

DEPARTMENT OF COMMERCE

Bureau of Standards

Certificate of Analyses

OF

STANDARD SAMPLE No. 63

PHOSPHOR BRONZE BEARING METAL

ANALYST*	Copper	Tin	Lead	Phosphorus		Anti-mony	Zinc	Iron	Arse-nic	Sul-phur	Alumi-num	Nickel
				Gravi-metric as Mg ₂ P ₂ O ₇	Alkali-metric method							
1.....	78.05	9.90	9.74	0.62	0.65	0.54	0.49	0.28	0.20	0.05	0.04	0.005
2.....	78.06	9.88	9.75	.65	.64	.58	.50	.30	.18	.06	.04
3.....	78.04	9.95	9.76	.64	.61	.53	.48	.25	.20	.06	.05	.01
4.....	78.09	9.85	9.82	.56	.58	.54	.45	.24	.19	.05	.06
.....	78.07	9.94	9.67	.56	.59	.52	.51	.24	.19	.06	.06	.008
6.....	78.06	9.90	9.7364	.57	.51	.28	.21	.06	.05	Trace.
7.....	78.03	9.91	9.78	.6455	.50	.28	.20	.05	.04	.006
8.....	78.04	9.92	9.80	.60	.62	.57	.47	.27	.18	.07	.04
9.....	78.10	9.87	9.70	.6256	.45	.26	.20	.06	.04	.008
10.....	78.01	9.89	9.72	.6155	.45	.32	.18	.06	.04
11.....	77.98	10.02	9.6862	.57	.52	.26	.18	.07	.07	.01
AVERAGES.....	78.05	9.91	9.74	.61	.62	.55	.48	.27	.19	.06	.05	.008
General Averages.....	78.05	9.91	9.74	0.62		.55	.48	.27	.19	.06	.05	.008

Analyst 5 reported 0.12 per cent of oxygen present as superficial oxide.

*LIST OF ANALYSTS

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George K. Burgess

Director.

Washington, D. C.

ANALYSIS OF THE BUREAU OF STANDARDS PHOSPHOR BRONZE BEARING METAL No. 63

The analysis of the Bureau of Standards phosphor bronze bearing metal No. 63 presents no special difficulties in the cases of copper, tin, lead, and phosphorus. The determination of antimony is complicated by the presence of appreciable arsenic, the interference of which must be considered if the permanganate titration method is used. The determinations of zinc, iron, arsenic, and sulphur are no more difficult than usual; good determinations of zinc and iron require recoveries of these elements in the nitric acid insoluble if tin, antimony, arsenic, and phosphorus are separated at the start by nitric acid attack. The correct determination of aluminum in this alloy is somewhat difficult, as shown by an average percentage error of 40 in the first analyses and of 24 in the final analyses; these high errors were partly the result of the use of too small samples. The chief concern in the determination of aluminum is the behavior of phosphorus during the analysis; for example, phosphorus may not be entirely caught with the metastannic acid if nitric acid attack is used, and in this event it will contaminate the ammonia precipitate; if no separation of phosphorus has been made, the ammonia precipitate will probably not be able to hold all of it and a determination of the amount of phosphorus in the precipitate must be made.

The first analyses which were reported by the analysts showed the following average deviations from the final average values given on the certificate: Copper, 0.12 per cent; tin, 0.19 per cent; lead, 0.1 per cent; phosphorus, 0.03 per cent; antimony, 0.1 per cent; zinc, 0.07 per cent; iron, 0.07 per cent; arsenic, 0.04 per cent; sulphur, 0.01 per cent; and aluminum, 0.02 per cent. The final analyses which are given on the certificate are, in several cases, the result of correspondence and reanalyses and show the following average deviations from the final average values: Copper, 0.025 per cent; tin, 0.033 per cent; lead, 0.037 per cent; phosphorus, 0.021 per cent; antimony, 0.015 per cent; zinc, 0.023 per cent; iron, 0.019 per cent; arsenic, 0.009 per cent; sulphur, 0.005 per cent; and aluminum 0.009 per cent. This summary is interesting in that it indicates the limits within which reasonably careful analyses (as indicated by the first analyses) and umpire analyses (as represented by the final analyses) should check.

OUTLINE OF METHODS USED AT BUREAU OF STANDARDS FOR ANALYSIS OF STANDARD SAMPLE No. 63.

Antimony and tin were determined in 2.5 g samples by the method described below. Because of uncertainty as to the completeness of the separation of the antimony and tin in the digestion with nitric acid, additional runs which gave the same results were made after the elements were separated as follows: 2 g samples were dissolved in a mixture of 20 cc of hydrochloric acid (sp. gr. 1.19), and 10 cc of nitric acid (sp. gr. 1.42). When action ceased the solution was thoroughly boiled, diluted to about 300 cc, and cautiously treated with dilute ammonium hydroxide (1 : 1) until the basic salts of copper were dissolved and the solution was deep blue in color. The solution was then boiled, the precipitate allowed to settle and the solution filtered. The precipitate was washed with a little dilute ammonium hydroxide, then with hot water, and treated in the same way as the residue obtained by digestion with nitric acid. In order to make sure of the antimony content, some check runs were made for this element after arsenic had been removed by distillation after reduction in strong hydrochloric acid solution.

Phosphorus was determined in 2 g samples by the gravimetric and volumetric methods described below, and, in addition, by Oettel's method—fusion with potassium cyanide of the precipitate obtained by digestion with nitric acid, extraction with water, and precipitation with magnesia mixture.

Arsenic was determined in 10 g samples by distillation, precipitation as sulphide, conversion to silver arsenate, and titration as in Volhard's method described below.

Sulphur was determined in 5 g samples, obtained by uniting five 1 g samples after preliminary separations consisting of solution and digestion in nitric acid, filtration, electrolytic separation of copper and lead, and repeated evaporation with hydrochloric acid as described below.

Copper, lead, zinc, iron, aluminum, and nickel were all determined by successive determinations in 5 g samples essentially as described below.

Suggested Methods of Analysis.—In ordinary analyses it appears desirable to combine the determinations of copper, lead, zinc, iron, aluminum, and nickel in a 5 g sample, to determine antimony and tin in a 2.5 g sample, and to use separate samples for each of the rest, phosphorus in a 2 g sample, arsenic in a 10 g sample, and sulphur in a solution obtained from five 1 g samples.

Separation of Copper, Lead, Zinc, Iron, Aluminum, and Nickel from Tin, Antimony, Phosphorus, and Arsenic.—Dissolve a 5 g sample in 50 cc of nitric acid (1 : 1), boil, dilute to about 150 cc with boiling water and digest on a steam bath for two hours, or preferably over night. Filter hot on a compact filter and wash with boiling hot water. Reserve the filtrate and washings, which should be clear. Transfer the filter paper and contents to a Kjeldahl flask, add 12 to 15 cc of sulphuric acid (sp. gr. 1.84), and about 5 g of pure sodium sulphate. Heat until all organic matter is decomposed. Transfer the digested solution to an 800 cc beaker, dilute to 400 cc, and add a solution of sodium hydroxide (30 to 50 per cent, and perfectly clear) until the solution is alkaline and the tin has dissolved. Add 20 cc of a 25 per cent solution of sodium sulphide, digest on a steam bath for several hours or until the supernatant liquid is clear. Filter, wash the residue with a dilute 3 to 5 per cent solution of colorless sodium sulphide, and, in very accurate work, dissolve the sulphides and repeat the above separation. Dissolve the final sulphides in nitric acid (1 : 1) and add the solution to the reserved nitric acid filtrate.

Determination of Lead.—Add 10 cc of sulphuric acid (sp. gr. 1.84), evaporate to fumes of sulphuric acid, cool, wash down the sides of the vessel with water, and again evaporate to fumes of sulphuric acid. Dilute to 150 cc, allow to digest for one to two hours, filter through a tared Gooch crucible, and wash the precipitate with a cool dilute solution of sulphuric acid (3 : 97). Reserve the filtrate and washings. Heat the precipitate to constant weight at very dull redness by suspending the crucible in a larger crucible so that an air space separates the two. Calculate the percentage of lead from the weight of $PbSO_4$ obtained.

Determination of Copper and Residual Lead.—Add 2 cc of nitric acid to the reserved filtrate and washings, dilute to about 200 cc, and electrolyze over night with weighed gauze electrodes at 0.5 ampere and 2 volts. Without interrupting the current, withdraw the electrolyte as the electrodes are well washed with water from a wash bottle. Reserve the electrolyte. Rinse the electrodes in alcohol, dry the cathode for a few minutes at 100° C., cool and weigh the deposit of copper. Dry the anode at 180° C., cool, weigh the PbO_2 and test it for impurities. Calculate lead by the factor 0.866 and add the recovery to the lead already found as sulphate.

Determination of Zinc and Residual Copper.—Evaporate the electrolyte and washings to small volume and heat to the appearance of fumes of sulphuric acid. Cool, dilute to 200 cc, and treat with hydrogen sulphide. If a precipitate appears, filter, wash with dilute sulphuric acid (1 per cent by volume) saturated with hydrogen sulphide, ignite, and weigh as CuO . Test for impurities, such as tin and antimony, and make appropriate corrections for any elements found.

Boil to expel hydrogen sulphide, add two drops of methyl orange solution, and neutralize with ammonium hydroxide. Finally, dilute to about 100 cc for every 0.1 g of zinc and make 0.01 *N* with sulphuric acid. Precipitate zinc as sulphide in cool solution, using a rapid current of hydrogen sulphide. Filter, wash with cold water, reserve the filtrate and ignite the wet filter contents very slowly in an oxidizing atmosphere until all carbon is destroyed. Gradually increase the temperature to 850° C. complete decomposition of the zinc sulphate formed. Do not heat above 900° C. Cool and weigh as ZnO.

Determination of Iron and Aluminum.—Boil the filtrate reserved from the determination of zinc until hydrogen sulphide and sulphur are expelled, using a few cc of nitric acid or a few crystals of ammonium persulphate at the end to make sure that all sulphur is gone and iron is oxidized. Add a few drops of methyl red and then dilute ammonium hydroxide (1 : 1) until the color of the solution changes to a distinct yellow. Boil the solution for one to two minutes, filter at once without washing, and reserve the filtrate. Dissolve the precipitate in a small amount of hot dilute hydrochloric acid (1 : 1), reprecipitate with ammonia as before, filter on the same paper, and wash slightly with hot ammonium chloride solution (2 per cent). Add this filtrate to the one already reserved. Ignite the wet paper and precipitate in a weighed platinum crucible in an oxidizing atmosphere, treat with a drop of dilute sulphuric acid (1 : 1), 1 to 5 cc of hydrofluoric acid, evaporate to dryness, again ignite, very slowly at first, and weigh as Fe₂O₃ plus Al₂O₃.

Fuse the small residue with 2 to 3 g of potassium pyrosulphate at as low a temperature as possible and dissolve the melt in 100 cc of water. Warm the solution, make slightly alkaline with ammonia and then only acid enough to hold iron in solution. Add enough of a saturated solution of ammonium bisulphite (made by passing sulphur dioxide into a cold solution of dilute ammonia (1 : 1) until the solution becomes yellow) to reduce the iron, 5 to 20 drops, and then a few drops of methyl orange. Quickly bring to neutrality with ammonia and then add 6 to 7 drops of dilute hydrochloric acid (1 : 1) in excess. Finally add from 1 to 3 cc of phenylhydrazine and stir until the precipitate becomes flaky. The supernatant liquid should be plainly acid to litmus although alkaline to methyl orange, and the precipitate may be colored owing to organic matter. Filter and wash the precipitate with phenylhydrazine wash solution. (Made by adding a saturated solution of sulphurous acid to a few cc of phenylhydrazine until the crystalline sulphite first formed is redissolved and then adding phenylhydrazine, drop by drop, with vigorous agitation, until the odor of sulphur dioxide is no longer perceptible. Five to ten cc of this solution are diluted with 100 cc of hot water and allowed to remain at the boiling point for a few minutes to remove the excess of sulphur dioxide.) In very accurate analyses or where there is reason to suspect that the precipitate is contaminated by iron, dissolve the precipitate in dilute hot hydrochloric acid and repeat the whole operation. Ignite the wet precipitate first at a moderate temperature until carbon is destroyed, then at approximately 1,100° C., cool, and weigh as Al₂O₃. Test the residue for iron and for phosphorus.

For other methods for the determination of aluminum see D. H. Brophy, *Ind. Eng. Chem.*, **16**, 963; 1924; and G. E. F. Lunnell and H. B. Knowles, same journal, **17**, 78; 1925.

Determination of Nickel.—Acidify the combined filtrates from the determinations of iron and aluminum, evaporate to approximately 300 cc, and filter if not absolutely clear. Nearly neutralize with ammonia, warm, add a 3 to 5 fold excess of a 1 per cent alcoholic solution of dimethyl-glyoxime and make slightly ammoniacal. Digest for one hour at the side of the steam bath, filter on a weighed Gooch crucible, wash with hot water, dry at 110 to 120° C. for about two hours, and weigh as the oxime salt which contains 20.31 per cent of nickel.

Separation of Antimony and Tin.—Dissolve a 2.5 g sample in 50 cc of nitric acid (1 : 1) and proceed as described in the separation of copper, etc., until the filter paper and contents have been dissolved and *all* organic matter has been destroyed. Cool, dilute the solution to 40 cc, add 0.5 to 1 g of sodium sulphite to reduce all of the antimony to the trivalent state, and boil until sulphur dioxide has been expelled.

Determination of Antimony.—Add 200 cc of distilled water, 20 cc of hydrochloric acid (sp. gr. 1.19), cool to about 10° C. and titrate with a standard solution of permanganate to a pink tint which persists for 10 seconds. Reserve the solution. The titration should be corrected by a blank determination (usually amounting to about 0.06 to 0.08 cc of a 0.1 *N* permanganate solution) made by treating a filter paper as in the determination. The theoretical antimony titre of the permanganate solution can be used when the amount of antimony is small; when much antimony is present it is better to standardize the solution against pure antimony, for the theoretical titre is usually a little low. Arsenic interferes because it also causes a consumption of permanganate solution. In ordinary analyses it is safer to titrate, calculate the apparent percentage of antimony, and then deduct 1.86 times the percentage of arsenic as separately determined.

Determination of Tin.—Pour the titrated solution into a 500 cc Erlenmeyer flask, add 80 cc of hydrochloric acid (sp. gr. 1.10) and 2 to 3 g of test lead. Insert a three-hole stopper carrying gas inlet and outlet tubes and having the third hole closed by a small stopper. It is advisable to use an air condenser about 15 to 20 cm in length in the gas outlet. Start a slow current of carbon dioxide from a Kipp generator or a cylinder, heat gradually to boiling and boil gently for 30 to 40 minutes. Finally cool in ice to about 10° C., after increasing the current of carbon dioxide to prevent back pressure. This may be detected by placing a bubble tube in the gas outlet. When the solution is cold, take out the stopper in the third hole, add 5 cc of clear starch solution from a pipette, and insert the tip of a burette containing standard iodine solution and titrate to a permanent blue tint. The titration should be corrected by a blank determination (usually amounting to 0.05 to 0.08 cc of 0.1 *N* iodine solution) carried through the steps of the procedure. The iodine solution should be standardized against pure tin by the method described, for the theoretical titre is usually a little low.

Determination of Phosphorus.—Dissolve 2 g of the sample in a 300 cc Erlenmeyer flask in a mixture of 15 cc of nitric acid (sp. gr. 1.42) and 5 cc of hydrochloric acid (sp. gr. 1.19). When solution is complete, add 15 cc of hot water, digest at 80 to 90° C. for 10 minutes, and dilute to 50 cc. Treat the hot solution with 100 cc of molybdate reagent. (Made as follows: Weigh 100 g of pure molybdic anhydride, mix it thoroughly in a beaker with 400 cc of cold distilled water, and add 80 cc of strong ammonium hydroxide (sp. gr. 0.90). When solution is complete, filter and pour the filtered solution slowly and with constant stirring into a mixture of 400 cc of strong nitric acid (sp. gr. 1.42), and 600 cc of water. Allow to settle for 24 hours and filter.) Shake the solution for 10 minutes, and allow the solution to stand for four to six hours or preferably over night when a gravimetric determination is to follow and for at least 30 minutes if the alkalimetric method is to be used.

Gravimetric Method.—Filter on a paper of close texture, keeping as much of the precipitate in the flask as possible, and wash the flask and the precipitate with not more than 50 cc of cold, dilute nitric acid (1 : 99). Set the filtrate and washings aside after thorough mixing and see whether further separation of phosphomolybdate occurs. Dissolve the precipitate on the filter in 20 cc of dilute ammonium hydroxide (1 : 1) to which have been added 2 g of citric acid, catch the solution in the original

flask containing the bulk of the yellow precipitate, and finally wash the filter several times with dilute hydrochloric acid (5 : 95). If the ammoniacal solution of the phosphomolybdate is not clear at this point, heat it to boiling, filter through the same paper, and wash the paper with hot water. In this case, preserve the paper and ignite it in a small platinum crucible with the paper reserved below. Render the ammoniacal solution acid with hydrochloric acid, add 20 cc of magnesia mixture and then ammonium hydroxide (sp. gr. 0.90) slowly until a crystalline precipitate appears, and then finally an excess of 3 to 5 per cent by vol. The volume of the solution at this point should not exceed 100 cc. Allow the solution to stand in a cool place for four to hours or preferably over night.

Filter, keeping as much of the precipitate as possible in the flask, and wash the flask and the precipitate moderately with dilute ammonium hydroxide (1 : 20). Set the filtrate and washings aside after thorough mixing and see whether further precipitation occurs. Dissolve the precipitate on the filter in 25 cc of dilute hydrochloric acid (1 : 1), catching the solution in the original flask containing the bulk of the precipitate. Wash the filter thoroughly with dilute hydrochloric acid (5 : 95). Add the paper to the one reserved above, ignite, fuse any residue with as little carbonate as possible (not over 0.5 g), extract the cooled melt with hot water, filter, and add the water solution to the hydrochloric acid solution. Transfer the solution to a 200 cc beaker. Treat with hydrogen sulphide, digest, filter, and wash with acidulated hydrogen sulphide water. Boil to expel hydrogen sulphide and to reduce the volume to 50 to 75 cc. Add 0.1 to 0.2 g of citric acid, 2 to 3 cc of magnesia mixture, make ammoniacal as above, and allow to stand in a cool place for 4 to 24 hours.

Filter, wash with dilute ammonium hydroxide (1 : 20), and ignite the precipitate carefully and at as low a temperature as possible until the carbon has been destroyed and the residue is white. Finally, ignite at approximately 1,000° C. to constant weight. Dissolve the ignited residue in 5 cc of dilute nitric acid (1 : 1) and 20 cc of water. Filter, wash the residue with hot water, ignite, and weigh. Treat with a few drops of hydrofluoric acid, evaporate to dryness, ignite, and reweigh. Subtract any loss of weight from the original weight of the pyrophosphate, and calculate on the basis of this purified magnesium pyrophosphate.

Alkalimetric Method.—Filter, wash the flask and precipitate with not more than 50 cc of cold dilute nitric acid (1.5 : 98.5) added in 5 cc portions, and then 10 times with 20 cc portions of a 1 per cent solution of potassium nitrate. Return the precipitate and paper to the original flask, add a 1 to 3 cc excess of a 0.1 N solution of sodium hydroxide (free from carbonate) and 25 cc of cool freshly distilled water. Shake until the precipitate has dissolved, dilute to 100 to 200 cc with cool freshly distilled water, add three drops of a 0.2 per cent solution of phenolphthalein, and discharge the pink color with standard acid. Finish the titration by adding standard alkali to the reappearance of the pink color. Subtract the volume of acid from the total volume of alkali used (solutions being equivalent) and calculate the percentage of phosphorus. The sodium hydroxide solution can be standardized by the use of the Bureau of Standards standard sample of benzoic acid No. 39b followed by calculation, using the ratio 23 NaOH : 1 P; until experience is gained in the analysis, it is well to check the phosphorus titre so found by runs on like material of known phosphorus content.

Determination of Arsenic.—Dissolve 10 g of sample in a 200 cc beaker in 30 to 40 cc of nitric acid (sp. gr. 1.42). When decomposition is complete, add 80 cc of sulphuric acid (sp. gr. 1.84), and evaporate until fumes of sulphuric acid appear. Cool, wash down the sides of the beaker, and again heat to fumes of sulphuric acid. (It is better to fume twice very moderately than to fume once very strongly, on account of the tendency to drive off small quantities of arsenic acid.) Carefully add 50 cc of water to the cooled residue and cool to room temperature. Now transfer to a distillation flask by means of 150 to 175 cc of concentrated hydrochloric acid, add 15 to 20 g of cuprous chloride or ferrous sulphate, and distill until about 75 to 100 cc of distillate have passed over. The outlet tube should be provided with a hole on the side about 1 cm from the opening at the end within the distillation flask. This allows for circulation in the outlet tube and prevents small particles of the material in the flask from passing over into the distillate. A vertical condenser should be used, with the end dipping into a beaker containing 150 cc of distilled water immersed in ice water. Remove the flame from the flask and add 15 cc of hypophosphorus acid and 20 cc of concentrated hydrochloric acid and continue the distillation until 15 to 20 cc more have passed over.

After the distillation is completed add 15 cc of strong ammonium hydroxide to the distillate and pass a rapid stream of hydrogen sulphide through the solution, which is still acid. Allow to stand for one hour or longer, filter on a weighed Gooch crucible, wash with alcohol, then with carbon disulphide, then with alcohol, dry at 100 to 105° C., and weigh as As_2S_3 .

The following modification is not subject to interference by moderate amounts of antimony and is more accurate for the determination of small amounts of arsenic.

After the filtration and washing of the sulphide, dissolve the sulphide in 2 to 3 cc of strong ammonium hydroxide or 2 to 3 cc of 10 per cent sodium hydroxide, wash the pad with the smallest possible quantity of water, evaporate nearly to dryness, add 10 cc of concentrated nitric acid and again evaporate nearly to dryness. Take up with 100 cc of water, and add 10 cc of silver nitrate solution (34 g per liter). Add dilute ammonium hydroxide (1 : 1) or 10 per cent sodium hydroxide drop by drop until a precipitate forms or the solution is alkaline to litmus. (If very small quantities of arsenic are present, a precipitate will not always form at this point and litmus must be used as a guide.) Now add dilute nitric acid (1 : 1) drop by drop until the precipitate just dissolves or until the litmus paper just turns red, and add 10 cc of a saturated solution of sodium or ammonium acetate. The arsenic will be completely precipitated as silver arsenate. Heat just to boiling to coagulate the precipitate, cool, filter on an asbestos pad, and wash with cold water. Dissolve the silver arsenate in nitric acid (1 : 1) and titrate the silver in this solution with standard potassium or ammonium sulphocyanate (N/100), using ferric alum as indicator (10 per cent). The potassium sulphocyanate may be standardized against pure silver or pure silver nitrate. The calculation of arsenic is based on $3Ag : 1As$.

Determination of Sulphur.—Dissolve five 1 g samples of the alloy in 250 cc beakers in 25 cc portions of nitric acid (sp. gr. 1.42). When solution is complete, boil until oxides of nitrogen are expelled and dilute to 100 cc with boiling water. Allow to digest for one hour or longer, filter hot and wash moderately with hot water. The filtrates and washings should be perfectly clear. Discard the precipitates. Electrolyze the solution separately with unweighed gauze electrodes and without, of course, any addition of sulphuric acid. Complete deposition of lead is essential; that of copper need not be complete. When electrolysis is finished, lower the electrolyte as the electrodes are washed and evaporate the solution nearly to dryness. Combine the five solutions, add hydrochloric acid, evaporate to dryness, and take up in a few cc of hydrochloric acid (sp. gr. 1.19). Dilute with 50 to 100 cc of hot water, warm for a few minutes to dissolve soluble salts, and filter if any insoluble matter is present. Again evaporate to dryness to complete the conversion of nitrate to chloride. Drench with 1 cc of concentrated hydrochloric acid (sp. gr. 1.19), add 100 cc of hot water, and filter if the solution is not clear. Heat the solution to boiling, add 10 cc of a 5 per cent solution of barium chloride, drop by drop, while stirring, and allow to settle for five hours or longer. Filter on a sand paper and wash with hot water until chlorides are removed. Do not wash immoderately, for barium sulphate is appreciably soluble. Heat in an oxidizing flame until carbon is destroyed and then at dull redness for 10 to 20 minutes. Cool and weigh as $BaSO_4$. Blank runs should be made and the results deducted, and care must be exercised throughout to prevent contamination by sulphur from gas or fumes in the laboratory.