

U. S. DEPARTMENT OF COMMERCE

# National Bureau of Standards

## Certificate of Analyses

OF

STANDARD SAMPLE 116A

### FERROTITANIUM

(LOW CARBON)

ANALYST*	Titanium	Carbon	Phosphorus	Silicon	Chromium	Vanadium	Aluminum
1.....	<sup>a</sup> 25.06	0.025	<sup>b</sup> 0.18	<sup>c</sup> 3.09	<sup>d</sup> 0.23	<sup>e</sup> 0.34	<sup>f</sup> 3.24
2.....	<sup>g</sup> 25.15	.025	.17	3.10	.24	.33	<sup>h</sup> 3.15
3.....	<sup>i</sup> 25.10	-----	-----	<sup>c</sup> 3.15	<sup>d</sup> .23	<sup>e</sup> .33	<sup>j</sup> 3.34
4.....	<sup>k</sup> 25.17	.020	-----	<sup>c</sup> 3.13	-----	-----	<sup>l</sup> 3.28
Averages.....	<sup>m</sup> 25.12	0.023	0.18	3.12	0.23	0.33	3.25

<sup>a</sup> One hundred milliliters of H<sub>2</sub>SO<sub>4</sub> (1:4) and 10 ml. of HF added to a 2.5-gm. sample in a covered platinum dish. Ten milliliters of HNO<sub>3</sub> added and solution digested, then evaporated to fumes of sulfuric acid and cooled. Walls of dish rinsed, solution again evaporated to fumes, cooled, diluted, and filtered. Filtrate reserved. Insoluble residue ignited, fused with K<sub>2</sub>S<sub>2</sub>O<sub>7</sub>, melt dissolved in water, solution made ammoniacal and filtered. Precipitate ignited, dissolved in H<sub>2</sub>SO<sub>4</sub>-HF, solution fumed and added to main reserved filtrate. Combined solutions diluted to 500 ml. One-hundred-milliliter aliquot portion taken and acid-sulfide and ammoniacal sulfide-tartrate separations made. Titanium twice precipitated with cupferron and ignited to TiO<sub>2</sub>. Gross weight corrected for (a) total V<sub>2</sub>O<sub>5</sub> (from V determined on a separate sample), (b) blank on entire procedure, (c) titanium retained in FeS (usually less than 0.1 mg.), determined colorimetrically. (Zirconium and iron not detected.)

<sup>b</sup> Molybdenum-blue photometric method.

<sup>c</sup> Double dehydration with sulfuric acid with intervening filtration.

<sup>d</sup> Silver nitrate-persulfate oxidation, potentiometric titration with ferrous ammonium sulfate and correction for vanadium.

<sup>e</sup> Nitric acid oxidation and potentiometric titration with ferrous ammonium sulfate.

<sup>f</sup> Two and a half-gram sample treated with sulfuric-nitric acids. Solution evaporated to fumes of sulfuric acid, diluted, and filtered. Filtrate reserved. Non-volatile residue, remaining after treatment with hydrofluoric acid, fused with both Na<sub>2</sub>CO<sub>3</sub> and K<sub>2</sub>S<sub>2</sub>O<sub>7</sub>. Melts dissolved in dilute H<sub>2</sub>SO<sub>4</sub> and added to reserved filtrate. Combined solutions diluted to 1,000 ml. Iron, titanium, and the like, twice precipitated with cupferron in a 200-ml. aliquot portion. Aluminum precipitated in the combined filtrates with 8-hydroxyquinoline. Precipitate decomposed with HNO<sub>3</sub>-H<sub>2</sub>SO<sub>4</sub>-HClO<sub>4</sub>. Aluminum precipitated with NH<sub>4</sub>OH. Ignited oxide examined and corrected for SiO<sub>2</sub>, P<sub>2</sub>O<sub>5</sub>, Cr<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub>, and TiO<sub>2</sub>.

<sup>g</sup> One-half-gram sample treated with hydrochloric-nitric-sulfuric acids. Solution evaporated to fumes of sulfuric acid, diluted, and filtered. Residue ignited, fused, and tested colorimetrically for titanium. Citric acid added to the filtrate and solution treated with H<sub>2</sub>S to remove acid-sulfide group, followed by removal of iron as sulfide in ammoniacal-citrate solution. Titanium precipitated with cupferron and the ignited oxide corrected for V<sub>2</sub>O<sub>5</sub> and traces of SiO<sub>2</sub> and Fe<sub>2</sub>O<sub>3</sub>.

<sup>h</sup> One-gram sample treated with hydrochloric-nitric-sulfuric acids. Residue fused and added to the main solution. Iron and titanium removed by precipitation with cupferron. Aluminum subsequently precipitated in the filtrate with NH<sub>4</sub>OH. Solution filtered, precipitate dissolved, and aluminum determined as AlPO<sub>4</sub>.

<sup>i</sup> Solution of a 0.5-gm. sample in a platinum dish with H<sub>2</sub>SO<sub>4</sub> (15 percent), H<sub>2</sub>O<sub>2</sub> (30 percent) and 1 to 2 ml. of HF. Boric acid added and acid-sulfides removed. Iron sulfide precipitated in ammoniacal-tartrate solution. Titanium precipitated with cupferron and the ignited residue corrected for V<sub>2</sub>O<sub>5</sub>.

<sup>j</sup> One-half-gram sample dissolved in sulfuric-hydrofluoric-nitric acids and the solution evaporated to dryness. Titanium and iron removed by double precipitation with cupferron. Aluminum precipitated twice with NH<sub>4</sub>OH and the ignited oxide corrected for P<sub>2</sub>O<sub>5</sub>.

<sup>k</sup> Sample dissolved in sulfuric-hydrofluoric-nitric acids and the solution evaporated to fumes of sulfuric acid. Vanadium and chromium separated by treatment with NaOH-H<sub>2</sub>O<sub>2</sub>. Hexamethylenetetramine separation in reduced solution to remove most of the iron. Precipitate dissolved, acid-sulfide and ammoniacal-sulfide separations made, and titanium twice precipitated with cupferron, ignited to the oxide and corrected for impurities.

<sup>l</sup> Cupferron-NH<sub>4</sub>OH-8-hydroxyquinoline-NH<sub>4</sub>OH. Ignited oxide corrected for blank.

<sup>m</sup> On the basis of special work at the National Bureau of Standards, it is recommended that the value 25.06 be used for the titanium content of this standard. Analyst 2 also reported 1.48 percent of manganese and 0.21 percent of copper.

#### \* LIST OF ANALYSTS

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The metal for the preparation of this standard was furnished by the Vanadium Corporation of America.

WASHINGTON, March 10, 1947.

E. U. CONDON, *Director*.