

U. S. DEPARTMENT OF COMMERCE

National Bureau of Standards

Certificate of Analyses

OF

STANDARD SAMPLE 53B

LEAD-BASE BEARING METAL

ANALYST*	Pb	Sb	Sn	COPPER	BISMUTH Internal electrolysis	ARSENIC Distillation	NICKEL Weighed as nickel dimethyl- glyoxime	SILVER Internal electrolysis	IRON	ALUMINUM Colorimetric
	Weighed as PbSO ₄		SnCl ₂ -Iodine							
1.....	^a 84.35	^b 10.30	^c 5.04	^d 0.206	^e 0.077	^f 0.039	0.005	0.003	^g 0.003	^h 0.0008
2.....	^a 84.48	ⁱ 10.32	^j 5.09	^k .21	^o .075	^l .044	.007	^m .002	ⁿ .001
3.....	84.48	10.19	5.05	.20	.078	.04	.006
4.....	^a 84.34	^{^o10.27 ^p10.28}	^q 5.06	^k .210	^r .077	^s .041	.006	.004	^t <.001	^h <.001
5.....	^a 84.36	^o 10.29	^u 5.04	^v .21	^w .076	^x .044	.008	^y .003	^z <.002
6.....	^{a1} 84.45	ⁱ 10.31	^{a2} 5.07	.22	.071	^l .048	.005	^y .002	^z .002	<.001
		^{^o10.34 ^p10.24}	^j 5.06	^k .209	^o .074	^l .037	.007	^{z4} .0015	^{z5} .0003
Averages.....	84.41	10.28	5.06	0.209	0.075	0.042	0.006	0.003	0.002	0.0007
Recommended values	84.35	10.28	5.06	0.209	0.075	0.042	0.006	0.003	0.002	0.0007

^a Lead precipitated as PbSO₄ from a nitric-sulfuric acid solution containing tartaric acid. Solution filtered through a hard paper of close texture and the precipitate transferred to the original beaker. Ten ml of H₂SO₄ added, solution evaporated to fumes, cooled, and 100 ml of water added. Solution filtered through a weighed fritted-porcelain crucible, and PbSO₄ dried to constant weight at 600° C. Lead recovered from both filtrates (see footnote d).

^b Determined by R. K. Bell. Antimony separated by distillation from a 1-gram sample, precipitated with H₂S, and titrated with KMnO₄ as described in J. Research NBS 21, 95 (1938) RP1116. KMnO₄ standardized on a synthetic alloy—0.85 g Pb, 0.1 g Sb, 0.05 Sn, and 0.01 g Cu—carried through the entire procedure.

^c Tin separated by distillation from a 4-g sample, precipitated with cupferron, and ignited to SnO₂, as described in J. Research NBS 33, 339 (1944) RP1010.

^d Combined filtrates from the PbSO₄ separations (footnote a) treated with KOH-Na₂S. Solution filtered and copper and residual lead determined by electrolysis.

^e Cathode deposit dissolved in dilute HNO₃ (1+1) and bismuth determined as BiOCl. See Ind. Eng. Chem. Anal. Ed. 8, 411 (1936).

^f Arsenic separated by double distillation from a 10-g sample, and titrated with 0.01 N iodine as described in J. Research NBS 21, 95 (1938) RP1116.

^g Metastannic acid separated from a 25-g sample. Lead removed from the filtrate as PbSO₄. Metastannic acid precipitate added to the filtrate from the PbSO₄ separation, and tin, antimony, and arsenic removed by HBr-Br₂ treatment. Iron precipitated as Fe(OH)₃, dissolved, and determined by the SnCl₂-K₂Cr₂O₇ method.

^h Aurin tricarboxylic acid method.

ⁱ Solution in H₂SO₄-KHSO₄. Titrated in cool dilute HCl with KMnO₄.

^j Tin reduced with iron.

^k Copper separated by internal electrolysis. Deposit dissolved and titrated with Na₂S₂O₃.

^l Arsenic determined as As₂S₃.

^m Iron reduced with zinc, and FeSO₄ titrated with KMnO₄.

ⁿ Tin, antimony, and arsenic removed by volatilization with HBr before precipitation of PbSO₄.

^o Solution in H₂SO₄-KHSO₄. Titrated in warm hydrochloric acid solution with KBrO₃, using methyl orange as an indicator.

^p H₂SO₄-H₂S₂O₈-KBrO₃ method.

^q Tin reduced with lead, and SnCl₂ titrated with KIO₃.

^r Deposit dissolved and bismuth determined photometrically by the thiourea method.

^s Arsenic separated by use of H₃PO₄, dissolved, distilled, and AsCl₃ titrated with KBrO₃.

^t Iron determined photometrically by the orthophenanthroline method.

^u Tin reduced with nickel, and SnCl₂ titrated with KIO₃.

^v Copper separated as CuCNS and determined by electrolysis.

^w Sample treated by fire assay method. Bismuth separated as bismuth formate and determined colorimetrically by the potassium iodide method.

^x Distillation-KIO₃ method.

^y Fire assay method.

^z KCNS colorimetric method.

^{z1} Lead separated as PbCl₂ in HCl solution containing ethyl alcohol, and determined as PbCrO₄.

^{z2} Tin reduced with nickel.

^{z3} Arsenic separated by double distillation, and AsCl₃ titrated with KBrO₃.

^{z4} Titrated with Ce(NH₄)₂(SO₄)₄ by the use of orthophenanthroline indicator.

^{z5} Volumetric, 8-hydroxyquinoline method.

*LIST OF ANALYSTS

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

WASHINGTON, October 15, 1944.

LYMAN J. BRIGGS, *Director.*