

# Certificate of Analysis

## Standard Reference Material 1169

### Steel

(Lead-Bearing)

ANALYST	C	Mn	P		S			Si	Cu	Ni	Cr	V	Mo	Pb
	Direct combustion	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 precipitation after reduction of iron)	Combustion Iodate titration	Evolution with HCl (1-1) ZnS-Iodine (theoretical sulfur titer) <sup>b</sup>	Perchloric acid dehydration	Photometric	Weighed as nickel dimethylglyoxime	FeSO <sub>4</sub> -KMnO <sub>4</sub> titration		Photometric	H <sub>2</sub> S-PbMoO <sub>4</sub>
1.....	0.076	<sup>o</sup> 0.996		<sup>d</sup> 0.062		<sup>e</sup> 0.325		<sup>f</sup> 0.012	<sup>g</sup> 0.085	0.030	<sup>h</sup> 0.015	<sup>i</sup> 0.001	0.008	0.231
2.....	.073	{ <sup>j</sup> 0.986 <sup>k</sup> 0.989	0.063	.064	0.314	<sup>l</sup> 0.311		<sup>m</sup> 0.011	<sup>n</sup> 0.088	{ <sup>o</sup> .031 <sup>p</sup> .032	<sup>q</sup> 0.016	<sup>r</sup> 0.001	.009	.230
3.....	.076	<sup>s</sup> 0.995		.066	.316	<sup>t</sup> 0.318		<sup>u</sup> 0.009	<sup>v</sup> 0.084	.032	<sup>w</sup> 0.019	<sup>x</sup> <.01	.011	.225
4.....	.077	<sup>y</sup> 0.991		.066			0.318	<sup>z</sup> 0.012	<sup>aa</sup> 0.080	{ <sup>ab</sup> .032 <sup>ac</sup> .031	<sup>ad</sup> 0.013	<sup>ae</sup> 0.001	.008	.218
5.....	.076	<sup>af</sup> 0.997		<sup>ag</sup> 0.066		.322		<sup>ah</sup> 0.011	<sup>ai</sup> 0.080	<sup>aj</sup> 0.033	<sup>ak</sup> 0.014	<sup>al</sup> 0.002	.006	.231
6.....	.081													
7.....	.078													
Averages.....	0.077	0.992	0.063	0.065	0.315	0.319	0.318	0.011	0.083	0.032	0.015	0.001	0.008	0.227
General averages.....	0.077	0.992	0.064			0.318		0.011	0.083	0.032	0.015	0.001	0.008	0.227

<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 23 NaOH:1P.  
<sup>b</sup> Value obtained by standardizing the titrating solution with sodium oxalate through KMnO<sub>4</sub> and Na<sub>2</sub>S<sub>2</sub>O<sub>8</sub> and the use of the ratio 21:1S.  
<sup>c</sup> Potentiometric titration.  
<sup>d</sup> Molybdenum-blue photometric method. See J. Res. NBS 26, 405 (1941) RP1386.  
<sup>e</sup> 1-g sample burned in oxygen at 1,425 °C and sulfur dioxide absorbed in starch-iodide solution. Iodine liberated from iodide by titration, during the combustion, with

standard KIO<sub>3</sub> solution. Titer based on 93 percent of the theoretical factor.  
<sup>f</sup> Double dehydration with intervening filtration.  
<sup>g</sup> Diethylthiocarbamate photometric method. See J. Res. NBS 47, 380 (1951) RP2265.  
<sup>h</sup> Chromium separated from the bulk of the iron by hydrolytic precipitation with NaHCO<sub>3</sub>, oxidized with persulfate, and titrated potentiometrically with ferrous ammonium sulfate.  
<sup>i</sup> Vanadium separated as in (h), oxidized with HNO<sub>3</sub> and titrated potentiometrically with ferrous ammonium sulfate.

<sup>j</sup> Bismuthate-FeSO<sub>4</sub>-KMnO<sub>4</sub> method.  
<sup>k</sup> Titrating solution standardized with a standard steel.  
<sup>l</sup> Sulfuric acid dehydration.  
<sup>m</sup> Photometric method.  
<sup>n</sup> Diphenylcarbazide photometric method.  
<sup>o</sup> Combustion gases absorbed in excess starch-iodide solution, and back titrated with Na<sub>2</sub>S<sub>2</sub>O<sub>8</sub>.  
<sup>p</sup> H<sub>2</sub>S-electrolytic method.  
<sup>q</sup> Spectrographic method.  
<sup>r</sup> H<sub>2</sub>S-CuS-CuO.  
<sup>s</sup> Perchloric acid oxidation.  
<sup>t</sup> Nitric acid oxidation, potentiometric titration with ferrous ammonium sulfate.

**SAMPLE CONDITION.**—The sample is supplied in the form of a disk 1 1/4 in. in diameter and 3/4 in. thick, obtained from rolled rod.

**HOMOGENEITY.**—Lead was found to be segregated both from center to outside of the cross-section, and from one end to the other longitudinally along the rod. The observed variation was about 0.005 percent lead in either direction. The homogeneity was investigated by metallographic examination, by optical emission and x-ray spectrochemical analysis, and by chemical analysis at the National Bureau of Standards.

**INTENDED USE.**—The sample is for application in opti-

#### List of Analysts

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| <ol style="list-style-type: none"> <li>1. James I. Shultz, E. June Maienthal, and T. W. Freeman, Division of Analytical Chemistry, National Bureau of Standards.</li> <li>2. A. J. Kielar, United States Steel Corporation, Lorain Works, National Tube Division, Lorain, Ohio.</li> <li>3. N. C. Bergstrom, United States Steel Corporation, Duluth Works, American Steel and Wire Division, Duluth, Minn.</li> </ol> | <ol style="list-style-type: none"> <li>4. C. A. Trathowen, Jones and Laughlin Steel Corporation, Pittsburgh Works, Pittsburgh, Pa.</li> <li>5. E. T. Saxer, Jones and Laughlin Steel Corporation, Cleveland Works, Cleveland, Ohio.</li> <li>6. O. W. Baldwin, United States Steel Corporation, Gary Works, Gary, Ind.</li> <li>7. L. M. Melnick, United States Steel Corporation, Applied Research Laboratory, Pittsburgh, Pa.</li> </ol> |
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The steel for the preparation of this standard was furnished by the American Steel and Wire Division, United States Steel Corporation, Cleveland, Ohio in the form of rods 1 1/8 in. in diameter. These rods were lathe cut at NBS to 1/4 in. in diameter.

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J. Paul Cali, Chief  
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cal emission and x-ray spectroscopic analysis. Metallographic examination shows a difference in structure for the transverse section as compared to the longitudinal (rolling direction) section. It is recommended that for both the standard sample and unknown samples the analyses be performed on the transverse cross-section. Because the lead is present as soft particles in a relatively hard steel matrix, care must be taken to insure the surface analyzed represents the metal. In optical emission analysis, the usual surface preparation will suffice although a longer preburn may be necessary. The surface preparation for x-ray analysis is more critical, and may require wet-finishing with a fine-grit paper.