Designation: ~~D6501 – 09~~ D6501 – 15

Standard Test Method for Phosphonate in Brines[1]

This standard is issued under the fixed designation D6501; 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.

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
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.

[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.

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).

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

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 concentration is the phosphonate concentration. The sample could contain orthophosphate naturally, or from decomposition of the phosphonate during processing or well treatment or from treating compounds containing molecular dehydrated phosphates.

6. Interferences

6.1 Sulfide interferes in this test method, but techniques described in the procedure (see 9.1.2) eliminate this interference. Concentrations less than 1000 mg/L copper (Cu^{+2}) and silica (SiO_2/SiO_3^{-2} /Si^{+4}); and less than 200 mg/L of iron (Fe^{+2}/Fe^{+3}) can be tolerated.

6.2 Produced brines can contain high concentrations of dissolved solids. Some of these dissolved solids tend to precipitate when produced brines reach new equilibria at atmospheric temperature and pressure. Phosphonate will coprecipitate or adsorb onto these newly formed solids and become unavailable for analysis. This problem can be minimized by acidifying the brine sample on-site with hydrochloric acid to pH below 2.

6.3 Glassware must be cleaned with phosphate free detergent and rinsed with 0.1 N hydrochloric acid to remove all residual phosphate or phosphonate.

6.4 The standard addition method in 12.6 is recommended for brine with high matrix interference.

7. Apparatus

7.1 *Pressure Cooker or Sterilizer* ~~*(Autoclave)*~~*(Autoclave).*[3]~~.~~

7.2 *Spectrophotometer,*[4] ~~,for~~ for measurement above 650 nm with 4-cm light path cells. A longer light path will yield a corresponding higher sensitivity (see 12.5.1). Spectrophotometer practices prescribed in this test method shall conform to Practice E275.

7.3 *Bottle Top Liquid Dispenser,*[5] ~~,20-mL~~ 20-mL capacity, <1 % accuracy, and <0.1 % precision.

7.4 *Pipetter,* automated,[6]~~,~~ 10-mL capacity with 0.2 to 0.5 % accuracy.

7.5 *Glass Bottles,*[7] ~~,60~~ 60 mL and 240 mL with Teflon-lined screw cap closure.

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~~Society.[8]~~.~~ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

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[3] Fisher Scientific No. 14-141-S has been satisfactory for this purpose, or equivalent, should be used. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

[4] Varian DMS-100 has been satisfactory for this purpose, or equivalent, should be used. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

[5] Fisher Scientific No. 13-687-21 REPIPET has been satisfactory for this purpose, or equivalent, should be used. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

[6] Fisher Scientific No. 21-279-25 Eppendorf Maxipipetter has been satisfactory for this purpose. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

[7] Fisher Scientific No. 03-326-3C and 03-326-3G have been satisfactory for this purpose. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

[8] *Reagent Chemicals, American Chemical Society Specifications* , American Chemical Society, Washington, DC. For suggestions on the testing of Reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

8.2 *Purity of Water*—Unless otherwise indicated, reference to water shall be understood to mean reagent water conforming to Specification D1193, Type I. Other reagent water types may be used provided it is first ascertained that the water is of sufficiently high purity to permit its use without adversely affecting the precision and bias of the test method. Type III water was specified at the time of round robin testing of this test method.

8.3 *Alcoholic Sulfuric Acid Solution*—Cautiously add 20 mL concentrated H_2SO_4 (sp. gr. 1.89) to 900 mL methyl alcohol (8.7) and dilute to 1 L with methyl alcohol. It is recommended to dispense the liquid with a bottle top liquid dispenser, which dispenses a 10-mL volume.

8.4 *Ammonium Molybdate Solution*—Dissolve 39.1 g $(NH_4)_6Mo_7O_{24} \cdot 4H_2O$ in 200 mL water. Cautiously add 210 mL concentrated HCl (sp. gr. 1.19) to 400 mL water. Cool, add molybdate solution, and dilute to 1 L. It is recommended to dispense the liquid with a liquid dispenser, which dispenses a 10-mL volume.

8.5 *Glycerol*—Reagent grade, 99 % or greater.

8.6 *Hydrochloric Acid (6N)*—Add 500 mL of concentrated HCl (sp. gr. 1.19) to 500 mL of water.

8.7 *Methyl Alcohol*—Reagent grade, 99 % or greater.

8.8 *Methyl Isobutyl Ketone/Cyclohexane Solvent*—Mix equal volumes of methyl isobutyl ketone (MIBK) and cyclohexane. ~~Warning:~~(**Warning—**This solvent is highly flammable. It is recommended to dispense the liquid with a bottle top liquid dispenser, which dispenses a 20-mL volume. ~~This solvent is highly flammable. It is recommended to dispense the liquid with a bottle top liquid dispenser, which dispenses a 20-mL volume.~~)

8.9 *Phosphate Solution,* standard (1.00 mL = 0.05 mg PO_4). Dissolve 71.6 mg anhydrous KH_2PO_4 in water and dilute to 1 L.

8.10 *Phosphonate Solution,* (50-mg/L phosphonate)—If the standard addition procedure (see 12.6) is to be used, a stock solution of 50 mg/L, as phosphonate, should be prepared. To prepare this solution, a concentrated sample of the phosphonate to be measured along with the wt/wt percent phosphonate concentration must be obtained from the manufacturer. The wt/wt percent phosphonate concentration also can be calibrated by this procedure as described in 12.2 and 12.3.

8.11 *Potassium Persulfate,* $K_2S_2O_8$.

8.12 *Sodium Chloride Solution (1.0 M, Synthetic Brine)*—Dissolve 58.44 g. NaCl in 800 mL water and dilute to 1 L. This solution is used as a synthetic brine.

8.13 *Sodium Hypochlorite,* (5.65–6 %).

8.14 *Stannous Chloride ~~Solution —~~Solution*—Mix 0.4 g $SnCl_2 \cdot 2H_2O$ in 100 mL glycerol (8.4). This reagent is stable for at least six months. The solution is stored in a dropper bottle.

9. Hazards

9.1 *Precautions:*

9.1.1 Most phosphonate inhibitors are strongly adsorbed to glass or metal; therefore, polyethylene beakers, flasks, pipets, etc., should be used to contain and transfer brine solutions from the field.

9.1.2 A glass bottle is recommended for use in the color development steps (see 12.2 and 12.3) for better visualization of the reaction. Since the reaction media is acidic, phosphonate will not adsorb to the glass surface.

9.1.3 Personnel performing this test must be familiar with all precautions for handling strong sulfuric acid, hydrochloric acid and sulfide-containing brine. Personnel should consult the material safety data sheet for handling strong acids. Protective clothing and latex gloves should be worn. The sulfide brine should be handled in the hood with good ventilation. Sulfide containing brine can be treated with sodium hypochlorite (8.13) prior to analysis to oxidize the hydrogen sulfide.

10. Sampling

10.1 Collect the sample in accordance with Practices D3370.

10.2 Preserve the samples immediately at the time of collection by adding 4 mL of 6 N hydrochloric acid 8.6 per 100-mL brine.

NOTE 1—Alternatively, the pH may be adjusted in the laboratory if the sample is returned within 14 days. However, acid must be added at least 24 hours before analysis to dissolve any metals that adsorb to the container walls. This could reduce hazards of working with acids in the field when appropriate.

11. Calibration and Standardization

11.1 Prepare standards by adding 2.0, 4.0, 6.0, 8.0, 10.0 mL each of phosphate standard solution (1.00 mL = 0.05 mg PO_4) (8.9) to separate 100-mL volumetric flasks. Dilute to 100 mL with 1 M sodium chloride solution (8.12). These solutions will contain 1.0, 2.0, 3.0, 4.0, 5.0 mg/L phosphate as PO_4. If the procedure in 12.5 is used for samples with low phosphonate concentrations, then solutions containing 0.2, 0.4, 0.6, 0.8, 1.0 mg/L phosphate as PO_4 should be used.

11.2 Follow the procedure in 12.2 and 12.3 to develop color, and determine the absorbance at 725 nm.

11.3 ~~Prepare~~ Read directly in concentration if this capability is provided with the instrument or prepare a calibration curve showing phosphate ion concentration in mg/L on the X axis with the corresponding absorbance (A) reading of the spectrophotometer on the Y axis of linear graph paper.

12. Procedure

12.1 The procedures in 12.2 and 12.3 are applicable to samples containing 0.5 to 5 mg/L phosphonate. For samples containing less than 0.5 mg/L phosphonate, a larger sample volume or a different light path cell can be used (see 12.5).

12.2 *Persulfate Digestion Procedure:*

12.2.1 Pipet 20 mL of the following samples (12.2.1.1, 12.2.1.2, 12.2.1.3) into separate 60-mL glass bottles, each containing 200 mg of potassium persulfate (8.11). Multiple samples can be digested at the same time.

12.2.1.1 Blank, 1-M sodium chloride (see 8.12).

12.2.1.2 Phosphate standards (see 11.1).

12.2.1.3 Samples of acidified brine.

12.2.2 Close the sample bottles loosely with Teflon-lined caps.

12.2.3 Heat the samples for 30 minutes in a pressure cooker or sterilizer at 100–120°C (103.4–137.9 kPa (15–20 ~~psig).~~psig)).

12.2.4 Make sure the samples are cooled to room temperature before proceeding to color development. The temperature of solution is critical in procedure 12.2.3. At this point in the procedure, all of the phosphonate has been oxidized to phosphate.

12.3 Color development and extraction procedure:

12.3.1 The timings specified in procedures 12.3.3, 12.3.4, and 12.3.7 are critical to the test. It is recommended to run small numbers of samples at a time in order to manage the timing.

12.3.2 Standard addition method (see 12.6) should be used for data quality control.

12.3.3 Add 20 mL MIBK/Cyclohexane solvent (8.8) and 10 mL ammonium molybdate solution (8.4) to the sample bottles, and immediately, vigorously shake each bottle for 15 s. At this point, the clear and electrically-neutral phosphomolybdate complex has been formed and extracted into the organic solvent phase.

12.3.4 Wait exactly five minutes to allow the aqueous and organic solvent phases to be separated, and withdraw 10.0 mL of liquid from the organic solvent layer into a clean 60-mL glass bottle using an automatic pipetter. Care should be taken not to disturb the solvent/water interface or accidentally withdraw some aqueous solution, since the excess molybdenum in the aqueous phase can also be reduced by stannous chloride to form a deep blue color.

12.3.5 Add 10 mL alcoholic H_2SO_4 solution (8.3) to the samples, and swirl to mix.

12.3.6 Add four drops stannous chloride solution (8.14) to each sample, and mix thoroughly.

12.3.7 After 10 minutes, but before 20 minutes, pour each sample into a 4-cm cell and read the absorbance against the blank at 725 nm. Absorbance readings also can be taken at 650 or 700 nm, but with reduced sensitivity. Use the sample blank as reference solution in measuring the sample.

12.3.8 Read the total phosphate concentration (C_T $-_{PO4}$) from a calibration curve prepared by analyzing known phosphate standards, as described in Section 11.

12.4 *Procedure for Analyzing Orthophosphate Concentration in the Brine:*

12.4.1 Pipet 20 mL of the acidified brine sample to a separate 60-mL glass bottle.

12.4.2 Follow the procedure in 12.3.3 – 12.3.7 to develop phosphomolybdate complex and to extract the complex to the organic liquid phase.

12.4.3 Read the orthophosphate concentration (C_F $-_{PO4}$) from a calibration curve prepared in Section 11.

12.5 Procedure for brines containing phosphonate concentrations outside the range(s) specified.

12.5.1 The above concentration range is specified for using a 4-cm light path cell. Longer light path cells are suitable for analyzing phosphonate at low concentrations (see the following):

Approximate Phosphonate Range (mg/L)	Light Path (cm)
0.1–2.0	10

12.5.2 Alternatively, the sample size can be adjusted to analyze brines containing low phosphonate concentration other than that specified in 12.1. An example of 100-mL sample size is given below.

12.5.2.1 Pipet 100 mL instead of 20 mL into a 240-mL bottle. The organic solvent phase in the 240-mL bottle will be a thin layer. Care should be taken not to disturb the solvent/water interface or accidentally withdraw aqueous solution when removing the phosphomolybdate complex from the organic solvent phase.

12.5.2.2 Add 1 g potassium persulfate (8.11) to the sample bottle.

12.5.2.3 Follow 12.2.2 – 12.3.8 to analyze for phosphate ion.

12.6 *Standard Additions Procedure:*

12.6.1 This procedure is recommended to determine the concentration of phosphonate in brine containing interfering components.

12.6.2 Prepare a blank and three samples, as in 12.2.1. Add 100 µL of 50 mg/L phosphonate standard solution (8.10) to one of the sample bottles. Add 200 µL of 50 mg/L phosphonate standard solution (8.10) to a second sample bottle.

12.6.3 Complete the procedures in 12.2.2 – 12.3.7 to digest phosphonate and to analyze for phosphate ion concentration.

12.6.4 Plot the absorbance versus concentration of added phosphonate. Draw a straight line through these three data points. Extend this line to intersect the X axis at a negative value of phosphonate concentration. The absolute value of this intersection is the concentration of phosphonate in the sample of interest.

13. Calculation

13.1 Calculate the phosphonate concentration in the sample (as mg/L PO_4) as follows:

$$\text{mg/L } PO_4 = \left[\left(C_{T\ -PO_4}\right) - \left(C_{F\ -PO_4}\right)\right]\left[\frac{\text{Volume of Standard}}{\text{Volume of Sample}}\right](\text{Field Dilution}) \quad (1)$$

where:

C_{T-PO_4} = Concentration of total phosphate (mg/L) read from calibration curve (see 12.3.8);
C_{F-PO_4} = Concentration of orthophosphate (mg/L) read from calibration curve (see 12.4.3);
Volume of Standard = Volume (mL) of standard used (see 12.2.1); and,
Volume of Sample = Volume (mL) of sample used (see 12.2.1, 12.5.2.1).

13.1.1 See 10.2 and as follows:

$$\text{Field Dilution} = \left(\frac{\text{Field Sample Volume(mL)} + \text{Acid Volume(mL)}}{\text{Field Sample Volume(mL)}}\right. \quad (2)$$

13.2 Use the following conversion factor to covert the mg/L PO_4, in Eq 2, to phosphonate:

$$\text{mg/L Phosphonate} = \frac{\text{mg/L } PO_4}{95\ \text{g/mol}} \times \frac{\text{Molecular Wt. of Phosphonate}}{\text{No. of phosphorus atoms phosphonate}} \quad (3)$$

13.2.1 For example, see Table 1.

14. Report

14.1 Report mg/L as phosphonate.

14.2 Report to one significant figure.

15. Precision and Bias

15.1 An interlaboratory study was conducted that involved eight laboratories analyzing samples at three different concentrations of phosphonate, each in a different brine concentration (see Table 2). The difference in brines was not expected to have any effect on the analytical results for phosphonate, but simulated different typical matrices. Each laboratory analyzed each sample in triplicate to provide a basis for estimating the single-operator standard deviation. It is recognized that the design of this study does not meet the requirements of D2777, but it is believed that the following statistical results are adequate to give the user's legitimate estimates of the precision and bias of the test method and for use as a basis for establishing generic quality control criteria to be used in the test method.

TABLE 1

Common Names[A]	Formula	Molecular Weight (g/mol)	No. of P-atoms/mole
~~ATMP, Dequest 2000®~~	~~$(H_2O_3PCH_2)_3N$~~	~~299 g/mol~~	~~3~~
~~DTPMP, Dequest 2060®~~	~~$\{(H_2O_3PCH_2)_2NCH_2CH_2\}_2N$-$CH_2PO_3H_2$~~	~~573 g/mol~~	~~5~~
~~HEPP, Dequest 2110®~~	~~$(H_2OO_3P)_2CCH_3OH$~~	~~206 g/mol~~	~~2~~

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HEPP, Dequest 2110[A]	$(H_2OO_3P)_2CCH_3OH$	206 g/mol	2

[A] ~~Dequest®~~ Dequest is a registered trade name of the Monsanto Company, St. Louis, MO 63167.

TABLE 2 Composition of Synthetic Brine Samples

	Brine 1, mg/L	Brine 2, mg/L	Brine 3, mg/L
NaCl	6.1	31	96.33
$CaCl_2$	3.9	15	54.47
$MgCl_2$	0.064	5	7.71
$CaCl_2 \cdot 2H_2O$	5.166	19.87	1696
$MgCl_2 \cdot 6H_2O$	0.166	10.674	386.3
Na_2SO_4	0.0739	0.037	0.0074
TDS, mg/L	10 000	51 000	157 000

15.2 Results from the interlaboratory study are given in Table 3. The outliers in Table 3 are underlined. Outliers were determined when a mean of replicates from a laboratory failed the *T*-test (see D2777) among related means or when an individual result failed the T test among related results. Statistical details are listed in Tables X1.1-X1.5 in the Appendix. Table 4 is a statistical data summary table of the interlaboratory study. The following statistical estimates were estimated from the retained data:

15.3 *Precision Estimates*—The overall and single operator precision for this test within the designed range is expressed as the following:

$$S_T = 0.0863*X + 0.0277 \quad (4)$$

The correlation coefficient for this equation is 0.97 (r^2).

$$S_O = 0.0303*X + 0.0129 \quad (5)$$

The correlation coefficient for this equation is 0.95 (r^2).

where:

S_T = overall precision;
S_O = single-operator precision; and,
X = true concentration of the phosphonate, mg/L.

15.4 *Bias Estimates*—The bias of the test method determined from the recoveries of known amounts of phosphonate ion in the synthetic brines is shown in Table 4.

15.5 Fig. 1 is a plot of the true concentration of PNA, mg/L versus mean concentration (outliers removed) of PNA, mg/L reported from the interlaboratory study. The unweighted least squares regression equation developed (Fig. 1) for mean concentration (XBAR) is as follows:

$$\text{XBAR} = 0.8848*X - 0.038 \quad (6)$$

The correlation coefficient for this equation is 0.0.96 (r^2).

where:

XBAR = mean concentration of PNA reported (outliers removed), mg/L; and,
X = true concentration of PNA, mg/L.

15.6 These collaborative test data were obtained on synthetic brine waters. For other matrices, these data may not apply. It is the user's responsibility to ensure the validity of this test method for waters of untested matrices.

15.7 Precision and bias for this test method conforms to Practice D2777–98, – 98, which was in place at the time of collaborative testing. Under the allowances made in 1.4 of D2777–08, – 13, these precision and bias data do meet existing requirements for interlaboratory studies of Committee D19 test methods.

TABLE 3 Results of Interlaboratory Study

Brine Matrix	1[A]			2[A]			3[A]		
True Concentration, PNA, mg/L	0.5	0.8	3	0.5	0.8	3	0.5	0.8	3
Laboratory	Reported Results mg/L								
A	0.35	0.84	1.34[A]	0.3	0.84	0.99	0.39	0.89	1.12[A]
B	0.24	0.83	2.58	0.28	0.83	2.6	0.18	0.86	2.56
C	0.3	0.9	2.8	0.3	0.9	2.8	0.3	0.9	2.7
D	0.2	0.7	3	0.1	0.7	3	0.1	0.7	3
E	0.33	0.88	2.46	0.3	0.76	2.13	0.35	0.84	2.55
G	0.36	0.95	2.71	0.37	0.95	2.7	0.37	0.94	2.71
H	0.19	0.48[A]	2.06	0.17	0.5	2.05	0.17	0.48[A]	2.1
I	0.3	0.83	2.7	0.35	0.85	2.5	0.39	0.83	2.8

[A] These results are outliers.