

UNITED STATES DEPARTMENT OF COMMERCE  
WASHINGTON

National Bureau of Standards  
Certificate of Analyses  
Standard Sample 124c  
Ounce Metal

ANALYST	COPPER Electrolytic	TIN SnCl <sub>2</sub> -KIO <sub>3</sub>	ZINC ZnS-ZnO	LEAD Weighed as PbO <sub>2</sub>	NICKEL Weighed as nickel dimethylglyoxime	ANTIMONY	IRON	SULFUR	PHOSPHORUS Colorimetric	SILICON Perchloric acid dehydration
1.....	<sup>a</sup> 84.22	<sup>b</sup> 5.13	4.93	<sup>o</sup> 4.71	0.60	<sup>d</sup> 0.20	<sup>o</sup> 0.105	<sup>t</sup> 0.046	<sup>u</sup> 0.024	<sup>b</sup> 0.002
2.....	<sup>h</sup> 84.23	<sup>x</sup> 5.12	4.91	4.73	{ <sup>i</sup> 0.60 <sup>p</sup> 0.61	<sup>n</sup> 0.20	<sup>e</sup> 0.110	<sup>f</sup> 0.045	<sup>o</sup> 0.024	-----
3.....	<sup>p</sup> 84.21	<sup>q</sup> 5.16	4.93	4.76	.59	<sup>r</sup> 0.20	<sup>e</sup> 0.108	<sup>s</sup> 0.052	<sup>t</sup> 0.023	-----
4.....	<sup>u</sup> 84.22	<sup>v</sup> 5.12	4.95	<sup>o, w</sup> 4.74	{ <sup>m</sup> 0.59 <sup>n</sup> 0.60	<sup>n, x</sup> 0.21	<sup>y</sup> 0.107	<sup>z</sup> 0.048	<sup>o</sup> 0.024	<sup>u</sup> 0.002
Average...	84.22	5.13	4.93	4.74	0.60	0.20	0.107	0.048	0.024	0.002

Five-gram sample dissolved in 55 ml of HNO<sub>3</sub>(1+1). Metastannic-acid precipitate filtered off, treated with HNO<sub>3</sub>-HClO<sub>4</sub>-HBr, and the residual solution added to the first filtrate. Two drops of 0.1 N HCl added, solution diluted to 325 ml and electrolyzed overnight, using a current density of 0.5 amp/dm<sup>2</sup>. H<sub>2</sub>SO<sub>4</sub> added to the electrolyte, solution evaporated to fumes of H<sub>2</sub>SO<sub>4</sub>, diluted, and residual copper precipitated as CuS and determined by the diethylthiocarbamate-colorimetric method.

<sup>b</sup> Three-gram sample dissolved in HCl-HNO<sub>3</sub>, iron added, and tin precipitated twice with NH<sub>4</sub>OH. Precipitate dissolved in HCl, tin reduced with nickel and titrated with KIO<sub>3</sub> standardized with pure tin. See ASTM method E54-49, Methods for Chemical Analysis of Metals, p. 267 (1950). American Society for Testing Materials, Philadelphia, Pa.

<sup>c</sup> Weighed as PbSO<sub>4</sub>.

<sup>d</sup> Antimony separated by distillation from a 5-g sample, precipitated with H<sub>2</sub>S, and titrated with KMnO<sub>4</sub> as described in J. Research NBS 21, 95 (1938) RP1116. KMnO<sub>4</sub> standardized with NBS standard sodium oxalate 40c.

<sup>e</sup> SnCl<sub>2</sub>-K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> method.

<sup>f</sup> Combustion-iodate method.

<sup>g</sup> Molybdenum-blue photometric method.

<sup>h</sup> Double dehydration with HClO<sub>4</sub> with intervening filtration.

<sup>i</sup> As in footnote (a) except 3-g sample used.

<sup>j</sup> Same value obtained by depositing copper in the presence of tin in an HNO<sub>3</sub>-HF solution of a 3-g sample.

<sup>k</sup> Tin reduced with iron in the presence of added antimony and titrated with KIO<sub>3</sub>.

<sup>l</sup> Nickel precipitated with dimethylglyoxime and titrated with KCN.

<sup>m</sup> Dimethylglyoxime-photometric method.

<sup>n</sup> Metastannic-acid precipitate separated and digested in H<sub>2</sub>SO<sub>4</sub>-HNO<sub>3</sub>-K<sub>2</sub>SO<sub>4</sub>. Antimony reduced with tartaric acid and titrated with KBrO<sub>3</sub>.

<sup>o</sup> Molybdivanadophosphoric-photometric method. See ASTM method E62-50T.

<sup>p</sup> Copper deposited in the presence of tin in an HNO<sub>3</sub>-HF solution.

<sup>q</sup> Tin reduced with lead in the presence of added antimony and titrated with KIO<sub>3</sub>.

<sup>r</sup> Antimony reduced with tartaric acid and titrated with KMnO<sub>4</sub>.

<sup>s</sup> Evolution method; titration with iodine.

<sup>t</sup> Phosphomolybdate-alkalimetric method.

<sup>u</sup> As in footnote (a), except residual copper determined by electrolysis.

<sup>v</sup> Tin reduced with iron and titrated with KIO<sub>3</sub>.

<sup>w</sup> Same value obtained by the PbCrO<sub>4</sub> method.

<sup>x</sup> Same value also obtained by the pyridine-iodide colorimetric method.

<sup>y</sup> Iron reduced with SnCl<sub>2</sub> and titrated with KMnO<sub>4</sub>. Same value obtained by the KCNS-colorimetric method.

<sup>z</sup> HBr evolution method; titration with KIO<sub>3</sub>. See ASTM method E54-50T.

<sup>aa</sup> Molybdisilicic acid-photometric method.

Analyst 2 reported 0.0016 percent aluminum by the aluminon-photometric method, and 0.002 percent arsenic by the KBrO<sub>3</sub> method.

List of Analysts

- |   |   |
|---|---|
| <p>1. Nonferrous Laboratory, National Bureau of Standards, R. K. Bell, in charge. Analysis by E. E. Maczkowske.</p> <p>2. A. B. Shapiro, H. Kramer and Co., Chicago, Ill.</p> | <p>3. Vincent Schwarz, Magnus Metal Division of National Lead Co., Chicago, Ill.</p> <p>4. John P. Brull, North American Smelting Co., Wilmington, Del.</p> |
|---|---|

WASHINGTON, D. C., April 30, 1954.

A. V. ASTIN, *Director*.