National Bureau of Standards

## Certificate of Analysis

## Standard Reference Material 124d Ounce Metal

ANALYST	COPPER Electrolytic	LEAD Weighed as PbO <sub>2</sub>	ZINC ZnS-ZnO	TIN SnCl <sub>2</sub> -K10 <sub>3</sub>	NICKEL Weighed as nickel dimethylglyoxime	IRON Photometric	ANTIMONY	SILVER Weighed as AgCl	PHOSPHORUS Photometric	ARSENIC Photometric	SULFUR
l	83.59a	5.16 <sup>b</sup>	5.07	4.53 <sup>e</sup>	0.99	0.18 <sup>d</sup>	0.16 <sup>e</sup>	0.021	0.022 <sup>f</sup>	0.015 <sup>g</sup>	0.096 <sup>h</sup>
2	83.62	5.15	5.11	4.55 <sup>i</sup>	1.00	.19 <sup>j</sup>	.17 <sup>k</sup>	.018	.025 <sup>l</sup>	.019 <sup>m</sup>	.094 <sup>h</sup>
3	83,64	5.20	5.03	4.48 <sup>n</sup>	0.980	.18 <sup>p</sup>	.19 <sup>q</sup>	.026	.022 <sup>l</sup>	.017	.092 <sup>h</sup>
4	83.63	5.13 <sup>r</sup>	5.07	4.57 <sup>8</sup>	1.00 <sup>t</sup>	.18 <sup>u</sup>	.16 <sup>v</sup>	.020	.025 <sup>l</sup>	${021^{w} \choose .025^{x}}$	.092 <sup>h</sup>
5	83.56	5.22	5.04	${4.57^{y} \atop 4.56^{z}}$	0.99	.19 <sup>z1</sup>	.17"2	.020	.026 <sup>z3</sup>		.092 <sup>h</sup>
6	83.60 <sup>z4</sup>	5.22	5.06	4.64 <sup>25</sup>	1.00°	.18 <sup>p</sup>	.19 <sup>z6</sup>		.023 <sup>l</sup>		.08 <sup>z7</sup>
Average	83,61	5.18	5.06	4.56	0.99	0.18	0.17	0.021	0.024	0.019	0.091

- d Sample dissolved in HNO<sub>3</sub> (1+2). Solution digested, filtered, and the precipitate washed with hot HNO<sub>3</sub> (1+99). Vertastannic-acid precipitate treated with HNO<sub>3</sub>-HClO<sub>4</sub>-HBr and the residual solution combined with the first filtrate. Solution diluted to 275 ml, electrolyzed for 2 hours, two drops of 0.1N HCl added, and electrolysis continued overnight, using a current density of 0.5 amp/dm<sup>2</sup>. Residual copper and lead in the electrolyte precipitated with H<sub>2</sub>S and determined by electrolysis. Correction made for silver in the cathode deposit.
- b Lead deposited electrolytically as PbO<sub>2</sub> and weighed as PbCrO<sub>4</sub>.
- <sup>c</sup> 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 test lead and titrated with KlO<sub>3</sub> standardized with high-purity tin.
- d SnCl<sub>2</sub>-K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> method.
- $^{\rm c}$  Antimony separated by distillation from a 10-g sample, precipitated with  $\rm H_2S$  and titrated with KMnO4 standardized against sodium oxalate.
- f Phosphomolybdenum blue photometric method,
- g Sodium hypophosphite-ammonium molybdate-photometric method.
- h Combustion-iodate titration method.
- <sup>1</sup> Tin reduced with nickel and titrated with KIO<sub>3</sub>.
- 1. 10-phenanthroline photometric method.
- k Metastannic-acid precipitate separated and digested in II<sub>2</sub>SO<sub>4</sub>-HNO<sub>3</sub>-Na<sub>2</sub>SO<sub>4</sub>. Antimony reduced with Na<sub>2</sub>SO<sub>3</sub> and titrated with KBrO<sub>3</sub>.
- <sup>1</sup> Molybdivanadophosphoric acid-photometric method.
- III Distillation-molybdenum blue-photometric method.
- <sup>11</sup> Tin reduced with lead in the presence of added antimony and titrated with iodine.

- O Dimethylglyoxime-photometric method.
- p NH<sub>4</sub>CNS-photometric method.
- <sup>q</sup> Antimony and tin separated from copper by the manganese coprecipitation method and digested in H<sub>2</sub>SO<sub>4</sub>·HNO<sub>3</sub>. Antimony reduced with hydrazine sulfate and titrated with KBrO<sub>3</sub>.
- r Weighed as PbSO<sub>4</sub>.
- 8 Tin reduced with iron and titrated with iodine.
- <sup>t</sup> Nickel precipitated with dimethylglyoxime.
- u Iron reduced with H2S and titrated with Ce(SO4)2.
- V Antimony reduced with H<sub>2</sub>SO<sub>3</sub> and titrated with KBrO<sub>3</sub>.
- WDistillation-As<sub>2</sub>S<sub>3</sub>-gravimetric method.
- X Arsenic separated by distillation and titrated with iodine.
- y Tin reduced with iron and zinc, and titrated with KIO3.
- $^{\rm Z}$  Tin reduced with iron-antimony alloy and titrated with KIO  $_{\rm 3}.$
- Z1 Metastannic-acid precipitate separated from a nitric acid solution and tin volatilized with HBr. The residual solution combined with the first filtrate and iron titrated with Ti<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub>.
- Antimony separated by the manganese coprecipitation method and titrated with KBrO<sub>3</sub>.
- 23 Phosphomolybdate-alkalimetric method.
- Copper deposited in the presence of tin in HNO<sub>3</sub>-HF solution.
- Tin reduced with iron in the presence of added antimony and titrated with KIO<sub>3</sub>.
- Antimony reduced with tartaric acid and titrated with  ${\rm KMnO_4}$ .
- <sup>27</sup> HBr evolution method-titration with KIO<sub>3</sub>.

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