



Standard Test Methods for Chemical Analysis of Molybdenum¹

This standard is issued under the fixed designation E 315; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last approval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 These test methods cover the chemical analysis of molybdenum and molybdenum alloys having chemical compositions within the following limits:

Element	Concentration Range, %
Carbon	0.010 to 0.040
Iron	0.020 max
Molybdenum	99.25 to 99.99
Nickel	0.010 max
Nitrogen	0.0010 max
Oxygen	0.0030 max
Silicon	0.010 max
Titanium	0.005 to 0.55
Zirconium	0.06 to 0.12

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

	Sections
Nickel by the Persulfate-Dimethylglyoxime Photometric Method	9-18
Iron by the 1,10-Phenanthroline Photometric Method	19-29
Silicon by the Molybdenum Blue-Extraction Photometric Method	30-40

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.* Specific precautionary information is given in Section 5.

2. Referenced Documents

2.1 ASTM Standards:²

- B 384 Specification for Molybdenum and Molybdenum Alloy Forgings
- B 385 Specification for Molybdenum and Molybdenum Alloy Billets for Reforging
- B 386 Specification for Molybdenum and Molybdenum Alloy Plate, Sheet, Strip, and Foil

¹ These test methods are under the jurisdiction of ASTM Committee E01 on Analytical Chemistry for Metals, Ores, and Related Materials and is the direct responsibility of Subcommittee E01.06 on Titanium, Zirconium, Wolfram, Molybdenum, Tantalum, Niobium, Hafnium.

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² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

- B 387 Specification for Molybdenum and Molybdenum Alloy Bar, Rod, and Wire
- E 29 Practice for Using Significant Digits in Test Data to Determine Conformance With Specifications
- E 50 Practices for Apparatus, Reagents, and Safety Considerations for Chemical Analysis of Metals, Ores, and Related Materials
- E 60 Practice for Molecular Absorption and Spectrometric Methods for Chemical Analysis of Metals, Ores, and Related Materials
- E 173 Practice for Conducting Interlaboratory Studies of Methods for Chemical Analysis of Metals
- E 1601 Practice for Conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method

3. Significance and Use

3.1 These test methods for the chemical analysis of metals and alloys are primarily intended to test such materials for compliance with compositional specifications. 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 sections, and other reagents used in more than one procedure are referred to by number and shall conform to the requirements prescribed in Practices E 50, except the photometers shall conform to the requirements prescribed in Practice E 60.

4.2 Photometric practice prescribed in these test methods shall conform to Practice E 60.

5. Hazards

5.1 For precautions to be observed in the use of certain reagents in these test methods, refer to Practices E 50.

6. Sampling

6.1 For procedures for sampling the material, refer to Specifications B 384, B 385, B 386, and B 387.

7. Rounding Calculated Values

7.1 Calculated values shall be rounded to the desired number of places as directed in 3.4 to 3.6 of Practice E 29.

8. Interlaboratory Studies

8.1 These test methods have been evaluated in accordance with Practice E 173, unless otherwise noted in the precision and bias section.

NICKEL BY THE PERSULFATE-DIMETHYLGLYOXIME PHOTOMETRIC TEST METHOD

9. Scope

9.1 This test method covers the determination of nickel in concentrations from 1 to 400 ppm. The range may be extended by suitably varying the sample weight, aliquot size, amount of reagents, and cell depth.

10. Summary of Test Method

10.1 A red soluble salt of trivalent nickel and dimethylglyoxime is formed. Photometric measurement is made at approximately 445 nm.

11. Concentration Range

11.1 The recommended concentration ranges are from 1.0 to 2.0 μg and from 10 to 100 μg of nickel for each 50 mL of solution, using cell depths of 5 and 2 cm respectively.

NOTE 1—This test method has been written for cells having 5 and 2-cm light paths. Cells having other dimensions may be used, provided suitable adjustments can be made in the amounts of sample and reagents used.

12. Stability of Color

12.1 The color develops in 5 min and is stable for 30 min.

13. Interferences

13.1 Provision has been made for the correction of any interfering elements present.

14. Reagents

14.1 *Ammonium Persulfate Solution* (150 g/L)—Dissolve 15 g of ammonium persulfate $[(\text{NH}_4)_2\text{S}_2\text{O}_8]$ in water and dilute to 100 mL. Prepare fresh as needed.

14.2 *Citric Acid Solution* (100 g/L)—Dissolve 10 g of citric acid in water and dilute to 100 mL.

14.3 *Nickel, Standard Solution A* (1 mL = 10 μg Ni)—Dissolve 0.1000 g of nickel (purity 99.9 % min) in 10 mL of water and 5 mL of HNO_3 in a 150-mL beaker. When dissolution is complete boil to remove oxides of nitrogen. Cool to room temperature, transfer to a 1-L volumetric flask, dilute to volume, and mix. Transfer a 50-mL aliquot of this solution to a 500-mL volumetric flask, dilute to volume, and mix.

14.4 *Nickel, Standard Solution B* (1 mL = 1 μg Ni)—Transfer a 10-mL aliquot of nickel solution A (1 mL = 10 μg Ni) to a 100-mL volumetric flask, dilute to volume, and mix.

14.5 *Sodium Dimethylglyoximate Solution* (30 g/L)—Dissolve 30 g of sodium dimethylglyoxime in water and dilute to 1 L.

15. Preparation of Calibration Curves

15.1 *Calibration Solutions:*

15.1.1 Transfer 1.00, 3.00, 4.00, 7.00, and 10.00 mL of nickel solution A (1 mL = 10 μg Ni) to five 50-mL volumetric flasks, dilute to 20 mL, and proceed as directed in 15.3.

15.1.2 Transfer 1.00, 5.00, 10.00, 15.00, and 20.00 mL of nickel solution B (1 mL = 1 μg Ni) to five 50-mL volumetric flasks, dilute to 20 mL, and proceed as directed in 15.3.

15.2 *Reference Solution*—Transfer 20 mL of water to a 50-mL volumetric flask and proceed as directed in 15.3.

15.3 *Color Development*—Add 4 mL of $(\text{NH}_4)_2\text{S}_2\text{O}_8$ solution, 5 mL of citric acid solution, and 10 mL of NH_4OH , and cool to room temperature. Add 1 mL of sodium dimethylglyoximate solution, dilute to volume, and mix.

15.4 *Photometry*—Transfer a suitable portion of the reference solution to an absorption cell with a 2 or a 5-cm light path, as appropriate (see 11.1), and adjust the photometer to the initial setting, using a light band centered at approximately 445 nm. While maintaining this adjustment, take the photometric readings of the calibration solutions.

15.5 *Calibration Curves*—Plot the photometric readings of the calibration solutions against micrograms of nickel per 50 mL of solution.

16. Procedure

16.1 *Test Solution:*

16.1.1 Select a sample weight in accordance with the following table:

Nickel, ppm	Sample Weight, g
1 to 50	1.00
45 to 120	0.50
115 to 400	0.25

16.1.2 Weigh duplicate samples of the size selected to the nearest 1 mg (Note 2), and transfer to 100-mL beakers. Add 10 mL of HCl and heat. Add HNO_3 dropwise until all the metal has decomposed (Note 3). Remove from the hot plate and cool.

NOTE 2—Duplicate samples are taken in order to have one available to determine the sample blank. For the higher concentrations of nickel, a larger sample may be dissolved and diluted to volume; duplicate aliquots may then be taken from a single sample.

NOTE 3—If any molybdenic oxide precipitates, add more HCl dropwise and boil gently until the molybdenum is completely in solution.

16.1.3 Transfer the solutions to 50-mL volumetric flasks and dilute to about 20 mL.

16.2 *Reference Solution*—Distilled water.

16.3 *Reagent Blank Solution*—Carry a reagent blank through the entire procedure using the same amount of all reagents.

16.4 *Sample Blank Solution*—Treat the duplicate as directed in 15.3 but omit the addition of the sodium dimethylglyoximate solution.

16.5 *Color Development*—Proceed as directed in 15.3.

16.6 *Photometry*—Take the photometric readings of the reagent blank solution, the sample blank solution, and the test solution as directed in 15.4.

17. Calculation

17.1 Convert the photometric readings of the test solution to micrograms of nickel, and of the reagent blank and sample