



Standard Test Methods for Chemical Analysis of Titanium and Titanium Alloys¹

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This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 These test methods cover procedures for the chemical analysis of titanium and titanium alloys containing the following elements in the concentration ranges listed:

Element	Concentration Range, %
Aluminum	1 to 10
Chloride	0.02 to 1.0
Chromium	0.005 to 20
Columbium	0.25 to 5.0
Copper	0.001 to 1.1
Iron	0.005 to 20
Magnesium	0.02 to 1.0
Manganese	0.005 to 20
Molybdenum	0.005 to 5
Nitrogen	0.005 to 0.20
Oxygen	0.03 to 0.50
Palladium	0.10 to 0.25
Silicon	0.005 to 5.0
Tantalum	0.25 to 5.0
Tin	0.25 to 10
Tungsten	0.005 to 1.0
Vanadium	0.5 to 20
Zirconium	1.0 to 20

1.2 The test methods in this standard are contained in the sections indicated as follows:

	Sections
Aluminum:	
8-Quinolinol (Gravimetric) Test Method ²	8-14
Chloride by the Gravimetric Test Method ²	15-22
Copper:	
Cuprizone (Photometric) Test Method	23-32
Diethyldithiocarbamate (Photometric) Test Method	33-42
Manganese:	
Periodate (Photometric) Test Method	43-52
Molybdenum	
Thiocyanate-Extraction (Photometric) Test Method	53-62
Thiocyanate 2-(2-Butoxyethoxy) Ethanol (Photometric) Test Method	63-72
Nitrogen by the Distillation-Titration Test Method ²	73-80
Palladium by the 1-Nitroso-2-Naphthol (Photometric) Test Method	81-90
Vanadium by the Potassium Permanganate (Titrimetric) Test Method	91-97
Zirconium by the Mandelic Acid (Gravimetric) Test Method	98-104

¹ These test methods are under the jurisdiction of ASTM Committee E-1 on Analytical Chemistry for Metals, Ores, and Related Materials and are the direct responsibility of Subcommittee E01.06 on Ti, Zr, W, Mo, Ta, Nb, Hf.

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1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

E 29 Practice for Using Significant Digits in Test Data to Determine Conformance With Specifications³

E 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals⁴

E 59 Practice for Sampling Steel and Iron for Determination of Chemical Composition⁴

E 60 Practice for Photometric and Spectrophotometric Methods for Chemical Analysis of Metals⁴

E 173 Practice for Conducting Interlaboratory Studies of Methods for Chemical Analysis of Metals⁴

3. Significance and Use

3.1 These test methods for the chemical analysis of metals and alloys are primarily intended for referee purposes. It is assumed that all who use these test methods will be trained analysts capable of performing common laboratory procedures skillfully and safely. It is expected that work will be performed in a properly equipped laboratory.

4. Apparatus, Reagents, and Photometric Practice

4.1 Apparatus and reagents required for each determination are listed in separate sections preceding the procedure. The apparatus, standard solutions, and certain reagents used in more than one procedure are referred to by number and shall conform to the requirements prescribed in Practices E 50, except that photometers shall conform to the requirements prescribed in Practice E 60.

5. Hazards

5.1 For hazards to be observed in these test methods,

² These test methods are based on the methods developed by the Panel on Methods of Analysis of the Metallurgical Advisory Committee on Titanium, Ordnance Corps, U. S. Army, and published in their *Bulletin T8*.

³ *Annual Book of ASTM Standards*, Vol 14.02.

⁴ *Annual Book of ASTM Standards*, Vol 03.05.

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reference shall be made to Practices E 50.

6. Sampling

6.1 No standardized sampling procedures are available, but the principles of Practice E 59 should be used as a guide to obtain representative samples, bearing in mind that with present melting practices titanium metal and titanium-base alloys may show greater inhomogeneities than are usually found in steels.

6.2 The sample used for the chloride determination shall not be subjected to any physical or mechanical process that breaks down or opens up significant portions of cells of the titanium sponge, or causes a localized heating of the sponge sample. Avoid briquetting, compacting, drilling, or combination thereof, of the titanium sponge to obtain a laboratory sample for analysis of chloride.

7. Rounding Calculated Values

7.1 Calculated values shall be rounded to the desired number of places in accordance with the rounding test method given in 3.4 and 3.5 of Practice E 29.

**ALUMINUM BY THE 8-QUINOLINOL
(GRAVIMETRIC) TEST METHOD**

8. Scope

8.1 This test method covers the determination of aluminum in titanium and titanium alloys in concentrations from 1 to 8 %.

9. Summary of Test Method

9.1 The sample is dissolved in hydrochloric and nitric acids and treated with sodium hydroxide to precipitate titanium and other metals. Aluminum in the filtrate is precipitated with 8-quinolinol in the presence of hydrogen peroxide, dried, and weighed.

10. Interferences

10.1 The elements ordinarily present do not interfere if their concentrations are under the maximum limits shown in 1.1.

11. Reagents

11.1 *8-Quinolinol Solution* (25 g/L)—Reagent No. 130.

11.2 *Sodium Hydroxide Solution A* (300 g/L)—Prepare a solution as described for Reagent No. 146, but use 300 g instead of the specified weight.

11.3 *Sodium Hydroxide Solution B* (100 g/L)—Reagent No. 146.

11.4 *Tartaric Acid Solution* (250 g/L)—Prepare a solution as described for Reagent No. 116, but use 250 g instead of the specified weight.

12. Procedure

12.1 Transfer a 1.0-g sample, weighed to the nearest 1 mg, to a 400-mL beaker.

12.2 Carry a reagent blank through the entire procedure, using the same amounts of all reagents with the sample omitted.

12.3 Add 100 mL of HCl (1 + 1), and heat gently until dissolution is complete. Add HNO₃ dropwise until the titanium is oxidized, and then boil gently to expel oxides of nitrogen.

12.4 Cool, nearly neutralize (Note 1) with NaOH Solution A, and pour the solution slowly, with stirring, into a 600-mL beaker containing 150 mL of hot NaOH Solution B. Boil for 1 min while stirring constantly, and cool to room temperature in a running water bath.

NOTE 1—Once the solution is made alkaline, it must be carried through the procedure without delay until it is acidified to litmus as directed in 12.6.

12.5 Transfer to a 500-mL volumetric flask, dilute to volume, and mix. Immediately transfer, without rinsing, to a dry 600-mL beaker. Filter through a dry 18.5-cm fluted filter paper into a dry 250-mL volumetric flask, discard the first 50 mL, and collect 250 mL of the filtrate.

12.6 Transfer to a 600-mL beaker and acidify to litmus with HCl (1 + 1). Add 25 mL of tartaric acid solution and adjust to pH 8.0 using NH₄OH and pH test paper. Add 10 mL of H₂O z(30 %) and heat to 50 to 60°C. While mechanically stirring, add 3.5 mL of 8-quinolinol solution for each 5 mg of aluminum present, plus 5 mL in excess. Add 5 mL of NH₄OH and continue stirring for 10 min.

12.7 Allow the precipitate to settle. Filter through a weighed medium-porosity fritted-glass crucible. Test for completeness of precipitation by the addition of 5 mL of 8-quinolinol solution to the filtrate. Wash the precipitate four times with warm NH₄OH (1 + 99). Dry at 135°C for 1½ h. Cool in a desiccator and weigh as aluminum quinolate.

13. Calculation

13.1 Calculate the percentage of aluminum as follows:

$$\text{Aluminum, \%} = (A - B) \times 0.0587 \times 100/C \quad (1)$$

where:

- A = grams of aluminum quinolate found in the aliquot used,
- B = grams of aluminum quinolate in the blank, and
- C = grams of sample represented in the 250-mL aliquot.

14. Precision and Bias

14.1 *Precision*—Seven laboratories cooperated in testing this test method on samples of Ti-6Al-2Sn-4Zr-2Mo and

TABLE 1 Precision Data for Aluminum by the 8-Quinolinol (Gavimetric) Method

Test Material	Aluminum Found, % by Laboratory No. ^A						
	1	2	3	4	5	6	7
(1) Titanium-base alloy, 2Sn-4Zr-2Mo	6.01	6.20	6.04	6.01	6.07	6.04	6.10
	6.00	6.16	6.01	6.02	6.13	6.05	5.95
(2) Titanium-base alloy, 13V-11Cr	3.11	3.19	3.27	3.09	2.91	3.22	3.06
	3.01	3.19	3.23	3.07	3.09	3.23	3.12

^A Each sample analyzed once on each of two days in each laboratory.

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Ti-13V-11Cr-3Al, and obtained the data summarized in Table 1.

14.2 *Bias*—No certified reference materials suitable for testing this test method were available when the interlaboratory testing program was conducted. The user of this test method is encouraged to employ accepted reference materials, if available, to determine the bias of this test method as applied in a specific laboratory.

CHLORIDE BY THE GRAVIMETRIC TEST METHOD

15. Scope

15.1 This test method covers the determination of chloride in titanium sponge in the range from 0.02 to 1 %.

16. Summary of Test Method

16.1 The sample is dissolved in dilute hydrofluoric acid and oxidized with nitric acid. The chloride is precipitated with silver nitrate and weighed as silver chloride.

17. Interferences

17.1 Elements normally present in titanium sponge do not interfere.

18. Apparatus

18.1 *Filtering Crucibles*, fritted-glass, fine-porosity, 30-mL, conforming to the requirements for Apparatus No. 2.

18.2 *Plastic Beakers*, 400 or 600-mL.

18.3 *Plastic Graduated Cylinder*, 25-mL.

19. Reagents

19.1 *Boric Acid* (H_3BO_3).

19.2 *Silver Nitrate Solution* (5.8 g/L)—Dissolve 5.8 g of silver nitrate ($AgNO_3$) in water and dilute to 1 L.

19.3 *Silver Nitrate Wash Solution* (0.05 g/L)—Dissolve 0.05 g of silver nitrate ($AgNO_3$) in 1 L of water.

20. Procedure

20.1 Transfer 20 mL of HF and 50 mL of water to a plastic beaker and mix. Weigh 5 g of the sample to the nearest 1 mg. Transfer 1 to 2 g of the sample, in small portions, to the dilute HF solution. (See 6.2 for guidelines for handling the sample.) Add 5 mL of HNO_3 and then add the remainder of the sample in small portions. After all of the metal has been added, allow to stand until dissolution is complete.

NOTE 2—Dissolution of the sample is usually complete in 5 min, with a colorless solution being obtained. If the solution is not colorless, add HNO_3 dropwise until the color disappears.

20.2 Carry a reagent blank through all steps of the procedure.

20.3 Add 5 g of H_3BO_3 to the solution, transfer to a 400-mL glass beaker, and dilute to 200 mL. Stir and heat gently until the H_3BO_3 dissolves. With stirring, slowly add 10 mL of the $AgNO_3$ solution. Heat to approximately 60°C and maintain at this temperature until the supernatant liquid clears. Test for completeness of precipitation by adding a few drops of the $AgNO_3$ solution. If additional precipitate forms, add an additional 5 mL of $AgNO_3$ solution and repeat the above operation. Allow to stand in a dark place for 2 h.

20.4 Filter, using a low-ash, fine-textured, paper, and wash thoroughly with the $AgNO_3$ wash solution. Dissolve the precipitate by pouring 50 mL of NH_4OH (1 + 1) in 10-mL increments through the filter, collecting the filtrate in the original beaker. If necessary, pour the NH_4OH through a second time to dissolve the silver chloride (Note 3). Wash the paper thoroughly with water.

NOTE 3—Residual titanium hydroxide on the paper should not be confused with silver chloride.

20.5 Make the filtrate slightly acid with HNO_3 , using methyl orange indicator. Add 5 mL of the $AgNO_3$ solution and stir to coagulate the precipitate. Check for complete precipitation as described in 20.3. Allow to stand in a dark place for 2 h. Filter through a weighed fritted-glass crucible. Police the beaker and stirring rod and wash thoroughly with the $AgNO_3$ wash solution. Finally, wash twice with water.

20.6 Dry the crucible and contents at 130 to 150°C to constant weight (approximately 2 h). Cool in a desiccator and weigh.

21. Calculation

21.1 Calculate the percentage of chloride as follows:

$$\text{Chloride, \%} = [(A - B) \times 0.2474] / C \times 100 \quad (2)$$

where:

A = grams of AgCl from the sample,

B = grams of AgCl from reagent blank, and

C = grams of sample used.

22. Precision and Bias

22.1 This test method was originally approved for publication before the inclusion of precision and bias statements within standards was mandated. The original interlaboratory test data for this test method are no longer available. The user is cautioned to verify by the use of reference materials, if available, that the precision and bias of this test method are adequate for the contemplated use.

COPPER BY CUPRIZONE (PHOTOMETRIC) TEST METHOD

23. Scope

23.1 This test method covers the determination of copper in concentrations from 0.4 to 1.1 %.

24. Summary of Test Method

24.1 Ammonium citrate is added to the sample solution, and the pH adjusted to about 8.5. Cuprizone is added to form a blue-colored complex with cupric copper. Photometric measurement is made at approximately 600 nm.

25. Concentration Range

25.1 The recommended concentration range is from 0.05 to 0.3 mg of copper/100 mL of solution using a 1-cm cell.

NOTE 4—This test method has been written for cells having a 1-cm light path. Cells having other dimensions may be used, provided suitable adjustments can be made in the amounts of sample and reagents used.

26. Stability of Color

26.1 The color develops in 20 min and is stable for 1 h.