

U. S. DEPARTMENT OF COMMERCE

# National Bureau of Standards

## Certificate of Analyses

OF

STANDARD SAMPLE 152

### BASIC OPEN-HEARTH STEEL 0.4% CARBON (TIN-BEARING)

ANALYST*	C	Mn		P	S			Si	COPPER RES-CuS-CuO	NICKEL Weighed as nickel dimethylglyoxime	CHROMIUM FeSO <sub>4</sub> -KMnO <sub>4</sub> titration	VANADIUM	TIN	MOLYBDENUM	NITROGEN
	Direct combustion	Bismuthate (FeSO <sub>4</sub> -KMnO <sub>4</sub> )	Persulfate-Arsenite	Gravimetric (weighed as Mg <sub>2</sub> P <sub>2</sub> O <sub>7</sub> after removal of arsenic)	Alkali-Molybdate <sup>a</sup>	Gravimetric (direct oxidation and final precipitation after reduction of iron)	Evolution with HCl (1:1) Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> -Iodine (theoretical sulfur titer) <sup>b</sup>	Combustion							
1	0.464	0.781	0.785	0.019	0.019	0.026	0.026	0.241	0.123	0.062	0.050	0.001	0.036	0.013	0.004
2	.471	<sup>k</sup> .782			<sup>l</sup> .020		<sup>m</sup> .029	<sup>n</sup> 0.028	<sup>o</sup> .250	<sup>p</sup> .120	<sup>q</sup> .066		<sup>r</sup> .036		
3	.468	<sup>k</sup> .788		<sup>s</sup> .019	.019	.028	.028	<sup>o</sup> .248	.13	.059					
4	.466		.782	.019	.019	.026	.026	<sup>o</sup> .241	.136	.064	<sup>t</sup> .045		<sup>r</sup> .039		
5	.462		<sup>u</sup> .773		.019	.028		<sup>v</sup> .25	<sup>w</sup> .127	<sup>x</sup> .065	.054		<sup>y</sup> .037		
6	.470		.783		<sup>l</sup> .022		.027	.026	.248	<sup>p</sup> .130	.059	.050		<sup>z</sup> .038	
7	.459	.781	.780	<sup>s</sup> .020	<sup>c</sup> .018	.026	.027	<sup>z</sup> 0.029	<sup>v</sup> .242	<sup>z</sup> 0.127	<sup>q</sup> .062	.050		<sup>z</sup> 0.038	
8	.468	.782	.783	.019	<sup>c</sup> .019	.028	.027	<sup>a</sup> 0.027	<sup>d</sup> .242	.127	.061	.049		<sup>z</sup> 0.035	
9	.465	<sup>z</sup> .79			.021		<sup>m</sup> .025	<sup>l</sup> 0.026	<sup>z</sup> 0.239	<sup>p</sup> .121	<sup>x</sup> .064	.051		<sup>z</sup> 0.035	
10	.470	.776			<sup>l</sup> .021	.029	<sup>m</sup> .029	<sup>v</sup> .243	<sup>p</sup> .128	.063	.049		<sup>z</sup> 0.035		
Averages	0.466	0.783	0.781	0.019	0.020	0.027	0.027	0.244	0.127	0.062	0.050		0.036		
General averages	0.466	0.782		0.019		0.027		0.244	0.127	0.062	0.050		0.036		

<sup>a</sup> Precipitated at 40° C, washed with a 1-percent solution of KNO<sub>3</sub> and titrated with alkali standardized by the use of acid potassium phthalate and the ratio 23NaOH:1P.

<sup>b</sup> Value obtained by standardizing the titrating solution by means of sodium oxalate through KMnO<sub>4</sub> and Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>, and the use of the ratio 2I:1S.

<sup>c</sup> Molybdenum-blue photometric method. See J. Research NBS 26, 405 (1941) RP1386.

<sup>d</sup> Double dehydration with intervening filtration.  
<sup>e</sup> Persulfate oxidation and potentiometric titration with ferrous ammonium sulfate.

<sup>f</sup> Vanadium separated from the bulk of iron in a 10-g sample by selective precipitation with sodium bicarbonate, then oxidized with nitric acid and titrated potentiometrically with ferrous ammonium sulfate.

<sup>g</sup> Sulfide-iodine method. See BS J. Research 8, 309 (1932) RP415.

<sup>h</sup> 50-g sample dissolved in 1,200 ml of diluted nitric acid (1:4), and acid-sulfides precipitated with H<sub>2</sub>S. Sulfides treated with HNO<sub>3</sub>-H<sub>2</sub>O<sub>2</sub>, tin subsequently distilled with HBr-HCl, precipitated with cupferron and ignited to SnO<sub>2</sub>.

See reference footnote g, and J. Research NBS 33, 307 (1944) RP1610. Determination made by R. K. Bell.

<sup>i</sup> SnCl<sub>2</sub>-KSCN colorimetric method.

<sup>j</sup> Determination made by J. T. Sterling, by the vacuum-fusion method. See BS J. Research 7, 375 (1931) RP346.

<sup>k</sup> Titration with sodium arsenite.

<sup>l</sup> Titrating solution standardized by use of a standard steel.

<sup>m</sup> Absorbed in ammoniacal cadmium chloride solution.

<sup>n</sup> Iodate-titration.

<sup>o</sup> Nitric-sulfuric acid dehydration.

<sup>p</sup> Finished by electrolysis.

<sup>q</sup> Dimethylglyoxime colorimetric method.

<sup>r</sup> Solution of the steel in diluted HNO<sub>3</sub>, tin precipitated as the sulfide, reduced with iron and titrated with KIO<sub>4</sub> standardized with high-purity tin.

<sup>s</sup> Weighed as ammonium phosphomolybdate.

<sup>t</sup> Diphenylcarbazide-colorimetric method.

<sup>u</sup> Perchloric acid method. See Iron Age 142, No. 15, 255 (1938).

<sup>v</sup> Perchloric acid dehydration.

<sup>w</sup> Copper precipitated as cyanate, precipitate ignited and copper titrated with KCN.

<sup>x</sup> Glyoxime-cyanide titration method.

<sup>y</sup> Solution in diluted HNO<sub>3</sub> and tin precipitated with H<sub>2</sub>S. Sulfides ignited to oxides, impurities removed, and tin again precipitated as the sulfide, ignited, and weighed as SnO<sub>2</sub>.

<sup>z</sup> Sulfur gases absorbed in NaOH-H<sub>2</sub>O<sub>2</sub> solution. Titration with H<sub>2</sub>SO<sub>4</sub>.

<sup>aa</sup> H<sub>2</sub>S-*a*-benzoinoxime-CuO method.

<sup>ab</sup> As in (r), but tin reduced with test lead and titrated with iodine standardized with a tin-free steel plus a known amount of tin, treated as in the procedure.

<sup>ac</sup> As in (r) but tin reduced with nickel and titrated with iodine using the theoretical titer.

<sup>ad</sup> Potentiometric titration with mercurous nitrate.

<sup>ae</sup> HCl dehydration.

<sup>af</sup> 10-g sample dissolved in 75 ml of HClO<sub>4</sub> (70%), plus a few crystals of KMnO<sub>4</sub>. Solution diluted and tin precipitated as sulfide, subsequently reduced with aluminum (plus antimony) and titrated with iodate, using the theoretical factor.

#### \*LIST OF ANALYSTS

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The steel for the preparation of this standard was furnished by the Weirton Steel Co.

WASHINGTON, February 27, 1947.

E. U. CONDON, Director.