

DEPARTMENT OF COMMERCE

Bureau of Standards

Certificate of Analyses

OF

STANDARD SAMPLE No. 62

MANGANESE BRONZE

ANALYST	Copper	Zinc	Man- gane- se	Iron	Alumi- num	Tin	Lead	Nickel
1.....	59.06	35.06	1.60	1.13	1.13	0.81	0.57	0.63
2.....	59.15	35.08	1.58	1.11	1.15	.83	.59	.66
3.....	59.05	35.07	1.61	1.15	1.12	.81	.56	.64
4.....	59.05	35.06	1.60	1.13	1.12	.82	.59	.59
5.....	59.06	35.08	1.58	1.12	1.15	.79	.57	.63
6.....	59.05	35.07	1.59	1.14	1.14	.82	.59	.64
.....	59.15	35.09	1.53	1.09	1.15	.82	.54	.65
.....	59.06	34.90	1.64	1.16	1.18	.84	.56	.63
.....	59.03	35.05	1.58	1.15	1.07	.85	.55	.65
10.....	59.10	35.08	1.56	1.11	1.13	.79	.58	.66
11.....	59.03	35.06	1.63	1.16	1.13	.82	.55	.65
12.....	59.07	35.12	1.58	1.16	1.11	.78	.52	.62
AVERAGES.....	59.07	35.06	1.59	1.13	1.13	.82	.56	.64

In addition to the above, Analyst 3 reported a trace of silicon and analyst 12 reported 0.003 per cent of phosphorus. Bureau analyses showed 0.02 per cent of silicon and confirmed the presence of phosphorus.

INDEX TO ANALYSTS

- | | |
|---|---|
| 1. J. A. Scherrer, Bureau of Standards. | 7. Pennsylvania Railroad System, Dr. M. E. McDonnell
chief chemist, Altoona, Pa. |
| 2. J. E. Virchow, Chicago, Burlington & Quincy Railroad
Co., Aurora, Ill. | 8. W. J. Brown, National Lead Co., Brooklyn, N. Y. |
| 3. J. Strauss, Naval Gun Factory, Washington, D. C. | 9. W. E. Baulieu, Bridgeport Brass Co., Bridgeport, Conn. |
| 4. S. A. Weigand, The Lunkenheimer Co., Cincinnati, Ohio. | 10. J. H. McKay, Lumen Bearing Co., Buffalo, N. Y. |
| 5. F. M. Barry and L. E. Lougee, Scovill Manufacturing
Co., Waterbury, Conn. | 11. H. A. Bedworth, E. A. Schroeder, and T. E. Lorenz,
The American Brass Co., Waterbury Conn. |
| 6. J. B. Mosley, The Ajax Metal Co., Philadelphia, Pa. | 12. W. F. Muehlberg, Newburg Steel Works, Cleveland, Ohio. |

Washington, D. C.
January, 1924

GEORGE K. BURGESS,
Director

THE ANALYSIS OF THE BUREAU OF STANDARDS

MANGANESE BRONZE SAMPLE No. 62.

The analysis of the Bureau of Standards Manganese Bronze Sample No. 62 is complicated by the high iron content of the sample. This prevents complete separation of tin by digestion with nitric acid, even though the solution is evaporated to dryness. The dissolved tin complicates the succeeding operations because it plates out in part with copper (sometimes also with lead dioxide) and interferes in other determinations made in the electrolyte if no precaution is taken, such as treatment with hydrogen sulphide in moderately acid solution.

The average errors in the first analyses which were received from the analysts were as follows: Copper 0.07 per cent, zinc 0.11 per cent, manganese 0.05 per cent, iron 0.04 per cent, aluminum 0.08 per cent, tin 0.05 per cent, lead 0.03 per cent, and nickel 0.01 per cent. The average percentage errors were: Aluminum 7.1, tin 6.1, lead 5.4, iron 3.5, manganese 3.1, nickel 1.6, zinc 0.31, and copper 0.12. The final analyses which are given in the certificate are the result of correspondence and reanalysis and show the following average errors: 0.03 per cent for copper and zinc, 0.02 per cent for manganese, iron, aluminum, tin, and lead; and 0.01 per cent for nickel. The summary is interesting in that it indicates the limits within which reasonably careful analyses (as indicated by the first analyses) and unimpure analyses (as represented by the final analyses) should check. It is apparent that aluminum and tin were the most difficult determinations; aluminum chiefly because the analysts had not had extended experience in its determination in this kind of material, and tin largely because of the disturbing iron effect. Lead, iron, and manganese presented equal difficulties, while the determination of zinc as usual was more difficult than that of copper. The determination of nickel offered no great trouble and the relatively high percentage error is merely a reflection of the small amount of nickel in the alloy.

OUTLINE OF THE METHODS USED AT THE BUREAU OF STANDARDS FOR THE ANALYSIS OF STANDARD SAMPLE No. 62.

The methods used at the Bureau of Standards were quite long and tedious and are presented more for the purpose of showing how the determinations were made than as models for ordinary analyses.

Tin was determined in a separate 5 g sample by the method described below.

Manganese was determined in a 1 g sample as described below and checked by the Ford-Williams method.

The determinations of copper, lead, aluminum, iron, and nickel were combined and made on 5 g samples. The sample was dissolved in aqua regia, the solution treated with an excess of ammonia and boiled to insure complete precipitation of tin. The solution was filtered and the precipitate washed with dilute ammonia and hot water. The ammoniacal filtrate and washings were acidified with hydrochloric acid and reserved (solution 1). The precipitate was dissolved in hydrochloric acid, the solution diluted properly, treated with hydrogen sulphide, filtered, and the precipitate washed with acidulated hydrogen sulphide water. The filtrate (which contained all of the original iron, the most of the aluminum, and some of the manganese, nickel, and zinc) was reserved (solution 2). The sulphides were dissolved in aqua regia, and two precipitations were made in sodium sulphide solution to completely separate the lead and copper from the tin, which last was discarded. The sulphides were dissolved in nitric acid and the solution was reserved (solution 3).

At this point, therefore, three solutions were on hand, namely: (1) The original acidified filtrate containing the most of the copper, zinc, manganese, and nickel together with small amounts of aluminum and lead; (2) a hydrochloric acid filtrate containing all of the iron, the most of the aluminum, and small amounts of manganese, zinc, and nickel; and (3) a nitric acid solution containing the most of the lead and a little copper.

Solution 1 was next treated with hydrogen sulphide, filtered, and the precipitate washed with dilute sulphuric acid (1 per cent by volume) saturated with hydrogen sulphide. This treatment served to separate copper and lead from zinc, etc., as well as from the large amount of ammonium salts which was present. The filtrate (solution 4) was reserved, while the precipitated sulphides were dissolved in nitric acid and the solution combined with the reserved solution 3. Lead was separated as sulphate from the combined solutions and weighed. Copper and residual lead were next obtained by electrolysis.

The electrolyte was treated with hydrogen sulphide to recover the last traces of copper and lead. The filtrate from this operation was combined with solutions 2 and 4 and the combined solutions were evaporated nearly to dryness. The solution was next treated with an excess of sulphuric acid, evaporated until hydrochloric and nitric acids were expelled, diluted to 200 cc, and neutralized to give an acidity of 0.01 *N*. Zinc was then thrown out as sulphide. In this particular run, the zinc sulphide was discarded. Zinc was determined by repeating the above procedure with a 1 g sample and finally igniting the sulphide to oxide, which was weighed.

Iron and aluminum were next separated by oxidizing the filtrate and precipitating twice with ammonia, methyl red being used as the indicator. The precipitate was ignited and weighed as $\text{Fe}_2\text{O}_3 + \text{Al}_2\text{O}_3$.