

UNITED STATES DEPARTMENT OF COMMERCE  
WASHINGTON

National Bureau of Standards  
Certificate of Analyses

Standard Sample 53d  
Lead-Base Bearing Metal

Replaced by  
53e Jan. 20, 1970

ANALYST	ANTIMONY	TIN	COPPER	BISMUTH	ARSENIC	NICKEL Colorimetric
1	{ <sup>a</sup> 9.88 <sup>b</sup> 9.92	{ <sup>c</sup> 4.93 <sup>d</sup> 4.94	{ <sup>e</sup> 0.266 <sup>f</sup> 0.268	<sup>g</sup> 0.140	<sup>h</sup> 0.047	0.002 <sub>3</sub>
2	<sup>i</sup> 9.94	<sup>j</sup> 4.92	<sup>k</sup> 0.274	<sup>l</sup> 0.143	<sup>m</sup> 0.050	.002
3	10.00	<sup>j</sup> 5.00	<sup>n</sup> 0.28	<sup>o</sup> 0.12	<sup>p</sup> 0.044	.001
4	<sup>q</sup> 9.87	<sup>d</sup> 4.93	<sup>r</sup> 0.253	<sup>a</sup> 0.143	<sup>t</sup> 0.044	.002 <sub>3</sub>
5	<sup>q</sup> 9.90	<sup>d</sup> 4.98	<sup>n</sup> 0.267	<sup>l</sup> 0.137	<sup>u</sup> 0.044	.002 <sub>4</sub>
6	<sup>q</sup> 9.92	<sup>v</sup> 4.94	{ <sup>w</sup> 0.268 <sup>x</sup> 0.271	<sup>l</sup> 0.121		.002 <sub>2</sub>
7	<sup>q</sup> 9.94	<sup>d</sup> 4.92	<sup>n</sup> 0.267	<sup>l</sup> 0.138	<sup>u</sup> 0.042	.002 <sub>0</sub>
Average	9.92	4.94	0.268	0.135	0.045	0.002 <sub>2</sub>

<sup>a</sup> 2-g sample treated with HNO<sub>3</sub>-HF. Lead separated as PbSO<sub>4</sub>. HF in the filtrate volatilized by heating to fumes of H<sub>2</sub>SO<sub>4</sub>. Antimony reduced with sulfur and titrated with KMnO<sub>4</sub> standardized on Na<sub>2</sub>C<sub>2</sub>O<sub>4</sub>. Correction made for arsenic.

<sup>b</sup> Antimony separated by distillation from a 1-g sample, precipitated with H<sub>2</sub>S, and titrated with KMnO<sub>4</sub>. KMnO<sub>4</sub> standardized on Na<sub>2</sub>C<sub>2</sub>O<sub>4</sub>. See J. Research NBS 21, 95 (1938) RP1116.

<sup>c</sup> Tin separated by distillation from a 4-g sample, precipitated with cupferron, and ignited to SnO<sub>2</sub>. See J. Research NBS 33, 339 (1944) RP1610.

<sup>d</sup> Tin reduced with nickel and titrated with KIO<sub>3</sub>.  
<sup>e</sup> 1-g sample treated with HBr-Br<sub>2</sub>-HClO<sub>4</sub>. Bismuth separated as BiOCl, and lead as PbSO<sub>4</sub>. Copper determined by the cupric bromide-photometric method.

<sup>f</sup> 5-g sample treated with HBr-Br<sub>2</sub>-HClO<sub>4</sub>. Bismuth separated as BiOCl, and lead as PbSO<sub>4</sub>. Copper determined by electrolysis.

<sup>g</sup> Bismuth in BiOCl separation (footnote f) determined photometrically with thiourea. See J. Research NBS 47, 252 (1951) RP2250.

<sup>h</sup> Arsenic precipitated with hypophosphorous acid and determined by the molybdenum blue-photometric method. See Anal. Chem. 20, 902 (1948).

<sup>i</sup> Arsenic removed by distillation and antimony titrated with KMnO<sub>4</sub>.

<sup>j</sup> Tin reduced with nickel and titrated with iodine.

<sup>k</sup> Lead precipitated as PbCl<sub>2</sub>. Copper and antimony precipitated with H<sub>2</sub>S in ammonium oxalate and oxalic acid solution, and antimony separated with K<sub>2</sub>S. Residual lead separated as PbSO<sub>4</sub> and copper determined by titration with cyanide.

<sup>l</sup> Thiourea-photometric method.

<sup>m</sup> Distillation-As<sub>2</sub>S<sub>3</sub> method.

<sup>n</sup> Cupric bromide-photometric method.

<sup>o</sup> Bismuth determined as BiOCl.

<sup>p</sup> Distillation-KIO<sub>3</sub> method.

<sup>q</sup> H<sub>2</sub>SO<sub>4</sub>-H<sub>2</sub>SO<sub>3</sub>-KBrO<sub>3</sub> method. See ASTM method E57-50T.

<sup>r</sup> Copper separated by internal electrolysis and determined by the iodide-thiosulfate method.

<sup>s</sup> Bismuth separated by internal electrolysis and determined photometrically with thiourea.

<sup>t</sup> Arsenic separated by double distillation and titrated with KBrO<sub>3</sub>. See ASTM method E57-50T.

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

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

<sup>w</sup> 5-g sample treated with HBr-Br<sub>2</sub> and lead separated as PbSO<sub>4</sub>. Copper deposited electrolytically, dissolved in HNO<sub>3</sub>, and the solution containing added iron treated with excess NH<sub>4</sub>OH. Copper in the filtrate determined electrolytically.

Analysts 2 and 6 reported 84.6 percent lead by the PbCrO<sub>4</sub> method. Iron is less than 0.001 percent, but is not certified because of uncertainty as to homogeneous distribution of this element.

List of Analysts

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- F. J. Oswiecimski, E. F. Wyanske, and H. W. Brummer, National Lead Co., Brooklyn, N. Y.
- John M. Janik, National Lead Co., Chicago, Ill.
- J. F. Jensen, Bell Telephone Laboratories, Murray Hill, N. J.

- D. C. Spindler and J. Danaczko, Western Electric Co., Chicago, Ill.
- J. P. Brull, North American Smelting Co., Wilmington, Del.
- M. Eisemann and T. W. Sattur, American Smelting and Refining Co., South Plainfield, N. J.

The metal for the preparation of this standard was furnished by the National Lead Co. and atomized by the Metals Disintegrating Co.

ASHINGTON, D. C., Sept. 21, 1956.

A. V. ASTIN, Director.