

# National Bureau of Standards

## Certificate of Analyses

OF

STANDARD SAMPLE 117

### FERROTITANIUM

(High Carbon)

ANALYST *	Titanium	Carbon	Silicon	Chromium	Vanadium	Aluminum
1	<sup>a</sup> 14. 58	<sup>b</sup> 5. 42	<sup>c</sup> 2. 55	<sup>d</sup> 0. 26	<sup>e</sup> 0. 07	<sup>f</sup> 0. 92
2	<sup>g</sup> 14. 69	<sup>h</sup> 5. 46				
3	<sup>a</sup> 14. 66	<sup>i</sup> 5. 50	<sup>c</sup> 2. 58	<sup>j</sup> . 23	<sup>k</sup> . 07	
4	<sup>k</sup> 14. 62	<sup>h</sup> 5. 45			<sup>l</sup> . 08	
5	<sup>m</sup> 14. 61	<sup>n</sup> 5. 45	<sup>o</sup> 2. 60	<sup>p</sup> . 27	<sup>q</sup> . 08	<sup>r</sup> . 97
6	<sup>s</sup> 14. 60	<sup>t</sup> 5. 42	<sup>o</sup> 2. 55	<sup>u</sup> . 24	<sup>v</sup> . 05	<sup>v</sup> . 97
7	<sup>w</sup> 14. 54	<sup>h</sup> 5. 50	<sup>x</sup> 2. 57	<sup>y</sup> . 27	<sup>z</sup> . 07	<sup>aa</sup> . 95
8	<sup>w</sup> 14. 69	5. 42				
Averages	14. 62	5. 45	2. 57	0. 25	0. 07	0. 95

Sample treated with sulfuric-nitric-hydrochloric acids. Solution evaporated fumes of sulfuric acid, diluted, and filtered. Ignited residue treated with HF-H<sub>2</sub>SO<sub>4</sub>, fused with K<sub>2</sub>S<sub>2</sub>O<sub>7</sub>, and melt dissolved in the silica filtrate. Iron precipitated in ammoniacal tartrate solution with (NH<sub>4</sub>)<sub>2</sub>S. Solution filtered, precipitate dissolved and iron again precipitated with (NH<sub>4</sub>)<sub>2</sub>S in the presence of tartaric acid. Titanium precipitated in the acidified combined filtrates with cupferron. Ignited oxide corrected for V<sub>2</sub>O<sub>5</sub> and traces of Fe<sub>2</sub>O<sub>3</sub> and ZrO<sub>2</sub>. For detailed discussion, see the text "Titanium," by W. M. Thornton, Jr., The Chemical Catalog Co. (1927).

- <sup>b</sup> 0.6-g sample plus 1-g of ingot iron.
- <sup>c</sup> Double dehydration with sulfuric acid.
- <sup>d</sup> Silver nitrate-persulfate oxidation and potentiometric titration with FeSO<sub>4</sub>.
- <sup>e</sup> Nitric acid oxidation and potentiometric titration.
- <sup>f</sup> Sample decomposed as in (a). Iron removed by electrolysis with a mercury cathode. Titanium and vanadium precipitated with cupferron. Nitric and perchloric acids added to filtrate. Solution evaporated to fumes of perchloric acid, diluted, and aluminum precipitated with ammonia. Solution filtered, and precipitate dissolved. Solution evaporated to fumes of perchloric acid, diluted, filtered, and aluminum precipitated with ammonia. Ignited oxide corrected for Fe<sub>2</sub>O<sub>3</sub> and SiO<sub>2</sub>.
- <sup>g</sup> Titanium separated from most of the iron by the cyanide-ammonia process. Solution filtered, precipitate dissolved, and residual iron separated in ammoniacal-citrate solution with (NH<sub>4</sub>)<sub>2</sub>S. Titanium precipitated in the acidified filtrate with cupferron. Ignited oxides corrected for V<sub>2</sub>O<sub>5</sub> and traces of Fe<sub>2</sub>O<sub>3</sub>.
- <sup>h</sup> Ingot iron used as an accelerator.
- <sup>i</sup> Tin used as an accelerator.
- <sup>j</sup> Na<sub>2</sub>O<sub>2</sub> fusion, melt leached with water, precipitate filtered, and chromium and vanadium precipitated in the filtrate with lead acetate. Solution filtered, precipitate dissolved, and lead removed as sulfate. Chromium determined by the silver nitrate-persulfate method and vanadium by the ferrous sulfate-persulfate method.

- <sup>k</sup> As in (a), except that vanadium was removed in the acid solution of the alloy by NaOH-Na<sub>2</sub>O<sub>2</sub> before the sulfide separation.
  - <sup>l</sup> Ferrous sulfate-persulfate method.
  - <sup>m</sup> Mercury cathode-cupferron. Ignited oxide corrected for V<sub>2</sub>O<sub>5</sub>.
  - <sup>n</sup> Red lead used as an accelerator.
  - <sup>o</sup> Sulfuric acid dehydration.
  - <sup>p</sup> Na<sub>2</sub>O<sub>2</sub> fusion, silver nitrate-persulfate method.
  - <sup>q</sup> Na<sub>2</sub>O<sub>2</sub> fusion, ferrous sulfate-persulfate method.
  - <sup>r</sup> Mercury cathode-cupferron-8 hydroxyquinoline.
  - <sup>s</sup> Acetic acid-thiosulfate method, see Sampling and Analysis of Ferro Alloys, by chemists of the U. S. Steel Corporation, Pittsburgh, Pa., p. 26.
  - <sup>t</sup> Copper oxide and ingot iron used as an accelerator.
  - <sup>u</sup> Vanadium reduced in perchloric acid solution with FeSO<sub>4</sub>. Excess FeSO<sub>4</sub> oxidized with 0.01 N KMnO<sub>4</sub>, using orthophenanthroline indicator. Phosphoric acid and K<sub>2</sub>HPO<sub>4</sub> added, and vanadium titrated with 0.01 N KMnO<sub>4</sub>.
  - <sup>v</sup> Phosphate method, after separation of titanium by the thiosulfate method as in footnote (s).
  - <sup>w</sup> Acid solution poured into an excess of sodium hydroxide solution (10 percent) containing some sodium peroxide. Solution boiled, cooled, and filtered. Precipitate dissolved and solution passed through a Jones reductor and titrated with 0.1 N ferric ammonium sulfate.
  - <sup>x</sup> Na<sub>2</sub>O<sub>2</sub> fusion, sulfuric acid dehydration.
  - <sup>y</sup> Na<sub>2</sub>O<sub>2</sub> fusion, colorimetric.
  - <sup>z</sup> Iron, titanium, and the like removed by a double NaOH-Na<sub>2</sub>O<sub>2</sub> treatment. Filtrate acidified and treated with cupferron. Ignited precipitate fused, and vanadium determined by the ferrous sulfate-persulfate method.
  - <sup>aa</sup> Iron, titanium, and the like precipitated with cupferron. Filtrate treated to destroy cupferron. Aluminum precipitated with ammonia. Solution filtered, precipitate dissolved, and aluminum determined as AlPO<sub>4</sub>.
- Analyst 1 also reported 0.01% of Zr. Analyst 5 also reported 0.31% of Mn and 0.20% of P. Analyst 7 also reported 0.32% of Mn, 0.20% of P, and 0.055% of S.

### \* LIST OF ANALYSTS

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|---|--|
| 1. Robert M. Fowler, National Bureau of Standards, Washington, D. C.        | 5. H. A. Kar, Timken Roller Bearing Co., Canton, Ohio.             |
| 2. C. J. Kinzie, The Titanium Alloy Manufacturing Co., Niagara Falls, N. Y. | 6. W. D. Brown, Carnegie-Illinois Steel Corporation, Duquesne, Pa. |
| 3. A. C. Schultz, Ohio Ferro-Alloys Corporation, Philo, Ohio.               | 7. L. E. Harper, Vanadium Corporation of America, Bridgeville, Pa. |
| 4. Thomas R. Cunningham, Electro Metallurgical Co., Niagara Falls, N. Y.    | 8. Fred R. Wetzling, Metal & Thermit Corporation, New York, N. Y.  |

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