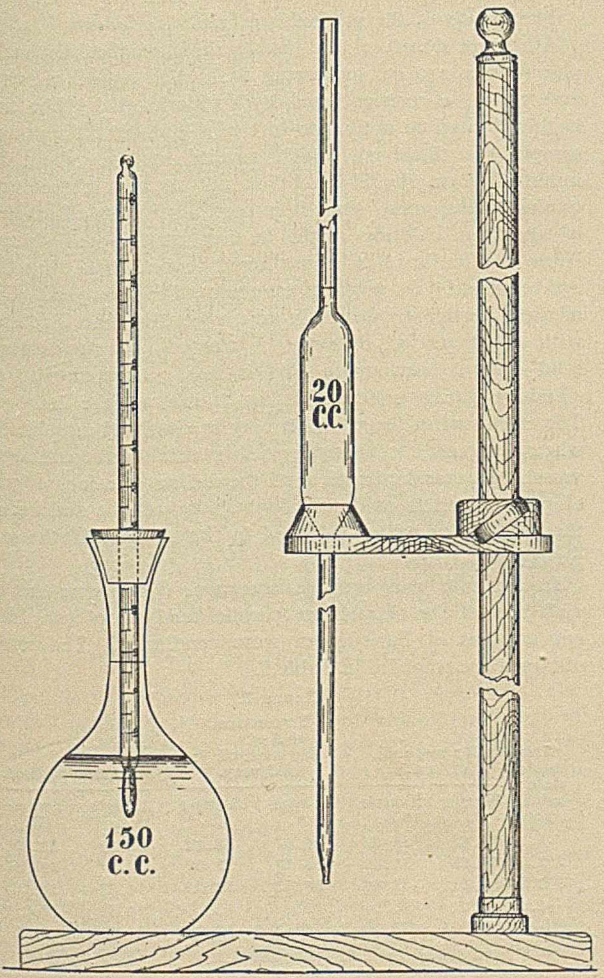
LOUISIANA EXPERIMENT STATION,
BATON ROUGE, LA.

SIMPLE SPECIFIC GRAVITY APPARATUS FOR PORTLAND CEMENT.

Apparatus.—The apparatus consists of a 150 cc. flask having a mark and the neck above flared out, a 20 cc. pipette, and a Fahrenheit thermometer inserted in a cork. The internal diameter of the neck of the flask at the graduation should not be less than 8 mm. nor more than 10 mm. The pipette is set upright in a filter stand to drain when not in use. The flask need not be accurate but the pipette must be calibrated.

Calibration.—Clean pipette perfectly, and dry. Make a mark 10 or 15 mm. above the graduation and another the same distance below the graduation. Fill to upper mark, with distilled water at 60° F. from a beaker and empty back again. Drain 10 minutes, blow out, and fill again. Empty into a weighed flask, drain 10 minutes, and blow out into flask. The weight of water delivered is that of the volume drawn up with the walls wet. Fill to lower mark, weigh, draining and blowing out as before. The difference between the two weights is the weight of the volume between the two marks. The position of the true graduation mark is then easily found and etched or scratched on, for example:

Distance between marks	20 mm.	
Volume to upper mark	20.32 cc.	
Volume to lower mark	19.76 cc.	
Volume between marks	0.56 cc.	
Volume for 1 mm. 20 (0.56)	0.028 cc.	
Volume of true mark above lowest mark	0.24 cc.	
Distance of true mark above lowest mark	0.028	(0.24) 8.5 mm.



Use.—Fill the flask nearly to the mark with kerosene oil. Take the temperature with thermometer, remove, and make up exactly, reading the bottom of the meniscus. Wipe the inside of neck free from oil. Blow out the pipette and withdraw 20 cc. of oil from flask, draining the outside of the delivery tube against neck near mark before removing, empty and hang up to drain. Weigh out 65 grams of cement to 0.1 g. and pour from glazed paper into flask until the oil nearly rises to the mark. Use a small camel's hair brush to brush the cement with all adhering oil down the neck. Rotate flask to remove air till all the cement is suspended and cautiously add cement till the mark is reached. Rotate again, take temperature, and weigh the residue to 0.1 g. The weight of cement used divided by 20 gives the specific gravity. Correct for temperature change, 1° F = 0.01 sp. gr. When the temperature rises add the correction; when it falls, subtract. The flask can now be shaken out, rinsed with oil, and filled nearly full as before, ready for the next determination. The pipette needs only to be cleaned when cement or dust is seen on the inside.

Results.—The difference in temperature between the pipette and the oil in the flask should not be too great. The cement is usually warmer than the oil and apparatus even after standing together some time, probably due to the slow hydration of lime. The effect of the difference in the coefficient of expansion of various samples of oil is negligible. The coefficient for 1° F. is taken as 0.00050, that of glass is neglected. A sample of oil in use gave a coefficient of 0.00053 with the pycnometer, using two temperatures. In calibrating, the correction in volume for the density of water at 60° F. is practically equal to and counter-balanced the difference in volume between oil and water retained in the pipette, so that both can be ignored.

Oil retained in pipette	0.06–0.07 cc.
Water retained in pipette	0.04–0.05 cc.
Correction for 20 gr. at 60° F.	0.02 cc.

Errors.—Any one of the following errors represents a difference of 0.01 sp. gr.

0.2 g. cement used.
0.065 cc. oil displaced.
1° F. change in flask.
6.5° F. change in pipette.
1 mm. meniscus above or below mark on flask.
0.00010 variation in coef. of exp. through 5° F. change.

FRANK GOTTSCH.

MT. PROSPECT LABORATORY,
Brooklyn, N. Y.

AN UNUSUAL INCRUSTATION ON GENERATOR COILS.

(Contribution from the Laboratory of the York Manufacturing Company, York, Penn.)

There was recently submitted to our laboratory, for analysis and explanation, a scale or incrustation of unusual character. It had been formed on the coils of pipe in the generator of a refrigeration plant built on the absorption system. The conditions in this plan of refrigeration are as follows. The generator is very similar in construction to a tubular boiler, the tubes being of two-inch iron pipe through which steam circulates. These steam coils are surrounded by strong aqua ammonia, 20° to 22° Bé. in strength, from which the heat of the steam coils drives the gaseous ammonia which is to be condensed and used as a refrigerant. The aqua ammonia comes to the generator at a temperature depending upon the cooling water used in the condenser, not often being lower than 55° Fah. and ranging at times up to 75° Fah.

The scale under consideration was formed on the outside of the steam coils and hence was deposited from the aqua ammonia and therefore represents materials dissolved by the latter. The incrustation had a maximum thickness of about one-sixteenth inch, was of an olive-green color on the outside surface, rusty from the iron pipes on the inside surface and of a somewhat varying light green on a broken cross section. Its texture presented a stony, somewhat crystalline appearance, its hardness was about 5, and its specific gravity 5.117.

The surprising feature of this material appeared upon analysis, which resulted as follows:

Insoluble matter	0.26%
Organic (oily matter)	0.59
Copper oxide	0.65
Ferric oxide	6.32
Zinc oxide	92.00
Total	99.98

It was found upon inquiry that at the plant in question some of the parts had been made of galvanized iron. The aqua ammonia coming in contact with these galvanized surfaces dissolved off some of the zinc and this in turn was deposited on the surface of the steam coils under the influence of the heat. The ferric oxide came from contact with the iron pipes.

In this connection it may be of interest to note that the action of anhydrous liquid ammonia upon galvanized and tinned iron pipe was made the subject of experimental investigation in our laboratory several years ago. The result of that inquiry, briefly stated, is that a galvanized surface is readily corroded by the ammonia while a tinned surface is but little affected.

The apparent lesson to be drawn is that galvanized iron should be avoided in all ammonia fittings.

C. H. Ehrenfeld.

TO DETERMINE FAT IN SWEETENED CONDENSED MILK.

Weigh out 25 grams of the sample, dissolve in water and make up to 100 cc. Transfer 6 cc. to a double tube milk flask provided with a small bore tube graduated to give percentage of fat for 5 cc. milk. Add 4 cc. of ether and 4 cc. acetic acid (80 per cent. or more absolute acetic acid). Acetic acid of this strength will dissolve the curd but has no effect on the sugar. Place the flask in a vessel of warm water and heat until the ether is expelled. A layer of milk fat will now be seen floating on the surface of a clear and colorless liquid. Fill the flask with hot water thus raising the fat into the graduated tube. The percentage of fat can now be read, the sample whirled in a centrifugal machine and another reading made. Multiply the reading by four.

C. B. Cochran.

A QUICK METHOD FOR THE DETERMINATION OF ARSENIOUS OXIDE IN PARIS GREEN AND OTHER INSECTICIDES.

Among the numerous methods that have been suggested for the analysis of the common insecticide, Paris green, are the methods of Thorne, Smith[1] and Avery.[2]

By the use of any of these methods it is possible to obtain very accurate results. In the Smith method, however, the determination of the end-point offers some difficulty, and in both the Smith and the Avery methods, the expenditure of considerable time is involved.

The following method is presented because of its simplicity, and because of the rapidity with which it may be carried out. With the standard iodine solution already at hand, it is possible to perform the analysis within about twenty minutes, or within one-fourth of the time required for the work when either of the other methods is employed.

The details of the method are briefly as follows:

A 2 gram sample of the insecticide is weighed out and is placed in a 250 cc. measuring flask with 25 cc. of water, and 25 cc. of concentrated hydrochloric acid. The flask is then heated to a temperature of about 80° C., until the sample has dissolved, which usually requires about five minutes. Care should be taken not to heat the solution to the boiling point, since it is possible that small amounts of arsenious chloride might be lost at this temperature. After the solution has been allowed to cool, it is diluted to 250 cc., and 25 cc. portions are removed for analysis. Each of these portions is placed in an evaporating dish, is diluted with about 300 cc. of water, is treated with an excess of solid sodium bicarbonate, and is finally titrated with the standard iodine solution, using starch as the indicator. The end point in the titration may be observed most readily when a porcelain evaporating dish is used as the containing vessel. In standardization of the iodine solution, 1 gram of pure arsenious oxide should be weighed out and carried through the procedure adopted in the analysis of the sample of Paris green.

In order to compare the accuracy of the new method with that of the two others, parallel analyses of two different samples of Paris green were conducted. The results obtained are recorded in Table I.

Table I.

Percentage of As_2O_3.

Number of determination.	By method of Smith.		By method of Avery.		By new method.	
	Sample A.	Sample B.	Sample A.	Sample B.	Sample A.	Sample B.
1..	54.47	52.02	54.62	52.10	54.51	52.00
2..	54.53	52.02	54.58	52.08	54.51	52.00
3..	54.51	52.16	54.68	52.08	54.52	52.16
4..	...	...	...	...	54.51	52.05
Ave.	54.50	52.10	54.62	52.08	54.51	52.05

---

[1] *J. Am. Chem. Soc.*, **21**, 769 (1899).

[2] Bureau of Chemistry, Bulletin No. 107, p. 26 (1907).

The results obtained in standardizing the iodine solution with pure aresenious oxide are given in Table II.

TABLE II.

Number of determination.	Grams of As_2O_3 equivalent to one cubic centimeter of the iodine solution.		
	By method of Smith.	By method of Avery.	By new method.
1..........	0.002894	0.002896	0.002898
2..........	0.002894	0.002896	0.002897
3..........	0.002895	0.002896	0.002898
Average..........	0.0028947	0.002896	0.0028977

C. C. HEDGES.

CORNELL UNIVERSITY,
December, 1908.

NEW PLANTS.

THE INDIANA STEEL COMPANY'S PLANT AT GARY, IND.[1]

BY W. D. RICHARDSON.

The commencement of operations in the largest metallurgical plant which has ever been planned and constructed is an event well worth recording not only in commercial, technological and engineering literature but in chemical literature as well. While to the superficial observer it may appear that in the working of a plant such as that at Gary, mechanical problems overshadow the chemical at every point, this is not strictly the case. The science of metallurgy embraces both mechanical and chemical methods, and while in the mills mechanical methods predominate, in the operation of blast furnace and open-hearth furnace chemistry comes to the fore. That the chemical operations of the blast furnace, although carefully studied for long periods of time, may yet be fundamentally improved, is well attested by the recent successful introduction of the Gayley dry blast in various furnaces throughout the country.

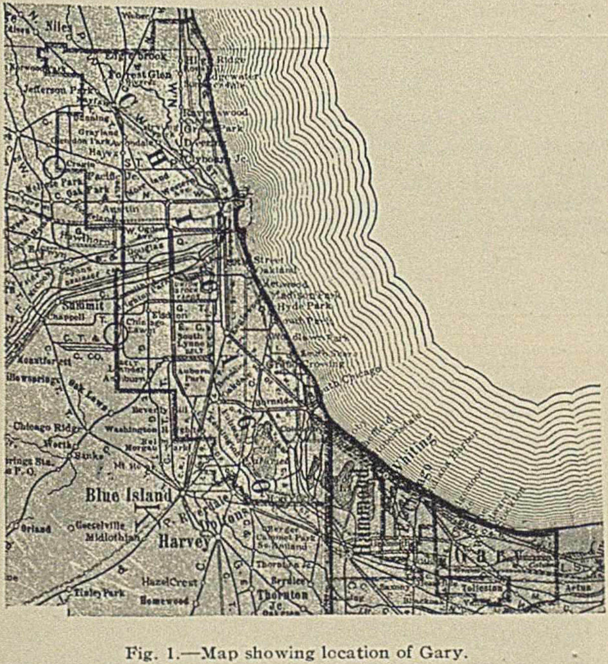


Fig. 1.—Map showing location of Gary.

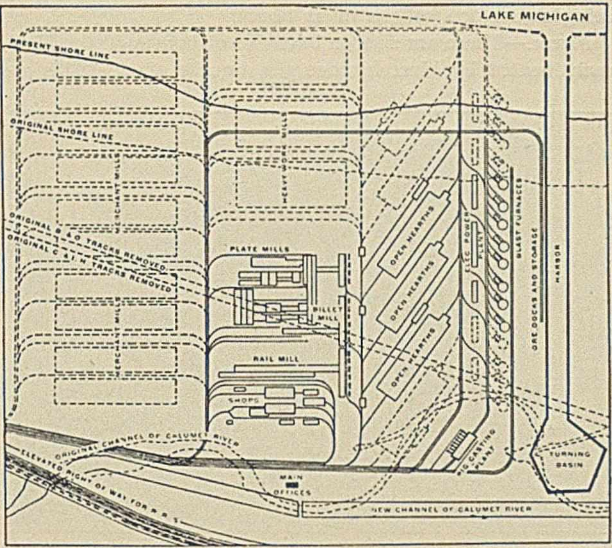


Fig. 2.—Sketch map of Gary—general plan of the new steel plant. Furnaces, mills and shops already completed or under way are indicated by solid black lines; future construction and extensions by dotted lines. To avoid confusion, duplicated railroad tracks are eliminated, the simple lines in and out of the open-hearth buildings representing six service tracks. Note how furnaces and mills have been placed to facilitate switching.

The builders of the Gary plant have done more than construct a steel works. They have in two or three years converted a waste of dune-sand and scrub oaks into a habitable region and laid the foundations of a prosperous and well-paved modern city.

The Indiana Steel Company is a subsidiary of the U. S. Steel Corporation. In 1905 the corporation set aside $10,000,000 for building the Gary works. In 1906, $21,500,000; in 1907, $18,500,000; a total of $50,000,000. No appropriation was made for 1908, inasmuch as the total amount expended even up to the present time is not greatly in excess of $40,000,000. The original announcement stated that an appropriation of $75,000,000 was contemplated from first to last. The Indiana Steel Company is a separate corporation, but is organized as a subsidiary of the Federal Steel Company. The operation of the plant, however, is carried on under lease to the Illinois Steel Company.

The President of the Indiana Steel Company is Mr. Eugene J. Buffington. The plant was designed and is being constructed under the direction of Mr. G. G. Thorp, Vice-President, Mr. A. B. Neumann, Chief Engineer, and Mr. W. P. Gleason, General Superintendent.

Location and Area.—The plant is located at the southern point of Lake Michigan, in Lake County, Indiana, about forty miles from the center of Chicago. The approximate area occupied at the present time is about 700 acres, and the area available for future extensions amounts to 1000 acres.

Water Supply.—The water supply is derived entirely from Lake Michigan, being taken from a crib one and one half miles from shore, through a six feet tunnel. The water gravitates to the pumping station, from which point it is supplied to the plant by means of horizontal-shaft direct-connected motor-driven centrifugal pumps. These are two

---

[1] For further descriptions, particularly of the mechanical features f the Gary plant, consult the *Iron Age*, Jan. 7th and Jan. 14th, and following numbers: The *Iron Trade Review* of Jan. 7th and succeeding numbers; *System*, Jan., '09, and *Engineering Record*, July 20, Aug. 17 and Nov. 2, 1907.

domestic pumps driven by 250 H. P. motors and two fire pumps driven by 175 H. P. motors, the combined capacity being 20,000,000 gallons in 24 hours.

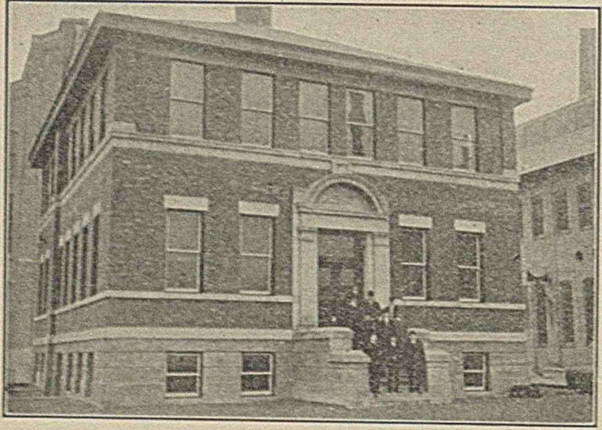


Fig. 3.—Chemical laboratory.

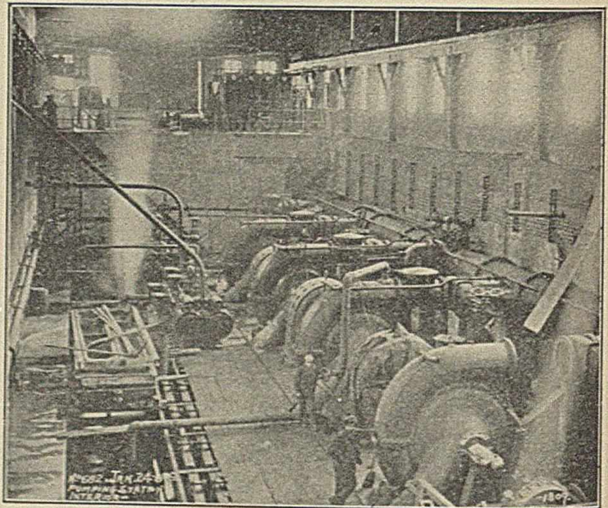


Fig. 4.—Pumping station.

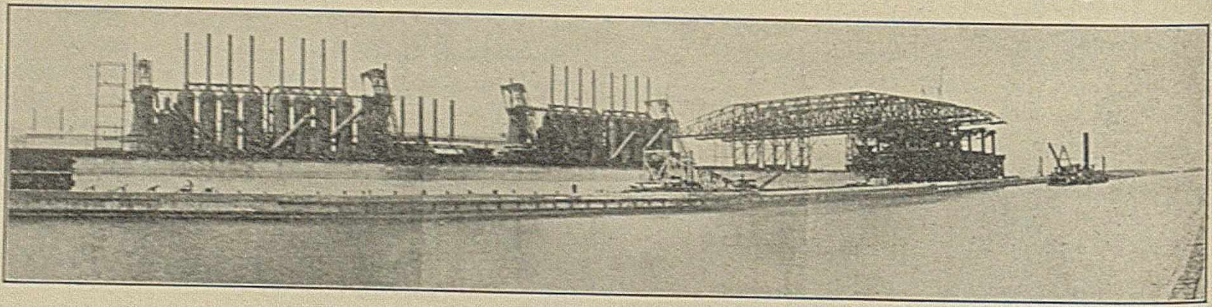


Fig. 5.—Blast furnaces from water front.

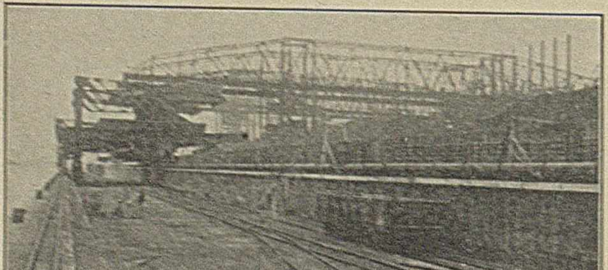


Fig. 6.—Unloaders and ore bridges.

Power.—There are sixteen batteries of boilers of 400 H. P. each, heated by blast furnace gas, on the plant. This gas averages in heat value 95 B. T. U. per cubic foot. Steam is used on the plant to drive two 2,000 K. W. turbines which were installed temporarily, and prior to the starting of the gas engine-driven generators. Steam is also used on the plant in auxiliary blowing engines, pumps, turbines, hammers, gas producers, etc. Of blowing engines for the blast furnaces there are eight Westinghouse and eight Allis-Chalmers horizontal twin-tandem of 2,000 H. P. each. They are operated by blast furnace gas and average 30,000 cubic feet of air per minute, each. The number planned for the complete plant is thirty-two, with a total H. P. of 64,000. The electric power plant equipment consists of horizontal twin-tandem Allis-Chalmers gas engines, direct-connected. At present seventeen of these engines are installed, with a total of 60,000 H. P., housed in a building 966 feet long by 105 feet wide. It is planned to duplicate this plant with another of the same size and this will give a total of thirty-four gas engines, with 120,000 H. P.

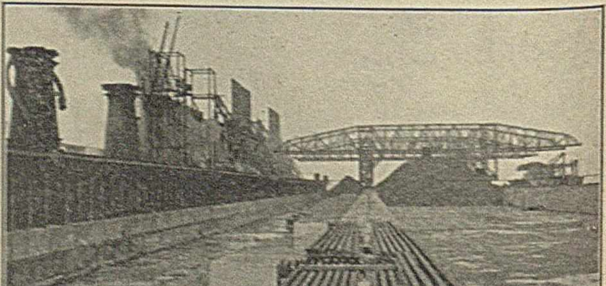


Fig. 7.—Ore bins.

Blast Furnaces.—Of a total of sixteen blast furnaces, each of a capacity of 450 tons per 24 hours, planned for the plant, four are operating at the present time and four are under construction. There are four stoves to each furnace and for the eight furnaces either operating or now under construction, there are provided fourteen dust catchers. The flue dust delivered by them amounts to about two carloads per furnace per day. The flue dust is not sintered. For each pair of furnaces there are provided three primary gas washers. From the primary washers the furnace gas passes through a large main to the secondary washers. Two types of these are used, vertical tower washers and centrifugal washers, and for each furnace there are provided two of each type.

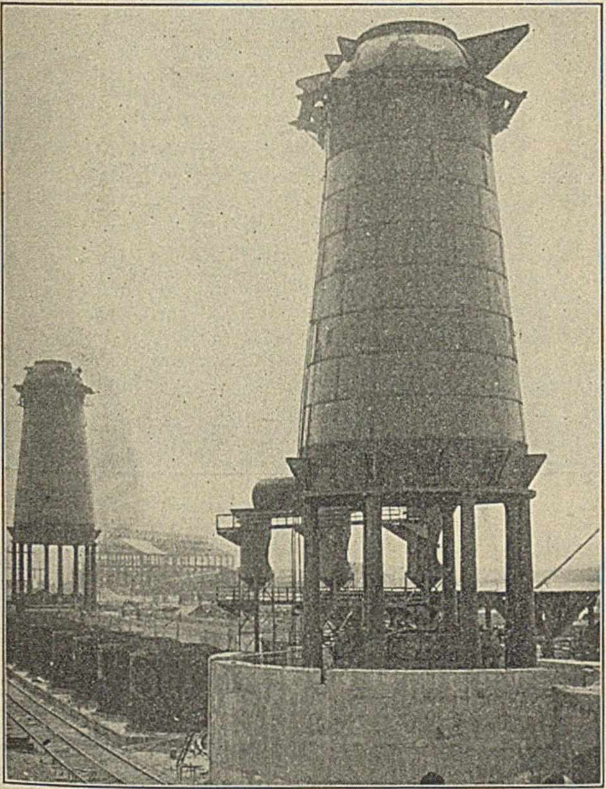


Fig. 8.—Blast furnaces under construction

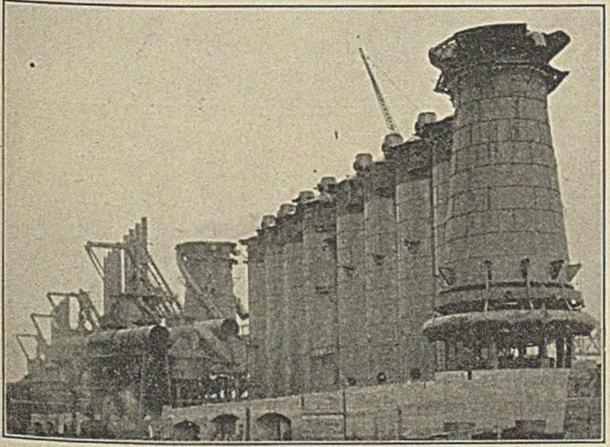


Fig. 9.—Blast furnaces and stoves under construction.

Supplementary to the blast furnace installation, a pig-casting equipment has been erected of sufficient capacity for handling the entire output of the blast furnaces.

The course of the gas after leaving the blast furnace is as follows: It first passes through the dust catchers, then through the primary washers, and after leaving the primary washers a fraction amounting to 30 per cent. of the total or 6,750,000 cubic feet per hour passes to the hot blast stoves. From the same point 7½ per cent. or 1,700,000 cubic feet per hour passes to the boiler house. The remainder passes to the secondary washers where 2½ per cent. or 600,000 cubic feet per hour is used up. From the secondary washers the gas enters the gas holder, of a capacity of 200,000 cubic

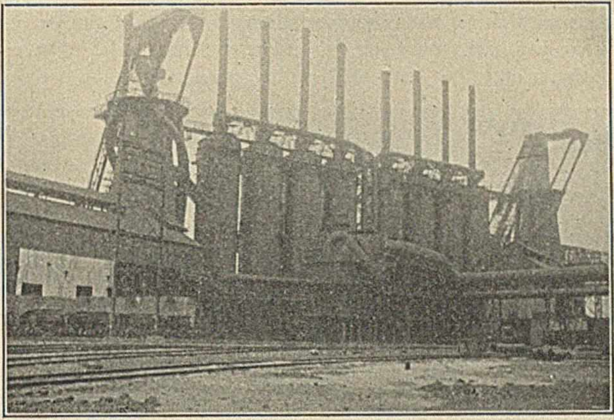


Fig. 10.—Furnaces, stoves and primary washers.

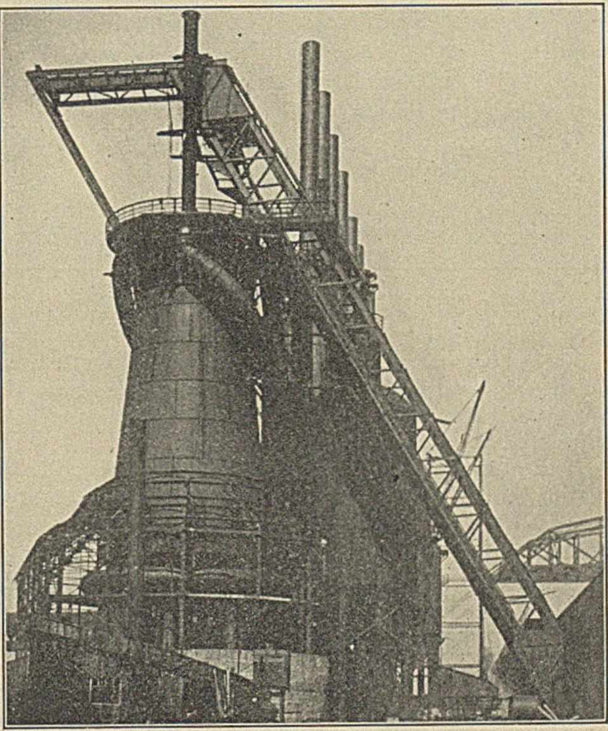


Fig. 11.—Blast furnace showing skip incline, stoves and dust catchers. Compliments of Allis-Chalmers Co.

feet. From the gas holder the gas passes by mains to the blowing engine house and the electric power station. In the former 12½ per cent. or 2,800,000 cubic feet per hour is used. 2½ per cent. or 600,000 cubic feet per hour is used for auxiliaries for blast furnaces themselves. The total consumption for blast furnaces then, exclusive of the stoves, is 15 per cent., or 3,400,000 cubic feet per hour. The remainder or 45 per cent. of the total, amounting to 10,000,000 cubic feet per hour, is used in the electric power station. The above figures are based on the production of eight blast furnaces delivering 3600 tons of pig iron per 24 hours. It is estimated that these eight furnaces will produce 22,450,000 cubic feet of gas per hour.

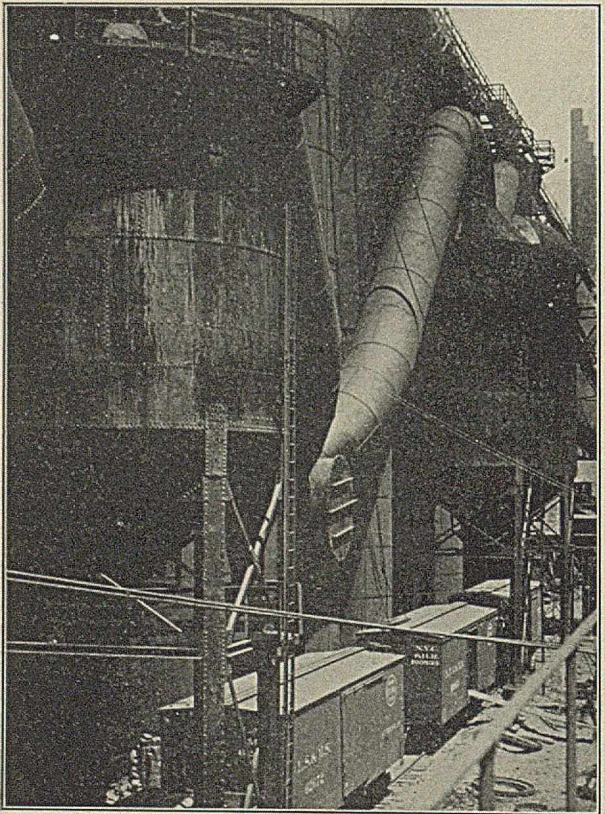


Fig. 12.—Dust catchers. Compliments of Allis-Chalmers Co.

The Gayley dry blast has not yet been introduced at Gary.

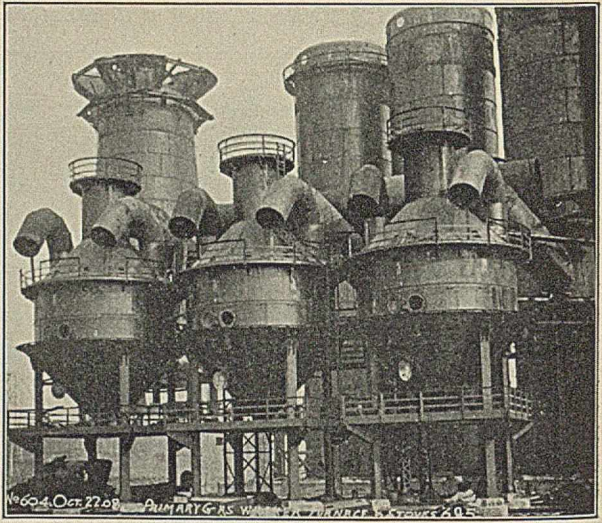


Fig. 13.—Primary gas washers.

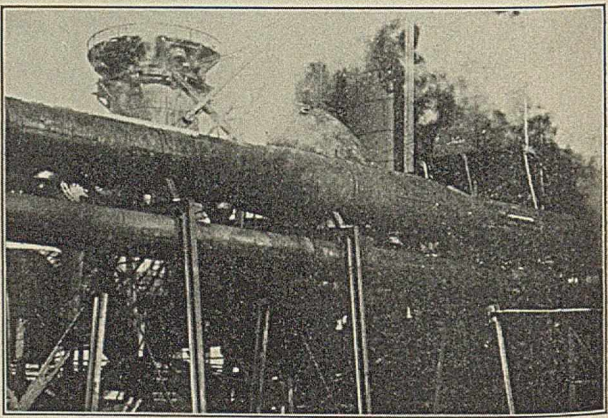


Fig. 14.—Back of blast furnaces. Gas mains leading to boiler house and secondary gas washers. Compliments of Allis-Chalmers Co.

Open-hearth Furnaces.—At present there are finished twenty-eight open-hearth furnaces each with a capacity of 60 tons. There are under construction fifty-six more which when completed, will give a total of eighty-four. For each open-hearth furnace are provided five Morgan gas producers. The first open-hearth plant will be in operation by the time this article is published. In the completed plant there will be a rail mill with a capacity of 100,000 tons per month. This is now ready to operate. A billet mill, an axle shop,

ANALYSES OF IRON ORES.

Ores.	Fe.	P.	Mn.	SiO_2.	Al_2O_3.	Fe_2O_3	CaO.	MgO.	MnO.	P_2O_5	Vol.	Moist.
					Dry Basis, Per cent.							
Chapin	59.46	0.066	0.21	5.50	1.60	85.66	1.50	3.24	0.27	0.151	2.40	...
Bernhart	56.66	0.059	0.26	13.30	1.80	80.94	1.25	0.28	0.34	0.135	2.20	...
Vermont	59.46	0.075	1.27	5.26	2.75	84.94	0.61	0.14	1.64	0.171	4.86	...
Group 4	57.58	0.063	1.10	6.20	2.53	82.26	0.50	0.36	1.42	0.144	6.66	...
Group 3	57.86	0.085	1.57	6.17	2.80	82.66	0.65	0.37	2.03	0.195	5.40	...
Vermil	65.61	0.145	0.10	3.86	1.01	93.73	0.57	0.35	0.13	0.332	0.30	...
					Original Basis.							
Chapin	56.10	0.062	0.20	5.15	1.50	80.14	1.40	3.03	0.26	0.142	2.25	6.43
Bernhart	51.59	0.054	0.24	12.11	1.64	73.70	1.14	0.25	0.31	0.124	2.00	8.95
Vermont	51.70	0.065	1.10	4.57	2.41	73.86	0.53	0.12	1.42	0.149	4.22	13.05
Group 4	49.34	0.054	0.94	5.31	2.17	70.49	0.43	0.31	1.21	0.124	5.71	14.31
Group 3	50.10	0.074	1.26	5.34	2.42	71.57	0.56	0.22	1.63	0.169	4.68	13.42
Vermil	65.04	0.144	0.10	3.83	1.00	92.91	0.56	0.35	0.13	0.330	0.30	0.87

a plate mill and a merchant mill are all under construction.

Raw Materials and Products.—The ores used in the plant will be from the Messaba, Menominee, Marquette and Vermillion districts. Average analyses from typical mines in these districts are as above, on the dry and original basis.

The ores are all delivered to the plant by the Steel Corporation's steamers. The coke used is from the Connellsville, Penna. district, and analyses about as follows:

	Per cent.
Vol.	0.83
Fixed carbon	88.42
Sulphur	0.75
SiO_2	5.98
Fe	0.60
Al_2O_3	0.45
P	0.01
Mn	0.04
CaO	0.17
MgO	0.22

At the present time dolomite is used in the blast furnaces and hence the slag cannot be used for cement-making purposes. In case it is desired to produce cement from the slag, the change to calcite can readily be made. The dolomite comes from McCook, Ill., and from Gary, Ill. The following is a typical analysis:

	Per cent.
Moisture	1.50
SiO_2	0.53
Fe_2O_3 and Al_2O_3	0.59
P_2O_5	0.01
CaO	29.60
MgO	21.60
S	0.041

The coal used in the producers at the open-hearth plant is Dering No. 2 lump, from Westville, Ill., and analyzes as follows:

	Per cent.
Moisture	8.50
Volatile com	37.01
Fixed carbon	46.88
Ash	7.61
S	1.44
B. T. U.	11,970

The limestone spalls for the open-hearth plant comes from the well-known quarries at Bedford, Ind. A typical analysis is as follows:

	Per cent.
SiO_2	0.63
Al_2O_3 and Fe_2O_3	0.46
P	0.008
CaO	54.90
MgO	0.51
S	0.055

Flue dust from the blast furnaces shows on analysis:

	Per cent.
SiO_2	7.63
Fe	53.88
Al_2O_3	3.51
P	0.062
Mn	0.82
CaO	0.52
MgO	0.27
C	8.36

Chemical Laboratory.—Mr. H. C. Thomas is chief chemist at the Gary plant and has been instrumental in designing one of the best iron and steel laboratories in the country. The chemical work, excepting the gas analysis work which is handled in the gas laboratory located at the blast furnaces, will be performed in one central laboratory. This is a three-story and basement brick and concrete building with 10,000 square feet of floor space. In the basement of the building are located the heating plant and power machinery for the laboratory, the acid storage, the photographic room and dark room, record room, vault and toilet room. On

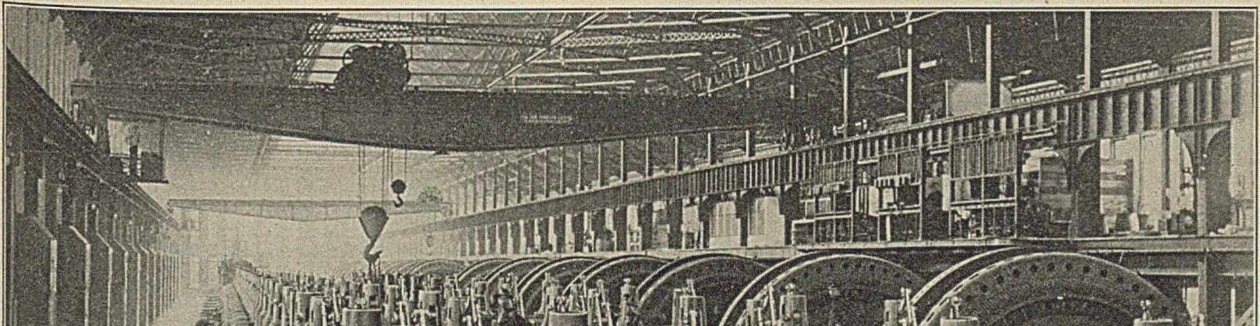


Fig. 15.—Partial view of installation where 17 Allis-Chalmers twin-tandem gas engines are direct-coupled to Allis-Chalmers generators, each unit being of 2,500 kw. capacity. Photograph taken during erection. The installation also includes 8 Allis-Chalmers twin-tandem, gas-driven blowing engines, making 25 units in all.

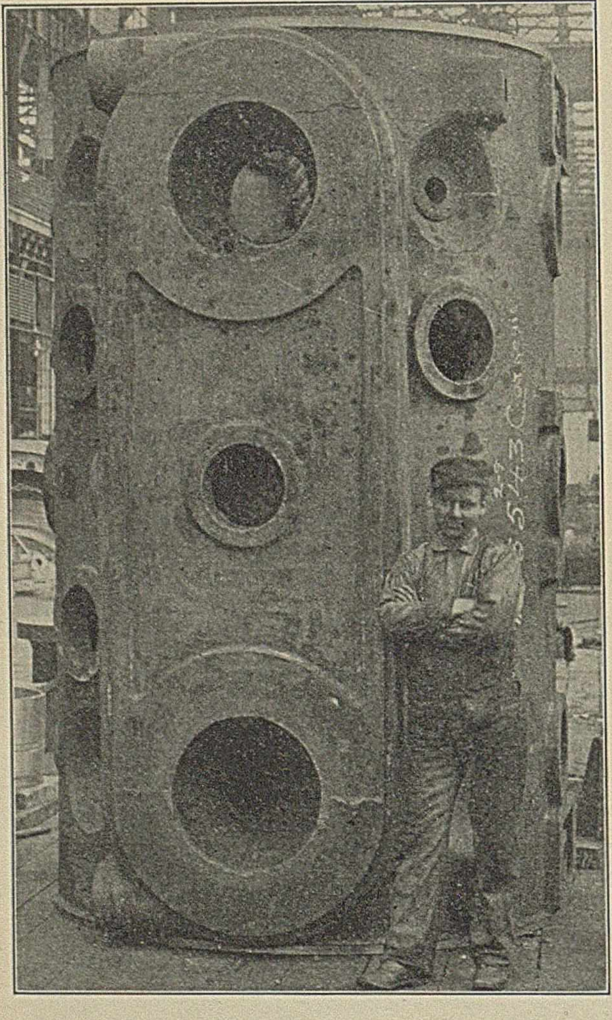


Fig. 16.—42-inch gas engine cylinder, Allis-Chalmers.

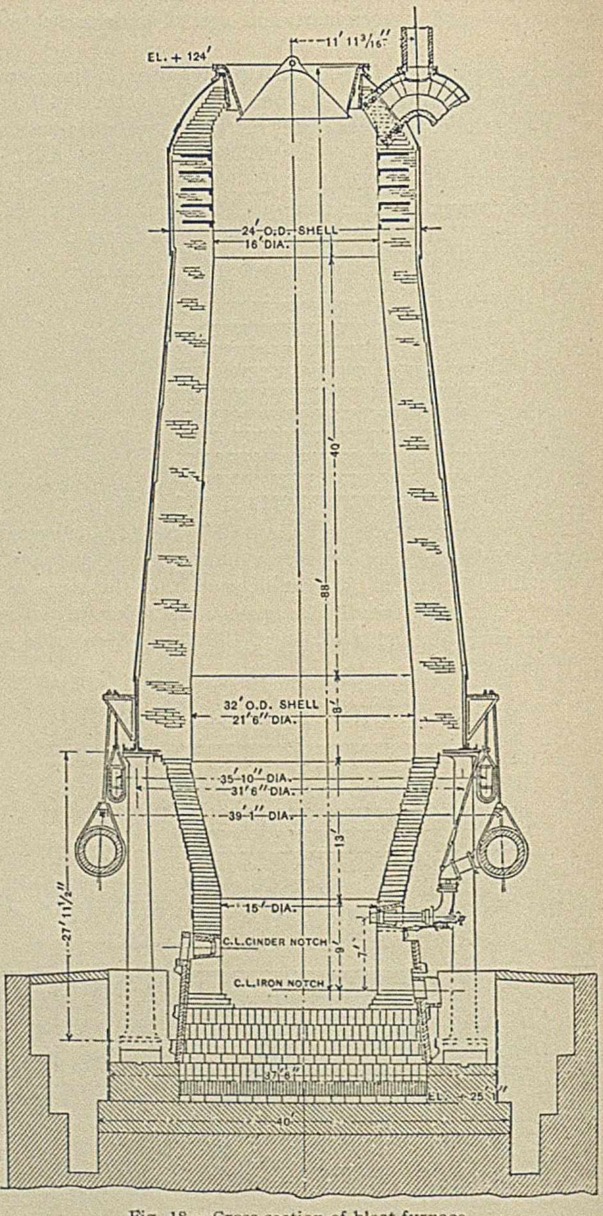


Fig. 18.—Cross-section of blast furnace.

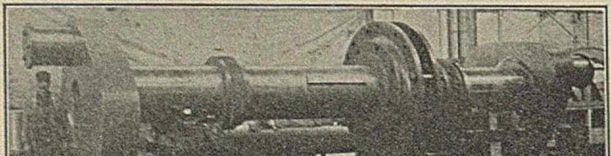


Fig. 17.—Crank shaft of 5000 H. P. Allis-Chalmers 4-cycle, double-acting twin-tandem gas engine—weight of this part, 85 tons.

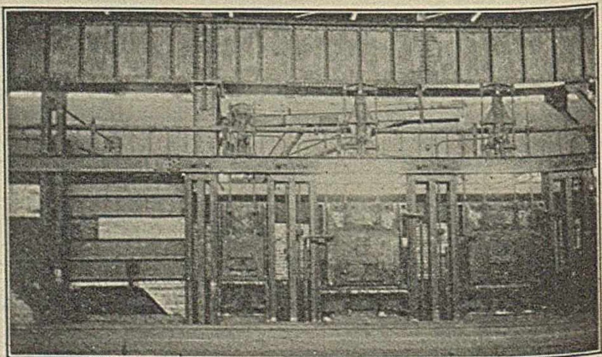


Fig. 19.—Open-hearth furnace, charging end.

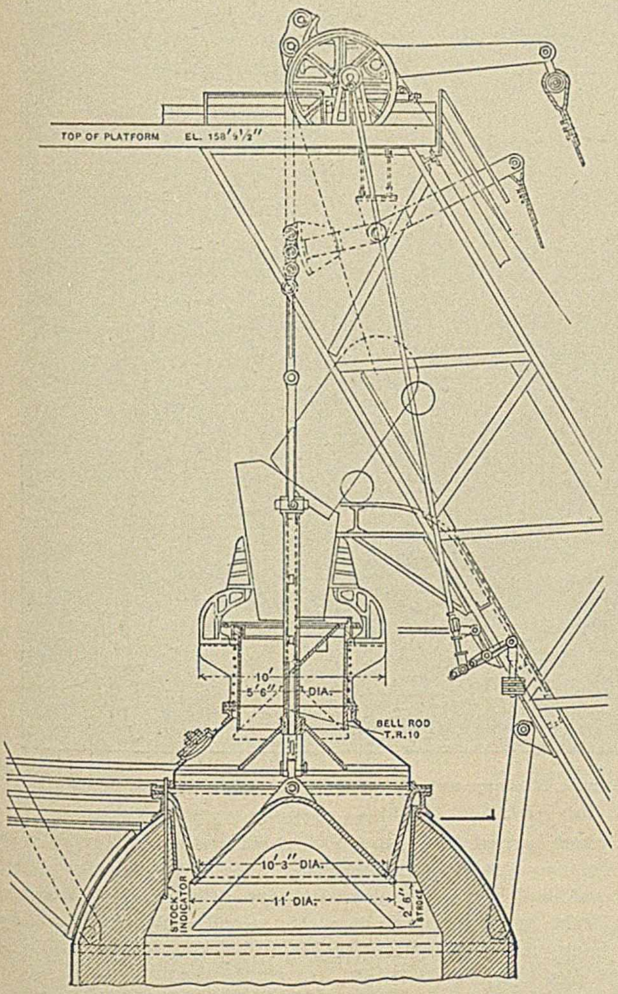


Fig. 20.—Top of blast furnace.

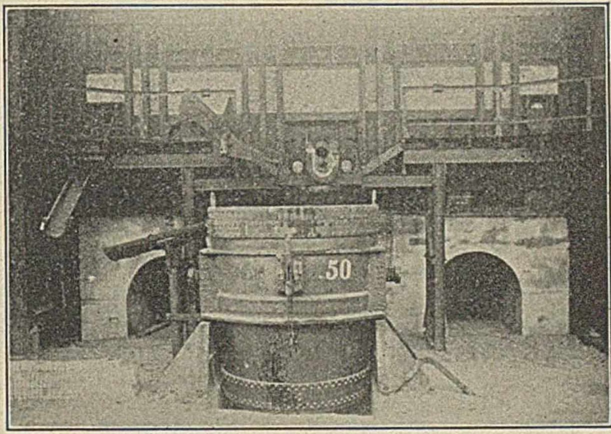


Fig. 21.—Open-hearth furnace, discharge end.

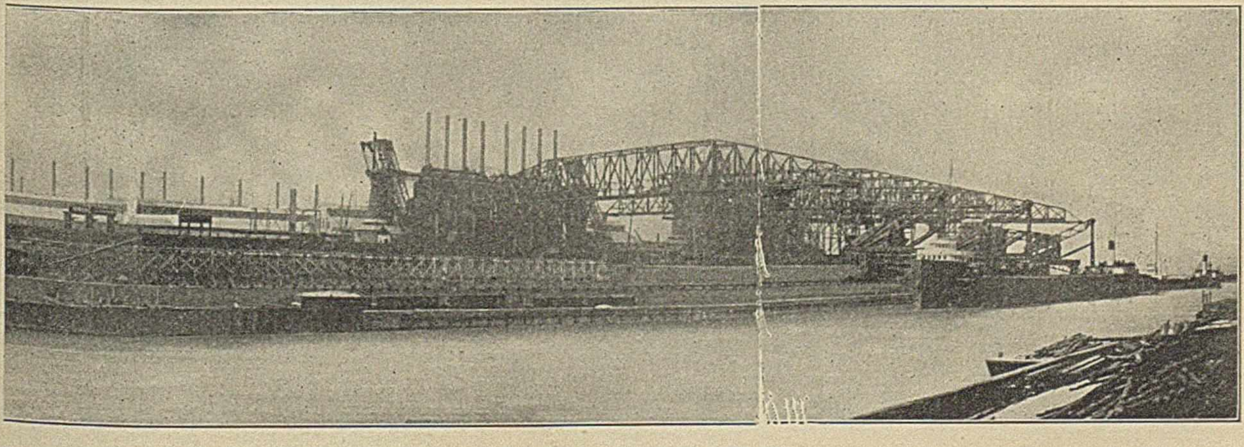


Fig. 22.—View from slip.

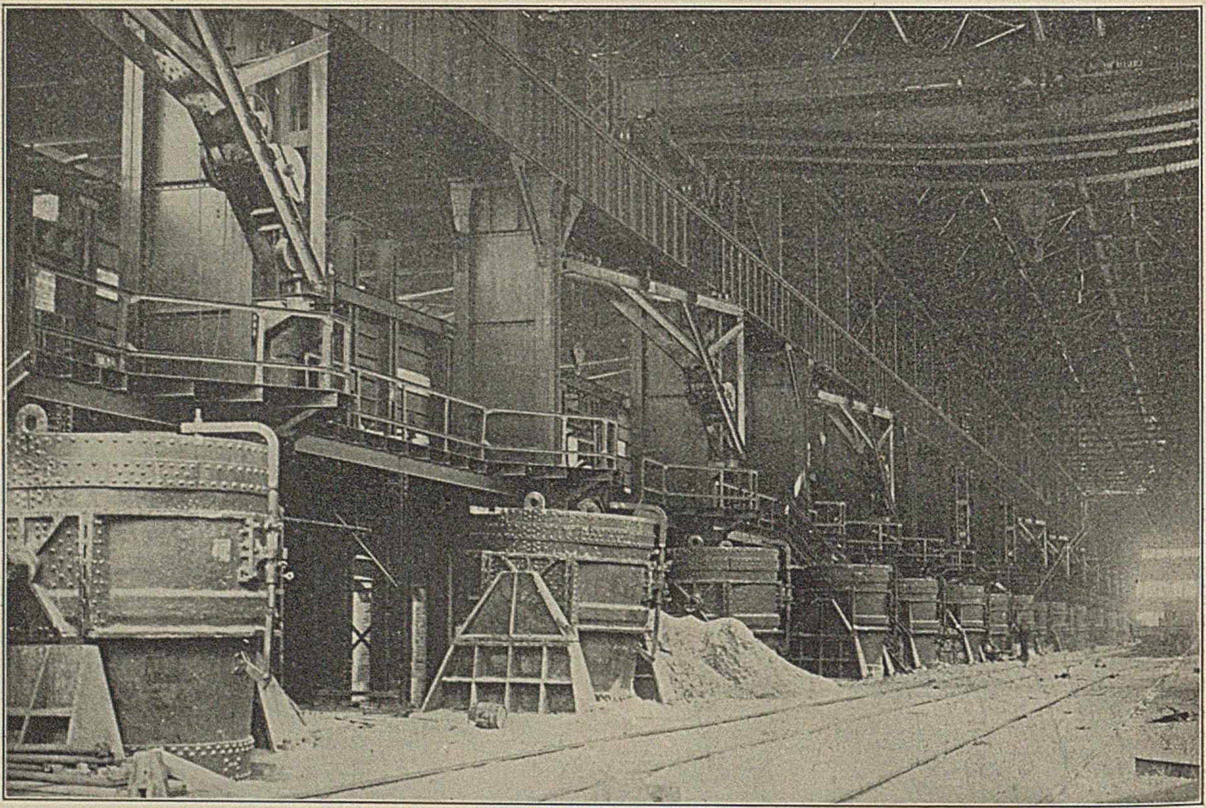
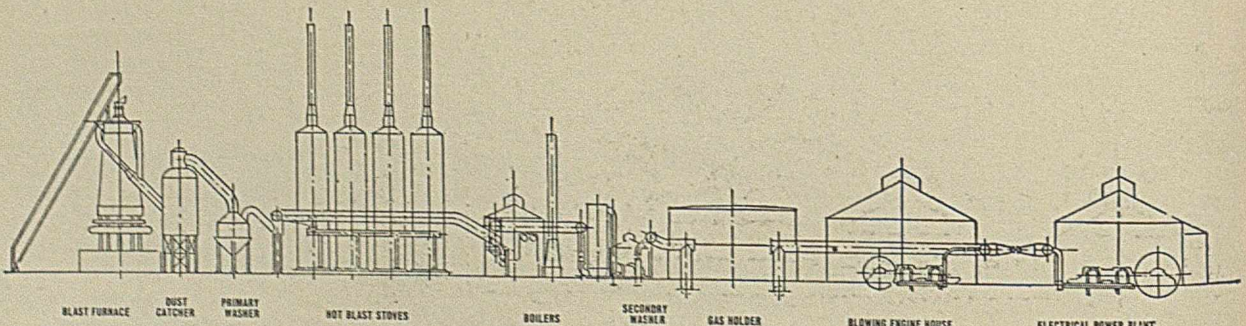


Fig. 23.—Open-hearth furnace, discharge end.

the first floor is the office of the chief chemist and the clerk's office, the main iron and steel laboratory, balance room and store room. On the second floor are located the laboratory for blast furnace and open-hearth materials; a laboratory for special work such as alloys, foundry work and waters; a laboratory for research work; a muffle room, balance room, store room, library, office and toilet room. The third floor is at present unfinished, but will be finished as the space is required. The building is so constructed that new stories can be added whenever necessary. No sampling is being done in the laboratory, but is handled in a special sampling plant located at the ore docks. This is a building 50 x 40 feet, containing 30 2-ton drying bins, 6 5x5 feet hot plates, 2 Allis-Chalmers grinders and one Allis-Chalmers and one Dodge crusher. Just now the sampling is performed by hand, the mechanical sampler not having as yet been installed.

The writer desires to express his thanks to Messrs. G. G. Thorp, Vice-President of the Indiana Steel Co., H. C. Thomas, Chief Chemist Indiana Steel Co., and Wm. Brady, Chief Chemist Illinois Steel Co., for the data furnished for this article.



8 Blast Furnaces=3600 T. Pig Iron per 24 Hrs. Total B. F. Gas prod. per Hr.=22,450,000 cu. ft.=100%. Equivalent to 250,000 B. H. P. (in Gas Engines.)

30% Used Here or 6,750,000 cu. ft. per Hr.

7.5% Used Here or 1,700,000 cu. ft. per Hr. For Spare Steam Blower, Pressure and other Pumps, steam for Gas Prod. heating of bldgs, etc.

2.5% Used Here and, in Primary Washers, or 600,000 cu. ft. per Hr.

12.5% Used Here or 2,800,000 cu. ft. per Hr. 2.5% used for auxiliaries for Blast Furnaces themselves (in gas engines) or 600,000 cu. ft. per Hr. Total for furnace operation 15 per cent. or 3,400,000 cu. ft. per Hr.

Remaining 45% available for other purposes or 10,000,000 cu. ft. per Hr. Equivalent to 110,000 B. H. P. (in gas engines) with gas at 90 B. T. U. per cu. ft. and 10,000 B. T. U. per B. H. P. hour gas consumption.

Fig. 24. Diagram for blast furnace gas.

THE FINK SMELTER.

By W. C. Ebaugh.

Much interest has been aroused in metallurgical circles by the test made with the Fink smelter at the Boston Consolidated Company's plant at Garfield, Utah. The successful operation of the experimental furnace during its trial run—when concentrates containing about twenty per cent. of copper were reduced to blister copper in a short space of time—has led the more enthusiastic and hopeful smeltermen to assert that a revolution in the treatment of fines has been effected, and that many properties that cannot be operated at a profit under present conditions will be made of value through the introduction of the new process of smelting. Whether these claims are too extravagant can be determined only after operations have been carried on under commercial conditions upon a larger scale.

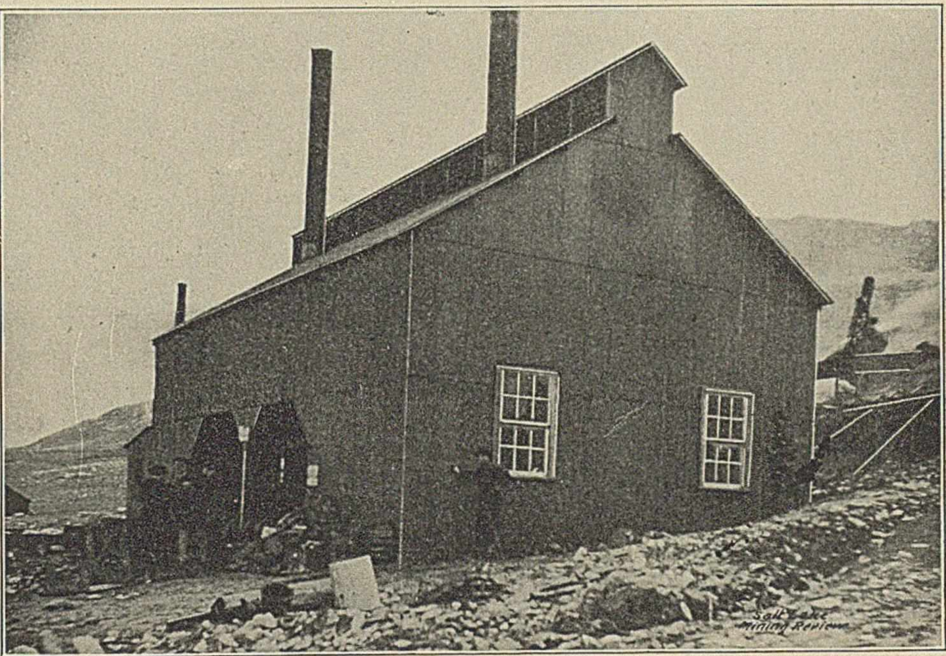


Fig. 1.—The Fink 100-ton smelter at Garfield, Utah.
Photograph by C. W. Higgins. Courtesy of *Salt Lake Mining Review*.

As shown by the accompanying illustrations the Fink plant consists essentially of two large rotating compartments, lined with refractories, between which is a flue for the passage of the gases from one compartment to another, as well as the openings for the introduction of the fuel. These smelting compartments or cylinders are made in the shape of truncated cones placed base to base, connected at one end with the central flue and at the other with a chimney. In the plant at Garfield, which is estimated to have a daily capacity of one hundred tons, these cylinders are nine feet in length, nine feet in maximum diameter, and taper to seven feet at the ends. The lining reduces the maximum internal diameter to about seven feet. Each end of the cylinder has an opening three feet in diameter. These cylinders are rotated by friction on trunnion wheels, but in future installations it is planned to have direct gear drives in order to avoid slipping.

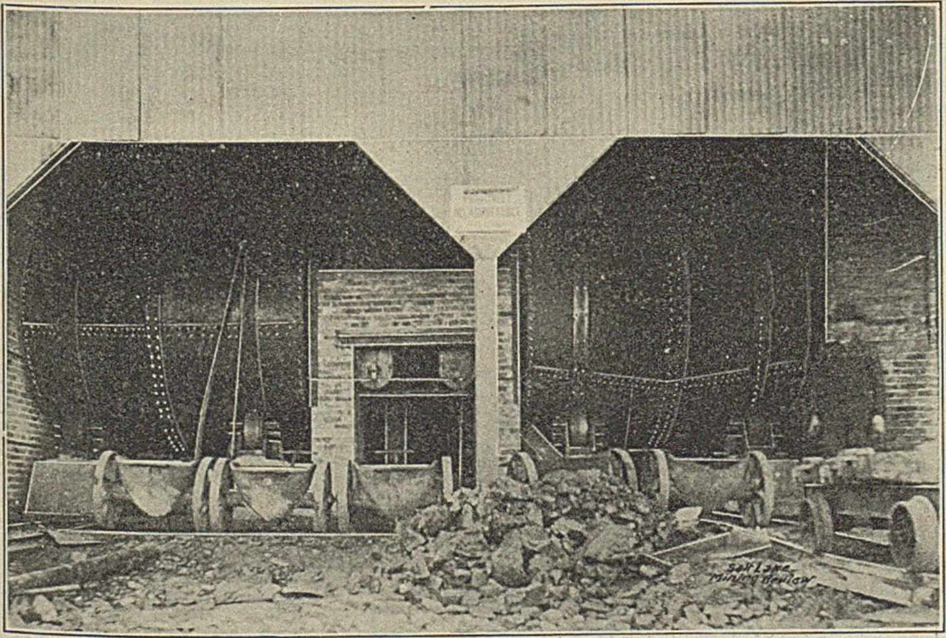


Fig 2.—Front view of the new Fink smelter.
Photograph by C. W. Higgins. Courtesy of *Salt Lake Mining Review*.

The fuel is introduced through cast iron, muffle-shaped pipes that can be seen below the central flue. Either crude oil or powdered coal can be employed, and as a matter of fact the Garfield plant was built to be run with oil, but failure to obtain a supply at the time of the test resulted in a modification of the feeding mechanism so that powdered coal could be used, and this fuel was found to be satisfactory. When it is remembered that coal and coke for smelting purposes are necessarily expensive, because of the high grades required, it is evident that a furnace that can use slack coal (either anthracitic or bituminous), crude oil or even sawdust as a fuel has a distinct advantage over the ordinary smelter.

Through the top of the central flue, tuyères, that reach into the charge in the cylinder, can be introduced. These tuyères are provided with magnesia tips and are water-jacketed. Screw conveyors carry the charge from the bins through the walls of the stack compartments at the ends of the cylinders and directly into the smelting cylinders. Around the middle line of each cylinder are three tap holes for the removal of slag, matte or metal.

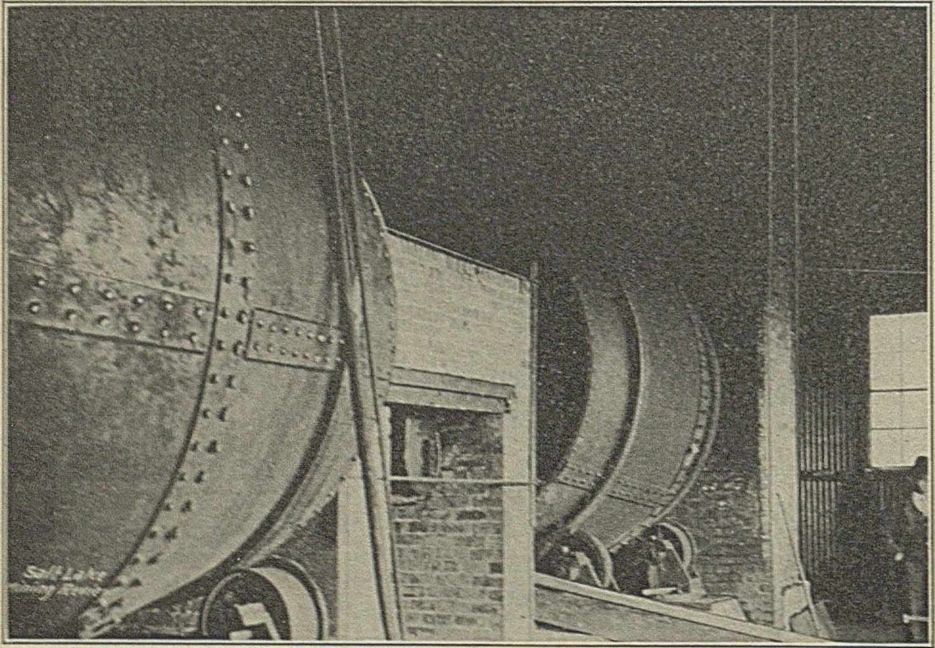


Fig. 3.—Fink smelter, showing revolving cylinders and chamber. Photograph by C. W. Higgins. Courtesy of *Salt Lake Mining Review*.

In the Fink smelter the inventor has applied the principle of the revolving roaster or black ash furnace, but with the important difference that in this instance there is no end fire box, the fuel being burnt in the cylinder itself. There is the additional advantage that when a bessemerizing action is wanted it is carried out in the same cylinder through the introduction of tuyères, as indicated in the preceding paragraph. It is possible, therefore, to first roast, then smelt to a matte, and finally bessemerize the matte without ever removing the charge from one cylinder. It is this economy of labor, as well as economy of fuel, that makes the new process attractive.

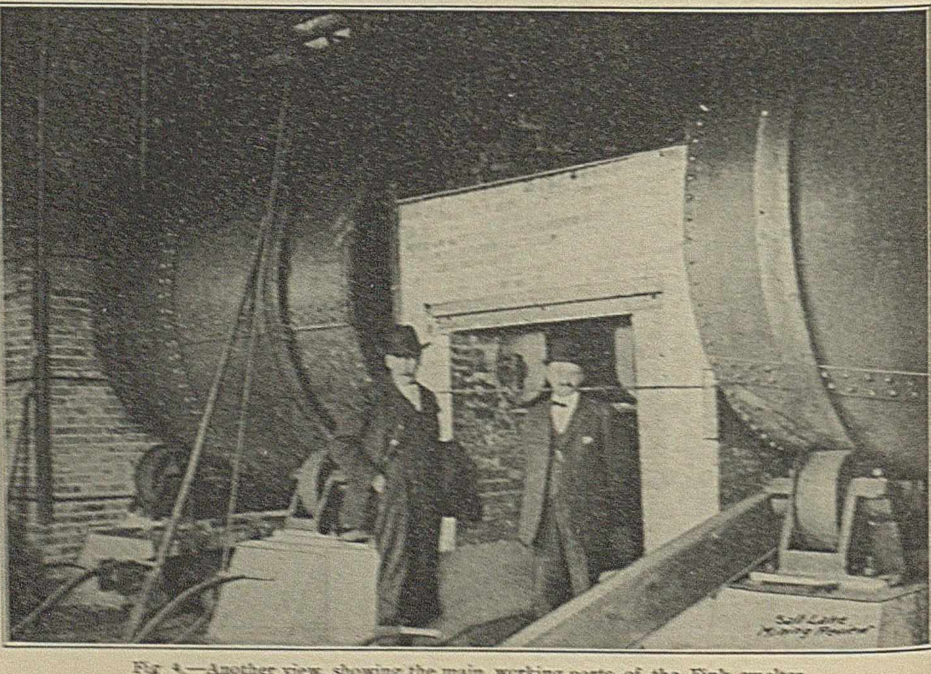


Fig. 4.—Another view, showing the main working parts of the Fink smelter. Photograph by C. W. Higgins. Courtesy of *Salt Lake Mining Review*.

In operating the furnace both cylinders are in use at the same time. The ores or concentrates are fed into one cylinder which still contains the slag from a preceding bessemerizing blow. The cylinder is then rotated, and the gases from the smelting operation going on in the other cylinder are led consecutively through the middle flue, the cylinder with the fresh ore, a dust chamber, and thence to the stack. No fuel is required directly for the roasting, the sulphur from the ore and the heated gases from the other cylinder sufficing. At the proper time the charge in the other cylinder is removed, and the now roasted ore, in the cylinder with which the explanation was started, is ready to be reduced to matte. Burning fuel is blown into the cylinder, the particles of ore in the finely divided condition are quickly reduced to matte, and ordinary slag is formed. Care must be taken to regulate the composition of the charge properly and to stop the operation at the right time. If it should happen that the blow had continued too long some fresh ore or flux could be introduced and restore the charge to its desired condition. This factor of ease in regulating the various operations in the cylinder is considered as another advantage of the process. Slag is tapped through one of the three tap holes along the middle line of the cylinder, and the remaining matte is bessemerized by having the tuyères introduced through the top of the central flue, through the hole at the end of the cylinder and directly into the molten matte. A silica flux is added to the matte to flux off the iron, thus avoiding the excessive corrosion of the lining of the cylinder. Either a high grade of matte (white metal) or bullion can be made at will. At the end of the blow the finished product is tapped off into molds, and the superincumbent slag, which will contain more or less copper, is retained to be worked up with a fresh charge of ore.

The tests carried out with the Fink process have been declared successful. Samuel Newhouse, whose word concerning metallurgical and mining matters carries weight, is quoted in the press as follows: "You are now at liberty to say for me that the Fink process of smelting ores is a demonstrated commercial success. You can make the statement just as strong as you like. * * * There is no question of its amounting to a revolution in the art of smelting; it is a triumph in the science of metallurgy that must command world-wide attention. The process will be the means of making mining profitable in hundreds of places and under innumerable conditions where and under which profit-making is now impossible." (Signed interview by Samuel Newhouse in the Salt Lake City *Herald* of January 18th, 1909.)

The advantages claimed for the process are: (1) that fines and concentrates can be smelted without preliminary treatment; (2) economy of fuel, for roasting is carried on by the aid of waste heat from the smelting operation, and either slack coal or crude oil can be used as fuel; (3) prevention of the loss of fines, for the green ore is not subjected to a blast while it is dry, but is run into the residual converter slag from a preceding operation; (4) complete conversion of ore into metal without removal from one compartment; (5) convenience for use in either intermittent or continuous smelting, thus adapting it to the small producer and to places where the ore supply is irregular; (6) repeated use of the furnace without relining; (7) low cost of installation.

Opposed to these advantages one must consider the following objections to the process: (1) its inability to compete with present blast furnace and reverberatory practice in handling large tonnages of ore obtainable in a regular supply; (2) that the desirability of trying to carry out three different such operations as roasting, smelting and bessemerizing in one piece of apparatus is open to question; (3) the possibility of obtaining clean slags without using some form of settler; (4) that the introduction of raw ore into a cylinder containing a mass of bessemer slag would result in a loss of roasting efficiency.

Until long runs are made under conditions that approximate those that are encountered in practice, judgment as to the cost of operation cannot be made with accuracy. It is said that such tests are to be carried out as soon as possible at Garfield and at the Cactus plant of the Newhouse Mines and Smelters Company.

UNIVERSITY OF UTAH,
January 29, 1909.

QUOTATIONS.

(From *Schimmel's Semi-Annual Report—Fall* 1908.)

PEPPERMINT OIL, AMERICAN.

We have again received a detailed report from our New York branch, whose expert has visited this year, as usual, all the peppermint districts. We abstract the following from this interesting information:

(1) WAYNE COUNTY, NEW YORK.

The area cultivated with peppermint is this year confined chiefly to the districts of Arcadia, Lyons, Sodus, Marion and Palmyra, while Williamson, Junius and Galen, as shown below have only very small plantations. The cultivation continues to decline from year to year, as all hopes of a permanent revival of the peppermint oil prices have after the experience of the last few years, been abandoned almost everywhere, and it is found more remunerative to grow wheat, buckwheat, sugar-beets, beans, or peas on the fields, and lately also willows for the manufacture of baskets. The bulk of the peppermint harvest this year is two years old, and experience shows that the quality of the oil in such case cannot exactly be called first-class. No less a quantity than 20,000 lbs. peppermint oil from last harvest is still unsold in the hands of the farmers, who are reported to be determined to hold back also the bulk of the new product, if better prices than in 1907–8 cannot be obtained this season.

The area under cultivation is made up as follows:

	This year's (1908)		Last year's (1907).	
Williamson.......	2	acres	4	acres
Marion..........	10	"	40	"
Palmyra.........	13½	"	18	"
Sodus...........	13	"	20½	"
Arcadia.........	43	"	113	"
Junius..........	3	"	11	"
Galen...........	6	"	10	"
Lyons...........	31	"	40	"
	121½	"	256½	"

The falling off in the cultivation is clearly shown by these figures.[1]

The result of the distillation of the whole area may be probably taken as follows:

121½ acres at 30 lbs. average.................	3645 lbs.
257½ acres at 12 lbs. average.................	3090 lbs.
	6735 lbs.
Add to this stocks amounting to about........	23000 lbs.
Makes a total available quantity of about......	30000 lbs.

It follows that in spite of the great shortage in the cultivation, there will be no scarcity of this oil, for the above quantity may be described as absolutely normal.

(2). MICHIGAN AND INDIANA.

In these districts also the distillation has to fall back chiefly on peppermint of last year's growth, as, owing to abundant rains in the spring, new plantations could be laid out only in a very few places. On the whole the fields are very weedy, so that the distillation material also leaves much to be desired. The stocks of 1907 oil are here also very considerable; our informant speaks of about 46,000 lbs., while others even mention 75,000 lbs. as having been held back by the farmers. The harvest is rather late this year, so that a second cut was out of the question.

The production of this year may bring the stocks still on hand to a quantity which approximately equals that available last year—as is the case in the state of New York—but the opinions of individual observers differ considerably on this point. Owing to the low prices the farmers in Mich-

---

[1] *Comp. Report*, Oct., 1907, **69**.

igan and Indiana have also resolved to grow very little peppermint next year, or none at all.

The following areas are under cultivation:

	This year's (1908).	Total area (1907).
Michigan—		
Berrien Co.	281 acres	1401 acres
St. Joseph Co.	250 "	1020 "
Branch Co.	65 "	215 "
Kalamazoo Co.	25 "	75 "
Van Buren Co	125 "	920 "
Allegan Co.	195 "	1685 "
Eaton Co.	30 "	735 "
Cass Co.	45 "	185 "
Indiana—		
South Bend.	...	200 "
Mishawaka.	130 "	585 "
Osceola.	74 "	450 "
	1220 acres	7471 acres

The peppermint oil trade in the United States has suffered during the last few months from severe depression; the general state of business was little inviting to speculative enterprise, and a large number of the well-to-do farmers having, as already stated, made up their minds not to dispose of their oil at the present low prices, the market was as quiet as possible. Only a very few transactions took place; and, contrary to what was usual in former years, it happened very rarely that any large parcels changed hands repeatedly already on the American market. For this reason it is all the more surprising that the European trade in this article during the last half-year was exceptionally brisk.

BOOK REVIEWS AND NOTICES.

A Short Pharmaceutic Chemistry, Inorganic and Organic. By I. V. Stanley Stanislaus, M.S., Ph.D., Professor of Pharmacy and Organic Chemistry and Dean of the School of Pharmacy of the Medico-Chirurgical College of Philadelphia, and Charles H. Kimberly, B.S. (in Pharmacy), Ph.D., Professor of Applied Chemistry in the School of Pharmaceutic Chemistry of the Medico-Chirurgical College of Philadelphia. Second Edition. P. Blakiston's Son & Co. Philadelphia. 1908. 610 pages. Price, $2.50 net.

The authors of this book are teachers in a school of pharmacy and presumably have had in mind the needs of students in schools of this class. It must be admitted that the presentation of the elements of chemistry to beginners in such schools is a difficult matter, as the young people usually enter upon their work with a relatively low intellectual equipment. Books for their guidance should be simple and clear, and need not be very extensive in scope. The aim should be to impart a knowledge of principles, rather than to present a great array of facts, however interesting and important the latter may finally appear. In the presentation of facts the book before us has gone far enough, possibly too far. But in the proper presentation of the foundation theories of general chemistry it is, in the opinion of the reviewer, very weak. The absurd paragraphs headed "The Ionic Theory" (p. 178) and "Physical Chemistry and Electro-Chemistry" (p. 179) are good illustrations of the authors' acquaintance with modern chemistry. Such awkward statements as this (p. 18) are not uncommon:

$$H_2O + \text{electrolysis} = H_2 + O.$$

Acids are treated as a group, without regard to their composition, and hence we find (p. 68 to 71) this order of paragraphs: nitrous acid, nitric acid, hydrocyanic acid, hypophosphorous acid, metaphosphoric acid, hydrochloric acid, nitromuriatic acid and the chlorine oxyacids. On the following pages we find dibasic, and then tribasic acids in a similar order.

The authors seem to feel more at home in the field of organic chemistry as they devote much space to wonderful graphic formulas. These look well in print, and impress the student. But think of the graphic formulas for chrysamine, Biebrich scarlet and crystal violet for beginners ! ! ! (pages 447 and 466). Of any proper appreciation of logical system there is but little, while ambiguous and inaccurate paragraphs like the following are common enough: "Substances which give reactions of two classes of substances are termed 'tautomeric' " (p. 395). On page 483 we find this bit of wisdom: "Benzoic acid is used as a preservative in foods, but its use should be prohibited, in that it is converted into phenol by the liver and acts as a systemic poison."

The book is full of typographical errors, some of which are listed in a table. But enough blemishes are left to condemn it, even for the overworked and not very particular pharmacy student. J. H. Long.

Chapters on Papermaking. Vol. V. Clayton Beadle. London: Crosby, Lockwood & Son. 1908. Small 8vo. pp. 182.

The present volume which is the fifth in the valuable series of which Mr. Clayton Beadle, the well-known English authority on papermaking, is the author, concerns itself with the theory and practice of "beating," which operation is aptly termed "the very centre of papermaking." The work of the Hollender or beating engine in opening out, bruising and shortening the fibers which are later to form the sheet and which have already undergone the preliminary mechanical and chemical processes of purification, is perhaps the most important operation in the whole art of papermaking, and the one which demands, if fine papers are to be produced, the highest degree of intelligence and discrimination in its direction. It is in fact a mill proverb that the paper is made or spoiled in the beater. In view of this it is a matter of surprise that papermakers generally have given so little attention to the underlying principles of this portion of their art and a subject for congratulation that so capable an investigator as Mr. Beadle has now devoted himself to their consideration. The volume in which his conclusions are recorded should be in the hands of every papermaker, and in the library of every chemist whose work has to do with papermaking. The studies, by dynamometer and direct-connected motors, of the amounts of power consumed by different types of beater during the several stages of the operation, are of especial interest even though their general application is necessarily limited by the great extent to which the several factors concerned in the problem may vary in different mills.

The advantages offered by stone beater rolls, so largely used abroad in the preparation of stock for grease-proof and translucent papers, have never been appreciated in this country. The relative merits of stone rolls and metal bars are here discussed at length. Attention should also be called to the studies of the length and condition of fibers at different stages of beating, and to the methods described for ascertaining the suitability of beaten stock for dense or bulky papers as the case may be by determination of its "wetness."

Although the text of this new volume is deserving of much praise, the same cannot be said for the illustrations which accompany it. These are generally of too small scale and from plates so worn that the impression is ragged and indistinct. A number of equally unsatisfactory photomicrographs of fibers are reproduced.

ARTHUR D. LITTLE.

The Methods of Textile Chemistry. By FREDERIC DANNERTH. New York: John Wiley & Sons. 164 pp. Price, $2.00.

From its title-page this little volume purports to be "the syllabus of a lecture course adapted for use in textile laboratories." By the latter term the author doubtless intends to signify the laboratories of textile *schools* rather than the general sense of laboratories in textile establishments, as these would scarcely permit of the delivery of a lecture course. The volume, though rather sketchy in character, contains some valuable matter, and in many cases is nicely systematized. In other cases, however, it rather states the problem than attempts to solve it; for instance, in a section on the "detection of mordants on the fiber," the author states that "the ignition method is found to be preferable. * * * The ash may contain aluminium, chromium, iron, manganese," etc., etc., but the student is left in the dark as to *how* these various substances may be detected. Further, "phosphates are detected by means of the ammonium molybdate reaction," but no hint is given as to what this reaction is or how it is carried out. Again, "tin compounds are looked for in the ash as given above;" on looking "above" we find the only reference to be "the ash may contain tin." In another section on the "detection of sizing on a fabric," the student who seeks for information on the subject is met by the rather unexplanatory statement that the "metals which may be present are magnesium, calcium, and zinc." No attempt is made to furnish a method for the proper detection of these substances. In the same section we are told that Irish moss may be detected in a sizing by the fact that it "gives no precipitate with the usual reagents"—a rather hopeless state of affairs. If the author had taken the pains to describe the "methods" which the title of the book would suggest, it would have far more value, but in the majority of instances, he merely states conditions or problems, and offers no method for their solution.

Another feature in this book, which we also believe to be common to many books of its class, is the mixed nature of the spelling employed. In some places we find "aniline," in others "anilin;" the author insists on "benzin" and "diphenylamin," but also gives us "chlorine," "iodine," etc. This is a transition period in the question of spelling chemical names, but an author should adopt either one side or the other, and adhere to it rigidly—or at least throughout one book.

On page 86, of this book, the figures given for the strength of cotton yarns would lead most readers astray; for instance, under 40's twisted American yarn, the average number of fibers in the cross-section of the yarn is given as 100, the strength of each fiber is stated as 140 grains; hence the calculated strength of the yarn is 160 pounds (*sic*). To the uninitiated this seems a rather remarkable calculation; though some solace is furnished in the further statement that the actual strength is only 36 pounds, yet even this figure would indicate that the 100 fibers acquired a marvelous increase in strength when spun into yarn. The author has omitted to state that the calculated and actual strengths of the yarn are based on a lea of 80 threads.

J. MERRITT MATTHEWS.

Tonindustrie-Kalender, 1909. Part I, Calendar; Part II, pages 406; Part III, pages 212. Berlin: Published by the Tonindustrie-Zeitung.

The first part of this work is merely a calendar, with spaces and blank pages for memoranda, and is satisfactory for the purpose, except that it might be just as well to omit the bright green pages devoted to advertising matter distributed indiscriminately through the volume. The third part consists of a classified book index and also contains an index of machinery manufacturers and dealers in materials of interest to the clay industries. The second part contains a large amount of information in connection with the raw materials, manufacture, analysis and testing of materials and products of the clay industries, a useful pocket companion to either the chemist or superintendent in the ceramic industry.

W. D. RICHARDSON.

American Handy-Book of the Brewing, Malting and Auxiliary Trades. By ROBERT WAHL and MAX HENIUS. Third edition. Two volumes. 12mo. flexible leather. pp. 1596. Chicago: Published by Wahl-Henius Institute. 1908. Price, $12.00.

This is a compendium of general and special information for the use of brewers. The first edition was published in 1901, the second edition in 1902. In the present edition several new chapters have been added and most of the old have been entirely rewritten with a view of bringing the work down to date. Volume I takes up the following subjects: Physics, Mechanics, Electricity, Power, Lubricants, Mensuration, Weights and Measures, Mathematical Tables, Chemistry, Brewer's Chemical Laboratory, Botany, Microorganisms, Brewer's Microscopical Laboratory, Legal Relations and Bibliography. In the chapter on Power, the following divisions are covered: steam boilers, combustion, pumps, steam engines, steam turbines, gas engines, transmission of power, air compressors, refrigeration, heating and ventilation, and dynamic electricity. Volume II takes up the malting and brewing processes proper, as follows: Brewing Materials, Barley, Malthouse, Malting Process, Brewery Buildings, Brewery Outfit, Diastase and Peptase, Yeasts, and Fermentation, Pure Yeast Culture, Brewing Operations, Utilization of By-products, Brewery Calculations, Beer Standards, Miscellaneous Information, Dictionary of Technical Terms in German and English. Under Brewing Operations are to be found the following subdivisions: fermenting cellar operations, special brewing systems, special American bottom-fermentation beers, production of thick-mash beers in Germany and Austria, chip-fermentation beers in the United Kingdom, America and Germany; combination of various beers, brewing losses from malt mill to platform, bottling of beer, treatment and protection of surfaces, and treatment of boiler water. The work is all the name implies and in point of usefulness may be compared to Kent in the engineer's library, or to Chemiker-Kalender and Lunge's pocket book, in the chemical laboratory. The printing, paper and binding are excellent and the index comprehensive. W. D. Richardson.

Handbook for Cement Works Chemists. Frank B. Gatehouse. J. B. Lippincott Co.: Philadelphia. Price, $1.75.

The author is chemist to the Dartford Cement Works, England. It is a work intended to serve a double purpose, namely, a guide to chemists without extended experience and at the same time a valuable reference book for the experienced chemist. C. N. Wiley.

Factory Glazes for Ceramic Engineers. By A. West, Riding Expert. E. and F. N. Spon, Ltd., 57, Waymarket, London. Price, 42 s. net.

Glazes for the temperature "of the soft porcelain kiln," using the following constituents: feldspar, Cornish stone, whiting, barium carbonate, oxide of zinc, china clay, and flint. Unfavorably reviewed in *Brit. Clayworker*, 17, 241–2. H. E. Ashley.

An important volume upon "The Design, Construction and Maintenance of Sewage Disposal Works," by Hugh P. Raikes, has recently appeared in England. (D. Van Nostrand Company, New York.) The problem of Sewage Disposal is one which is receiving necessarily much attention in this country, and which is certain to be an increasing factor in the well-being of all thickly settled communities. This work, therefore, of Mr. Raikes is a valuable manual and as it is a practical and complete treatise it will certainly demand the attention of our civil engineers generally as well as the municipal officers of our towns and cities. It forms an 8 vo. volume of over 400 pages, well illustrated, and price, $4.00, net.

Dr. George F. Lunge, the well-known author of the standard work on "Sulphuric Acid, Ammonia," etc., which has been translated into English, also author of "The Alkalies Makers' Hand Book," in collaboration with the late Dr. Ferdinand Hurter, has recently prepared a new and revised edition of this latter work under the title of the "Technical Chemist's Hand Book." Practically, this may be stated as an entirely new work, inasmuch as the additions and the general revision have been so extensive and thorough. A more complete treatment of the subject matter was published in the German, in 1904–5, and a translation of this, edited by Dr. Chas. A. Keane, is in course of publication under the title of "Technical Methods of Chemical Analysis," of which volume 1 (in 2 parts) has appeared (price, $15.00). The publishers in this country of these works are "The D. Van Nostrand Company."

Prof. S. P. Thompson has in preparation a new book on "The Electric Production of Nitrates from the Atmosphere." There are no important works on the subject at present and the new volume will be welcomed by the many engineers and chemists interested, especially as Prof. Thompson has taken a foremost part in the investigations carried on along this line. Two German works have previously appeared on this subject: Donath and Frenzel, *Die technische Ausnutzung des Atmosphärischen Stickstoffs*, 1907; and Vageler, *Die Bindung des Atmosphärischen Stickstoffs in Natur und Technik*, 1908.

Salter's translation of Gregorius's Manual on "Mineral Waxes," is somewhat important as the very scanty literature, at least in English, on the subject is apparent. The manufacturing industries, however, in the line of paraffin, ceresine, and more recently mineral wax, rank among the important branches of technical chemical production, and therefore this work will probably prove of value to all those engaged in such industry. Price is $3.00, net.

NEW BOOKS.

Anleitung zur mikrochemischen Analyse der wichtigsten organischen Verbindungen. By H. Behrens. (4 Hefte) Heft 2: Die wichtigsten organischen Faserstoffe. 2. Aufl. Hamburg, 1908. 8vo. mit 3 Farbendrucktafeln u. 20 Figuren. Price, M. 5.

An Introduction to Pharmacodynamics Based on the Study of the Carbon Compounds. By F. F. and J. M. Brickdale London: Edw. Arnold. 1908. pp. 372. Price, $4.

Traité d'Analyse chimique quantitative. By R. Frésénius. Huitième édition français. 1652 pp. 430 fig. 18 fr. Lib. Masson et Cie. Paris. 1909.

Handbook for Cement Work Chemists. By F. B. Gatehouse. Philadelphia: J. B. Lippincott Co. Price, $1.75.

Laboratory Outline for Determinations in Quantitative Chemical Analysis. By Albert F. Gilman. 1908. Easton, Pa.: Chemical Publishing Co. Price, 90c. net.

Text-book of Medical Chemistry and Toxicology. By J. W. Holland. Saunders. Price, $3.00 net.

Die Pharmokologie eine biologische Wissenschaft. By C. Jacobi. Leipzig. 1908. 8. Price, M. 1.50.

Agenda Dunod pour 1909: Chimie a l'usage des Chemistes et Ingenieurs. By Emile Javet. 400 pages de texte et 128 pages blanches pour notes. 2 fr. 50. Lib. H. Dunod et E. Pinot, Paris, 1909.

Die Chemie im täglichen Leben. By Lassar-Cohn. Gemeinverständliche Vorträge. 6 Aufl. Hamburg. 1908. 8°, 352 Ss. 24 Fig. Price, M. 4.

L'Evolution souterraine. By E. A. Martel. Paris: E. Flammarion. 388 pp., 80 fig.

Notes on Qualitative Analysis; for Students of the Rensselaer Polytechnic Institute. By W. P. Mason, 5th ed. 57 pp., tabs. Easton, Pa.: Chemical Publishing Co. Price, 80c.

Text-book of Quantitative Chemical Analysis. By J. C. Olsen. New York: D. Van Nostrand Co. 3rd ed., rev. 1908. (D 12) c., ill, tabs., 8°. Price, $4.00.

Thermoelemente und Thermosäulen. By Dr. Franz Peters. Ihre Herstellung und Anwendung. Halle a. S: Verlag von Wilhelm Knapp. 1908. pp. 180.

Synthèse et constitution des albuminoides. By Emm. Pozzi-Escot. 110 pp. 1 fr. 50. Lib. Paris: Jules Rousset. 1908.

Some Electro-Chemical Centres. By J. N. PRING. Manchester: Sherratt and Hughes, University Press. 1908. 8°, 136 pp. Price, 1s. 6d.

Préparation mécanique des minerais. By C. RATEL. Paris: 1908. gr. in-8 574 pp av 11 planches et 190 figures. Price, M. 18.80.

Laboratory Notes on Industrial Water Analysis. By ELLEN H. RICHARDS. New York: John Wiley & Sons, 1908 (1909). c. 3+49 pp. tabs., 8°, cl. Price, 50c.

Laboratory Guide of Industrial Chemistry. ALLEN ROGERS. New York: D. Van Nostrand Co. c. il. tabs., 8°. Price, $1.50 net.

Laboratory Manual of Qualitative Analysis. By W. SEGERBLOM. London, 1908. 8°. Price, $1.00.

La Chimie dans l'art militaire moderne. By SERRANT-BELLENOUX. 1 vol. in-8°. 131 pp. 2 fr. 50. Paris: Lib. H. Desforges. 1908.

Story of Iron and Steel. By J. R. SMITH. London, 1908. 8°, 206 pp., with figures. Price, 75c.

Factory Glazes for Ceramic Engineers: A West Riding Expert. By E. AND F. N. SPON. Ltd., London. Price, 42 s. net.

Practical Metallurgy. By THOMAS TURNER. London: Charles Griffin & Co., Ltd. 1908.

Cyanide Processes. E. B. WILSON. 4th ed. New York: John Wiley & Sons. London: Chapman and Hall, Ltd. 1908.

SCIENTIFIC AND INDUSTRIAL SOCIETIES.

AMERICAN INSTITUTE OF CHEMICAL ENGINEERS.

Abstracts of papers read at the Pittsburg meeting Dec. 28 and 29, 1908, in complete form for publication in the annual volume of the Institute.

Modern Electric Pyrometry.

BY EDWIN F. NORTHRUP.

This paper gives a discussion of the various conditions under which electric pyrometers could be used and the accuracy of temperature measurements which could be obtained from the temperature of liquid air to 1200° C. The resistance thermometer is more accurate and reliable than the thermocouple. Between 1200°–1600° C. the platinum plus 10 per cent. rhodium thermocouple will take its place. The accuracy even at 1200° C. is within 2 to 5 per cent., while ordinary temperatures can be registered to 100th of a degree.

But few chemicals or manufacturing processes are independent of temperature changes which vitally influence the money value of the product. It is impossible to obtain an accurate control of the temperature of the processes without some form of pyrometer or other temperature measuring device. The objection to pyrometers of apparent unreliability is usually due to improper usage in the hands of inexperienced operators, to uses at ranges other than that for which they are intended, or to cheap and unreliable wiring systems. The resistance pyrometer is superior to the thermocouple for temperatures from that of liquid air to 1200° C. because of the greater available energy obtained from a battery.

No fixed design for pyrometers is possible, each manufacturer requiring instruments designed according to his needs. Any number of thermometers may be read by a single indicator at a convenient location or the record of any instrument may be kept on an automatic recorder which is accurate and reliable.

A few of the extensive uses to which pyrometers may be put are air temperatures, lead kettles, steel annealing furnaces, sulphur acid manufacture, etc. The principles and construction of electrical pyrometers is well understood and obtaining successful and accurate results now depend on the intelligence used in their installation and operation.

The Use of Pulverized Fuel for Heating Industrial Furnaces.

BY RICHARD K. MEADE, NAZARETH, PA.

This paper discussed the advisability of using pulverized fuel in place of producer gas for heating metallurgical and industrial furnaces. It also called attention to the precautions which are necessary to guard against explosions and fires in the preparation of pulverized fuel. The various forms of dryers used to free coal from moisture, of the mills used to pulverize the fuel, the apparatus employed for injecting the coal into the furnace were described and illustrated with lantern slides. The paper closed with an estimate of the cost of a model fuel plant and some views of particularly up to date installations.

The Examination of Flue Gases in Boiler Tests.

BY H. A. HUNICKE.

It was shown that the oxygen equivalent, which the authors define as the sum of the volumes of oxygen, carbonic anhydride and carbonic oxide corrected to their respective equivalent volumes of oxygen is a convenient means of studying combustion phenomena. The data of the Fuel Testing Plant of the United States Geological Survey are plotted on a chart and discussed. The author concludes that for the present we cannot hope to gain much more information from the flue gases than that most valuable instrument, the carbonic acid recorder, already reveals.

An Apparatus for Testing Liquefied Ammonia Gas.

BY F. W. FRERICHS, ST. LOUIS, MO.

A review was given of the apparatus now in use in the United States and in Europe. It was pointed out that one source of error inherent in some of the apparatus arose from the moisture contained in the atmosphere, which could not be excluded from the sample. The new apparatus excludes this error entirely and its accuracy was demonstrated by repeating the experiments of Lange and Heffter (Chemische Industrie 1898, p. 2) with liquefied ammonia gas of greatest purity and also with mixtures of this ammonia with known quantities of benzole, pyridine, alcohol and water. The liquefied ammonia gas used in these tests had been made from sulphate of ammonium previously purified from all volatile carbon compounds. For this reason it was certain that no carbon compounds like benzole, pyridine, alcohol, etc., could be present and the amount of water was ascertained (by treatment with metallic sodium and measuring the hydrogen evolved) to be less than 0.002%. The average of the five analyses made in the new apparatus of this ammonia showed it to leave upon evaporation a residue of not more than 0.00175% consisting principally of water, iron oxide and lubricating oil; while Lange and Heffter, using Urbans method, have worked with liquefied ammonia gas leaving upon evaporation 0.2% of a non-volatile residue.

Evaporation test of pure liquefied ammonia gas containing known quantities of benzole, pyridine, ethyl alcohol or water.

			Addition evaporated	
Substances present.	Liq. NH_3. grams.	Addition, grams.	by Urban. Per cent.	by F. F. Per cent.
C_6H_6 and C_5H_5N...	99.0	1.0	00	15
Benzole..........	98.5	1.5	41	59
Pyridine..........	99.0	1.0	33	24
Ethyl alcohol......	99.0	1.0	20	17
Water............	98.9	1.1	17	11
Used ammonia, leaving upon evaporation			0.2	none

The limit of accuracy of testing with the new apparatus has been ascertained to be as follows:

For water 0.002% For alcohol 0.006%
For pyridine 0.009% For benzole 0.110%

PERSONAL NOTES.

On Feb. 13, in the Academy of Music, Philadelphia, the degree of L.L.D. was conferred on Mr. Swank by Temple University. This honor was conferred on Mr. Swank as a recognition of his great service to the business interests of the country and also as a tribute to his literary attainments as shown in his books including "Iron in all Ages," "Notes and Comments" and "Progressive Pennsylvania."

S. B. Sheldon, General Superintendent of the Lackawanna Steel Company, Buffalo, N. Y., has resigned and accepted a position with the Bethlehem Steel Co., Bethlehem, Pa.

Mr. Geo. F. Docons, Assistant General Superintendent, has been appointed General Superintendent, Lackawanna Steel Company.

Mr. Thos. Mathias, Superintendent of rolls, has been appointed Assistant General Superintendent, Lackawanna Steel Company.

Jose Francisco de Navarro, founder of the Atlas Portland Cement Co. at Northampton, died at his home in New York, February 3rd, at the age of 71 years.

"Purification of Ground Waters Containing Iron and Manganese" is the title of a paper by Robert Spurr Weston, who is employed by the Board of Water Commissioners, Reading, Mass. The article is reported in *Engineering Record* of Feb. 3rd, and was read before the meeting of American Society of Civil Engineers at their December meeting.

Mr. G. L. Norris, after a long service with the Standard Steel Works, Burnham, Pa., as chemist and engineer of tests, has resigned to accept the position of engineer of tests with the American Vanadium Company, 324 Frick Building, Pittsburg, Pa., works at Bridgeville, Pa. Mr. Norris in 1890 was First Asst. Chemist, Illinois Steel Company, South Chicago.

INDUSTRIAL AND TRADE NOTES.

The *Daily Consular Report* of January 30, 1909, states that Consul Paul Lang reports from Sherbrooke that asbestos has been discovered near Eastman, Quebec, and that several companies have organized to develop the new mines. The consul continues:

The Orford Mountain Railroad is near these properties, and a spur will be built from the main line to the mills, which are already in course of construction. This discovery is important, as bearing on the extent of the asbestos fields in this province. The mines near Danville, Quebec, some 65 miles from this new find, and at Thetford Mines, Black Lake, and Broughton, Quebec, and several smaller places on the line of the Quebec Central Railway, about 100 miles from Eastman, are now the only producers of this mineral on this continent. [With the exception of a small output in Vermont and a new mine in Georgia.—B. of M.] The development of these new mines will induce more thorough prospecting all over the eastern townships.

Representatives of the Millers' National Federation met in the Auditorium Hotel, Chicago, January 8th, to consider means of obtaining the withdrawal of an order of the Department of Agriculture against the bleaching of flour. It is probable that Secretary Wilson will be asked to lay the matter before a board of referees composed of milling experts for final decision.

It was reported from Bisbee. Ariz., January 4th, that work on the Calumet and Arizona smelters at Douglas was progressing rapidly. The new 500 ton stack was working over time and producing nearly 60 tons a day. All of the old furnaces will be increased and will practically double their present output. The furnaces are now 300 tons capacity but this will be increased to 500 tons.

The production of copper in the United States during 1908, as compiled by L. C. Gaton, of the U. S. Geological Survey, shows a considerable increase over 1907. The increase amounts to about 50,000,000 pounds, or between 5 per cent. and 6 per cent. more than the production of 1907, which was 8,996,491 pounds.

It is reported from Butte, Mont., that although no complaints are made about the Great Falls smelter, the Amalgamated Co. is spending $500,000 there in building a stack and dust chambers, for the purpose of saving flue dust and arsenic, and rendering the sulphur fumes as little harmful as possible. The stack was completed some time ago. It is hoped that it will save flue dust to the value of $180,000 per year. It is 506 feet high and has an outside diameter at the top of 50 feet. The flue dust will be precipitated by means of a new system consisting of a forest of fine wires strung from the ceiling of the dust chamber. There are 1,300,000 of these wires and more than 1000 hoppers have been built to receive the dust. The hoppers discharge automatically into cars which carry the dust direct to the reverberatory furnaces, where it is smelted. The tracks under the great dust chamber resemble a freight-yard.

For eleven months in 1908, Germany imported 147,114 tons of copper and exported 8,034 tons. The consumption was 139,080 tons as against 105,317 tons for the same period in 1907. Of the quantity consumed in 1908, 134,906 tons were imported from the United States.

A new $2,500,000 steel company has been organized for Chicago, with New York and Chicago capital. The plant will be located at Chicago Heights and construction will be commenced during the present month. The company will be known as the Inter Ocean Steel Company and will manufacture steel tires for locomotive wheels, car equipment, rolled steel shells and other circular sections used in mining machinery. It will be independent of the U. S. Steel Corporation. The Board of Directors is composed of: E. C. Converse, president Bankers' Trust Company, New York; W. T. Graham, president American Can Company; Frederick H. Easton, president American Car & Foundry Company; Julian Kennedy, engineer, Pittsburg; John S. Runnells, vice-president Pullman Company, Chicago; Arthur Meeker, Armour & Co., Chicago; Mason B. Starring, president Northwestern Elevated Railway, Chicago; W. V. D. Wright and W. L. Jacoby, respectively treasurer and president of the new company.

Julian Kennedy, for many years general manager of the Carnegie Steel Company at Homestead, Pa., and an engineer famous the world over, is designing the plant. There will be open-hearth furnaces and rolling mills included in the equipment.

The second case against the Standard Oil Company of Indiana was brought to trial before Judge A. V. Anderson at Chicago, on February 23. This is the world famous case, in which at the conclusion of the first trial, a $29, 000,000 fine was assessed by Judge K. M. Landis.

The work of the U. S. Geological Survey, as shown by its 1908 report, shows it to be of the most diverse character, ranging from that of the explorer and geographer making surveys in interior Alaska to that of the mining engineer, investigating gas and coal dust explosions and mine explosives, together with the considerable work of a purely geological nature. The appropriations for the year amounted to $1,782,770 and more than 150 parties were in the field.

The metal output of Utah during 1908 had a value of not much more than half that of 1907. Several causes contributed to this result. While the production held up well, prices fell off to a large extent, and further difficulty was caused by the closing of smelters by court order on account of damage by smelter fumes to agricultural products and stock. The copper production for 1908 was 74,000,000 lbs. as against 98,800,000 lbs. in 1907; the price for 1908 was 13 1/8 cents per lb. as against 20 cents in 1907. The total value for 1908 was slightly under $10,000,000 as against $19,760,000 in 1907. Gold production dropped from 750,802 ounces, valued at $11,416,040 in 1907, to 250,000, ounces valued at less than $5,000,000 in 1908. The silver produced amounted to $17,204,100 ounces in 1907, which was valued at $11,354,651; whereas in 1908 the production was about 10,000,000 ounces, of a total value of less than $5,000,000. The lead production dropped from 142,176,000 lbs. in 1907 to 103,000,000 lbs. in 1908, and the value from $7,108,800 to about $3,350,000.

On January 25, there was formed at the Waldorf Astoria in New York an organization of manufacturers of foods, to oppose the use of chemical preservatives and chemicals in foods. The organization is called the American Association for the Promotion of Purity in Food Products. Resolutions were adopted, stating that since "certain reactionary interests" opposed to the enactment of a national pure food law are now opposing its enforcement and by concerted action have been able to nullify in great measure some of the important provisions of the law, the members will give their moral and financial support and their undivided influence toward upholding "the proper and legitimate efforts of the regularly constituted officials charged with the administration of all laws looking to the elevation of the food-producing interests of the country.

The total copper production in the United States during 1908 amounted to 868,996,491 lbs., an increase of 50,000,000 over 1907. Imports decreased about 13 per cent. and exports increased about 30 per cent.

The output of petroleum in the state of California for 1908 amounted to 50,000,000 barrels, The Kern river district produced 13,000,000 barrels; Coalings district 12, 000,000 barrels; Santa Barbara produced 9,000,000 barrels; Fullerton 4,000,000 and Salt Lake 4,000,000. Other districts run as follows: McKittrick, 3,500,000; Sunset, 1,500, 000; Whittier, 750,000; Los Angeles city, 475,000; Midway, 400,000, and Ventura, 300,000.

The total production of bituminous coal in the United States amounted to 330,000,000 tons in 1908 as against 400,000,000 in 1907.

The report of the Food Referee Board, consisting of Dr. Ira Remsen, president of Johns Hopkins University; Russel H. Chittenden, director of the Sheffield Science School of Yale University; John H. Long, professor of chemistry, medical school, North Western University; C. A. Herter, professor of physiological chemistry, College of Physicians and Surgeons, New York, and Alonze E. Taylor, professor of pathology, University of California, and appointed last March by the Secretary of Agriculture at the suggestion of President Roosevelt, was handed to the Secretary January 23. The questions submitted by the President to the Board were as follows: 1. Does a food to which there has been added benzoic acid or any of its salts, contain any added poisonous or other added delectorious ingredient which may render the said food injurious to health? In large quantities? In small quantities?

2. If benzoic acid or any of its salts be mixed or packed with a food is the quality or strength of said food thereby reduced, lowered, or injuriously affected? In large quantities? In small quantities?

"To make this experimental inquiry as thorough as possible," says the report of the board, "and to minimize the personal equation, three independent investigations have been carried out: One at the medical school of Northwestern University in Chicago, a second at the private laboratory of Christian A. Herter of Columbia University, New York, and the third at the Sheffield Scientific School of Yale University.

"The same general plan of procedure was followed in all three experiments. A certain number of healthy young men were selected as subjects and during a period of four months these men under definite conditions of diet, etc., with and without sodium benzoate, were subjected to thorough clinical and medical observation, while the daily food and the excretions were carefully analyzed and otherwise studied and comparison made on clinical, chemical bacteriological and other data collected. In this manner material has been brought together which makes possible conclusions regarding the effect of small and large doses of sodium benzoate upon the human system."

The board says that the fact should be emphasized that the results obtained from the three separate investigations are in close agreement in essential features. The main general conclusions reached by the board are:

First—Sodium benzoate in small doses (under 0.5 gram per day) mixed with food is not deleterious or poison, and is not injurious to health.

Second—Sodium benzoate in large doses (up to 4 grams per day) mixed with the food has not been found to exert any deleterious effect on the health nor to act as a poison in the general acceptation of the term. In some directions there were slight modifications in certain physiological processes, the exact signification of which modifications is not known.

Third—The admixture of sodium benzoate with food in small or large doses has not been found to injuriously affect or impair the quality or nutritive value of such food.

Dr. Wiley as the result of his experiments says[1] that "from a careful study of the data in individual cases and of the summaries of the results, it is evident that the administration of benzoic acid, either as such or in the form of benzoate of soda, is highly objectionable and produces a very serious disturbance of the metabolic functions, attended with injury to digestion and health."

"These injurious effects," says Dr. Wiley, "are evident in the medical and clinical data which show grave disturbances of digestion attended by phenomena which are clearly indicative of irritation, naseau, headache and in a few cases vomiting."

In concluding, Dr. Wiley said: "Coming to the final consideration of all these different phases of the subject, there is only one conclusion to be drawn from the data which have been presented and that is in the interests of health both benzoic acid and benzoate of soda should be excluded from food products. This conclusion is reached independently of any consideration of the conditions which it is alleged surround the processes of manufacture and which result in the demands of manufacturers to be allowed to continue the use of these substances. This is a subject which must be discussed from an entirely different point of view and has no bearing whatever upon the general conclusions which have been reached, namely, that both benzoic acid and benzoate of soda are bodies which when added to foods are injurious to health."

It is reported that the experiments thus far made in the testing laboratory of the U. S. Geological Survey at Pittsburg, have shown that mine explosions can be minimized or prevented by forcing steam through the air currents produced by the ventilating fan of the mine. A miniature mine was constructed at the testing plant, and the conditions inside were made as nearly as possible like those in a real mine with a quantity of dust in it. In the presence of mine superintendents, foremen and engineers, steam was turned into the dust-laden air of the miniature mine, and it was found impossible to cause an explosion. Cold dry air was then forced into the mine, and the explosion took place almost instantly. The same experiments were tried repeatedly, with the same results.

This use of exhaust steam was suggested two years ago and put into effect by H. A. Kuhn, president of the Pittsburgh & Westmoreland Coal Co. Since resorting to this measure as a precaution explosions have been unknown in the Pittsburgh–Westmoreland mines.

Further tests by the government experts are to be made, with the idea of trying out the theory in every known form.

On January 22nd President Roosevelt sent to Congress a special message transmitting the report of the National Conservation Commission, which was appointed by him in June, 1908, to inquire into and advise him as to the condition of the country's natural resources. The report discusses lands, forests and waters. "The facts set forth in this report constitute an imperative call to action," says the President in his letter of transmittal. "The situation they disclose, demands that we, neglecting for a time, if need be, smaller and less vital questions, shall concentrate an effective part of our attention upon the great material foundations of national existence, progress and prosperity."

He discusses the various recommendations approvingly, and concludes with the suggestion that an appropriation of at least $50,000 be made to maintain a national conservation commission. "This is a very small sum," he says. "I know of no other way in which the appropriation of so small a sum would result in so large a benefit to the whole nation."

On Jan. 18th, the Supreme Court affirmed the judgment of the Texas courts imposing a fine aggregating $1,623,900 on the Waters-Pierce Oil Co. for violating the anti-trust laws of that state.

The National Tube Co. of Lorain, Ohio, have completed their new open-hearth plant of seven furnaces.

The new rail mill of the Indiana Steel Co. at Gary was tried out Jan. 17th, and rails were produced from steel sent from the Illinois Steel Co. at South Chicago.

The Illinois Steel Co.'s plant at South Chicago has nearly resumed full capacity. Blast furnace No. 10, idle since Nov., 1907, was blown in Jan. 31st. Blast furnace E, idle since Jan., 1908, was blown in Feb. 8th. The steel mill and rail mill, idle since Dec. 31st, 1908, were started again

---

[1] Bull. 84, Part 3, Bur. Chemistry.

Feb. 8th. Cement plant No. 2, located at South Chicago, idle since Sept. 1908, started producing again Feb. 1st.

It is reported that a new $200,000,000 organization, the North American Portland Cement Company, which includes plants with an output of nearly 40,000,000 barrels, has been formed.

A bill was introduced in the House of Representatives, sixtieth congress, first session, by Mr. Currier, No. H. R. 21455, entitled "A Bill to Establish a United States Court of Patent Appeals and for Other Purposes." If this bill is passed, it will do away with the grievous conditions that exist now in patent litigations before the circuit courts (see address by Louis C. Raegener in this number). The bill has been favorably reported upon, as will be seen by reading the arguments before the Committee on Patents of the House of Representatives, March 18th, 1908, number H.R. 14047. All chemists, all engineers, all inventors, as well as the public at large are interested in this subject and it is very desirable that the bill should pass. The members of the Committees of the House and of the Senate who will have the bill under consideration are as follows:

Committee on Judiciary of the House of Representatives: John J. Jenkins, of Wisconsin, *Chairman*; De Alva S. Alexander, of New York; Charles Q. Tirrell, of Massachusetts; John H. Foster, of Indiana; Reuben O. Moon, of Pennsylvania; George R. Malby, of New York; David A. DeArmond, of Missouri; Robert L. Henry, of Texas; Charles C. Reid, of Arkansas; Richard Wayne Parker, of New Jersey; Charles E. Littlefield, of Maine; John A. Sterling, of Illinois; Henry T. Bannon, of Ohio; Garrit J. Diekema, of Michigan; Henry S. Caulfield, of Missouri; Henry D. Clayton, of Alabama; William G. Brantly, of Georgia; Edwin Y. Webb, of North Carolina.

Senate Committee on Patents: Hon. Reed Smoot, of Utah; William H. Milton, of Florida; Frank B. Brandegee, of Connecticut; Frank B. Gary, of South Carolina; Alfred B. Kittridge, of South Dakota; Murphy J. Foster, of Louisiana; Moses E. Clapp, of Minnesota.

According to the press reports of January 26th, Federal Judge Hunt has declined to issue an injunction against the Washoe smelter of the Anaconda Copper Mining Co. at Anaconda, Mont. The complaint of the plaintiff, who in reality represents the Deer Lodge Valley Association. is not dismissed, but the court desires additional information before final disposition of the case is made. February 15th was named by Judge Hunt as the time for the hearing of new testimony, and E. P. Mathewson, manager of the smelter, and others were ordered to appear in court at that time.

This case is of particular interest as it represents the largest suit growing out of the alleged damage to vegetation and stock by smelter smoke, and is the first suit in the west that has not been decided in favor of the complainants. Had the injunction been granted the paralysis of the mining and smelting industries of the Butte district—as well as of many other industries directly or indirectly dependent upon them—would have been assured.

The H. C. Frick Coke Co., the coking company of the United States Steel Corporation, has decided to abandon work on additional coke ovens in Pennsylvania, which were to be built at a cost of $3,000,000, and in their stead construct an enormous coking by-product plant at Gary, Ind. It will take the place of 700 ovens, which were to have been built at Filbert, 500 at Ralph, and 500 at Sarah, Pa. In the meantime the company is opening new mines to supply the ovens with coal.

It was the intention of the Steel Corporation to build more beehive ovens in the Connellsville region in Pennsylvania, but it has decided to build an extensive series of by-product ovens instead, thus saving all the gas now wasted in coking coal, besides saving the ammonia and other coal tar products.

Platinum Mining in Russia.—Consul-General Hunter Sharp, of Moscow, advises that owing to the depression in the platinum-mining industry in Russia, the platinum miners have applied to the Government asking that the industry be upheld until it is definitely decided whether the mining of platinum is to be made a monopoly of the Russian Government. The platinum miners have petitioned the Government for temporary assistance; that is, to permit the Russian Imperial Bank to grant loans on platinum ore containing 83 per cent. of pure platinum, at the valuation of 16,000 rubles per pood ($8,240 per 36 pounds), with interest at the rate of 5 per cent. per annum.—*U. S. Consular Report*, Jan. 26, 1909.

Nitrate of Soda Supply.—According to Consul Rea Hanna, of Iquique, the Delegacion Fiscal, or Official Board of Engineers, states the following in its annual report to the Government of Chile:

It is the current opinion in Europe and the United States that our nitrate deposits will be exhausted after a maximum period of forty years, and so widely spread is this error that the subject has been considered in the annual report of the Secretary of Agriculture presented to the Congress of the United States. Perhaps the data submitted by the delegation in former years caused the lack of confidence, but the data then given were the results of this office up to that time, and at present the aspect is entirely different. The experience of the last ten years has shown that the former figures do not nearly approximate the actual conditions. The report of 1896 gave the following figures in Spanish quintals of 101.61 pounds, which were confirmed later in 1906:

	Quintals.
Tarapaca: Nitrate of soda contained in the fields according to the private and official measurements and estimates	573,000,000
Toco: Nitrate of soda contained in the fields according to the private and official measurements and estimates	225,000,000
Antofagasta, Aguas Blancas, and Taltal: Private measurements and estimates only (not official)	305,000,000
Amount calculated in unworked official fields	500,000,000
Total possible production in nitrate zone	1,603,000,000

With a probable annual exportation of 35,000,000 quintals this estimate gave, in 1899, enough nitrate to last forty-six years before being exhausted.

After a detailed discussion of the shortcomings of the former estimates and the increasing importance of the Antofagasta Pampa on account of recent developments the delegation reports:

We have then the following total:

	Quintals.
Tarapaca	740,000,000
Antofagasta	4,103,000,000
Total	4,843,000,000

FUTURE SUPPLY OF NITRATE OF SODA IN CHILE.

There are then undeniably 4,843,000,000 Spanish quintals of nitrate of soda in sight, which by the methods in use at the present time may be produced from the lands measured and estimated in the nitrate region. With an annual exportation of 35,000,000 quintals, which is more than that exported in 1907, there is sufficient to satisfy the entire consumption of the world for one hundred and thirty years.

[The complete official report from which the foregoing was taken may be seen at the Bureau of Manufactures.]—*U. S. Consular Report*, Feb. 10, 1909.

Rubber Production, Mexico.—Consul Charles M. Freeman, of Durango, sends the following in response to an inquiry from Boston as to the opportunities in that Mexican State for an American with a few thousand dollars to engage in rubber production, particularly from the palo colorado tree:

The first absolute necessity for a person contemplating the rubber or any other business in Mexico is a knowledge of the Spanish language. While there are many in Mexico engaged in business who have no knowledge of Spanish, they are handicapped in their operations and, being dependent on an interpreter, their ultimate success is doubtful. Only one rubber-producing plant in northern Mexico, the guayule, has been developed into a commercial success. This and all others grow far from civilization, in localities where the English language is never spoken. The guayule rubber ground is well covered by companies capitalized to the extent of millions of dollars. There is no opportunity for a person of limited means to compete in the guayule region.

The production of rubber from palo colorado and other mild rubber-producing plants, aside from guayule, is in an experimental stage. The exploiters feel certain that they have a good thing, but as they are not getting out any gum it is not possible to give positive statements covering all of the business. From the number of inquiries received at the consulate from consumers of raw rubber it would appear that the market is not only ready, but waiting for the gum of the palo colorado. Further developments are awaiting the verdict of chemists who are working on the raw material to determine exactly its adaptability to certain uses and its commercial value. Should these points be settled satisfactorily, an extensive business in the gum of the palo colorado may be expected.

POSSIBLE RESULTS FROM PALO COLORADO.

The tree grows at least four days' journey from this city, in the Sierra Madre Mountains, to which point all material must be packed on mules or burros, and from which gum must be freighted in the same manner.

Approximately, the cost of production in American currency per kilo (2.2 pounds) is as follows: Employment of natives for the purpose of tapping the trees and delivering the gum at some central station, 12 1/2 cents; freight from central station to railroad, some three or four days' journey, 2 1/2 cents; freight from Mexican railroad station to market in the United States, 5 cents; total, 20 cents per kilo or about 10 cents per pound.

The foregoing does not include the proportionate share to be placed on each pound of gum produced for the interest on the capital invested in leasing or buying land on which the tree grows, nor the proportionate share for management, high-priced labor, government taxes, and other necessary expenses.

Attention is called to the difference in cost between laborers and management. Good laborers can be hired for $10 a month, while a person capable of managing labor, who can speak both English and Spanish, can not be had under $100 American currency.

Guayule sells in the markets of the United States for about 25 cents American currency per pound, and is said to contain 10 per cent. of pure rubber. The palo colorado at the same price should be a good investment, provided the chemists' analysis is favorable. In fact, if, as the exploiters claim, the palo colorado contains over 30 per cent. of pure rubber, then the price should be much greater for the palo colorado than for guayule.

The first work in the rubber business is to locate the trees and to buy the land or lease the privilege of tapping the trees. To do this work is not so simple a matter as in the United States and one finds many obstacles to be overcome. The most satisfactory results will be obtained by employing some person acquainted with Mexican laws and localities to act, under supervision, as agent in this matter. There are reliable persons who, to a certain extent, make that their business.

There seem to be possibilities of success with the palo colorado. If the tree is to be found in sufficient numbers, and if the chemists' report should be favorable, the business will be an assured success. If a success, the first to put the gum on the market will get the profits. However, in the light of present developments, the business is very much of a venture and likely to prove for a person not fully acquainted with all that has been accomplished an unprofitable one. It is estimated that it would take $5,000, possibly twice that amount, before a pound could be marketed.

Barytes in Germany.—Consul-General Robert P. Skinner, of Hamburg, writes that he has been asked to supply information respecting the cost of barytes, which in hearings before Congress was said to be worth $2.78 per ton at the foreign seaports in 1885. Mr. Skinner reports:

This material is used almost exclusively in the paint trade, being ground and mixed with white paint to give it body. The best grades, from the Harz and Thuringian mountains, are naturally white, and can be used without first being bleached. To some extent these grades are utilized in the porcelain and white-glass industries. The cheaper grades of barytes come from the Rhine, are shipped in crude form, and these, both hard and soft, are exported to the United States, where they are treated chemically and made white. Barytes from the Rhine and from Newfoundland contain 96 per cent. of barium sulphate.

The exporting points for barytes in Europe are Rotterdam, Antwerp, and Hamburg. There are many mining firms in the business, and they have formed a pool to maintain prices and apportion the tonnage. It is said that exporters get the same quotations from all producers, who turn back orders when their quota is reached.

Hamburg exporters invariably buy on terms delivered at seaboard. The prices now (December 1) are from $5.47 to $5.71 per ton. The ocean freight from seaboard to New York runs from $1.70 to $2.19 per ton. The German rate from mine to seaboard, shipments being made in large barges, is from $1.19 to $1.42 per ton. This would bring the present price at the mine from $4.05 to $4.52. Since the pooling arrangement referred to prices have been advanced from 50 to 75 cents per ton, although labor cost, it is stated, has also advanced.—*U. S. Consular Report*, Feb. 1, 1909.

Australia's Mineral Wealth.—Consul-General John P. Bray writes from Sydney that the mineral output of Australia up to the end of 1907 reached $3,350,969,703, and that as there was a further production in 1908 amounting to probably no less than $97,330,000, the total is now about three and a half billion dollars, of which fully two and a half billion dollars represent gold. Mr. Bray continues:

For a long time after the gold discoveries in 1851 that metal, in the main, represented Australia's mineral output, but after the total of $62,841,114 was reached in 1856 there was a steady decline until, in 1886, the gold output was only $21,548,862. With the opening of the Western Australian gold fields this position was materially altered and the 1856 production was exceeded in 1899. The maximum output was reached in 1903. The following table gives the production of metals and minerals since 1895:

Year.	Gold.	Silver-lead.	Copper.
1895	$37,510,982	$9,285,282	$1,951,466
1896	38,625,410	9,966,592	2,155,859
1897	47,492,173	9,392,345	4,199,789
1898	56,991,581	9,713,534	4,433,381
1899	71,347,756	12,044,587	10,097,987
1900	66,680,783	13,981,454	8,662,370
1901	69,055,635	10,433,776	10,219,650
1902	72,106,930	9,416,677	7,703,669
1903	79,294,751	9,003,025	8,263,317
1904	77,576,876	12,584,769	7,922,662
1905	75,722,740	13,927,923	11,071,287
1906	71,206,628	17,636,196	16,278,442
1907	65,751,281	22,969,880	17,096,014

Year.	Tin.	Coal.	Other.
1895	$1,153,360	$6,618,440	$885,703
1896	1,377,219	6,379,981	656,977
1897	978,166	7,275,417	1,250,690
1898	997,632	7,528,475	1,338,287
1899	2,311,587	8,068,657	4,170,590
1900	2,219,124	9,835,196	2,769,038
1901	1,121,794	12,764,829	2,550,046
1902	2,087,728	12,896,225	2,919,900
1903	3,357,885	12,808,628	3,017,230
1904	4,014,862	11,334,078	3,946,731
1905	4,995,699	11,377,877	4,297,119
1906	71,206,628	12,993,555	4,199,789
1907	7,309,483	16,074,049	8,842,430

U. S. Consular Report, Jan. 30, 1909.

Peanut-oil Industry.—Consul-General Robert P. Skinner, of Hamburg, after referring to his former reports from France on the peanut-oil industry and expressing the belief that interest in this subject will not subside until mills have been erected in the United States and a peanut crop assured large enough to supply the demand, gives the following additional information:

The Department of Agriculture at Washington is now at work on this subject and is experimenting with African seed nuts which were shipped from this consulate to the United States some months ago. At the present time, and probably contrary to the American impression, the annual crop of groundnuts is not always sufficient to supply the cravings of the domestic appetite. It frequently happens that the west coast of Africa is called upon to make good this shortage, although there is no reason whatever why a forage plant so useful as the peanut, and a kernel so valuable, should not be produced in the United States in practically unlimited quantities. It happens also, that an American oil industry is rendered difficult of establishment, not only because of the uncertainty of the crop, but because the American nut is less rich in oil than the African. The Department of Agriculture is now studying this phase of the matter, hoping, no doubt, to develop an edible nut which will be as palatable as the varieties now on the market, and as rich in oil as the African kernels.

The first step to be taken, naturally, in building up an American peanut-oil trade is to increase the crop of peanuts. In 1900, according to the census returns, the American crop was 11,964,109 bushels, or roundly, 326,294 tons. As Marseille alone imports upward of 200,000 tons of these nuts for French oil mills, it can be judged that the total American crop is a comparatively small affair. Hamburg has come into the importing market more recently, receiving only 11,903 tons in 1907. London is also an importing center. Marseille, therefore, retains its preeminence as the European oil center, and American investigators can learn more of interest and importance there than elsewhere.

West African nuts (Ruffisque, Cayor, Sine, Gambia, Bas de Cote) are usually exported in the shell, and produce edible oil of the highest grade. Other kernels are usually, but not always, received decorticated. The percentages in oil of the best-known grades run as follows: Senegal 51, East African 49, American 42, Bombay 44, and Madras 43.

The oil is obtained by pressure, the first application being while the material is cold, or to be more exact, very slightly heated, and the second and third pressures hot Upon reaching the mill the nuts are first winnowed and then shelled. The kernels are next broken into large pieces and at the same time freed from the red skin to some extent, and from foreign matter completely. The material is then reduced to a fine flour, placed in bags, and pressed cold during one hour. The cake, after the first pressing, is broken, heated to 70 degrees C., and pressed a second and third time. The resulting products per hundredweight of decorticated nuts run in percentages about as follows: First-class oil, 31.5; second-class oil, 10; oil cake, 54.5; losses, etc., 4.

In Europe a first-class peanut oil is the most highly esteemed of vegetable oils after olive oil. It is also used in the manufacture of butter substitutes. The low-grade oils are taken up by the soap trade.

Raw material enters France free of duty, but the oils are dutiable at the rate of 6 francs ($1.16) per 100 kilos (220 pounds) It would be impossible, probably, for peanut oil of foreign manufacture to compete in the Marseille market with the domestic article.

The prices per 100 kilos at Marseille, on November 20, 1908, were as follows: Edible oil, $13.51–15.44; soap oil, $11.97; Bombay peanuts, shelled, $5.84; Mozambique peanuts, shelled, $7.14; Ruffisque nuts, in the shell, $4.58; Bas de Cote, $5.35; peanut cake, $3.13. During ten months of 1908 Marseille received 92,707 tons of shelled peanuts and 94,737 tons in the shell.

German manufacturers press peanuts three times, twice for edible oil and once for soap oil. Ruffisque kernels are chiefly utilized. It would cost in this country about $23,800 to erect a mill of 10 tons' capacity and about $83,300 for one of 50 tons' capacity, exclusive of the cost of real property and buildings.

Prices to-day (November 27, 1908) per 220 pounds in Hamburg for peanuts in the shell are $5.24; shelled, $5.71; oil from $14.76 to $21.42. German peanut cake commands from $35.70 to $38.08 per 2,204.6 pounds. The duty on peanuts in Germany is 2 marks ($0.476) per 220 pounds; that on oil is 10 marks ($2.38) per 220 pounds.

[Consul-General Skinner appends a list of the best-known firms in France and in Germany manufacturing oil presses, which is placed in the files of the Bureau of Manufactures.]

U. S. Consular Report, Jan. 23, 1909.

Rubber-tanned Leather.—A British publication states that a new syndicate has established a factory in London and is placing upon the market a new form of leather, for which it is claimed there is a big future, and which it thus describes:

The leather is submitted to a tanning process of the chrome variety, which preserves it; rubber solution is then worked into the interstices, rendering the hide thoroughly waterproof. The elasticity of the rubber permits of perfect flexibility and extraordinary toughness of some of the skins, especially of rabbit, goat, and sheep skins. It is impossible to forecast the many uses for which this process may be available. Tests have been made for motor tires, soles for boots and shoe, pump washers, machine belting, miner's boots, etc., and it is probable that the multitude of articles that can be made of rubberized leather will, in time, create a further demand for rubber. This new material, it is thought, may also prove useful for motor and cycle tires, as it is almost impossible to puncture it, while it is said to be much more resilient and waterproof than ordinary leather.—

U. S. Consular Report, Jan. 23, 1909.

The Universal Portland Cement Co. is about to start construction on an addition to Plant No. 5 at Universal, Pa. This plant at present has an output of 4,500 barrels per day. When the new addition is completed, which will be about January, 1910, the total output of this plant will be 10,000 barrels per day or 3,000,000 barrels per year. This will give the Company a total production of 23,000 barrels per day or 8,000,000 barrels per year.

OFFICIAL REGULATIONS AND RULINGS.

FOOD INSPECTION DECISION, 103.

The Labeling of Turpentine.—The Department has received a number of letters with reference to the proper labeling of the product generally known as "wood turpentine," etc., obtained by steam distilling or destructively distilling woods. Food Inspection Decision 58 recognizes that—

Products used in the arts and for technical purposes are not subject to the Food and Drugs Act * * * when plainly marked so as to indicate that they are not to be employed for food or medicinal purposes.

It is held, therefore, that when wood turpentine is labeled "Not for Medicinal Use," etc., it is not subject to the food and drugs act. When not so labeled it is in violation of section 7 of the food and drugs act unless labeled "wood" or "stump" turpentine. Articles labeled "turpentine," "spirits of turpentine," or "gum terpentine," etc., must comply with pharmacopoeial requirements; that is, they must be light oils of certain properties made by distilling the oleoresin of various species of Pinus. The word "wood" or "stump" should be in the same type and on the same background as the word "turpentine," thus being given equal prominence.

H. W. WILEY,
F. L. DUNLAP,
GEO. P. MCCABE,
Board of Food and Drug Inspection.

Approved:
JAMES WILSON,
Secretary of Agriculture,
WASHINGTON, D. C., *January 22, 1909.*

Under date of January 30, the Board of Food and Drug Inspection has issued a notice of judgment No. 36, Food and Drugs Act, Misbranding of Canned Apples and Blackberries. Under the same date judgment No. 37, Food and Drugs Act, Adulteration of Milk (Water).

TREASURY DEPARTMENT, *January 4, 1909.*

(T. D. 29455) *Casein.*—Decision of the United States court of appeals, second circuit (suit 4586; T. D. 29374), United States *v.* W. M. Brownell, acquiesced in.

TREASURY DEPARTMENT, *January 8, 1909.*

(T. D. 29461) *Drawback on lubricating oils.*—Drawback on lubricating oils manufactured by the Swan & French Company, of New York City, wholly or in part from imported sperm oil, Russian oil, and Japan oil.—T. D. 17355 of August 1, 1896, extended.

(T. D. 1449). *Method of sampling butter.*—Instructions to internal-revenue officers to discontinue use of butter "trier" in taking samples and describing new method to be used in lieu thereof as follows:

TREASURY DEPARTMENT,
OFFICE OF COMMISSIONER OF INTERNAL REVENUE,
WASHINGTON, D. C., January 2, 1909.

To collectors and other internal-revenue officers and employees:

The act of Congress approved May 9, 1902, imposes an internal-revenue tax of 10 cents per pound on adulterated butter, as described in the act, and special taxes on any person engaged in the business of manufacturing adulterated butter or in the sale of the same.

The enforcement of this law necessitates the examination of the product found on the market or in storage by revenue officers, who are required to procure samples from the packages so found and submit the same for chemical analysis, by which process it is determined whether any chemicals have been used or any substances foreign to genuine butter have been added, or any process of manipulation has been resorted to with the effect of causing the absorption of an abnormal quantity of water, milk, or cream. Upon the results of these analyses it is determined what butter is subject to the tax herein referred to.

Heretofore, while acting under instructions of this office, care has been taken by field officers to obtain truly representative samples of the contents of the tub or package of butter without particular reference to the method of taking the samples, and complaints have reached this office concerning all these methods, more especially where a "butter trier" has been used. It is claimed that this is especially objectionable for the reason that it disfigures the surface of the contents of the package and interferes with the division of the same into prints. Obtaining the samples from the top, bottom, or sides of the package by means of a spoon or other instrument has been criticized, as not giving representative samples of the contents of the tubs.

Hereafter, in procuring these samples, the use of a "butter trier" will be entirely abandoned. The quantity required from each package will be obtained from the top and bottom or sides of the butter contained in the tubs by cutting a V or wedge shaped strip about 2 inches wide, 1½ inches deep, and of sufficient length to make up the required weight of the sample, which must be approximately 1 pound. This can be accomplished by removing the butter from the tub. A V-shaped strip of this character should be taken from each opposite sides of the package of butter, or from both the top and the bottom of same, but it is regarded as sufficient if two such strips are obtained—that is, from the top and bottom in one case and from the two sides in the other instance.

Butter put up in prints will be sampled by taking a 1-pound print from the box or other container.

All officers and employees engaged in securing samples of butter for investigation are admonished to perform this duty with as little trouble and friction as possible, leaving the packages sampled as nearly in their normal condition as may be practicable.

Any labor required in the handling of packages to be sampled will be performed by the officers, or, if assistance is necessary, by persons employed for that work by the officers in charge of the investigation. The business of merchants or of storage houses should be interfered with as little as possible by officers while in discharge of these duties. In return, it is assumed that the owners of butter will interpose no unreasonable obstacle in the way of officers who seek only to carry out their instructions.

Great care must be exercised in the packing, sealing, labeling, and shipping of samples of butter, following existing instructions as contained in circulars now in force.

JOHN G. CAPERS, *Commissioner.*

TREASURY DEPARTMENT,
January 9, 1909.

(T. D. 29463). *Drawback on zinc shavings.*—Drawback on zinc shavings manufactured by F. W. Braun, of Los Angeles, Cal., from imported zinc in sheets.

(T. D. 1450). Denatured alcohol produced in and imported from Porto Rico is subject to same tax as alcohol not denatured.

TREASURY DEPARTMENT,
OFFICE OF COMMISSIONER OF INTERNAL REVENUE,
Washington D. C., January 11, 1909.

SIR: This office is in receipt of your letter of the 9th instant, relative to the manufacture and exportation of the United States of denatured alcohol produced in Porto Rico.

In reply, you are informed that the law authorizing the withdrawal of distilled spirits from bonded warehouse, free of tax for denaturation in the United States, does not apply to Porto Rico, and that importations of denatured alcohol from Porto Rico would be subject to the same tax as the alcohol not denatured.

Respectfully,
J. C. WHEELER,
Deputy Commissioner.

Mr. ——— ———, *Arecibo, P. R.*

(T. D. 29471). January 14, 1909.—Drawback on Babbitt metal manufactured by the Frictionless Metal Company at its factories at Chattanooga, Tenn., and Richmond, Va., with the use of imported lead and antimony.—T. D. 22963 of April 16, 1901, extended.

(T. D. 29497—G. A. 6857). U. S. General Appraisers, New York, January 26, 1909.

COLOR—OXIDE OF IRON—LEVIGATED IRON ORE.

Oxide of iron, or hematite iron ore, which is in a powdered state resulting from a process of grinding and levigation, and which is used in the manufacture of paint and in coloring leather board, etc., is dutiable under paragraph 58, tariff act of 1897, relating to colors and pigments, rather than under paragraph 121 as iron ore.—Hill *v.* Francklyn (162 Fed. Rep., 880; T. D. 29074) distinguished.

(T. D. 1453). Adulterated butter.—Revenue officers are enjoined to show utmost consideration for the rights of legitimate business in investigating questions of liability under the act of May 9, 1902.

TREASURY DEPARTMENT,
OFFICE OF COMMISSIONER OF INTERNAL REVENUE,
Washington, D. C., January 16, 1909.

To collectors and other internal-revenue officers:

My attention has been called to an article in a recent issue of a trade journal relative to the methods pursued by revenue officers in taking samples of suspected butter, and holding up various lots of butter in stores and warehouses.

The article in question cites various instances of butter being held under detention or suspicion for an unreasonable length of time before being released as normal, or seized as adulterated, and is illustrated with a cut made from a photograph of certain tubes released after examination, showing wholly unnecessary mutilation as a result of the methods used in the taking of samples.

The article is doubtless exaggerated, but contains enough truth, judging from certain instances which have come to my notice, to warrant me in again calling attention of all revenue officers to the necessity of using the utmost care and consideration for the rights of legitimate business in making investigations of this character.

Where the field force have facilities for making such tests preliminary tests of suspected butter should be made, and samples showing 15½ per cent. or more of moisture should be at once forwarded to this office, and all tubs from which the samples were taken showing less than that per cent. should be promptly relieved from suspicion, only the tubs showing 15½ per cent. or more being detained to await the results of the official analysis, which will be forwarded with all possible dispatch.

Should the official chemist's report show that any of the detained tubs of butter contain butter of less than 16 per cent. moisture, such tubs should be promptly released and formal seizure made of the tubs found by tests made in the laboratory here to contain 16 per cent. or more of moisture.

The two principal grievances complained of are the injury to the appearance of the packages through the taking of samples and the delay in determining the classification of the suspected article, and it is believed that a careful compliance with T. D. 1449 will minimize the first, and all officers are enjoined to use the utmost effort to avoid the second.

John G. Capers,
Commissioner.

(T. D. 29499) *Fire brick.*—United States *v.* Behrend.—United States *v.* Wing.—U. S. Circuit Court of Appeals, Second Circuit. January 12, 1909. Nos. 90 and 91 (suits 4304 and 4585).

1. Fire Brick—Retort Settings—Similitude.

Retort settings more than 10 pounds in weight are dutiable by similitude as "fire-brick weighing not more than ten pounds each," under paragraph 87, tariff act of 1897.

2. Same—Mineral Substances—Susceptibility to Decoration.

Fire brick, which can be but never are decorated, are not susceptible of decoration so as to be covered by paragraph 97, tariff act of 1897, which through relating to articles composed of earthy or mineral substances, is limited to such as are susceptible of decoration.

3. Similitude—Points of Resemblance—Identity.

Retort settings more than 10 pounds in weight resemble fire brick weighing not more than 10 pounds, in material, quality, texture, and use, within the meaning of the similitude clause in section 7, tariff act of 1897. Identity ordinarily would exclude all question of similarity, but not here, because of the distinction in weight.

4. Similitude—Tests of Resemblance—Incongruity of Assessment.

The amount of duty is not one of the test prescribed for the application of the similitude clause in section 7, tariff act of 1897. If incongruity results, the inference is not that the tests should be abandoned, but the Congress failed to express its intention.

Noyes, Circuit Judge, dissents.

Appeals from the circuit court of the United States for the southern district of New York.

[Decision adverse to the Government].

For decision below see T. D. 28718, in which the circuit court, on the authority of Wing *v.* United States (119 Fed. Rep., 479), affirmed decisions of the Board of United States General Appraisers, which are reported as G. A. 6382 (T. D. 27422) and Abstract 13038 (T. D. 27649), and which sustained protests of Fred Behrend and Wing & Evans against the assessment of duty by the collector of customs at the port of New York.

(T. D. 29512—G. A. 6859) *Magnesia rings.*

1. Magnesia Rings—Bisque.

So-called magnesia rings, used as part of gas-light burners, held upon the evidence to be bisque, and hence dutiable under the specific provisions of paragraph 96, tariff act of 1897.

2. Cases Reviewed.

Previous decisions of the Board and the courts reviewed, and Crawford *v.* United States (T. D. 28539) distinguished upon the facts.

(T. D. 29516). January 29, 1909.—No. 20562.—Resin Pitch.—Protests 291529, etc., of R. F. Lang (New York).

So-called resin pitch classified as a chemical compound under paragraph 3, tariff act of 1897, was claimed to be dutiable as an unenumerated manufactured article under section 6. Protests sustained.

Chamberlain, *General Appraiser*: * * * Samples of the merchandise were submitted, at the request of the importer, to the United States chemist at the port of New York for analysis; and based upon his evidence, we find the merchandise to be a mixture of oxidized resin and vegetable oils, the product having no well-recognized composition of chemical formula. It is our opinion, therefore, that the merchandise is not a chemical compound, for it has been repeatedly held that a mixture where no chemical union results is not a chemical compound within the meaning of paragraph 3. G. A. 5832 (T. D. 25733); G. A. 6269 (T. D. 27051); G. A. 5718 (T. D. 25410).

(T. D. 29518) *Glycerophoshate of lime.*—Klipstein *v.* United States.—U. S. Circuit Court Appeals, Second Circuit. January 12, 1909. No. 127 (suit 5205).

Glycerophosphate of Lime—Chemical Compound—Medicinal Preparation.

Glycerophosphate of lime, which, though occasionally dispensed medicinally in its imported form, is almost always used in combination with other drugs in the preparation of elixirs, is not a medicinal preparation within the meaning of paragraph 67, tariff act of 1897, but is dutiable as a chemical compound under paragraph 3.

Appeal from the circuit court of the United States for the southern district of New York.

[Decision adverse to the Government.]

The decision below (T. D. 29100) affirmed a decision by the Board of United States General Appraisers, which had affirmed the assessment of duty by the collector of customs at the port of New York on merchandise imported by A. Kilpstein & Co. Abstract 17921 (T. D. 28687).