

U. S. DEPARTMENT OF COMMERCE

# National Bureau of Standards

## Certificate of Analyses

OF

### STANDARD SAMPLE 123

### 18 CHROMIUM—11 NICKEL STEEL

(Columbium-bearing)

ANALYST*	Cb		Ta	P		S		Si	TITANIUM	VANADIUM
	Direct determination	Weight of mixed oxides calculated to Cb, using the ratio 2CH/Cb <sub>2</sub> O <sub>5</sub>		Gravimetric (weighed as Mg <sub>2</sub> P <sub>2</sub> O <sub>7</sub> after removal of arsenic)	Alkali-molybdate	Gravimetric (direct oxidation and final precipitation in reduced solution)	Evolution with HCl ZnS-iodine (theoretical sulfur titre) <sup>a</sup>	Perchloric acid dehydration		
1	<sup>b</sup> 0. 436		<sup>b</sup> 0. 027	0. 006		0. 004	0. 003	<sup>o</sup> 0. 383	<sup>d</sup> 0. 004	<sup>e</sup> 0. 021
2	<sup>f</sup> 0. 434		<sup>k</sup> 0. 024	. 010		. 008		. 385		
3		<sup>b</sup> 0. 441			0. 007	. 008	. 006	. 39		
4		<sup>i</sup> . 44			. 006	. 006	. 006	. 376		
5	<sup>f</sup> 0. 427		<sup>j</sup> 0. 027		. 006	. 003	. 003	<sup>c</sup> 0. 381	<sup>d</sup> 0. 004	
6	<sup>f</sup> . 44		<sup>k</sup> 0. 037					<sup>c</sup> . 39		
7		<sup>k</sup> 0. 443				. 006	<sup>l</sup> 0. 007	. 382		
8		. 47			. 005		<sup>l</sup> 0. 004	<sup>m</sup> 0. 378		
9	<sup>f</sup> 0. 425	<sup>h</sup> 0. 467		. 007	. 005		<sup>n</sup> 0. 008	. 384		
10		<sup>h</sup> 0. 453			. 007		. 008	. 388		
11		<sup>h</sup> 0. 45			. 006		. 006	. 381		
12	<sup>f</sup> . 44				. 008	. 003	. 005	. 384		
13	<sup>f</sup> 0. 444			. 007	. 005	. 008		. 388		
	<sup>f</sup> . 43				. 007	<sup>o</sup> 0. 009	<sup>l</sup> 0. 009	<sup>c</sup> 0. 390		
16	. 42				. 009		<sup>n</sup> 0. 006	. 378		
		<sup>p</sup> 0. 46			. 009			<sup>c</sup> 0. 383		
<b>Averages</b>	<b>. 433</b>	<b>0. 453</b>	<b>. 029</b>	<b>. 007</b>	<b>. 007</b>	<b>. 006</b>	<b>. 006</b>	<b>. 384</b>	<b>. 004</b>	<b>. 021</b>
<b>Recommended values</b>	<b>0. 433</b>		<b>0. 027</b>	<b>0. 007</b>	<b>. 007</b>	<b>0. 005</b>	<b>0. 384</b>	<b>0. 004</b>	<b>0. 021</b>	

Values for constituents not as accurately determined as the above are: Carbon 0.06; manganese 0.38; nickel 11.55; and chromium 17.67 percent.

<sup>a</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>b</sup> Columbium and tantalum precipitated from a hydrochloric acid solution of a 5-g sample with cupferron. The solution filtered, and the precipitate ignited, and fused with K<sub>2</sub>S<sub>2</sub>O<sub>7</sub>. The cooled melt dissolved in tartaric-sulfuric acid solution, the solution treated with H<sub>2</sub>S and the solution filtered. The filtrate made ammoniacal, treated with H<sub>2</sub>S and filtered. The filtrate boiled, cooled, acidified, and treated with cupferron. The precipitate ignited, the combined oxides from four 5-g samples fused, and tantalum determined by the tannin procedure of Schoeller and Powell. Columbium determined in the tannin filtrates by precipitating with tannin and correcting for impurities. (Cf. The Analyti-

cal Chemistry of Tantalum and Niobium. Chapman and Hall, Ltd., London, 1937.)

<sup>c</sup> Double dehydration.

<sup>d</sup> Colorimetric.

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

<sup>f</sup> Columbium and tantalum precipitated by hydrolysis with H<sub>2</sub>SO<sub>4</sub>. Solution filtered, precipitate ignited, and fused with K<sub>2</sub>S<sub>2</sub>O<sub>7</sub>. Cooled melt dissolved and precipitation by SO<sub>2</sub> hydrolysis repeated. Solution filtered, precipitate ignited, fused with

K<sub>2</sub>S<sub>2</sub>O<sub>7</sub>, and columbium determined by reduction in the Jones reductor. (Cf. T. R. Cunningham, Ind. Eng. Chem. Anal. Ed., 10, 233 (1938).)

<sup>g</sup> By difference, Cb<sub>2</sub>O<sub>5</sub>+Ta<sub>2</sub>O<sub>5</sub> minus Cb<sub>2</sub>O<sub>5</sub> calculated to Ta.

<sup>h</sup> Sulfurous acid hydrolysis from HCl or HClO<sub>4</sub> solution; reported as columbium plus tantalum.

<sup>i</sup> Precipitated with cupferron; reported as columbium plus tantalum.

<sup>j</sup> Tantalum determined in the mixed oxides by the tannin procedure. (See reference in footnote f.)

<sup>k</sup> Nitric-hydrochloric acid dehydration.

<sup>l</sup> Sample ignited in a stream of oxygen, gases passed into H<sub>2</sub>O<sub>2</sub> and H<sub>2</sub>SO<sub>4</sub> titrated or precipitated as BaSO<sub>4</sub>.

<sup>m</sup> Double dehydration with sulfuric acid.

<sup>n</sup> Solution in diluted HCl (1+1).

<sup>o</sup> Meineke's method.

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

<sup>q</sup> If the equivalent of 0.027 tantalum is subtracted (.453-.027) a columbium value of 0.490 is obtained.

#### \* LIST OF ANALYSTS

1. Ferrous laboratory, National Bureau of Standards; analysis by Waino H. Jukkola and John L. Hague.
2. D. P. Bartell, Allegheny Steel Co., Brackenridge, Pa.
3. J. A. Wiley, The Midvale Co., Philadelphia, Pa.
4. H. N. Austin, The Babcock and Wilcox Tube Co., Beaver Falls Pa.
5. T. R. Cunningham and R. M. Fowler, Union Carbide and Carbon Research Laboratories, Niagara Falls, N. Y.
6. C. M. Johnson, The Crucible Steel Co. of America, Park Works, Pittsburgh, Pa.
7. Chemical Laboratory of the Timken Steel and Tube Division, The Timken Roller Bearing Co., Canton, Ohio.
8. F. W. Dillon, The Carpenter Steel Co., Reading, Pa.

9. Armco Research Laboratories, A. H. Thomas, in charge; analysis by O. B. Ellis and S. A. Lapham, Middletown, Ohio.
10. W. C. Maurer, Carnegie-Illinois Steel Corporation, Wood Works, McKeesport, Pa.
11. W. D. Brown, Carnegie-Illinois Steel Corporation, Duquesne Works, Duquesne, Pa.
12. W. F. Muehlberg, American Steel and Wire Co., Cleveland District Laboratory, Cleveland, Ohio.
13. W. M. Davidson, National Tube Co., Ellwood Works, Ellwood City, Pa.
14. L. P. Chase, Carnegie-Illinois Steel Corporation, South Works, Chicago, Ill.
15. G. P. Burks, Carnegie-Illinois Steel Corporation, Gary Works, Gary, Ind.
16. S. S. Heide, Tennessee Coal, Iron and Railroad Co., Ensley, Ala.

Washington, D. C.  
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LYMAN J. BRIGGS,  
Director.