

DEPARTMENT OF COMMERCE

CIRCULAR
OF THE
BUREAU OF STANDARDS

S. W. STRATTON, DIRECTOR

No. 25

STANDARD SAMPLES—GENERAL INFORMATION

[6th Edition]

(This circular supersedes former editions numbered 1 to 5 under title
"Standard Analyzed Samples—General Information")

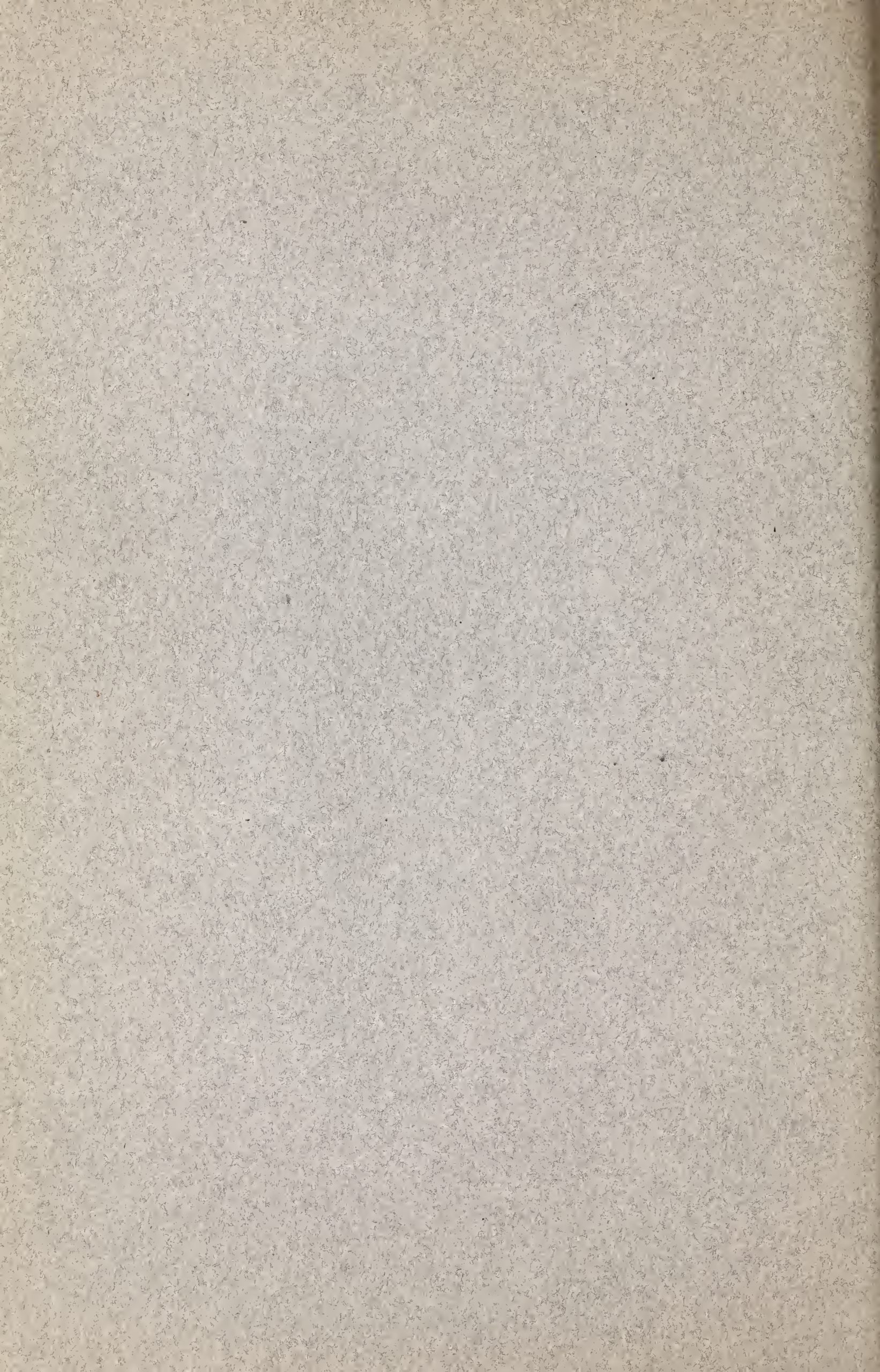
Issued June 20, 1917



PRICE, 5 CENTS

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I. INTRODUCTION

In the past 15 or 20 years analytical chemistry has taken a place of such importance in many manufacturing industries that not only is the analyst called upon to determine the policy of the works in buying raw material and in grading finished products, but often his figures are required at every step of the process. This has necessitated many changes and advances in this branch of the science of chemistry, for on the one hand the great complexity of many industrial products from a chemical point of view has increased the difficulty of securing good determinations, and on the other hand it has been necessary to obtain results much more promptly than heretofore.

Hence has arisen a need for special aids by which the ends named can be reached with the minimum expenditure of time.

Other aids, not involving analytical operations, are needed for the grading of many materials. These needs can be met, often, by the employment of special substances of accurately determined composition, or which without knowledge of their exact composition are suitable for a special purpose. Such substances are termed standards for the particular end they are to serve.

II. FUNCTION OF STANDARD SAMPLES

1. ANALYZED SAMPLES

The variable factors in analytical chemistry are many. The purity of reagents used, the influence of other elements on the determination of a particular element, the influence of temperature, changes of concentration, solubility of precipitates, catalytic phenomena recognized and unrecognized, solubility of glass vessels, etc., these are but a few of the long list with which the chemist has to contend. It is obvious that the technical chemist when called upon to devise new and rapid methods to fit complex materials of a hitherto uninvestigated nature can not generally avail himself of the methods of the research chemist in handling but one or two of these variables at a time. The time at his disposal for investigation is necessarily limited and research must be conducted along lines of greatest efficiency. The result has been the evolution of the standard analyzed sample. This may be defined as a material resembling as closely as possible the chemical and physical nature of the material with which the chemist expects to deal, and which has been analyzed by a sufficient number of methods and analysts to establish its average composition with considerable certainty. The use of such a sample is quite obvious where the chemist has at hand a material which when subjected to the usual operation of analysis will behave like the stock material he expects to analyze. Errors arising from solubility of precipitates, varying concentrations, effects of one element upon the precipitation of another, etc., will affect both sample and standard alike. Hence, if standard and sample are analyzed at the same time (and under practically identical conditions) and results are obtained for the standard corresponding to those obtained by the other analysts, the presumption is strong that the figures obtained for the unknown material are equally accurate, whereas wide divergence from the determinations made by the other chemists on the standard sample shows at once something radically wrong, such as improper method, personal equation, or faulty reagents. Thus, in the simplest possible manner and in the shortest possible

time a large number of variable factors have been simultaneously investigated with almost the same result as though each had been taken up separately.

2. SAMPLES OF GREAT PURITY

For many purposes substances of fixed and definite chemical composition are required. These may be chemical elements or they may be chemical compounds, each of the highest attainable purity. Being pure they have a fixed and definite composition. They find use as standards for volumetric analysis, for calibrating calorimetric bombs, polarimeters, thermometers, pyrometers, and other instruments.

3. OTHER SAMPLES

Closely related to pure metals are metallic alloys of definite composition and free from all other elements than those which it is desired to have present. Still other samples owe their value to the possession of certain definite physical properties. Such, for example, are materials graded according to fineness of grain and intended for sieving and other tests.

III. HISTORY OF THE BUREAU OF STANDARDS' SAMPLES

When the chemical division of the Bureau of Standards was organized it was but natural that it should sooner or later be confronted with this requirement of the industries of the country. In 1905 the American Foundrymen's Association turned over to the Bureau its standardized pig-iron samples, comprising four sets and covering a considerable range of compositions. The distribution of these was carried on after they had been reanalyzed by the Bureau chemists and by some commercial testing laboratories. The Bureau, however, had not participated long as a distributor of these irons before it was realized from the numerous inquiries received that the preparation of new materials for the steel trade was in order, and consequently, aided by valuable suggestions and advice from various steel companies and from A. L. Colby, at that time a member of the visiting committee of the Bureau, and others, the issuing of various kinds of steel samples was begun and is being actively continued at the present time. In 1905 the committees of the American Chemical Society upon uniformity of technical analysis, and upon zinc ore analysis, assigned their samples to the Bureau of Standards for distribution. Along with the demand for steel and iron products there has developed demand for other alloys, for ores, and numerous other products, which is also being met as fast as the facilities of the Bureau permit.

IV. CHOICE OF STANDARD MATERIALS

1. METALS AND ALLOYS

(a) *Standards for Analysis.*—Theoretically, there should be provided for the chemist at a works as many standard samples as he has different kinds of materials to deal with. Thus, in a steel works making steel by the Bessemer, basic open-hearth, and crucible processes, in addition to standards for all the raw materials, there should be as many steel standards as there are processes, and a standard sample for each different composition produced by the same process if there is much range. Sometimes, however, there need not be absolute similarity in all respects between the product and standard; for instance, sometimes a steel sample is ordered on the basis of its carbon content without very much regard to the percentage of phosphorus present. This is a matter that must be determined, however, for each individual case and no general rule can be given. The Bureau in determining the question as to the composition of samples to be issued is guided by the number and kind of requests received and by the advice of those engaged in the industries interested.

Metal samples intended as checks on analysis should be in grains of small and uniform size, such that attack by chemical reagents is comparatively rapid; the size and the heat treatment must be such as to produce this result. It is especially important to have uniformity of size and freedom from dust, for it is only in this way that it is possible to obtain and maintain a perfect mixture. The works in preparing raw materials for analyzed samples must see that they possess extraordinary homogeneity. This is secured by casting, forging, heat treatment, rolling, etc., under expert supervision. In general, the greatest homogeneity is possible with metals whose constituents form solid solutions. Wherever eutectics separate there is danger of segregation, which can be avoided only by the greatest care throughout subsequent operations. In the course of preparing the chips by machining of various kinds the particles of the sample must not be heated by the tool so much as to cause superficial oxidation, nor must they be exposed to the danger of corrosion by moist air after their preparation.

(b) *Samples for Physical Measurements.*—If a pure substance is to be used as a means of defining some physical constant, such as heat of combustion or melting point, the material chosen should be stable and such that it may be obtained in a pure state or readily purifiable by further treatment. The physical constant

defined by use of the material should be of a useful magnitude; for example, of combustion samples one having about the same heat of combustion as the average coal usually would be preferred. It is also desirable that the physical constant should be accurately defined by the material, so that results obtained will not be sensibly affected by slight variations in procedure; and finally, it is desirable that the physical property should be only slightly affected by impurities in the material.

2. ORES AND SIMILAR MATERIALS

(a) *Ores*.—The ideal ore sample is one which is practically a chemically pure compound of the type which is being smelted, usually an oxide or a sulphide. Occasionally ores are found in nature which satisfy this condition. If an ore sample is a blend, the difficulty of uniform mixing is a prominent one, and where it is a question of the most thorough mixing of 200 or 300 pounds of such a blend under conditions of chemical cleanliness the problem is very complex. Another desirable quality in an ore is permanency with regard to its hygroscopic condition, the hardest of all to meet. In the case of ores containing 60 or 70 per cent of the metal which is to be recovered by smelting, differences of a few tenths of a per cent of moisture make very considerable differences in the determination of the metal. A fine state of division is even more important here than in the case of metals, owing to the oftentimes difficult solubility, but this is incompatible in many cases with the desired hygroscopic qualities, for the readiness with which moisture is taken up by powdered materials is a function of their fineness. Hence, not all ores are suitable as analytical standards, but the defect of varying moisture content becomes less the lower the content of the important constituent. It is especially undesirable that the mineral components of an ore should differ markedly in density, for if they do segregation after mixing is inevitable.

(b) *Cement*.—In many industries a control of the fineness of pulverized materials is essential. For this purpose sieves of 100 and 200 meshes to the linear inch are most commonly used, although still finer sieves are obtainable. In the case of very fine sieves, however, it is impossible to obtain wire cloth sufficiently uniform in mesh to insure reliable fineness determinations, and a similar condition holds, in lesser degree, for the 100 and 200 mesh sieves. At the present time the 200-mesh sieve is generally

regarded as the practicable limit of fineness in wire cloth for so-called standard sieves.

The methods of standardization of 200-mesh sieves have been carefully studied, and it has been demonstrated that both microscopic examination of the sieve cloth and actual sieving tests are required to insure the quality and performance of these sieves.

The sieving tests are all-important if the sieve cloth meets specification tolerances covering the permissible variation in size of mesh and diameter of wire. The sieving tests may be made with any suitable finely ground material which has been carefully standardized; for example, pulverized quartz, marble dust, or cement. Investigation has shown that cement of normal manufacture, which has been carefully dried, mixed, and stored in hermetically sealed jars will maintain its original state of subdivision without appreciable change for an indefinite period. Samples of cement so prepared are therefore used by the Bureau in all tests of 200-mesh sieves, and they are also furnished at a nominal price to users of such sieves who desire to make their own standardization tests.

The cement samples are furnished in two degrees of fineness, one a normally ground cement of which approximately 78 per cent passes the 200-mesh sieve, the other a more finely ground cement of which approximately 88 per cent passes the 200-mesh sieve. Both these samples have been carefully standardized on the fundamental standard sieves of the Bureau.

V. PREPARATION OF MATERIALS

1. METALS AND ALLOYS

(a) *Standards for Analysis.*—The raw material for the steel samples is supplied in the form of round bars about 5 inches in diameter, which have been carefully rolled or forged. These are cut into convenient lengths in the shop and, in order to see whether they are sufficiently homogeneous, samples are taken by drilling at intervals across the ends of the bars and analyzed for carbon. If these preliminary tests show the bars to be sufficiently uniform, they are turned into chips of the desired form. The amount of sample prepared in the past has ranged from 200 to 300 pounds. Sometimes it is not possible to produce directly on the lathe exactly the form of chip desired, and it then becomes necessary to grind the material in order to break up clumps of chips. The process of preparing the metals as outlined is somewhat wasteful both of time and material, but so far as steel is concerned it seems the only prac-

licable one at present. Of course, in the preparation of these samples it is necessary to take the utmost precautions to prevent contamination by oil, dust, etc., and the storage of the finished samples requires similar precautions.

Pig-iron samples are not very difficult to prepare. Chips are cut from hollow cylinders 20 inches in diameter, with walls about 2 inches thick, which have been cast according to methods devised by the American Foundrymen's Association with a view to preventing segregation. The greatest difficulty experienced here is the separation of graphite, which can not be kept uniformly mixed with the metal particles. In the samples prepared at the Bureau this difficulty has been overcome by blowing a strong current of air through the material, which is caused to fall in a thin layer at right angles to the air current.

As said before, the iron samples were originally obtained by transfer from the American Foundrymen's Association. As these approached exhaustion, efforts were made to replace them through the original source, but without success. It should be said that this failure was due to no lack of interest on the part of those who had originally prepared the castings. It was some time before the Bureau was able to make arrangements for procuring and mixing materials that would afford a product sufficiently near the originals in composition and for casting this product under such expert supervision as to insure a proper degree of homogeneity.

To insufficient experience on the part of the Bureau and the foundry undertaking the casting may, perhaps, be due the inhomogeneity that manifested itself in one of the iron samples thus prepared. This defect, as shown by analysis, was so great, notwithstanding careful sifting and long mixing, that the whole operation had to be repeated.

A sample of rolled brass has been prepared by clamping together in a vise a number of sheets of the material, averaging about one one-hundredth of an inch in thickness, and cutting chips from these with a milling cutter. This method can not be employed for steel and iron samples, owing to the oxidation of the surfaces of rolled sheets and to the limitation of composition thus imposed.

The preparation of sufficiently homogeneous castings of non-ferrous alloys has encountered difficulties which have not yet been overcome. If entirely homogeneous castings were available, it would not be necessary to mix the fine chips obtained from them. No process having been devised for attaining the requisite homogeneity, subsequent mixing is necessary; but here

again difficulties arise which have not been entirely surmounted. Discoloration, caused chiefly by oxidation, even selective oxidation of a single component, is the main obstacle encountered. Hence, it has not been possible to extend the list of nonferrous alloys as desired. It is hoped, however, that the delay will be but temporary.

(b) *Samples for Physical Measurements.*—Metals and alloys intended for checking physical properties in particular do not, as a rule, need to be reduced to small particles like the analytical standards. Their mode of preparation varies with the nature of the material. Commercial materials, usually of high quality, serve as the starting point and are refined in a variety of ways—such as by electrolytic deposition or distillation, followed in some instances by appropriate casting.¹

2. ORES, CEMENTS, ETC., GRINDING AND MIXING

Ores and similar materials are prepared with comparative ease by grinding in a pebble mill lined with enameled brick and supplied with flint or porcelain pebbles.

With such mills a subsequent sifting of the powder is imperative, for the balls chip (particularly those of flint) and the chips produced in the latter stage of grinding are not reduced to powder. In general, the grinding of ores in contact with iron or steel surfaces is objectionable, sometimes quite inadmissible, for it always results in the incorporation of considerable amounts of metallic particles with the sample, and these are especially objectionable in that they are never uniformly distributed. Unfortunately the sifting above referred to occasions more or less segregation, hence thorough remixing must supervene under conditions that will permit no entrance of foreign matter. This thorough mixing applies to irons and steels as well as to ores and cements. A mixer built of two hollow, internally enameled, cast-iron cones, bolted together at their bases and turning about a hollow axle at right angles to their common axis, has been devised at the Bureau and is used for mixing metal samples. A blast of air may be directed through the axle if desired.

3. STORING, BOTTLING, AND ANALYSIS OF FINE-GRAINED SAMPLES

The mixed samples are transferred to numbered Mason jars (glass) and from these to the small bottles in which they are to be finally distributed.

¹ For more detailed description of the preparation of these samples see Bureau of Standards Circular 66, "Standard Samples for Thermometric Fixed Points."

In order to expedite the process of transferring the finished material to the sample bottles, an apparatus has been devised whereby automatic delivery of any determined weight is made into the bottle through a funnel. The outlet of the latter is closed electrically the moment the required amount has passed. But, before the final bottling, samples for analysis are removed from the jars and sent, one each, to a number of analysts. Each sample sent bears the number of the jar from which it was taken. In general, three types of analysts are chosen—commercial chemists, works chemists, and chemists of the Bureau. When all the analytical results have been received they are inspected and, if not sufficiently concordant, analysts are sometimes requested to repeat the determinations without knowing in which direction from the average their value lies.

4. PURE CHEMICALS

In the selection of pure chemicals which may serve as primary standards for various purposes, the effort has been made to choose stable substances of the highest purity that can be obtained at a reasonable expense. Where products of the requisite purity can not be obtained from the manufacturers, methods and apparatus for effecting the purification on a semiindustrial scale have been developed at this Bureau. So far as possible, the impurities in the final products are reduced to such an amount as to be outside the present limits of experimental error of the method which it is intended to standardize; that is, the materials may be considered entirely pure for the purposes under consideration. In addition, however, careful tests are made to detect and if possible determine quantitatively any impurities present in order that corrections may be made if necessary. Thus far the following samples have been issued:

(a) *Sucrose*.—Calorimetric and saccharimetric standard: The raw material for the standard sucrose samples is drawn from the purest granulated sugar of commerce. A comparatively dilute solution is filtered and boiled in an improved vacuum apparatus to high concentration at temperatures below 35° C. Minute crystals form under the influence of constant stirring and are separated by a centrifugal machine. The process is repeated until analysis shows a negligible amount of ash and reducing substances. The final crystals are ground in an agate mortar and dried in a vacuum over lime. The substance is only slightly hygroscopic, but should be kept tightly stoppered in a dry compartment, at a

temperature not much above 20° C., in order to prevent a slight spontaneous decomposition. Standard sucrose samples prepared by a modified alcohol-precipitation method may also be obtained.

(b) *Dextrose*.—Standard reducing sugar: High-grade glucose of commerce, containing about 87 per cent of dextrose, is washed with aqueous alcohol, dissolved in a small quantity of hot water, and treated with alcohol to precipitate the dextrans. The filtered solution is boiled in a vacuum to a concentration of 60 per cent and finally rotated in a shaking machine for 24 hours to permit crystallization. The crystals are centrifuged and washed with alcohol. The entire process is repeated until satisfactory purity is attained. The crystals obtained in this fashion contain one molecule of water of crystallization, but the substance loses this at 60° C., a temperature considerably below the melting point. The remaining traces of moisture are removed by exposing to a temperature of 80 to 100° in a vacuum over freshly ignited lime. The dried substance, in spite of its finely divided condition, is not very hygroscopic, but should be kept in a desiccator. If the sample for analysis is weighed in such manner as to avoid exposure to a damp atmosphere, the moisture content is less than 0.01 per cent.

(c) *Benzoic Acid*.—Issued primarily as a calorimetric standard, but may be also employed in acidimetry: Benzoic acid, as purchased in the open market, may contain chlorine compounds if made synthetically, or gums and related organic acids if made from natural sources. The standard samples are prepared from the purest synthetic benzoic acid obtainable, by two crystallizations from ethyl alcohol, followed by one crystallization from water and fractional sublimation in a vacuum. The progress of purification is followed by titration with standard alkali, and the final product is tested for chlorine and sulphur, and the melting point and acid and calorimetric values are determined. Benzoic acid is neither appreciably volatile nor hygroscopic,² and when prepared as above and kept in stoppered bottles can be used for all but the most accurate work without preliminary drying.

For details regarding the use of combustion samples, consult Bureau of Standards Circular No. 11.

(d) *Sodium Oxalate*.—Issued primarily as an oxidimetric standard, but may be used for acidimetry: This material was prepared, upon rigid specifications, by the Mallinckrodt Chemical Works,

² It has been found, however, to take up moisture from the air very slowly. Old samples may contain as much as 0.1 per cent of water, which can be easily removed by careful fusion. See "Note on the use of benzoic acid as a standard material," by E. R. Weaver, in *J. Am. Chem. Soc.*, 35, p. 1309; 1913.

St. Louis, Mo., and was carefully tested at this Bureau. For details regarding the use of sodium oxalate, consult Bureau of Standards Circular No. 40 on "Sodium oxalate as a standard in volumetric analysis."

(e) *Naphthalene*.—Calorimetric standard: The most common impurities in commercial naphthalene are sulphur compounds, which are eliminated with difficulty. The standard naphthalene samples are prepared from material as free from sulphur as possible by two crystallizations from benzol, followed by one crystallization from alcohol and sublimation in a vacuum. The purification is followed by determination of the sulphur content (which is below 0.05 per cent in the final product), the melting point and the calorific value. Naphthalene is not hygroscopic but is appreciably volatile, and care must be taken in accurate work to prevent error from the latter source.

VI. USES OF THE STANDARD SAMPLES

The principal uses of the Bureau's analyzed samples appear to be as follows:

First. In the settling and avoiding of disputes between buyers and sellers. Such disputes very frequently arise through the selection of inappropriate or faulty methods of analysis by one of the chemists, and the analysis of a standard material usually brings these to light before the case comes to court.

Second. In standardizing volumetric solutions; for example, the determination of sulphur and manganese in iron and steel; iron ores are used for establishing the iron standard for permanganate or bichromate solutions, from which the standards for most other titrations are calculated.

Third. As material for trying new methods. The use in this connection is obvious.

Fourth. As practice material for new and inexperienced chemists. They have quite an extended use in universities in this connection.

Fifth. The combustion samples for standardizing calorimeters and the sugars for the same purpose as well as for polarimeters.

Sixth. The pure metals for melting point and other determinations of physical properties.

Seventh. The cements for checking the sieving value of standard sieves.

VII. GENERAL CONSIDERATIONS

In conclusion, it may be said that the Bureau's work in providing analyzed and other samples has attracted considerable attention and interest, partly because of its intrinsic merit and partly because the Bureau has sought, through brief mention in the leading chemical and engineering journals of the country, to secure the attention and interest of those who need this kind of service. The result has been the hearty and oftentimes unsolicited cooperation of some of the leading technical societies of the country. Among these are the American Foundrymen's Association, the American Brass Founders' Association, the American Steel Manufacturers' Association, the United States Steel Corporation, the American Chemical Society, the Institute of Metals, the Portland Cement Association, the American Society of Civil Engineers, and the American Society for Testing Materials. The latter society has committees on which the Bureau is represented. It will be one of the functions of these committees, which include in their personnel members of the technical staffs of some of the leading manufacturers of the country, to suggest some of the future lines of activity of the Bureau in the work it has begun on standard samples and to aid it in selecting and securing the necessary raw materials.

VIII. STANDARD SAMPLES ISSUED OR IN PREPARATION

The samples are numbered consecutively nearly in the order of issuance of the first representative of each kind. Renewals of an analyzed sample are indicated by the original number, with an added letter to denote its intended relation. Thus, 4a is the first and 5b the second renewal of irons B and C, respectively. In this way a given number will always represent a material of fixed, or approximately fixed, composition. Numbers missing from the series in the table in the "Supplement to Bureau of Standards Circular No. 25" represent samples of which the supply has become exhausted and which it is not the present intention to replace.

The sample numbers and their analyses in the "Supplement" are subject to correction from time to time to take account of the somewhat different composition of such "Renewal" samples as differ slightly from the original.

IX. ORDERING AND SHIPPING REGULATIONS

(a) *Ordering.*—Orders should give both the number and name of the sample wanted. Example: No. 9a Steel, Bessemer 0.2 C. The list of standard samples, their numbers, prices, and analyses are to be found in the "Supplement to Bureau of Standards Circular No. 25."

(b) *Terms.*—Prepayment in full is required by the regulations of this Department before samples can be shipped. Payment may be by check, draft, postal or express money order, and should be payable to the "Secretary of Commerce."

(c) *Discount.*—Ten per cent discount is allowed on the list price when four or more samples (alike or different) are ordered at one time. No samples of smaller size than those listed are distributed.

(d) *Shipping.*—Samples, if not numerous, are sent by mail under Government frank without expense to the purchaser, unless other instructions are given. If sent by express, it is at the expense of the purchaser.

Iron, steel, and ore samples are sent in screw-capped glass bottles, and organic samples in glass-stoppered bottles, under seal.

Detailed certificates of analysis, and also gummed labels with the summary of analysis, together with the corresponding descriptive circulars, are sent with certain samples. In the case of new or renewal samples, provisional typewritten certificates will be supplied until they can be replaced by the printed certificates and labels when ready.

(e) *Samples Out of Stock.*—The preparation of "Renewal" samples is intended to be complete at the time each kind of sample becomes exhausted, but owing to delays encountered in obtaining a proper grade of material and for other reasons this is not always possible. In case of orders for samples thus out of stock, but soon to be replaced, the fee covering the entire order will be charged unless other instructions are sent, and the "Renewal" sample will be sent when ready, without further notice. Should a long delay be necessary, refund will be made, and notice mailed when the "Renewal" is ready. The "Renewal" of an analyzed sample will have a composition more or less different from that of its predecessor, but as regards the characteristic constituent or constituents will pattern after it closely.

(f) *New Samples.*—When new samples are issued, announcement will be made in the following journals: Iron Age, Journal of

Industrial and Engineering Chemistry, and Metallurgical and Chemical Engineer.

(g) *Mixing*.—In order to overcome the effect of any segregation of granular samples in shipment the contents of each bottle (except the organic samples) *should be thoroughly mixed before any is used for analysis*.

S. W. STRATTON,
Director.

Approved:

WILLIAM C. REDFIELD,
Secretary.

