Stratton, S. W.

DEPARTMENT OF COMMERCE AND LABOR

CIRCULAR

OF THE

BUREAU OF STANDARDS

S. W. STRATTON, DIRECTOR

No. 25

STANDARD ANALYZED SAMPLES—GENERAL INFORMATION

[4th Edition]
Issued November 1, 1912

WASHINGTON
GOVERNMENT PRINTING OFFICE
1912

DEPARTMENT OF COMMERCE AND LABOR

CIRCULAR

OF THE

BUREAU OF STANDARDS

S. W. STRATTON, DIRECTOR

No. 25

STANDARD ANALYZED SAMPLES— GENERAL INFORMATION

[4th Edition]
Issued November 1, 1912

WASHINGTON
GOVERNMENT PRINTING OFFICE
1912

STANDARD ANALYZED SAMPLES—GENERAL INFORMATION

CONTENTS

I. INTRODUCTION

In the past fifteen or twenty 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 science, 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.

II. FUNCTION OF STANDARD 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 univestigated 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 or 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.

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 recognized 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. THE CHOICE OF STANDARD MATERIALS

1. METALS

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 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 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 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 only be avoided 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.

2. ORES AND SIMILAR MATERIALS

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. Frequently 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 chemically pure 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 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.

V. PREPARATION OF MATERIALS

1. METALS

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 if they are sufficiently homogeneous, cuts are taken on the lathe to different depths 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 practicable 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.

One of the brass samples has been prepared by clamping together in a vise a number of sheets of the material, of average thickness about one one-hundredth of an inch, 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.

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.

2. DIFFICULTIES ENCOUNTERED IN RENEWAL OF METAL SAMPLES

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 long 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.

3. ORES, 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 later 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. 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.

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.

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, work 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 the direction from the mean in which 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 reasonable expense. Where products of the requisite purity can not be obtained from the manufacturers, methods and apparatus for effecting the purification on a semicommercial 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; i. e., 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:

Sugar.—Calorimetric and saccharimetric standard. The raw material for the standard sugar 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, which are separated by a powerful centrifugal machine. This 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 sugar samples prepared by a modified alcohol-precipitation method may also be obtained.

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 vacuo. 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 as determined by combustion. Naphthalene is not hygroscopic but is appreciably volatile, and care must be taken in accurate work to prevent error from the latter source.

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 acid chlorides 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 vacuo. 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, 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.

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, 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."

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 out 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 sugar for the same purpose as well as for polariscopes.

VII. GENERAL CONSIDERATIONS

In conclusion it may be said that the Bureau's work in providing analyzed 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 testing 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, and the American Society for Testing Materials. The latter society has recently organized two new committees—a committee on nonferrous alloys and a committee on alloy steels—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 analyzed samples and to aid it in selecting and securing the necessary raw materials.

The table below gives information regarding the samples at present being distributed by the Bureau and those in course of preparation. The samples are numbered consecutively nearly in the order of issuance of the first representative of each kind. Renewals of a sample are indicated by the original number, with an added letter to denote its intended relation. Thus, 5a is the first renewal by the Bureau of Standards of the original Iron C. In this way a given number will always represent a material of fixed, or approximately fixed, composition. Numbers missing from the series represent samples of which the supply has become exhausted, and which it is not the present intention to replace.

VIII. STANDARD ANALYZED SAMPLES ISSUED OR IN PREPARATION

SCHEDULE OF FEES (See page 15 for discounts)

Sample number	Name	Constituents determined	Weight of sample in grams	Fee per sample with certificate[1]
1	Argillaceous limestone	Complete analysis	100	$1.00
2	Zinc Ore D	Zinc	100	1.00
4a	Iron B, Renewal	C, Si, Ti, P, S, Mn	150	2.00
5a	Iron C, Renewal	C, Si, Ti, P, S, Mn, Cu	150	2.00
6a	Iron D, Renewal	C, Si, Ti, P, S, Mn, Cu	150	2.00
8a	Steel, Bessemer, 0.1 C Renewal	C, Si, P, S, Mn	150	2.00
9a	Steel, " 0.2 C Renewal	C, Si, P, S, Mn	150	2.00
10a	Steel, " 0.4 C Renewal	C, Si, P, S, Mn	150	2.00
11a	Steel, B. O. H., 0.2 C Renewal	C, Si, P, S, Mn	150	2.00
12a	Steel, B. O. H., 0.4 C Renewal	C, Si, P, S, Mn	150	2.00
13a	Steel, B. O. H,. 0.6 C Renewal	C, Si, P, S, Mn	150	2.00
14a	Steel, B. O. H., 0.8 C Renewal	C, Si, P, S, Mn	150	2.00
15a	Steel, B. O. H., 0.1 C Renewal	C, Si, P, S, Mn	150	2.00
16	Steel, B. O. H., 1.0 C	C, Si, P, S, Mn	150	2.00
17	Sugar	Calorimetric and saccharimetric value	60	2.00
18	Steel, A. O. H., 0.1 C	C, Si, P, S, Mn	150	2.00
19a	Steel, A. O. H., 0.2 C Renewal	C, Si, P, S, Mn (Cu, Cr, Mo, V)	150	2.00
20	Steel, A. O. H., 0.4 C	C, Si, P, S, Mn	150	2.00
21	Steel, A. O. H., 0.6 C	C, Si, P, S, Mn	150	2.00
22	Steel, Bessemer, 0.6 C	C, Si, P, S, Mn	150	2.00
23	Steel, Bessemer, 0.8 C	C, Si, P, S, Mn	150	2.00
24	Steel, Vanadium, 0.15 V	C, Si, P, S, Mn, V (Ni, Cr, Cu, Mo)	150	2.50
25	Manganese Ore	Mn, Available O	100	1.50
26	Crescent Iron Ore	Al_2O_3, CaO, MgO	100	1.50
27	Sibley Iron Ore	SiO_2, P, Fe	150	2.00
28	Norrie Iron Ore	Mn (low)	100	1.50
29	Magnetite Iron Ore (titaniferous)	Full analysis	150	2.00
30	Steel, Chrome-vanadium	C, Si, P, S, Mn, Cr, V (Ni, Cu, Mo)	150	2.50
31	Steel, Chrome-tungsten	C, Si, P, S, Mn, Cr, W (Ni, Cu, Mo, W)	150	2.50
32	Steel, Chrome-nickel	C, Si, P, S, Mn, Cr, Ni (Co, Cu, Mo)	150	2.50
33	Steel, Nickel	C, Si, P, S, Mn, Ni (Co, Cr, Cu, W, Mo)	150	2.50
34	Steel, A. O. H., 0.8 C	In preparation		
35	Steel, A. O. H., 1.0 C	In preparation		
36	Brass, Red Casting	In preparation		
37	Brass, Rolling Mill	In preparation		
38	Naphthalene	Calorimetric value	50	2.00
39	Benzoic acid	Calorimetric value	50	2.00
40	Sodium oxalate	Oxidimetric value	120	2.00
			200	3.00

[1] For date of effect, see first paragraph under section X.

IX. SUMMARY OF ANALYSES

In general the values here given represent the averages of all determinations. In certain cases, for reasons explained on the certificates, other values are given in these tables and are recommended by the Bureau of Standards.

AVERAGED ANALYSES

Irons

Number	Sample	Total carbon	Graphite	Combined carbon	Silicon	Titanium	Phosphorus gravimetric
4a	B			0.67	1.37	0.062	0.102
5a	C	2.77	2.22	0.55	1.84	0.074	0.189
6a	D	2.46	1.85	0.61	2.57	0.081	0.526

Number	Sample	Phosphorus permanganate titration	Phosphorus alkali titration	Sulphur by oxidation	Sulphur by evolution	Manganese	Copper
4a	B	0.105	0.104	0.039	0.036	1.04	
5a	C	0.196	0.193	0.0354	0.0335	0.744	0.06
6a	D	0.540	0.545	0.044	0.034	1.54	0.043

Steels

Number	Kind of sample with approximate carbon content	Carbon			Silicon	Phosphorus	Sulphur		Manganese
		Direct combustion	Solution and combustion	Colorimetric			By oxidation	Evolved as hydrogen-sulphide	
	BESSEMER								
8a	0.1	0.084	0.083	0.088	0.005	0.094	0.082	0.082	0.336
9a	0.2	0.253	0.257	0.250	0.027	0.112	0.063	0.062	0.918
10a	0.4	0.448	0.459	0.450	0.087	0.102	0.069	0.066	0.916
22	0.6	0.591	0.587	0.605	0.077	0.102	0.066	0.063	0.708
23	0.8	0.805	0.806	0.803	0.150	0.109	0.061	0.057	0.775
	BASIC OPEN-HEARTH								
15a	0.1	0.109	0.115	0.107	0.009	0.0066	0.028	0.030	0.372
11a	0.2	0.223	0.227	0.227	0.097	0.008	0.041	0.039	0.620
12a	0.4	0.363	0.366	0.360	0.012	0.012	0.035	0.036	0.419
13a	0.6	0.579	0.581	0.585	0.116	0.014	0.032	0.032	0.635
14a	0.8	0.813	0.814	0.83	0.10	0.025	0.039	0.038	0.320
16	1.0	1.049	1.054	1.100	0.153	0.045	0.027	0.028	0.405

Steels—Continued

Number	Kind of sample with approximate carbon content	Carbon			Silicon	Phosphorus	Sulphur		Manganese
		Direct combustion	Solution and combustion	Colorimetric			By oxidation	Evolved as hydrogen-sulphide	
	ACID OPEN-HEARTH								
18	0.1	0.101	0.104	0.097	0.016	0.056	0.057	0.057	0.412
19a	0.2	0.205	0.214	0.20	0.032	0.084	0.071	0.068	0.85
20	0.4	0.378	0.376	0.390	0.059	0.031	0.044	0.044	0.486
21	0.6	0.591	0.592	0.590	0.081	0.025	0.050	0.049	0.559
33	0.8								
34	1.0								

Alloy Steels

Number	Kind	Carbon [2]		Silicon	Phosphorus	Sulphur	Manganese	Vanadium	Chromium	Nickel	Tungsten
		Direct combustion	Solution and combustion								
24	Vanadium	0.350	0.348	0.303	0.035	0.027	0.669	0.15	0.007	0.009	
30	Chrome-vanadium	0.372	0.374	0.114	0.043	0.029	0.563	0.21	1.35	0.117	
31	Chrome-tungsten	0.599		0.11	0.013	0.019	0.154	Trace	3.50	Trace	19.55
32	Chrome-nickel	0.375	0.370	0.096	0.018	0.025	0.214		0.89	1.62	
33	Nickel	0.278	0.280	0.104	0.026	0.038	0.551		0.12	3.33	0.15

Argillaceous Limestone

[Cf. Jour. Am. Chem. Soc. 28, 223; 1906.]

Number	SiO_2	TiO_2	Al_2O_3	P_2O_5	Fe_2O_3	MnO	CaO	MgO	K_2O
1	18.15	0.22	5.70	0.18	1.72	0.04	37.65	1.94	1.15

Number	Na_2O	H_2O 100°—	H_2O 100°+	S	SO_3	CO_2	C	Total	Ign. loss
1	0.33	0.16	1.51	0.27	0.013	30.68	0.65	100.25	32.27

[2] It is believed that these results for carbon may be slightly lower than the truth. The matter is being investigated at the Bureau and elsewhere.

Zinc Ore

[Cf. Jour. Am. Chem. Soc. 29, 262; 1907.]

Number	Zinc—General average	
2	31.43	

Manganese Ore

Number	Total manganese	Available oxygen	Calculated MnO_2
25	56.4	16.1	87.5

Lake Superior Iron Ores

Number	Name	SiO_2	TiO_2	P	Al_2O_3	Fe	Mn	CaO	MgO
26	Crescent	[3] 5.03	[3] 0.07	[3] 0.040	1.02	[3] 58.62		2.56	3.27
27	Sibley	0.76		0.037		69.20			
28	Norrie						0.465		

Magnetite Iron Ore

Number	SiO_2	TiO_2	Al_2O_3	V_2O_3	FeO	Fe_2O_3	Fe	MnO
29	12.02	0.99	1.91	0.08	24.78	52.20	[55.75]	0.09

Number	CaO	MgO	K_2O	Na_2O	H_2O+	CO_2	P_2O_5	S
29	2.90	2.01	0.51	0.45	0.47	0.68	1.01	0.025

PURE CHEMICALS

Sugar

Number	Sucrose by polarization	Moisture	Reducing substances	Ash
17	99.98	<0.01	0.004	0.003

Naphthalene

Number	S	
38	<0.05	Cf. method of purification, pp. 8–9

[3] Values derived from a small number of determinations at the Bureau of Standards, and not so well established as the other values.

Benzoic Acid

Number	
39	No impurities could be detected Cf. method of purification, p. 9

Sodium Oxalate

Number	Water		$NaHC_2O_4$	S	K	Fe	Cl	Organic impurity
	105°	240°						
40	0.005	0.027	<0.01	Very faint trace	Very faint trace	None	None	None

X. ORDERING AND SHIPPING

The information contained in this circular supersedes that of all previous circulars.

On all orders for four or more samples (alike or different) a reduction of 10 per cent will be made in view of the decreased cost of handling larger orders. Samples 1 and 2 will no longer be supplied gratis. Orders for samples should be accompanied by a remittance, which may be by check, draft, or post-office order, and should be payable to the Bureau of Standards. Samples will be sent by mail in glass bottles with screw cap (organic samples in glass-stoppered bottles), each bottle being in a separate mailing case.

Detailed certificates of analysis, and also gummed labels with the summary of analysis, will be sent with samples ordered.

Descriptive circulars Nos. 14 and 26, with a summary of the methods of analysis, will be sent to those ordering samples other than organic.

This circular will be revised from time to time, and a copy of each new edition will be sent to all who have ordered samples within twelve months.

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.

In order to overcome the effect of any segregation 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:
Benj. S. Cable,
Acting Secretary.