



Standard Test Method for Molybdenum in Water¹

This standard is issued under the fixed designation D 3372; 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 reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

1. Scope

1.1 This test method² covers the determination of dissolved and total recoverable molybdenum in most waters, wastewaters, and brines by atomic absorption spectroscopy.

1.2 This test method is applicable in the range from 1 to 25 µg/L of molybdenum. The range may be extended by dilution of the sample.

1.3 This test method has been used successfully with natural and reagent waters. It is the user's responsibility to ensure the validity of this test method for waters of untested matrices.

1.4 *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.* For specific precautionary statements, see Note 3 and Note 9.

2. Referenced Documents

2.1 ASTM Standards:

D 1129 Terminology Relating to Water³

D 1193 Specification for Reagent Water³

D 2777 Practice for Determination of Precision and Bias of Applicable Test Methods of Committee D-19 on Water³

D 3370 Practices for Sampling Water from Closed Conduits³

D 4691 Practice for Measuring Elements in Water by Flame Atomic Absorption Spectrophotometry³

D 4841 Practice for Estimation of Holding Time for Water Samples Containing Organic and Inorganic Constituents³

3. Terminology

3.1 **Definitions**—For definitions of terms used in this test method, refer to Terminology D 1129.

3.2 Definition of Term Specific to This Standard:

3.2.1 **total recoverable molybdenum**—an arbitrary analyti-

cal term relating to the forms of molybdenum that are determinable by the digestion procedure described in this test method.

4. Summary of Test Method

4.1 Molybdenum is determined by atomic-absorption spectrophotometry. The element is chelated with 8-hydroxyquinoline, extracted with methyl isobutyl ketone, and the extract aspirated into the nitrous oxide-acetylene flame of the spectrophotometer.

5. Significance and Use

5.1 Molybdenum can be found in waste that results from chemical cleaning of components in which the metal is alloyed.

5.2 NPDES permits or other standards, or both, require monitoring pollutants in waste discharged onto the water shed of, or into, navigable waters, and those disposed of in such a manner that eventual contamination of underground water could result.

5.3 This test method affords an accurate and sensitive means of determining compliance/noncompliance with those permits.

6. Interferences

6.1 Vanadium (V) and iron (III) enhance the absorption, while chromium (VI) and tungsten (VI) suppress it. These interferences are eliminated by the addition of ascorbic acid.

7. Apparatus

7.1 **Atomic-Absorption Spectrophotometer**, for use at 313.3 nm. A general guide for the use of flame atomic absorption applications is given in Practice D 4691.

NOTE 1—The manufacturer's instructions should be followed for all instrumental parameters.

7.1.1 Molybdenum Hollow-Cathode Lamp.

7.2 **Pressure-Reducing Valves**—The supplies of fuel and oxidant shall be maintained at a pressure somewhat higher than the controlled operating pressure of the instrument by suitable valves.

8. Reagents and Materials

8.1 **Purity of Reagents**—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society,

¹ This test method is under the jurisdiction of ASTM Committee D-19 on Water and is the direct responsibility of Subcommittee D19.05 on Inorganic Constituents in Water.

Current edition approved June 15, 1992. Published October 1992. Originally published as D 3372 – 75. Last previous edition D 3372 – 86.

² Chau, Y. K., and Lum-Shue-Chan, K., "Atomic Absorption Determination of Microgram Quantities of Molybdenum in Lake Waters," *Analytica Chimica Acta*, Vol 48, 1969, p. 205.

³ *Annual Book of ASTM Standards*, Vol 11.01.