Designation: D7524 – 10 (Reapproved 2015)

IP 568/08

Standard Test Method for Determination of Static Dissipater Additives (SDA) in Aviation Turbine Fuel and Middle Distillate Fuels—High Performance Liquid Chromatograph (HPLC) Method[1]

This standard is issued under the fixed designation D7524; 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 determination of static dissipater additive (SDA) content of aviation turbine fuel and middle distillate fuels.

1.2 The precision of this test method has been established for aviation turbine fuel over the concentration range of 1 mg/L to 12 mg/L. Higher concentrations can be determined by dilution, but the precision of the test method will not apply.

NOTE 1—The SDA used to develop this test method was STADIS 450[2] for aviation fuels and STADIS 450 and 425[2] for middle distillates.

1.3 The test method includes a procedure to concentrate the sulfonic acid component in the SDA prior to analysis.

1.4 The test method only applies to SDAs that contain alkyl substituted sulfonic acid.

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

1.6 *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:*[3]

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products

2.2 *ISO Standards:*[4]

EN ISO 3696 Water for Analytical Laboratory Use—Specifications and Test Methods

2.3 *Energy Institute Standards:*[5]

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3. Terminology

3.1 *Definitions:*

3.1.1 *middle distillate fuels, n*—generic refinery/supplier term that usually denotes a fuel primarily intended for use in compression ignition/diesel engine applications, and also in non-aviation gas turbine engines and other non-automotive applications such as a burner fuel.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *aviation turbine fuel, n*—fuel used for powering jet and turbo-prop engine aircraft.

3.2.2 *conductivity improver additive, n*—material added to a fuel in very small amounts to increase its electrical conductivity and thereby reduce relaxation time.

3.2.2.1 *Discussion*—Conductivity improver additives are also known as static dissipater additives (SDAs) or antistatic additives.

4. Summary of Test Method

4.1 A solid phase extraction procedure is used to concentrate the sulfonic acid component of SDA present in an aviation turbine fuel or middle distillate fuel sample prior to analysis. A

[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.04.0C on Liquid Chromatography.

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[2] Stadis 450 and 425 are registered trademarks marketed by Innospec, Inc., Innospec Manufacturing Park, Oil Sites Road, Ellesmere Port, Cheshire Ch65 4EY, UK.

[3] 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.

[4] Available from International Organization for Standardization (ISO), 1, ch. de la Voie-Creuse, Case postale 56, CH-1211, Geneva 20, Switzerland, http://www.iso.ch.

[5] Available from Energy Institute, 61 New Cavendish St., London, WIG 7AR, U.K., http://www.energyinst.org.uk.

fixed volume of the concentrated test fraction is injected into a calibrated high performance liquid chromatograph. An analytical column is used to separate the sample components of the test fraction by polarity.

4.2 The analytical column is attached to a liquid chromatography detector where the sulfonic acid components are readily detected by UV absorption as they elute from the column. The electronic signal from the liquid chromatography detector is continually monitored by a chromatography data system. The amplitudes of the signal (peak area) from the sulfonic acids are compared with those obtained from previously measured calibration standards in order to calculate the percent m/V SDA present in the sample.

4.3 The test method only applies to SDAs that contain alkyl substituted sulfonic acids.

5. Significance and Use

5.1 This test method will allow the determination of static dissipater additive in jet and middle distillate. These additives reduce the hazardous effects of static electricity generated by transfer and movement of jet and middle distillate fuels.

6. Apparatus

6.1 *High Performance Liquid Chromatograph (HPLC)*—Any HPLC capable of pumping an isocratic mobile phase at flow rates between 0.1 mL/min and 1.5 mL/min, with a precision better than 0.5 % and a pulsation of < 1 % full scale deflection under the test method conditions.

6.2 *Variable Wavelength Ultraviolet Photometric Detector or Photometric Diode Array Detector*—Capable of operation at 225 nm and 234 nm.

6.3 *Manual or Automatic Sample Injection Valve*—Capable of injecting 10 µL to 25 µL, using either partial or full loop mode, with a repeatability ±1 %.

6.3.1 An equal and constant volume of the calibration and sample solutions is injected into the chromatograph. Both manual and automatic sample injection systems (using either complete or partial filling of the sample loop) will, when used correctly, meet the repeatability requirements specified in 6.3.

NOTE 2—When using the partial loop-filling mode, it is recommended that the injection volume should be less than half the total loop volume. For complete filling of the loop, best results are obtained by overfilling the loop at least six times.

6.4 *Chromatography Data System*—Any data system can be used, provided it is compatible with the liquid chromatography detector, has a minimum sampling rate of 1 Hz, and is able to measure peak areas and retention times and perform post-run data processing such as baseline correction and re-integration.

6.5 *Analytical Column*[6]—Any stainless steel HPLC column packed with C_6 alkyl-bonded reversed-phase, 5 µm particle size, 250 mm × 4.6 mm ID is suitable, provided that it meets the resolution requirements specified in 9.3.

6.6 *HPLC Column Oven*—Any suitable HPC column oven block heating or air circulating) capable of maintaining a constant temperature of ±1 °C within the range from 20 °C to 40 °C.

NOTE 3—Alternative forms of temperature control are permitted, for example, temperature-controlled laboratories.

6.7 *Analytical Balance*—Accurate to ±0.0001 g.

6.8 *Solid Phase Extraction (SPE) Columns Reservoirs*[6]—Amino bonded silica, 6500 mg (A) or 100 mg (B) capacity.

6.8.1 *Reservoirs with Connectors*—Approximately 60 mL capacity and connectors.

6.9 *Solid Phase Extraction Vacuum Manifold*—Optional.

6.10 *Volumetric Flasks*—Class A, of 2 mL, 5 mL, 10 mL, 25 mL, and 100 mL capacity.

6.11 *Graduated Pipette*—Class A, of 1 mL, 2 mL, 10 mL, and 50 mL capacity. Capable of delivering volumes of the range 0.5 mL to 4.0 mL with an accuracy of ±0.0005 mL.

6.12 *Measuring Cylinder*—50 mL and 500 mL capacity.

6.13 pH meter.

7. Reagents and Materials

7.1 *Dinonylnaphthalene Sulfonic Acid (DINNSA)*—50 % (m/m) solution in heptane.

7.2 *Dodecylbenzene Sulfonic Acid (DDBSA)*—70 % (m/m) solution in 2-propanol.

7.3 *Tetrahydrofuran*—HPLC grade. (**Warning—**HPLC grade tetrahydrofuran does not contain inhibitor, hence explosive peroxides may form. Highly flammable and may cause irritation by inhalation, ingestion or skin contact.)

7.4 *Hydrochloric Acid*—37 %.

7.5 *Methanol*—HPLC grade. (**Warning—** Methanol is highly flammable and toxic by inhalation, ingestion or skin contact.)

7.6 *Methanolic Hydrochloric Acid*—Mix approximately 1 mL of hydrochloric acid (see 7.4) with approximately 9 mL of methanol (see 7.5).

7.7 *Orthophosphoric Acid.*

7.8 *Sodium Hydroxide Pellets.*

7.9 *Sodium Hydroxide Solution*—Approximately 1 M. Dissolve approximately 4 g of sodium hydroxide (see 7.8) in approximately 100 mL of water.

7.10 *Buffered Phosphoric Acid*—Add approximately 2 mL of orthophosphoric acid (see 7.7) to approximately 11 mL of water and buffer to 2.5 pH using sodium hydroxide solution (see 7.9).

7.11 *Isohexane*—HPLC grade. (**Warning—** Isohexane is highly flammable, and may cause irritation by inhalation, ingestion or skin contact.)

7.12 *Solid Phase Extraction (SPE) Columns and Cartridges*—Amino-bonded silica, 500 mg and 100 mg.

[6] The sole source of supply of the apparatus known to the committee at this time is Waters Corp. 34 Maple St., Milford, MA 01757. 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.

7.13 *Mobile Phase*—Mix approximately 400 mL methanol (see 7.5), approximately 400 mL THF (see 7.3), and approximately 50 mL of buffered phosphoric acid (see 7.10).

7.14 *Nitrogen*—Optional.

NOTE 4—It is recommended practice to degas HPLC mobile phase before use; this can be done conveniently, on-line or off-line, by helium sparging, vacuum degassing, or ultrasonic agitation. A failure to degas the mobile phase may lead to negative peaks.

7.15 *Calibration Stock Solutions*—Accurately weigh, to the nearest 0.0001 g, between 0.078 g and 0.082 g of DINNSA solution (7.1) into a 100 mL volumetric flask and make up to the mark with heptane or mobile phase (7.13). Accurately weigh, to the nearest 0.0001 g, between 0.028 g to 0.032 g of DDBSA solution (7.2) into a 100 mL volumetric flask and make up to the mark with heptane or mobile phase (7.13).

7.16 *Calibration Standards*—From the calibration stock solutions (7.15), prepare a set of five calibration standards in the mobile phase (7.13) to cover the concentration ranges indicated in the table:

SPE Column Size	DINNSA	DDBSA
500 mg	0.8 mg/mL to 16 mg/mL	0.7 mg/mL to 8.5 mg/mL
100 mg	0.32 mg/mL to 9 mg/mL	0.35 mg/mL to 4.5 mg/mL

NOTE 5—A suggested procedure to prepare these standards is given in Annex A1.

7.17 *Calibration Check Solutions*—2.0 mg/L DINNSA and 2.8 mg/L DBSA.

7.17.1 Prepare a 500 mg/L DINNSA solution by accurately weighing, to the nearest 0.0001 g, about 0.05 g DINNSA (7.1) into a 50 mL volumetric flask and making up to the mark with heptane. Pipette 1 mL of this solution into a 25 mL volumetric flask and make up to the mark with heptane (20 mg/L DINNSA solution). Pipette 1 mL of the 20 mg/L DINNSA solution into a 10 mL volumetric flask and make up to volume with mobile phase (7.13) to give a 2.0 mg/L check solution.

7.17.2 Prepare a 700 mg/L DDBSA solution by accurately weighing, to the nearest 0.0001 g, 0.05 g DDBSA (7.2) into a 50 mL volumetric flask and making up to the mark with heptane. Pipette 1 mL of this solution into a 25 mL volumetric flask and make up to the mark with heptane (28 mg/L DDBSA solution). Pipette 1 mL of the 28 mg/L DDBSA solution into a 10 mL volumetric flask and make up to volume with mobile phase (7.13) to give a 2.8 mg/L check solution.

7.18 *Water*—Grade 3 of EN ISO 3696.

8. Sampling

8.1 Use only representative samples obtained as described in Practice D4057 or D4177, unless otherwise specified.

9. Sample Preparation

9.1 Clamp the SPE column, 6.8(A) or 6.8(B), vertically or attach to a vacuum manifold if used. Add a 60 mL reservoir to the 500 mg SPE column (6.8(A)) using a connector (6.8.1); the 100 mg SPE column has an integral reservoir.

9.2 With a pipette, transfer the test specimen (see Table 1) to the SPE reservoir and allow the fuel to percolate through the column under gravity or vacuum at a flow rate of 2 mL/min or less. Discard the eluate.

TABLE 1 SPE Sample, Wash, and Elution Volumes

	500 mg SPE Column	100 mg SPE Column
Sample volume	50 mL	10 mL
Isohexane/heptane wash	2 mL × 5 mL	2 mL × 2 mL
Methanol wash	5 mL	2 mL
DINNSA/DDBSA eluate	5 mL	2 mL

9.3 After all the fuel has eluted from the SPE column, rinse the reservoir and SPE adsorbent with portions of isohexane (or heptane) and then methanol, discarding the eluate (see Table 1).

9.4 Elute the sulfonic acid (DINNSA or DDBSA) from the 100 mg or 500 mg SPE column using approximately 2 mL or 5 mL of mobile phase (7.13), respectively, and collect the eluate in a 2 mL or 5 mL volumetric flask (6.10), making up to the mark with mobile phase (7.13) if necessary. Replace stopper in volumetric flask and shake well.

9.5 Sample and eluent volumes are summarized in Table 1.

10. Preparation of Apparatus

10.1 Set up the pump, injector, detector, and data system according to the manufacturer's instructions. Set the UV detector to 234 nm for DINNSA and 225 nm for DDBSA.

10.2 Install the HPLC column and set the mobile phase flow rate to 0.5 mL/min.

NOTE 6—Set the temperature of the column oven, if used, to at least 10 °C above ambient, for example, 40 °C.

10.3 When operating conditions are steady, inject a fixed volume (10 µL to 25 µL) of the middle calibration standard (7.16), and ensure the chromatogram resembles those shown in Fig. 1 (DINNSA) or Fig. 2 (DDBSA).

NOTE 7—The sulfonic acid peak around 4 min may exhibit some broadening due to the presence of isomers but no fine structure.

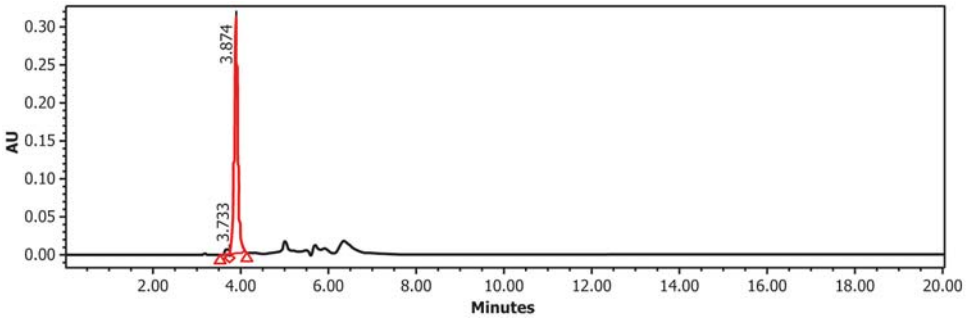
NOTE 8—Small adjustments in the buffered phosphoric acid content of the mobile phase may improve the chromatographic profile of the sulfonic acid.

10.4 Inject a fixed volume (10 µL to 25 µL) of the lowest concentration calibration standard (7.16) to check system sensitivity. The detector shall have a signal to noise ratio (S/N) greater than 10 for the lowest concentration calibration standard; increase the injection volume if necessary.

10.5 Inject the same volume (10 µL to 25 µL) of middle calibration standard (7.16) five times to check system repeatability (less than 1 %).

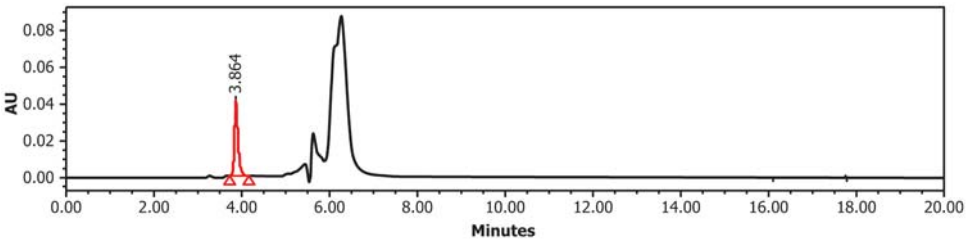
10.6 Inject the same volume (10 µL to 25 µL) of all calibration standards (7.16) to check system linearity (correlation co-efficient greater than 0.995).

10.7 Inject the same (10 µL to 25 µL) of the highest concentration standard (7.16) followed by blank (mobile phase) injection to check for analyte carryover (less than 0.05 %).



NOTE 1—Detector wavelength set at 234 nm.

FIG. 1 Chromatogram of DINNSA Calibration Standard



NOTE 1—Detector wavelength set at 225 nm.

FIG. 2 Chromatogram of DDBSA Calibration Standard

10.8 *Determination of Injection Volume*—When operating conditions are steady, as indicated by a stable horizontal baseline of the liquid chromatography detector, inject a fixed volume (10 µL to 25 µL) of the lowest calibration standard (E) and record the chromatogram with the chromatography data system to check system sensitivity. Select a fixed injection volume that provides a detector response (that is, signal to baseline noise ratio) of at least 10 for standard (E). Use the same selected injection volume for all analyses in this test method.

NOTE 9—At a mobile phase flow rate of 0.5 mL/min, the analysis time should be approximately 20 min.

10.9 *Evaluate Injection System Repeatability*—Inject a fixed volume of middle calibration (C) standard five times, recording each chromatogram with the chromatography data system to check system repeatability. Use Fig. 1 to help identify the DINNSA peak in each recorded chromatogram. Measure the area of the DINNSA peak of each recorded chromatogram and ensure that the repeatability for the calibration standard (C) is within ±1 %.

NOTE 10—If repeatability is poor, check to see that the injection system is working optimally and the baseline is stable (minimal drift) and noise-free.

10.10 *Check for Contamination between Sample Injections*—Inject a fixed volume of highest concentration standard (A) followed by blank (mobile phase) to check for analyte carryover. Record each of these chromatograms with the data system. Ensure that there is no response for DINNSA in the blank mobile phase injection.

NOTE 11—If a detector response for DINNSA is found in the blank injection then evaluate, fix or optimize the sample injection system according to the manufacturer's suggestions.

10.11 *Check for System Linearity*—Inject a fixed volume (10 µL to 25 µL) of all calibration standards (7.16) to check system linearity (correlation coefficient should be greater than 0.995).

10.12 *Construction of Calibration Graph*—Inject a fixed volume (10 µL to 25 µL) of all the calibration standards and integrate the peak areas using the data system. Construct the calibration graph by plotting DINNSA (or DDBSA) concentration (mg/L) as *y*-axis and peak areas along *x*-axis.

11. Procedure

11.1 *Calibration:*

11.1.1 Inject a fixed volume of DINNSA calibration standards (A to E), and measure peak areas with data system.

11.1.2 Construct a calibration graph by plotting DINNSA concentration (mg/L) as *y*-axis and peak areas along *x*-axis. A linear calibration plot is required with a correlation coefficient greater than 0.999 and an intercept of less than ±0.01 g/100 mL.

NOTE 12—A computer or a data system may be used to interpret these calibrations. It should only be necessary to calibrate the detector on a daily basis. It is recommended that a reference sample or one of the calibration standards be run after every ten samples to check the stability of the chromatographic system.

11.1.3 Ensure that the liquid chromatography detector wavelength is set for 225 nm. Inject a fixed volume of DDBSA calibration standards (F to J); record and measure peak areas with data system. An example chromatograph of a DDBSA calibration standard is shown in Fig. 2.

11.1.4 Construct a calibration graph by plotting DDBSA concentration (mg/L) as *y*-axis and peak areas along *x*-axis. A linear calibration plot is required with a correlation coefficient greater than 0.999 and an intercept of less than ± 0.01 g/100 mL.

11.2 *Analysis of Samples:*

NOTE 13—It is recommended samples are run in duplicate by taking a fresh test specimen and repeating the full test procedure from 9.1 onwards. Duplicate values should be within the repeatability of this test method, and the mean value should be reported. If duplicate values fall outside the repeatability of the test method then a verification fuel sample (for example, Jet A1 spiked with a known amount of SDA) should be prepared and analyzed by the full test procedure. It is also recommended that a verification fuel sample be used periodically for quality assurance purposes.

11.2.1 Inject same fixed volume of the test portion, and measure the peak area using the data system.

11.2.2 Repeat 10.3 for all samples.

11.2.3 At least every five samples, inject the same volume of mobile phase as a blank.

11.2.4 At least every ten samples, inject the same volume of the calibration check standard (7.16).

11.2.5 Check that the sample chromatograms for DINNSA or DDBSA are similar to Fig. 1 or Fig. 2, respectively.

12. Calculation

12.1 Compare test portion peak areas with calibration graph and calculate concentration of sulfonic acid in accordance with the following equations:

12.2 *Sulfonic Acid Concentration (DINNSA-based):*

$$\text{Sulfonic acid concentration (DINNSA based)} = \frac{\text{Concentration from calibration graph of DINNSA mg/L}}{\text{Concentration Factor}} \quad (1)$$

where:

Concentration factor = 10 for 500 mg SPE columns.
Concentration factor = 5 for 100 mg SPE columns.

$$\text{Concentration of SDA} = \text{sulfonic acid concentration} \times 7.952 \quad (2)$$

where:

7.952 = SDA formulation/standard factor.

12.3 *Sulfonic Acid Concentration (DDBSA-based):*

$$\text{Sulfonic acid concentration (DDBSA based)} = \frac{\text{Concentration from calibration graph of DDBSA mg/L}}{\text{Concentration Factor}} \quad (3)$$

where:

Concentration factor = 10 for 500 mg SPE columns.
Concentration factor = 5 for 100 mg SPE columns.

$$\text{Concentration of SDA} = \text{sulfonic acid concentration} \times 14.29 \quad (4)$$

where:

14.29 = SDA formulation/standard factor.

TABLE 2 Precision Values

Range % (m/m)	Repeatability	Reproducibility
0.4–11.8	0.5991	1.779

13. Report

13.1 Report the SDA content in mg/L to the nearest 0.1 mg/L.

13.2 Report the following information in the test report:

13.2.1 A reference to Test Method D7524.

13.2.2 The type and the complete identification of the product tested.

13.2.3 The result of the test (see Section 11).

13.2.4 Any deviation, by agreement or otherwise, from the procedure specified.

13.2.5 The date of the test.

14. Precision and Bias[7]

14.1 The precision was obtained by statistical analysis of results from an interlaboratory study involving nine laboratories and eleven fuels (including nine aviation turbine fuels and two ULSD).

14.2 *Precision*—The following criteria should be used for judging the acceptability of results (95 % probability).

14.3 *Repeatability*—The difference between two results obtained by the same operator on the same apparatus under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following values given in Table 2 only in one case in twenty.

Range	Repeatability
0.4 – 11.8	0.5991

14.4 *Reproducibility*—The difference between two single and independent results obtained by different operators working in different laboratories on identical test materials would, in the long run, in the normal and correct operation of the test method, exceed the following values given in Table 2 only in one case in twenty.

Range	Reproducibility
0.4–11.8	1.1779

NOTE 14—Note that the precision study only applies to DINNSA based SDA.

14.5 *Bias*—No information can be presented on the bias of the procedure in Test Method D7524 for measuring static dissipater additives in aviation turbine fuels or middle distillates because no material having an accepted reference value is available.

15. Keywords

15.1 aviation turbine fuel; high performance liquid chromatography; middle distillate; solid phase extraction; static dissipater additive

[7] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1685.

ANNEX

(Mandatory Information)

A1. RECOMMENDED PROCEDURE FOR MAKING UP CALIBRATION STANDARDS

A1.1 DINNSA Calibration Solutions

NOTE A1.1—SDA contains 12.58 % (m/m) DINNSA.

A1.1.1 Pipette 1 mL DINNSA stock solution (7.1) into a 25 mL volumetric flask and make up to the mark with mobile phase (7.13)—Calibration Standard A (about 16 mg/L).

A1.1.2 Pipette 1 mL DINNSA stock solution (7.1) into a 50 mL volumetric flask and make up to the mark with mobile phase (7.13)—Calibration Standard B (about 8 mg/L).

A1.1.3 Pipette 2 mL Calibration Standard A (7.16) into a 10 mL volumetric flask and make up to the mark with mobile phase (7.13)—Calibration Standard C (about 3.2 mg/L).

A1.1.4 Pipette 1 mL Calibration Standard A (7.16) into a 10 mL volumetric flask and make up to the mark with mobile phase (7.13)—Calibration Standard D (about 1.6 mg/L).

A1.1.5 Pipette 1 mL Calibration Standard B (7.16) into a 10 mL volumetric flask and make up to the mark with mobile phase (7.13)—Calibration Standard E (about 0.8 mg/L).

A1.1.6 Pipette 1 mL Calibration Standard C (7.16) into a 10 mL volumetric flask and make up to the mark with mobile phase (7.13)—Calibration Standard F (about 0.32 mg/L).

A1.1.7 For 500 mg SPE columns and 50 mL test specimens, use calibration standards A, B, C, D, E.

A1.1.8 For 100 mg SPE columns and 10 mL test specimens, use calibration standards B, C, D, E, F.

A1.2 DDBSA Calibration Solutions

NOTE A1.2—SDA contains 7.0 % (m/m) DDBSA.

A1.2.1 Pipette 1 mL DDBSA stock solution (7.2) into a 25 mL volumetric flask and make up to the mark with mobile phase (7.13)—Calibration Standard A (about 7.4 mg/L).

A1.2.2 Pipette 1 mL DDBSA stock solution (7.2) into a 50 mL volumetric flask and make up to the mark with mobile phase (7.13)—Calibration Standard B (about 3.9 mg/L).

A1.2.3 Pipette 2 mL Calibration Standard A (7.16) into a 10 mL volumetric flask and make up to the mark with mobile phase (7.13)—Calibration Standard C (about 1.68 mg/L).

A1.2.4 Pipette 1 mL Calibration Standard A (7.16) into a 10 mL volumetric flask and make up to the mark with mobile phase (7.13)—Calibration Standard D (about 0.84 mg/L).

A1.2.5 Pipette 1 mL Calibration Standard B (7.16) into a 10 mL volumetric flask and make up to the mark with mobile phase (7.13)—Calibration Standard E (about 0.78 mg/L).

A1.2.6 Pipette 1 mL Calibration Standard C (7.16) into a 10 mL volumetric flask and make up to the mark with mobile phase (7.13)—Calibration Standard E (about 0.16 mg/L).

A1.2.7 For 500 mg SPE columns and 50 mL test specimens, use calibration standards A, B, C, D, E.

A1.2.8 For 100 mg SPE columns and 10 mL test specimens, use calibration standards B, C, D, E, F.