



Standard Test Method for Phosphonate in Brines¹

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1. Scope*

1.1 This test method covers the colorimetric determination of phosphonate (PNA) in brines from gas and oil production operations in the range from 0.1 to 5 mg/L.

1.2 This phosphonate method is intended for use to analyze low concentration of phosphonate in brine containing interfering elements. This test method is most useful for analyzing phosphonate at 0.1 to 1 mg/L range in brines with interfering elements; however, it requires personnel with good analytical skill.

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

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.5 *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 hazard statements, see 9.1.3.

2. Referenced Documents

2.1 ASTM Standards:²

D1129 Terminology Relating to Water

D1193 Specification for Reagent Water

D2777 Practice for Determination of Precision and Bias of Applicable Test Methods of Committee D19 on Water

D3370 Practices for Sampling Water from Closed Conduits

D3856 Guide for Management Systems in Laboratories Engaged in Analysis of Water

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

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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.

D4375 Practice for Basic Statistics in Committee D19 on Water

D5810 Guide for Spiking into Aqueous Samples

D5847 Practice for Writing Quality Control Specifications for Standard Test Methods for Water Analysis

E275 Practice for Describing and Measuring Performance of Ultraviolet and Visible Spectrophotometers

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, refer to Terminology D1129.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *phosphonate, n*—a group of organophosphorus compounds typically used for mineral scale and corrosion control, as cleaning agents, dispersants, and chelants.

3.2.1.1 *Discussion*—Typical phosphonate compounds include, but are not limited to, the following phosphonic acid and their neutralized salts: Aminotri(methylenephosphonic acid), 1-hydroxyethylidene-1,1-diphosphonic acid, ethylenediaminetetra (methylenephosphonic acid), hexamethylenediaminetetra (methylenephosphonic acid), and diethylenetriaminepenta (methylenephosphonic acid).

4. Summary of Test Method

4.1 Phosphonate materials are converted to orthophosphate by potassium persulfate digestion. The orthophosphate is then reacted with ammonium molybdate to form a phosphomolybdate complex. The complex is extracted with a methyl isobutyl ketone/cyclohexane mixture and measured colorimetrically.

5. Significance and Use

5.1 This test method is useful for the determination of trace level phosphonate residues in brines. Chemical treatment which contain phosphonates are used as mineral scale and corrosion inhibitors in gas and oil drilling and production operations; and other industrial applications. Often, the decision for treatment is based on the ability to measure low phosphonate concentration and not upon performance criteria. Phosphonate concentrations as low as 0.16 mg/L have been shown effective in carbonate scale treatment. This test method enables the measurement of sub-mg/L phosphonate concentration in brines containing interfering elements.

5.2 The procedure includes measuring total (see 12.3.8) and free orthophosphate (see 12.4.3) ions and the difference in

*A Summary of Changes section appears at the end of this standard