

## U. S. DEPARTMENT OF COMMERCE

# National Bureau of Standards

## Certificate of Analyses

OF  
STANDARD SAMPLE 101A  
18 CHROMIUM—8 NICKEL STEEL

ANALYST*	C	Mn	P		S		Si		Ni	Cr				
	Direct combustion 1,300° to 1,375° C		Gravimetric (Weighed as MgP <sub>2</sub> O <sub>7</sub> after re- moval of arsenic)	Alkali-molybdate <sup>a</sup>	Gravimetric (Direct oxidation and final precipitation in re- duced solution)	Evolution with HCl ZnS-Iodine (theo- retical sulfur titre) <sup>b</sup>	Perechloric acid dehy- dration	COPPER H <sub>2</sub> S-CuS-CuO	Weighed as nickel di- methylglyoxime	FeSO <sub>4</sub> -KMnO <sub>4</sub> titra- tion	VANADIUM	MOLYBDENUM (colorimetric)	COBALT	NITROGEN
1.....	0.047	0.466 <sup>e</sup>	0.017	0.019	0.008	0.010	0.338 <sup>d</sup>	0.050 <sup>e</sup>	8.98	18.33 <sup>f</sup>	0.029 <sup>g</sup>	0.012	0.071 <sup>h</sup>	0.044 <sup>i</sup>
2.....	.046 <sup>j</sup>	.47 <sup>k</sup>		.016		.008	.333 <sup>d</sup>	.05	9.02 <sup>l</sup>	18.33				
3.....	.049	.473 <sup>k</sup>		.016	.008	.009	.344 <sup>d</sup>	.046 <sup>e</sup>	8.95	18.32				.046 <sup>m</sup>
4.....	.051 <sup>j</sup>	.460 <sup>n</sup>		.014	.008	.010	.341 <sup>d</sup>	.053 <sup>e</sup>	8.98 <sup>l</sup>	18.30 <sup>o</sup>				.044
5.....	.047	.460 <sup>e</sup>	.016	.017		.010	.349	.059	9.00	18.30 <sup>f</sup>	.027 <sup>g</sup>	.010	.062 <sup>p</sup>	
6.....	.052	.460 <sup>k</sup>		.016		.009	.331	.050 <sup>q</sup>	8.94	18.35				
7.....	.052 <sup>j</sup>	.45 <sup>r</sup>		.021	.010	.012	.333 <sup>d</sup>	.054	8.98 <sup>l</sup>	18.34				.043 <sup>s</sup>
8.....	.045 <sup>j</sup>	.466 <sup>t</sup>		.018		.009	.330	.054	8.97 <sup>l</sup>	18.31 <sup>o</sup>	.038 <sup>u</sup>	.011	.080 <sup>v</sup>	.045 <sup>w</sup>
9.....	.048 <sup>j</sup>	.466 <sup>x</sup>	.018	.019	.011	.010 <sup>y</sup>	.339 <sup>d</sup>	.049	9.01	18.36	.040 <sup>z</sup>	.008	.065 <sup>v</sup>	
10.....	.048 <sup>j</sup>	.474 <sup>x</sup>	.014	.015	.010	.011	.337	.049 <sup>e</sup>	9.02	18.36	.040 <sup>z1</sup>	.009	.060 <sup>v</sup>	
11.....	.051 <sup>j</sup>	.464 <sup>x</sup>	.017	.018	.008	.009	.344 <sup>d</sup>	.046	8.96	18.32				
12.....	.049 <sup>z2</sup>	.460		.021			.339		9.02 <sup>l</sup>	18.34				
Averages.....	0.049	0.465	0.016	0.018	0.009	0.010	0.338	0.051	8.99	18.33	0.034	0.010	0.068	0.044
Recommended values.....	0.049	0.465	0.017		0.009		0.338	0.051	8.99	18.33	0.030	0.010	0.070	0.044

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

<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>5</sub>.

<sup>c</sup> Bismuthate (FeSO<sub>4</sub>-KMnO<sub>4</sub>) method after ZnO separation.

<sup>d</sup> Double dehydration.

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

<sup>f</sup> Persulfate oxidation, potentiometric titration with FeSO<sub>4</sub> standardized with K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>.

<sup>g</sup> 10-g sample dissolved in 120 ml of diluted HCl(1+2). Sufficient ZnO added to precipitate all the chromium and vanadium. Solution filtered and the precipitate dissolved in diluted HNO<sub>3</sub>. 0.6-g of Na<sub>2</sub>HPO<sub>4</sub>·12H<sub>2</sub>O added, and phosphorus and vanadium precipitated with ammonium molybdate. Solution filtered and the precipitate dissolved in H<sub>2</sub>SO<sub>4</sub>-HNO<sub>3</sub> and evaporated to fumes. Solution diluted, treated with SO<sub>2</sub> to reduce any oxidized chromium; vanadium then oxidized with HNO<sub>3</sub> and titrated potentiometrically with FeSO<sub>4</sub>.

<sup>h</sup> 10-g sample dissolved in diluted HCl(1+1). Bulk of the iron separated by extraction with ether.

Residual iron and chromium in extracted acid-portion separated from cobalt by double precipitation with ZnO. Cobalt precipitated twice with  $\alpha$ -nitroso- $\beta$ -naphthol, ignited, and weighed as Co<sub>2</sub>O<sub>3</sub>.

<sup>i</sup> Determination made by Vernon C. Holm, by the vacuum-fusion method. See BS J. Research 7, 375 (1931) RP346.

<sup>j</sup> Burned with tin at 1,100° to 1,300° C.

<sup>k</sup> Persulfate-arsenite method after ZnO separation.

<sup>l</sup> Titrated with standard KCN solution.

<sup>m</sup> Solution-distillation (Allen) method. Sample dissolved in diluted HCl(1+1).

<sup>n</sup> Bismuthate-arsenite method after ZnO separation.

<sup>o</sup> Perechlorate acid oxidation.

<sup>p</sup> Bulk of iron removed with ether. Chromium separated as PbCrO<sub>4</sub> from perechloric acid solution. Solution treated with cupferron, and cobalt precipitated in filtrate with  $\alpha$ -nitroso- $\beta$ -naphthol and weighed as Co<sub>2</sub>O<sub>3</sub>.

<sup>q</sup> KI-Na<sub>2</sub>S<sub>2</sub>O<sub>5</sub> titration.

<sup>r</sup> Persulfate-arsenite in presence of chromium. See Iron Age 142, No. 26; 16 (1938).

<sup>s</sup> Average value obtained by the solution-distillation method after solution of sample in (1) diluted HCl(1+1), (2) diluted H<sub>2</sub>SO<sub>4</sub>(1+3), and (3) diluted

HCl(1+1), followed by addition of HClO<sub>4</sub> and evaporation to fumes of HClO<sub>4</sub>.

<sup>t</sup> Bismuthate (FeSO<sub>4</sub>-KMnO<sub>4</sub>) method after separation of chromium as PbCrO<sub>4</sub>.

<sup>u</sup> Chromium separated as PbCrO<sub>4</sub>. Vanadium determined by differential titration with FeSO<sub>4</sub>-KMnO<sub>4</sub> using o-phenanthroline indicator. See Sampling and Analysis of Carbon and Alloy Steels, Chemists of the U. S. Steel Corporation, p. 160-161 (1938).

<sup>v</sup> ZnO- $\alpha$ -nitroso- $\beta$ -naphthol method. Precipitate ignited and weighed as Co<sub>2</sub>O<sub>3</sub>.

<sup>w</sup> Solution-distillation method. Sample dissolved in sulfuric acid.

<sup>x</sup> Persulfate-arsenite method after removal of chromium as CrO<sub>2</sub>Cl<sub>2</sub>. (See Ind. Eng. Chem., Anal. Ed. 10, 360 (1938).)

<sup>y</sup> Sample ignited in oxygen, gasses passed into H<sub>2</sub>O<sub>2</sub> and H<sub>2</sub>SO<sub>4</sub> titrated.

<sup>z</sup> Mercury cathode separation. Vanadium reduced with SO<sub>2</sub>, and titrated with KMnO<sub>4</sub> after removal of excess SO<sub>2</sub>. See reference in footnote u, p. 164-165.

<sup>z1</sup> FeSO<sub>4</sub>-KMnO<sub>4</sub> titration after removal of chromium as CrO<sub>2</sub>Cl<sub>2</sub>. See reference in footnote u, p. 167-160.

<sup>z2</sup> Burned with PbO<sub>2</sub> at 1,300° C.

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