Cover Sheet for

Environmental Chemistry Method

Pesticide Name: **Pyroxsulam (XDE 742)**

MRID#: **469085-01**

Matrix: **Water**

Analysis: **LC/MS/MS**

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If you have difficulties in downloading the method, or further questions concerning the methods, you may contact Elizabeth Flynt at 228-688-2410 or via email at flynt.elizabeth@epa.gov.

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Dow AgroSciences

Determination of Residues of XDE-742 and its Metabolites in Drinking Water, Ground Water, and Surface Water by Liquid Chromatography with Tandem Mass Spectrometry

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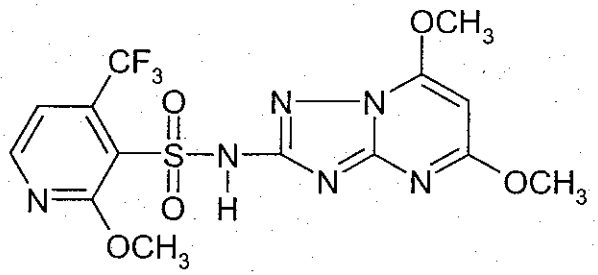
1. SCOPE

This method is applicable for the quantitative determination of residues of XDE-742 and its metabolites in drinking water, ground water, and surface water. The method was validated over the concentration range of 0.05-5.0 µg/L with a validated limit of quantitation of 0.05 µg/L.



XDE-742
CAS No. 422556-08-9

Common and chemical names, molecular formulas, and the nominal masses for XDE-742 and its metabolites are given in Table 1.

2. PRINCIPLE

Residues of XDE-742 and its metabolites are analyzed without need for prior extraction, concentration or cleanup. The parent compound, XDE-742 and its 7-OH-XDE-742, ADTP and ATSA metabolites are analyzed directly by liquid chromatography with positive-ion electrospray tandem mass spectrometry (LC/MS/MS). The XDE-742 sulfinic acid and sulfonic acid metabolites are analyzed by liquid chromatography with negative-ion electrospray tandem mass spectrometry.

3. SAFETY PRECAUTIONS

3.1. Each analyst must be acquainted with the potential hazards of the reagents, products, and solvents used in this method before commencing laboratory work. SOURCES OF INFORMATION INCLUDE: MATERIAL SAFETY DATA SHEETS, LITERATURE, AND OTHER RELATED DATA. Safety information on non Dow AgroSciences LLC products should be obtained from the container label or from the supplier. Disposal of reagents, reactants, and solvents must be in compliance with local, state, and federal laws and regulations.

3.2. Acetonitrile and methanol are flammable and should be used in well-ventilated areas away from ignition sources.

3.3. Formic acid is corrosive and can cause severe burns. Liquid nitrogen can also cause severe burns. It is imperative that proper eye and personal protection equipment be used when handling all chemicals.

4. EQUIPMENT (Note 12.1.)

4.1. Laboratory Equipment

4.1.1. Balance, analytical, Model AE100, Mettler-Toledo Inc., Columbus, OH 43240.

4.1.2. Pipetter, adjustable, Eppendorf, 10-100 µL, catalog number 05-402-48, Brinkmann Instruments, Inc., Westbury, NY 11590. (Note 12.2.)

4.1.3. Pipetter, adjustable, Eppendorf, 50-1000 µL, catalog number 21-378-83, Brinkmann Instruments, Inc. (Note 12.2.)

4.1.4. Pipetter, adjustable, Eppendorf, 1000-5000 µL, catalog number 22-46-134-6, Brinkmann Instruments, Inc. (Note 12.2.)

4.1.5. Pipet, positive-displacement, 10-100 µL capacity, model number M100, catalog number F148504, Gilson Inc., Middleton, WI 53562.

4.1.6. Pipet, positive-displacement, 100-1000 µL capacity, model number M1000, catalog number F148506, Gilson Inc.

4.1.7. Ultrasonic cleaner, Model 1200, Branson Cleaning Equipment Company, Shelton, CT 06484.

4.1.8. Vortex mixer, Model G-560, Scientific Industries Inc., Bohemia, NY 11716.

4.2. Chromatographic System

4.2.1. Column, analytical, Synergi Hydro RP 80A, 2.0 x 50 mm, 4.0-µm, catalog number 008-4252-B0, Phenomenex, Torrance, CA 90501.

4.2.2. Liquid chromatograph, Symbiosis Pharma, Spark Holland Inc., Plainsboro, NJ 08536.

4.2.3. Mass spectrometer, Model API 4000, Applied Biosystems, Foster City, CA 94404.

4.2.4. Mass spectrometer data system, Analyst 1.4.1, Applied Biosystems.

5. GLASSWARE AND MATERIALS (Note 12.1.)

5.1. Bottle, 1.0-L, media bottle, catalog number 06-423-3D, Fisher Scientific, Pittsburgh, PA 15275.

5.2. Collection plate, 96-well, 2-mL, catalog number 121-5203, Argonaut Technologies, Inc., Redwood City, CA 94063.

5.3. Collection plate sealing cap, catalog number 121-5205, Argonaut Technologies, Inc.

5.4. Flask, volumetric, 100-mL, catalog number 5640-100, Corning Inc., Acton, MA 01720.

5.5. Flask, volumetric, 200-mL, catalog number 5640-200, Corning Inc.

5.6. Cylinder, graduated, 100-mL, catalog number 20024-100, Kimble/Kontes, Vineland, NJ 08360.

5.7. Cylinder, graduated, 500-mL, catalog number 20024-500, Kimble/Kontes.

5.8. Cylinder, graduated, 1000-mL, catalog number 20024-1000, Kimble/Kontes.

5.9. Pipet, polyethylene disposable transfer, 3-mL, catalog number, 13-711-7, Fisher Scientific.

5.10. Pipet, volumetric, 3.0-mL, catalog number 13-650-2D, Fisher Scientific Company.

5.11. Pipet, volumetric, 5.0-mL, catalog number 13-650-2F, Fisher Scientific Company.

5.12. Pipet, volumetric, 9.0-mL, catalog number 13-650-2K, Fisher Scientific Company.

5.13. Pipet, volumetric, 10.0-mL, catalog number 13-650-2L, Fisher Scientific Company.

5.14. Pipet, volumetric, 20.0-mL, catalog number 13-650-2N, Fisher Scientific Company.

5.15. Pipet, volumetric, 30.0-mL, catalog number 13-650-2Q, Fisher Scientific Company.

5.16. Pipetter tips, Brinkmann Eppendorf, 1-200-µL tip, catalog number 22351371, Brinkmann Instruments, Inc.

5.17. Pipetter tips, Brinkmann Eppendorf, 300-µL tip, catalog number 22351419, Brinkmann Instruments, Inc.

5.18. Pipetter tips, Brinkmann Eppendorf, 1000-µL tip, catalog number 22350901, Brinkmann Instruments, Inc.

5.19. Pipet tip, positive-displacement, 100-µL capacity, capillary piston number CP100, catalog number F148414, Gilson Inc.

5.20. Pipet tip, positive-displacement, 1000-µL capacity, capillary piston number CP1000, catalog number F148560, Gilson Inc.

5.21. Vial, autosampler, 2-mL, catalog number C4000-1W, National Scientific Company, Duluth, GA 30097.

5.22. Vial, 40-mL, with PTFE-lined screw cap, catalog number B7800-6, National Scientific Company.

5.23. Vial cap, for autosampler vial, catalog number C4000-54B, National Scientific Company.

6. REAGENTS, STANDARDS, AND PREPARED SOLUTIONS (Note 12.1.)

6.1. Reagents

6.1.1. Acetonitrile, ChromAR HPLC grade, catalog number 2856, Mallinckrodt Baker, Inc., Paris, KY 40361.

6.1.2. AmQuel+Plus®, instant water detoxifier, item number 33411 (1 oz.) or 33444 (4 oz.), Kordon® (Division of Novalek, Inc.), Hayward, CA 94545-1114. (Note 12.3.)

6.1.3. Formic acid, ACS reagent grade, 96% purity, catalog number 25,136-4, Sigma-Aldrich Inc., St. Louis, MO 63103.

6.1.4. Methanol, ChromAR HPLC grade, catalog number 3041, Mallinckrodt-Baker Inc.

6.1.5. Nitrogen, refrigerated liquid, catalog number LQNI, BOC Gases, New Providence, NJ 07974.

6.1.6. Water, OmniSolv grade, catalog number WX-0004-1, EMD Chemicals Inc., Gibbstown, NJ 08027.

6.2. Standards

6.2.1. XDE-742, *N*-(5,7-dimethoxy[1,2,4]triazolo[1,5-*a*]pyrimidin-2-yl)-2-methoxy-4-(trifluoromethyl)-3-pyridinesulfonamide

6.2.2. 7-OH-XDE-742, *N*-(7-hydroxy-5-methoxy[1,2,4]triazolo[1,5-*a*]pyrimidin-2-yl)-2-methoxy-4-(trifluoromethyl)-3-pyridinesulfonamide

6.2.3. ADTP metabolite of XDE-742, 5,7-dimethoxy[1,2,4]triazolo[1,5-*a*]pyrimidin-2-amine

6.2.4. ATSA metabolite of XDE-742, *N*-(5-amino-1H-1,2,4-triazol-3-yl)-2-methoxy-4-(trifluoromethyl)pyridine-3-sulfonamide

6.2.5. sulfinic acid metabolite of XDE-742, 2-methoxy-4-(trifluoromethyl)pyridine-3-sulfinic acid

6.2.6. sulfonic acid metabolite of XDE-742, 2-methoxy-4-(trifluoromethyl)pyridine-3-sulfonic acid

Obtain all of the above from the Test Substance Coordinator, Dow AgroSciences LLC, 9330 Zionsville Road, Building 304, Indianapolis, IN 46268.

6.3. Prepared Solutions

6.3.1. acetonitrile + 0.3% formic acid (initial sample diluent)

Measure 1000 mL of acetonitrile using a 1-L graduated cylinder and transfer to a 1-L bottle. Pipet 3.0 mL of formic acid (96%) into the same 1-L bottle. Cap the bottle and mix. Allow the solution to equilibrate to room temperature before use.

6.3.2. acetonitrile + 0.01% formic acid (mobile phase B)

Measure 1000 mL of acetonitrile using a 1-L graduated cylinder and transfer to a 1-L bottle. Pipet 0.1 mL of formic acid (96%) into the same 1-L bottle. Cap the bottle and mix. Allow the solution to equilibrate to room temperature before use.

6.3.3. acetonitrile/water (80:20) + 0.01% formic acid (autosampler wash solution)

Measure 800 mL of acetonitrile using a 1-L graduated cylinder and transfer to a 1-L bottle. Measure 200 mL of HPLC-grade water using a 500-mL graduated cylinder and transfer to the same 1-L bottle. Pipet 0.1 mL of formic acid (96%) into the 1-L bottle. Cap the bottle and mix. Allow the solution to equilibrate to room temperature before use.

6.3.4. AmQuel+Plus, 12.5-µl/mL aqueous dilution (instant water detoxifier)

Pipet 10 mL of HPLC-grade water into a 40-mL vial. Pipet 125 µL of the AmQuel+Plus product into the same vial. Cap the sample vial with a PTFE-lined cap and vortex mix for 5-10 seconds.

6.3.5. water + 0.01% formic acid (mobile phase A)

Measure 1000 mL of HPLC-grade water using a 1-L graduated cylinder and transfer to a 1-L bottle. Pipet 0.1 mL of formic acid (96%) into the same 1-L bottle. Cap the bottle and mix. Allow the solution to equilibrate to room temperature before use.

6.3.6. water + 0.03% formic acid (final diluent for the calibration standards)

Measure 1000 mL of HPLC-grade water using a 1-L graduated cylinder and transfer to a 1-L bottle. Pipet 0.3 mL of formic acid (96%) into the same 1-L bottle. Cap the bottle and mix. Allow the solution to equilibrate to room temperature before use.

6.3.7. water/acetonitrile (90:10) + 0.03% formic acid (used for final sample dilution when further dilution is needed) (Note: This solution will only need to be prepared for sample dilution if the analyte concentration is found to be outside the range of the calibration curve.)

Measure 900 mL of HPLC-grade water using a 1-L graduated cylinder and transfer to a 1-L bottle. Measure 100 mL of acetonitrile using a 100-mL graduated cylinder and transfer to the same 1-L bottle. Pipet 0.3 mL of formic acid (96%) into a 1-L bottle. Cap the bottle and mix. Allow the solution to equilibrate to room temperature before use.

7. PREPARATION OF STANDARDS (Note 12.4. and 12.5.)

7.1. Preparation of Fortification Solutions

7.1.1. Weigh 0.0100 g of XDE-742 analytical standard and quantitatively transfer to a 100-mL volumetric flask with methanol. Dilute to volume with methanol to obtain a 100-µg/mL stock solution.

7.1.2. Weigh 0.0100 g of 7-OH-XDE-742 analytical standard and quantitatively transfer to a 100-mL volumetric flask with methanol. Dilute to volume with methanol to obtain a 100-µg/mL stock solution.

7.1.3. Weigh 0.0100 g of ADTP metabolite of XDE-742 analytical standard and quantitatively transfer to a 100-mL volumetric flask with methanol. Dilute to volume with methanol to obtain a 100-µg/mL stock solution.

7.1.4. Weigh 0.0100 g of ATSA metabolite of XDE-742 analytical standard and quantitatively transfer to a 100-mL volumetric flask with methanol. Dilute to volume with methanol to obtain a 100-µg/mL stock solution.

7.1.5. Weigh 0.0102 g (corrected for the molecular weight as the lithium salt) of sulfinic acid metabolite of XDE-742, lithium salt analytical standard and quantitatively transfer to a 100-mL volumetric flask with methanol. Dilute to volume with methanol to obtain a 100-µg/mL stock solution.

7.1.6. Weigh 0.0100 g of sulfonic acid metabolite of XDE-742 analytical standard and quantitatively transfer to a 100-mL volumetric flask with methanol. Dilute to volume with methanol to obtain a 100-µg/mL stock solution.

7.1.7. Pipet 10.0 mL of each of the 100-µg/mL solutions prepared in Sections 7.1.1 through 7.1.6 into a single 200-mL volumetric flask and dilute to volume with acetonitrile to obtain a mixed stock solution containing 5.0 µg/mL of each compound.

7.1.8. Pipet 10.0 mL of the 5.0-µg/mL solution prepared in Section 7.1.7 into a 100-mL volumetric flask and dilute to volume with acetonitrile to obtain a mixed fortification solution containing 0.50 µg/mL of each compound.

7.1.9. Pipet 10.0 mL of the 0.50-µg/mL mixed solution in Section 7.1.8 into a 100-mL volumetric flask and dilute to volume with acetonitrile to obtain a 0.050-µg/mL mixed fortification solution.

7.1.10. Pipet 10.0 mL of the 0.05-µg/mL solution in Section 7.1.9 into a 100-mL volumetric flask and dilute to volume with acetonitrile to obtain a 0.005-µg/mL mixed fortification solution.

7.1.11. Prepare fortification solutions by diluting the above stock solutions from Sections 7.1.8-7.1.10 with acetonitrile as follows:

Concentration of Stock Soln.	Aliquot of Stock Soln.	Final Soln. Volume	Spiking Soln. Final Conc.	Equivalent Sample Conc.[a]
µg/mL	mL	mL	µg/mL	µg/L
0.005	30.0	100	0.0015	0.015
0.005	- - -	- - -	0.005	0.050
0.050	- - -	- - -	0.050	0.500
0.500	- - -	- - -	0.500	5.00

[a] The equivalent sample concentration is based on fortifying a 9.0-mL control water sample with 90 µL of spiking solution.

7.2. Preparation of Calibration Standards for Quantitation (Note 12.6.)

7.2.1. Prepare mixed calibration solution 10X concentrates by pipetting the specified volume of the mixed fortification solutions (Sections 7.1.8 - 7.1.10) and diluting each to a final volume of 100 mL with acetonitrile. The concentrations of the mixed stock calibration standard concentrates are as follows:

Concentration of Stock Calibration Soln.	Aliquot of Stock Calibration Soln.	Final Soln. Volume	Calibration Soln. Concentrate Final Conc.[a]	Equivalent Sample Conc.[b]
µg/mL	mL	mL	ng/mL	µg/L
0.005	3.0	100.0	0.15	0.0135
0.005	10.0	100.0	0.50	0.045
0.050	5.0	100.0	2.50	0.225
0.050	10.0	100.0	5.00	0.45
0.050	20.0	100.0	10.0	0.90
0.50	5.0	100.0	25.0	2.25
0.50	10.0	100.0	50.0	4.5
0.50	20.0	100.0	100.0	9.0

[a] Note that the mixed calibration solution concentrates are 10X the concentration of the final calibration standards. A 1.0-mL aliquot of each of these concentrates is diluted with 9.0 mL of water containing 0.03% formic acid in order to prepare the final calibration standards. This dilution to prepare the final calibration standards should be performed daily or with each new sample set.

[b] The equivalent sample concentration is based on taking a 9.0-mL water sample which will be diluted to a final volume of 10.0 mL.

8. LIQUID CHROMATOGRAPHY/TANDEM MASS SPECTROMETRY (LC/MS/MS) CONDITIONS

8.1. Typical Instrumental Conditions for XDE-742 and its 7-OH-XDE-742, ADTP and ATSA Metabolites using Positive-ion Electrospray (Note 12.7.)

Instrumentation:	Spark Holland Symbiosis Pharma MDS/Sciex API 4000 LC/MS/MS System MDS/Sciex Analyst® 1.4.1 data system
Column:	Phenomenex Synergi Hydro-RP 80A 50 x 2.00 mm, 4-µm
Column Temperature:	Ambient
Injection Volume:	100 µL
Autosampler Wash Program:	Autosampler loop and needle washed with: 1) 500 µL of acetonitrile/water (80:20) containing 0.1% formic acid 2) 500 µL of acetonitrile/water (80:20) containing 0.1% formic acid with valve wash 3) 500 µL of methanol with valve wash 4) 500 µL of water with valve wash 5) 500 µL of water
Run Time:	Approximately 15.0 minutes
Mobile Phase:	A – water containing 0.01% formic acid B – acetonitrile containing 0.01% formic acid
Flow Rate:	300 µL/min

Gradient:

Time, min	Solvent A, %	Solvent B, %
0.01	100	0
1.01	100	0
7.01	50	50
8.00	10	90
10.00	10	90
11.00	100	0
15.00	100	0

Flow Diverter:	Time, min	Direction
	0.0→3.9	waste
	3.9→8.0	source
	8.0→end of run	waste

Ionization Mode:	electrospray
Polarity:	positive
Scan Type:	MRM
Resolution:	Q1 – unit, Q3 – low
Curtain Gas (CUR):	30 psi
Collision Gas (CAD):	4 psi
Temperature (TEM):	450 °C
Ion Source Gas 1 (GS1):	45 psi
Ion Source Gas 2 (GS2):	75 psi
Acquisition Time Delay:	4.0 minutes
Period Duration	4.0 minutes
IonSpray Voltage (IS):	+5000 volts

Analytes:	Precursor Ion Q1	Product Ion Q3	Dwell Time, ms	Collision Energy, v
XDE-742 (quantitation)	435.1	195.1	100	37
XDE-742 (confirmation)	435.1	82.0	100	70
7-OH-XDE-742 (quantitation)	420.9	181.0	100	37
7-OH-XDE-742 (confirmation)	420.9	148.1	100	59
ADTP (quantitation)	196.2	115.1	100	29
ADTP (confirmation)	196.2	163.9	100	29
ATSA (quantitation)	339.0	99.1	100	33
ATSA (confirmation)	339.0	57.2	100	81

8.2. Typical Instrumental Conditions for the XDE-742 Sulfinic Acid and Sulfonic Acid Metabolites using Negative-ion Electrospray (Note 12.7.)

Instrumentation:	Spark Holland Symbiosis Pharma MDS/Sciex API 4000 LC/MS/MS System MDS/Sciex Analyst® 1.4.1 data system
Column:	Phenomenex Synergi Hydro-RP 80A 50 x 2.00 mm, 4-µm
Column Temperature:	Ambient
Injection Volume: Autosampler Wash Program:	25 µL Autosampler loop and needle washed with: 1) 500 µL of acetonitrile/water (80:20) containing 0.1% formic acid 2) 2 x 500 µL of acetonitrile/water (80:20) containing 0.1% formic acid with valve wash 3) 500 µL of methanol with valve wash 4) 500 µL of water with valve wash
Run Time:	Approximately 10.0 minutes
Mobile Phase:	A – water containing 0.01% formic acid B – acetonitrile containing 0.01% formic acid
Flow Rate:	300 µL/min

Gradient:

Time, min	Solvent A, %	Solvent B, %
0.01	100	0
5.01	10	90
6.01	10	90
6.15	100	0
10.15	100	0

Flow Diverter Program:

Time, min	Direction
0.0→1.9	waste
1.9→6.0	source
6.0→end of run	waste

Ionization Mode:	electrospray
Polarity:	negative
Scan Type:	MRM
Resolution:	Q1 – unit, Q3 – unit
Curtain Gas (CUR):	30 psi
Collision Gas (CAD):	4 psi
Temperature (TEM):	450 °C
Ion Source Gas 1 (GS1):	45 psi
Ion Source Gas 2 (GS2):	75 psi
Acquisition Time Delay:	2.0 minutes
Period Duration	4.0 minutes
IonSpray Voltage (IS):	-4200 volts

Analytes:	Precursor Ion Q1	Product Ion Q3	Dwell Time, ms	Collision Energy, v
Sulfinic acid (quantitation)	239.9	175.8	150	-12
Sulfinic acid (confirmation)	239.9	155.7	150	-20
Sulfonic acid (quantitation)	255.7	149.0	150	-40
Sulfonic acid (confirmation)	255.7	79.7	150	-54

8.3. Mass Spectra

Full-scan and product-ion mass spectra of XDE-742 and its metabolites are shown in Figures 1-6.

8.4. Typical Calibration Curves

Typical calibration curves for the determination of XDE-742 and its metabolites in water are shown in Figures 7-18.

8.5. Typical Chromatograms

Typical chromatograms of a standard, a control sample, and a 0.05-µg/L (LOQ) recovery sample for the determination of XDE-742 and each of its metabolites in drinking water are illustrated in Figures 19-24. (Chromatograms for ground water and surface water samples were similar to those for drinking water.)

9. DETERMINATION OF RECOVERY OF XDE-742 AND ITS METABOLITES FROM WATER

9.1. Method Validation

Validate the analytical procedure given in Section 9.3 by analyzing the following with each sample set:

At least one reagent blank.
At least one unfortified control.
At least one control fortified at the limit of detection.
At least two controls fortified at the limit of quantitation.
At least two controls fortified at the expected residue concentration in the samples.

9.2. Sample Preparation

It is recommended that field samples be treated with AmQuel+Plus, as per the instructions on the bottle, at the time of collection in order to stabilize residues prior to analysis. The manufacturer's standard treatment dose is 5 mL of AmQuel+Plus per each 10 gallons (40 liters) of water. No other prior preparation is needed for water samples. Samples may be stored refrigerated or frozen prior to analysis.

9.3. Sample Analysis

9.3.1. Pipet 9.0-mL portions of a well-mixed water sample into a series of 40-mL vials. (Critical Step: Proceed to Step 9.3.5 for field samples that have been previously treated with AmQuel+Plus, as per the instructions on the bottle, at the time of collection.)

9.3.2. For preparing unfortified or fortified control samples (and/or for samples that have not been previously treated with AmQuel+Plus), add 100 µL of a diluted solution of AmQuel+Plus (Section 6.3.4). (Critical Step: For fortified samples, it is critical that the diluted solution of AmQuel+Plus be added prior to fortification.)

9.3.3. Cap the sample vial with a PTFE-lined cap and vortex mix the sample for 5-10 seconds.

9.3.4. Additionally, for preparing fortified control samples, add a 90-µL aliquot of the appropriate spiking solution (Section 7.1.11) to control water samples to obtain concentrations ranging from 0.015 to 5.0 µg/L.

9.3.5. Add 1.0 mL of acetonitrile containing 0.3% formic acid to each sample.

9.3.6. Cap the sample vial with a PTFE-lined cap and then vortex mix the sample for 5-10 seconds.

9.3.7. Prepare the calibration standards by pipetting a 1.0-mL aliquot of each of the mixed calibration solution concentrates (Section 7.2.1) into a series of 40-mL vials.

9.3.8. Pipet 9.0 mL of HPLC-grade water containing 0.03% formic acid (as prepared in Section 6.3.6.) into each of the vials (from Step 9.3.7) in order to prepare the final mixed calibration standards from the concentrates. (Critical Step: This dilution step for preparation of the final calibration standards should be performed with each new sample set or at least daily.)

9.3.9. Cap the vials (Step 9.3.8) with PTFE-lined caps and then vortex mix the final calibration standards for 5-10 seconds.

9.3.10. Transfer a portion of the sample (Step 9.3.6) and portions of the final calibration standards (Step 9.3.9) to 2-mL autosampler vials and seal the vials with a cap, or transfer portions to a 96-well plate and cap the plate.

9.3.11. For the determination of XDE-742, and its 7-OH-XDE-742, ADTP and ATSA metabolites, analyze the calibration standards and samples by HPLC with positive-ion electrospray tandem mass spectrometry as described in Sections 8.1. For the determination of the XDE-742 sulfinic acid and sulfonic acid metabolites, analyze the calibration standards and samples a second time by HPLC with negative-ion electrospray tandem mass spectrometry as described in Section 8.2. Determine the suitability of the chromatographic system using the following performance criteria:

a. Standard curve linearity: Determine that the coefficient of determination (r^2) equals or exceeds 0.995 for the least squares equation which describes the detector response as a function of standard curve concentration.

b. Peak resolution: Visually determine that sufficient resolution has been achieved for the analyte relative to background interferences.

c. Appearance of chromatograms: Visually determine that the chromatograms resemble those shown in Figures 19-24 with respect to peak response, baseline noise, and background interference. Visually determine that a minimum signal-to-noise ratio of 10:1 has been attained for each analyte in the 0.05-ng/mL calibration standard.

9.3.12. If any sample concentrations exceed the range of the standard calibration curve, use a water/acetonitrile solution (90:10) containing 0.03% formic acid to proportionally dilute those samples in order to obtain responses within the range of the calibration curve.

10. CALCULATIONS

10.1. Calculation of Standard Calibration Curves for XDE-742 and its Metabolites in Water

10.1.1. Inject a series of calibration standards (Section 7.2.1) using the conditions described in Section 8 and determine the peak areas for XDE-742 and its metabolites as indicated below:

XDE-742	*m/z* Q1/Q3 435/195 (quantitation) *m/z* Q1/Q3 435/82 (confirmation)
7-OH-XDE-742	*m/z* Q1/Q3 421/181 (quantitation) *m/z* Q1/Q3 421/148 (confirmation)
ADTP metabolite of XDE-742	*m/z* Q1/Q3 196/115 (quantitation) *m/z* Q1/Q3 196/164 (confirmation)
ATSA metabolite of XDE-742	*m/z* Q1/Q3 339/99 (quantitation) *m/z* Q1/Q3 339/57 (confirmation)
sulfinic acid metabolite of XDE-742	*m/z* Q1/Q3 240/176 (quantitation) *m/z* Q1/Q3 240/156 (confirmation)
sulfonic acid metabolite of XDE-742	*m/z* Q1/Q3 256/149 (quantitation) *m/z* Q1/Q3 256/80 (confirmation)

10.1.2. Prepare a standard curve by plotting the concentration of XDE-742 and its metabolites on the abscissa (x-axis), and the respective peak area on the ordinate (y-axis), as shown in Figures 7-18. Using linear regression analysis (13.1.) with a 1/x weighting (13.2.), determine the equation for the curve with respect to the abscissa. (Note 12.8.)

For example, using XDE-742 data from Figure 7:

$$X = \left(\frac{Y - \text{intercept}}{\text{slope}}\right)$$

$$\text{XDE - 742 conc. (ng/mL)} = \left(\frac{\text{XDE - 742 peak area} - \text{intercept}}{\text{slope}}\right)$$

$$\text{XDE - 742 conc. (ng/mL)} = \left(\frac{\text{XDE - 742 peak area} - (30225.8)}{2804644}\right)$$

10.2. Calculation of Percent Recovery for XDE-742 and its Metabolites

10.2.1. Determine the gross concentration in each recovery sample by substituting the peak area obtained into the above equation and solving for the concentration.

For example, using the data for XDE-742 from the injection of the drinking water sample fortified at 0.050 µg/L, Figure 19 (c):

$$\text{XDE - 742 conc. (ng/mL)} = \left(\frac{\text{XDE - 742 peak area} - (30225.8087)}{2804644}\right)$$

$$\text{XDE - 742 conc. (gross ng/mL)} = \left(\frac{146523 - (30225.8087)}{2804644}\right)$$

$$\text{XDE - 742 conc. (gross)} = 0.04147 \text{ ng/mL}$$

Correct the concentration of XDE-742 found in the final sample prepared for analysis diluted to a volume of 10.0 mL by accounting for the original water sample aliquot volume of 9.0 mL as follows:

$$\text{XDE - 742 conc. (gross ng/mL)} = 0.04147 \text{ ng/mL} \times \frac{\text{Final sample vol. (mL)}}{\text{Initial sample vol. (mL)}} \text{ x 1 (dil)}$$

$$\text{XDE - 742 conc. (gross ng/mL)} = 0.04147 \text{ ng/mL} \times \frac{10.0 \text{ mL}}{9.0 \text{ mL}} \text{ x 1 (dil)}$$

$$\text{XDE - 742 conc. (gross)} = 0.0460 \text{ ng/mL (or 0.0460 µg/L)}$$

where:

Aliquot taken (initial) = 9.0 mL
Final volume of sample = 10.0 mL
Dilution (dil) factor (of final sample volume, if needed) = 1

10.2.2. Determine the net concentration of XDE-742 and its metabolites in each recovery sample by subtracting any contribution found at the retention time of the analyte in the unfortified control sample from that of the gross analyte concentration found in the recovery sample.

For example, using the data for XDE-742 from Figure 19:

$$\begin{array}{lcl} \text{XDE-742 conc. (net µg/L)} & = & \text{XDE-742 conc. (gross µg/L)} - \text{XDE-742 conc. (control µg/L)} \\ \text{XDE-742 conc. (net µg/L)} & = & 0.0460\ \mu g/L - 0.0000\ \mu g/L \\ \text{XDE-742 conc. (net)} & = & 0.0460\ \mu g/L \end{array}$$

10.2.3. Determine the percent recovery by dividing the net concentration of each recovery sample by the theoretical concentration added.

$$\text{Recovery} = \frac{\text{conc. found}}{\text{conc. added}} \times 100\%$$

$$\text{Recovery} = \frac{0.0460\,\mu g/L}{0.050\,\mu g/L} \times 100\%$$

$$\text{Recovery} = 92\%$$

10.3. Determination of XDE-742 and its Metabolites in Water Samples

10.3.1. Determine the gross concentration of XDE-742 and its metabolites in each water sample by substituting the respective peak area obtained into the equation for the standard calibration curve and calculating the uncorrected residue result as described in Section 10.2.1.

10.3.2. For those samples that require correction for method recovery, use the average recovery of all the recovery samples from a given sample set to correct for method efficiency. For example, continuing with the data from Figure 19 and the average recovery from Table 2 for the samples analyzed on 20-Jan-2006:

$$\text{XDE - 742 conc. (corrected µg/L)} = \text{XDE - 742 conc. (gross µg/L)} \times \left(\frac{100}{\text{Average \% Recovery}} \right)$$

$$\text{XDE - 742 conc. (corrected µg/L)} = 0.0460\ \mu g/L \times \frac{100}{95}$$

$$\text{XDE - 742 conc. (corrected)} = 0.0484\ \mu g/L$$

11. RESULTS AND DISCUSSION

11.1. Method Validation

11.1.1. Recovery Levels and Precision

A method validation study was conducted to determine the recovery levels and the precision of the method for the determination of XDE-742 and its metabolites in drinking water, ground water, and surface water. The individual quantitative and confirmative results are listed in Tables 2-19 and they are summarized in Tables 20-31.

For all six of the analytes, the individual quantitative recoveries from the validation study were between 72 and 119%, while the average quantitative recoveries for each of the six analytes at each fortification level were between 87 and 113%.

11.1.2. Standard Curve Linearity

For the linear regression analysis with 1/x weighting, the coefficients of determination (r^2) were all greater than or equal to 0.9972 for all of the calibration curve determinations during the method validation for XDE-742 and its metabolites. The results indicate linearity of the detector response as a function of the standard concentration.

11.1.3. Calculated Limits of Quantitation and Detection

Following established guidelines (13.3), the limits of quantitation (LOQ) and detection (LOD) for the determination of XDE-742 and its metabolites in water were calculated using the standard deviation from the 0.050-µg/L recovery results. The LOQ was calculated as ten times the standard deviation (10*s*), and the LOD was calculated as three times the standard deviation (3*s*) of the results. The results are summarized in Tables 32-43.

The calculated LOQ derived by using the results from the quantitative-ion transitions for all of the analytes ranged from 0.0095 to 0.0455 µg/L with one exception. The calculated LOQ for the sulfonic acid in drinking water was 0.0535 µg/L. The calculated LOQ supports the method LOQ of 0.050 µg/L for each analyte. In a similar fashion, the calculated LOD ranged from 0.0029 to 0.0137 µg/L with one exception. The calculated LOD for the sulfonic acid in drinking water was 0.0161 µg/L. The calculated LOD supports the method validation study LOD of 0.015 µg/L.

In actual residue samples, numerical results should be reported as less than the LOQ (<0.05 µg/L) for residues that are equal to or above the LOD but less than the validated LOQ, indicating that the results are being reported at a lower confidence level. For residues less than the LOD, results should be reported as not detected.

11.2. Specificity of Method and Confirmation of Residue Identity

The method is specific for the determination of XDE-742 and its metabolites by virtue of the chromatographic separation and selective detection system used. Further confirmation should not be necessary due to the highly specific nature of the MS/MS transitions. Further confirmation can be achieved, if necessary, by monitoring additional MS/MS transitions as described in Sections 8.1 and 8.2.

11.2.1. Inject the series of calibration standards described in Section 7.2.1 and determine the peak areas for the analytes as indicated below.

XDE-742	*m/z* Q1/Q3 435/195 (quantitation) *m/z* Q1/Q3 435/82 (confirmation)
7-OH-XDE-742	*m/z* Q1/Q3 421/181 (quantitation) *m/z* Q1/Q3 421/148 (confirmation)
ADTP metabolite of XDE-742	*m/z* Q1/Q3 196/115 (quantitation) *m/z* Q1/Q3 196/164 (confirmation)
ATSA metabolite of XDE-742	*m/z* Q1/Q3 339/99 (quantitation) *m/z* Q1/Q3 339/57 (confirmation)
sulfinic acid metabolite of XDE-742	*m/z* Q1/Q3 240/176 (quantitation) *m/z* Q1/Q3 240/156 (confirmation)
sulfonic acid metabolite of XDE-742	*m/z* Q1/Q3 256/149 (quantitation) *m/z* Q1/Q3 256/80 (confirmation)

11.2.2. For each standard, calculate each analyte's confirmation ratio. Use the average confirmation ratio for each analyte to confirm the presence of the analyte in the water samples.

$$\text{confirmation ratio} = \frac{\text{peak area of confirmation transition}}{\text{peak area of quantitation transition}}$$

For example, using the data for XDE-742 from Figure 19a:

$$\text{confirmation ratio (}m/z\text{ 435.1/82.0 / }m/z\text{ 435.1/195.1)} = \frac{\text{peak area at }m/z\text{ 435.1/82.0}}{\text{peak area at }m/z\text{ 435.1/195.1}}$$

$$\text{confirmation ratio (}m/z\text{ 435.1/82.0 / }m/z\text{ 435.1/195.1)} = \frac{17430}{168747} = 0.1033$$

Confirmation of the presence of analytes is indicated when the retention time of the samples matches that of the standards and the confirmation ratio for the sample is within the range of ±20% of the average found for the standards.

11.3. Assay Time

A typical analytical run would consist of a minimum of eight standards encompassing the expected range of sample concentrations, a reagent blank, a control (a non-fortified sample), a minimum of three fortified controls (two of which must be at the LOQ), and 25 samples. This typical analytical run can be prepared in approximately 3 hours, followed by the chromatographic analysis.

Since the preparation of samples for analysis is relatively short, there are no acceptable "stopping points" described in the method, where sample preparation (Section 9) may be suspended, upon completion of a step, without deleterious effects on the sample analysis.

12. NOTES

12.1. Equipment, glassware, materials, reagents, and chemicals considered to be equivalent to those specified may be substituted with the understanding that their performance must be confirmed by appropriate tests. Common laboratory supplies are assumed to be readily available and are, therefore, not listed.

12.2. Electronic pipets are used only for pipetting aqueous solutions. If they are used for pipetting non-aqueous solutions, the pipets should be calibrated following the manufacturer's instruction manual and Standard Operating Procedures (13.4).

12.3. The product AmQuel+Plus, is a proprietary solution containing sodium hydroxymethanesulfonate according to the manufacturer.

12.4. Section 7 provides suggested concentrations for the preparation of fortification and calibration standards. Other dilution schemes may be followed.

12.5. Sonication is required to ensure that some of the analytical standards thoroughly dissolve in the initial methanol solution.

12.6. The dilution of the calibration solutions for use should be made fresh daily or with each sample set as the sufinic acid is known to slowly degrade to the sulfonic acid in an aqueous environment.

12.7. The instrumental conditions may be modified to obtain optimal chromatographic separation and sensitivity.

12.8. Other regression techniques may also be used based on detector response.

13. REFERENCES

13.1. Freund, J. E.; Williams, F. J. *Dictionary/Outline of Basic Statistics*; Dover: New York, 1991; p 170.

13.2. Neter, J.; Kutner, M. H.; Nachtssheim, C. J. ; Wasserman, W. *Applied Linear Regression Models*; The McGraw-Hill Company: New York, 1996; p 409.

13.3. Keith, L. H.; Crummett, W.; Deegan, J., Jr.; Libby, R. A.; Taylor, J. K.; Wentler, G. *Anal. Chem.* **1983**, *55*, 2210-2218.

13.4. *Standard Operating Procedure for Pipettes*; Brinkmann/Eppendorf SOP 5101-C20, Brinkmann Instruments, Inc., Westbury, NY, 2001.

Table 1. Identity and Structures of XDE-742 and Its Metabolites

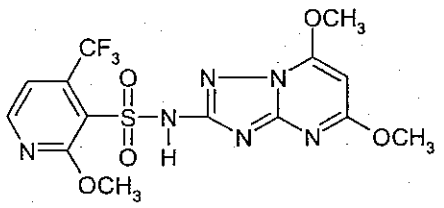
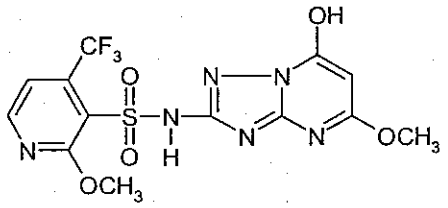
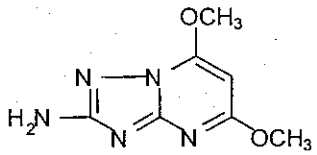
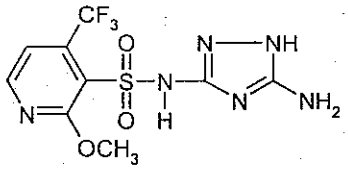
Common Name of Compound	Structural Formula and Chemical Name (IUPAC)
XDE-742 Molecular Formula: $C_{14}H_{13}F_3N_6O_5S$ Formula Weight: 434.4 g/mole Nominal Mass: 434 CAS Number 422556-08-9	 *N*-(5,7-dimethoxy[1,2,4]triazolo[1,5-a]pyrimidin-2-yl)-2-methoxy-4-(trifluoromethyl)pyridine-3-sulfonamide
7-OH-XDE-742 Molecular Formula: $C_{13}H_{11}F_3N_6O_5S$ Formula Weight: 420.3 g/mole Nominal Mass: 420 CAS Number NA	 *N*-(7-hydroxy-5-methoxy[1,2,4]triazolo[1,5-*a*]pyrimidin-2-yl)-2-methoxy-4-(trifluoromethyl)-3-pyridinesulfonamide
ADTP metabolite of XDE-742 Molecular Formula: $C_7H_9N_5O_2$ Formula Weight: 195.2 g/mole Nominal Mass: 195 CAS Number NA	 5,7-dimethoxy[1,2,4]triazolo[1,5-a]pyrimidin-2-amine
ATSA metabolite of XDE-742 Molecular Formula: $C_9H_9F_3N_6O_3S$ Formula Weight: 338.27 g/mol Nominal Mass: 338 CAS Number NA	 *N*-(5-amino-1H-1,2,4-triazol-3-yl)-2-methoxy-4-(trifluoromethyl)pyridine-3-sulfonamide

Table 1. (Cont.) Identity and Structures of XDE-742 and Its Metabolites

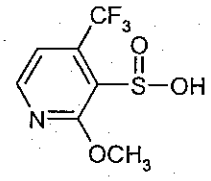
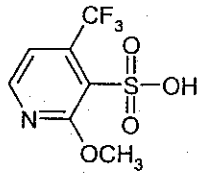
sulfinic acid metabolite of XDE-742 Molecular Formula: $C_7H_6F_3NO_3S$ Formula Weight: 241.19 g/mol Nominal Mass: 241 CAS Number NA	 2-methoxy-4-(trifluoromethyl)pyridine-3-sulfinic acid
sulfonic acid metabolite of XDE-742 Molecular Formula: $C_7H_6F_3NO_4S$ Formula Weight: 257.19 g/mol Nominal Mass: 257 CAS Number NA	 2-methoxy-4-(trifluoromethyl)pyridine-3-sulfonic acid

Table 2. Recovery of XDE-742 from Drinking Water

Sample Number	Sample Matrix	Date of Analysis[a]	Analyte Added (µg/L)	XDE-742 Quant (Q1/Q3 *m/z* 435.1/195.1) Found (µg/L)[b]	XDE-742 Quant (Q1/Q3 *m/z* 435.1/195.1) % Recovery	XDE-742 Confirm (Q1/Q3 *m/z* 435.1/82.0) Found (µg/L)[b]	XDE-742 Confirm (Q1/Q3 *m/z* 435.1/82.0) % Recovery[c]
189-0001	drinking water	20-Jan-2006	0.000	ND[d]	--	ND	--
194-0001	drinking water	23-Jan-2006	0.000	ND	--	0.0005	--
189-0001	drinking water	20-Jan-2006	0.015	0.0085[e]	NA[f]	0.0105	NA
194-0001	drinking water	23-Jan-2006	0.015	0.0108	NA	0.0143	NA
189-0001	drinking water	20-Jan-2006	0.050	0.0460	92	0.0523	105
189-0001	drinking water	20-Jan-2006	0.050	0.0464	93	0.0487	97
189-0001	drinking water	20-Jan-2006	0.050	0.0443	89	0.0439	88
194-0001	drinking water	23-Jan-2006	0.050	0.0466	93	0.0499	100
194-0001	drinking water	23-Jan-2006	0.050	0.0471	94	0.0478	96
194-0001	drinking water	23-Jan-2006	0.050	0.0471	94	0.0467	93
189-0001	drinking water	20-Jan-2006	5.00	4.921	98	4.750	95
189-0001	drinking water	20-Jan-2006	5.00	4.903	98	4.698	94
189-0001	drinking water	20-Jan-2006	5.00	4.919	98	4.731	95
194-0001	drinking water	23-Jan-2006	5.00	5.022	100	4.804	96
194-0001	drinking water	23-Jan-2006	5.00	5.144	103	4.908	98
194-0001	drinking water	23-Jan-2006	5.00	5.051	101	4.871	97

[a] The 'Date of Analysis' indicates the date that the samples were prepared for analysis.
[b] All calculations were performed using Microsoft Excel 2002 with full precision.
[c] Corrected for contribution from the control sample where appropriate.
[d] ND = not detected. The residue was below the 0.015-µg/L limit of detection and no background subtraction was performed.
[e] Samples fortified at the method's limit of detection of 0.015 µg/L are reported with a lower degree of confidence than values above the limit of quantitation.
[f] NA = not applicable. Samples were fortified below the method's limit of quantitation of 0.050 µg/L.

Table 3. Recovery of XDE-742 from Ground Water

Sample Number	Sample Matrix	Date of Analysis[a]	Analyte Added (µg/L)	XDE-742 Quant (Q1/Q3 *m/z* 435.1/195.1) Found (µg/L)[b]	XDE-742 Quant (Q1/Q3 *m/z* 435.1/195.1) % Recovery	XDE-742 Confirm (Q1/Q3 *m/z* 435.1/82.0) Found (µg/L)[b]	XDE-742 Confirm (Q1/Q3 *m/z* 435.1/82.0) % Recovery[c]
190-0001	ground water	20-Jan-2006	0.000	ND[d]	--	ND	--
193-0001	ground water	23-Jan-2006	0.000	ND	--	0.0005	--
190-0001	ground water	20-Jan-2006	0.015	0.0083[e]	NA[f]	0.0117	NA
193-0001	ground water	23-Jan-2006	0.015	0.0109	NA	0.0142	NA
190-0001	ground water	20-Jan-2006	0.050	0.0464	93	0.0467	93
190-0001	ground water	20-Jan-2006	0.050	0.0447	89	0.0479	96
190-0001	ground water	20-Jan-2006	0.050	0.0461	92	0.0458	92
193-0001	ground water	23-Jan-2006	0.050	0.0468	94	0.0468	94
193-0001	ground water	23-Jan-2006	0.050	0.0481	96	0.0507	101
193-0001	ground water	23-Jan-2006	0.050	0.0464	93	0.0496	99
190-0001	ground water	20-Jan-2006	5.00	4.865	97	4.703	94
190-0001	ground water	20-Jan-2006	5.00	4.946	99	4.739	95
190-0001	ground water	20-Jan-2006	5.00	4.915	98	4.700	94
193-0001	ground water	23-Jan-2006	5.00	5.033	101	4.788	96
193-0001	ground water	23-Jan-2006	5.00	4.969	99	4.850	97
193-0001	ground water	23-Jan-2006	5.00	5.055	101	4.806	96

[a] The 'Date of Analysis' indicates the date that the samples were prepared for analysis.
[b] All calculations were performed using Microsoft Excel 2002 with full precision.
[c] Corrected for contribution from the control sample where appropriate.
[d] ND = not detected. The residue was below the 0.015-µg/L limit of detection and no background subtraction was performed.
[e] Samples fortified at the method's limit of detection of 0.015 µg/L are reported with a lower degree of confidence than values above the limit of quantitation.
[f] NA = not applicable.. Samples were fortified below the method's limit of quantitation of 0.050 µg/L.

Table 4. Recovery of XDE-742 from Surface Water

Sample Number	Sample Matrix	Date of Analysis[a]	Analyte Added (µg/L)	XDE-742 Quant (Q1/Q3 *m/z* 435.1/195.1) Found (µg/L)[b]	XDE-742 Quant (Q1/Q3 *m/z* 435.1/195.1) % Recovery	XDE-742 Confirm (Q1/Q3 *m/z* 435.1/82.0) Found (µg/L)[b]	XDE-742 Confirm (Q1/Q3 *m/z* 435.1/82.0) % Recovery
191-0001	surface water	20-Jan-2006	0.000	ND[c]	--	ND	--
192-0001	surface water	23-Jan-2006	0.000	ND	--	ND	--
191-0001	surface water	20-Jan-2006	0.015	0.0092[d]	NA[e]	0.0140	NA
192-0001	surface water	23-Jan-2006	0.015	0.0114	NA	0.0148	NA
191-0001	surface water	20-Jan-2006	0.050	0.0461	92	0.0473	95
191-0001	surface water	20-Jan-2006	0.050	0.0450	90	0.0470	94
191-0001	surface water	20-Jan-2006	0.050	0.0464	93	0.0487	97
192-0001	surface water	23-Jan-2006	0.050	0.0499	100	0.0509	102
192-0001	surface water	23-Jan-2006	0.050	0.0494	99	0.0507	101
192-0001	surface water	23-Jan-2006	0.050	0.0509	102	0.0502	100
191-0001	surface water	20-Jan-2006	5.00	4.976	100	4.700	94
191-0001	surface water	20-Jan-2006	5.00	5.007	100	4.778	96
191-0001	surface water	20-Jan-2006	5.00	4.950	99	4.777	96
192-0001	surface water	23-Jan-2006	5.00	4.767	95	4.595	92
192-0001	surface water	23-Jan-2006	5.00	5.196	104	4.964	99
192-0001	surface water	23-Jan-2006	5.00	5.159	103	5.019	100

[a] The 'Date of Analysis' indicates the date that the samples were prepared for analysis.
[b] All calculations were performed using Microsoft Excel 2002 with full precision.
[c] ND = not detected. The residue was below the 0.015-µg/L limit of detection and no background subtraction was performed.
[d] Samples fortified at the method's limit of detection of 0.015 µg/L are reported with a lower degree of confidence than values above the limit of quantitation.
[e] NA = not applicable. Samples were fortified below the method's limit of quantitation of 0.050 µg/L.

Table 5. Recovery of 7-OH-XDE-742 Metabolite from Drinking Water

Sample Number	Sample Matrix	Date of Analysis[a]	Analyte Added (µg/L)	7-OH-XDE-742 Quant (Q1/Q3 *m/z* 420.9/181.0) Found (µg/L)[b]	% Recovery	7-OH-XDE-742 Confirm (Q1/Q3 *m/z* 420.9/148.1) Found (µg/L)[b]	% Recovery[c]
189-0001	drinking water	20-Jan-2006	0.000	ND[d]	--	ND	--
194-0001	drinking water	23-Jan-2006	0.000	ND	--	0.0058	--
189-0001	drinking water	20-Jan-2006	0.015	0.0162[e]	NA[f]	0.0182	NA
194-0001	drinking water	23-Jan-2006	0.015	0.0179	NA	0.0132	NA
189-0001	drinking water	20-Jan-2006	0.050	0.0516	103	0.0502	100
189-0001	drinking water	20-Jan-2006	0.050	0.0501	100	0.0535	107
189-0001	drinking water	20-Jan-2006	0.050	0.0529	106	0.0538	108
194-0001	drinking water	23-Jan-2006	0.050	0.0523	105	0.0460	92
194-0001	drinking water	23-Jan-2006	0.050	0.0549	110	0.0485	97
194-0001	drinking water	23-Jan-2006	0.050	0.0534	107	0.0456	91
189-0001	drinking water	20-Jan-2006	5.00	4.992	100	5.014	100
189-0001	drinking water	20-Jan-2006	5.00	4.932	99	4.925	98
189-0001	drinking water	20-Jan-2006	5.00	4.977	100	4.925	98
194-0001	drinking water	23-Jan-2006	5.00	5.114	102	5.079	102
194-0001	drinking water	23-Jan-2006	5.00	5.192	104	5.175	104
194-0001	drinking water	23-Jan-2006	5.00	5.154	103	5.163	103

[a] The 'Date of Analysis' indicates the date that the samples were prepared for analysis.
[b] All calculations were performed using Microsoft Excel 2002 with full precision.
[c] Corrected for contribution from the control sample where appropriate.
[d] ND = not detected. The residue was below the 0.015-µg/L limit of detection and no background subtraction was performed.
[e] Samples fortified at the method's limit of detection of 0.015 µg/L are reported with a lower degree of confidence than values above the limit of quantitation.
[f] NA = not applicable. Samples were fortified below the method's limit of quantitation of 0.050 µg/L.

Table 6. Recovery of 7-OH-XDE-742 Metabolite from Ground Water

Sample Number	Sample Matrix	Date of Analysis[a]	Analyte Added (µg/L)	7-OH-XDE-742 Quant (Q1/Q3 *m/z* 420.9/181.0) Found (µg/L)[b]	% Recovery[c]	7-OH-XDE-742 Confirm (Q1/Q3 *m/z* 420.9/148.1) Found (µg/L)[b]	% Recovery[c]
190-0001	ground water	20-Jan-2006	0.000	0.0036	--	ND[d]	--
193-0001	ground water	23-Jan-2006	0.000	0.0042	--	0.0049	--
190-0001	ground water	20-Jan-2006	0.015	0.0110[e]	NA[f]	0.0182	NA
193-0001	ground water	23-Jan-2006	0.015	0.0122	NA	0.0146	NA
190-0001	ground water	20-Jan-2006	0.050	0.0457	91	0.0513	103
190-0001	ground water	20-Jan-2006	0.050	0.0434	87	0.0469	94
190-0001	ground water	20-Jan-2006	0.050	0.0442	88	0.0528	106
193-0001	ground water	23-Jan-2006	0.050	0.0465	93	0.0467	93
193-0001	ground water	23-Jan-2006	0.050	0.0472	94	0.0495	99
193-0001	ground water	23-Jan-2006	0.050	0.0480	96	0.0438	88
190-0001	ground water	20-Jan-2006	5.00	4.831	97	4.848	97
190-0001	ground water	20-Jan-2006	5.00	4.894	98	4.962	99
190-0001	ground water	20-Jan-2006	5.00	4.923	98	4.969	99
193-0001	ground water	23-Jan-2006	5.00	5.038	101	5.060	101
193-0001	ground water	23-Jan-2006	5.00	5.034	101	5.056	101
193-0001	ground water	23-Jan-2006	5.00	5.024	100	4.993	100

[a] The 'Date of Analysis' indicates the date that the samples were prepared for analysis.
[b] All calculations were performed using Microsoft Excel 2002 with full precision.
[c] Corrected for contribution from the control sample where appropriate.
[d] ND = not detected. The residue was below the 0.015-µg/L limit of detection and no background subtraction was performed.
[e] Samples fortified at the method's limit of detection of 0.015 µg/L are reported with a lower degree of confidence than values above the limit of quantitation.
[f] NA = not applicable. Samples were fortified below the method's limit of quantitation of 0.050 µg/L.

Table 7. Recovery of 7-OH-XDE-742 Metabolite from Surface Water

Sample Number	Sample Matrix	Date of Analysis[a]	Analyte Added (µg/L)	7-OH-XDE-742 Quant (Q1/Q3 *m/z* 420.9/181.0) Found (µg/L)[b]	% Recovery[c]	7-OH-XDE-742 Confirm (Q1/Q3 *m/z* 420.9/148.1) Found (µg/L)[b]	% Recovery[c]
191-0001	surface water	20-Jan-2006	0.000	0.0037	--	ND[d]	--
192-0001	surface water	23-Jan-2006	0.000	0.0042	--	0.0071	--
191-0001	surface water	20-Jan-2006	0.015	0.0116[e]	NA[f]	0.0183	NA
192-0001	surface water	23-Jan-2006	0.015	0.0142	NA	0.0124	NA
191-0001	surface water	20-Jan-2006	0.050	0.0480	96	0.0524	105
191-0001	surface water	20-Jan-2006	0.050	0.0463	93	0.0490	98
191-0001	surface water	20-Jan-2006	0.050	0.0461	92	0.0543	109
192-0001	surface water	23-Jan-2006	0.050	0.0532	106	0.0517	103
192-0001	surface water	23-Jan-2006	0.050	0.0539	108	0.0498	100
192-0001	surface water	23-Jan-2006	0.050	0.0540	108	0.0549	110
191-0001	surface water	20-Jan-2006	5.00	4.962	99	4.934	99
191-0001	surface water	20-Jan-2006	5.00	5.020	100	5.055	101
191-0001	surface water	20-Jan-2006	5.00	5.070	101	5.087	102
192-0001	surface water	23-Jan-2006	5.00	5.195	104	5.099	102
192-0001	surface water	23-Jan-2006	5.00	5.652	113	5.562	111
192-0001	surface water	23-Jan-2006	5.00	5.620	112	5.661	113

[a] The 'Date of Analysis' indicates the date that the samples were prepared for analysis.
[b] All calculations were performed using Microsoft Excel 2002 with full precision.
[c] Corrected for contribution from the control sample where appropriate.
[d] ND = not detected. The residue was below the 0.015-µg/L limit of detection and no background subtraction was performed.
[e] Samples fortified at the method's limit of detection of 0.015 µg/L are reported with a lower degree of confidence than values above the limit of quantitation.
[f] NA = not applicable. Samples were fortified below the method's limit of quantitation of 0.050 µg/L.

Table 8. Recovery of the ADTP Metabolite of XDE-742 from Drinking Water

Sample Number	Sample Matrix	Date of Analysis[a]	Analyte Added (µg/L)	ADTP Quant (Q1/Q3 *m/z* 196.2/115.1) Found (µg/L)[b]	ADTP Quant (Q1/Q3 *m/z* 196.2/115.1) % Recovery	ADTP Confirm (Q1/Q3 *m/z* 196.2/163.9) Found (µg/L)[b]	ADTP Confirm (Q1/Q3 *m/z* 196.2/163.9) % Recovery
189-0001	drinking water	20-Jan-2006	0.000	ND[c]	--	ND	--
194-0001	drinking water	23-Jan-2006	0.000	ND	--	ND	--
189-0001	drinking water	20-Jan-2006	0.015	0.0121[d]	NA[e]	0.0107	NA
194-0001	drinking water	23-Jan-2006	0.015	0.0120	NA	0.0127	NA
189-0001	drinking water	20-Jan-2006	0.050	0.0505	101	0.0475	95
189-0001	drinking water	20-Jan-2006	0.050	0.0492	98	0.0469	94
189-0001	drinking water	20-Jan-2006	0.050	0.0442	88	0.0423	85
194-0001	drinking water	23-Jan-2006	0.050	0.0480	96	0.0475	95
194-0001	drinking water	23-Jan-2006	0.050	0.0493	99	0.0479	96
194-0001	drinking water	23-Jan-2006	0.050	0.0504	101	0.0516	103
189-0001	drinking water	20-Jan-2006	5.00	4.908	98	4.867	97
189-0001	drinking water	20-Jan-2006	5.00	4.823	96	4.845	97
189-0001	drinking water	20-Jan-2006	5.00	4.839	97	4.832	97
194-0001	drinking water	23-Jan-2006	5.00	5.042	101	5.182	104
194-0001	drinking water	23-Jan-2006	5.00	5.224	104	5.159	103
194-0001	drinking water	23-Jan-2006	5.00	5.157	103	5.140	103

[a] The 'Date of Analysis' indicates the date that the samples were prepared for analysis.
[b] All calculations were performed using Microsoft Excel 2002 with full precision.
[c] ND = not detected. The residue was below the 0.015-µg/L limit of detection and no background subtraction was performed.
[d] Samples fortified at the method's limit of detection of 0.015 µg/L are reported with a lower degree of confidence than values above the limit of quantitation.
[e] NA = not applicable. Samples were fortified below the method's limit of quantitation of 0.050 µg/L.

Table 9. Recovery of the ADTP Metabolite of XDE-742 from Ground Water

Sample Number	Sample Matrix	Date of Analysis[a]	Analyte Added (µg/L)	ADTP Quant (Q1/Q3 *m/z* 196.2/115.1) Found (µg/L)[b]	ADTP Quant (Q1/Q3 *m/z* 196.2/115.1) % Recovery	ADTP Confirm (Q1/Q3 *m/z* 196.2/163.9) Found (µg/L)[b]	ADTP Confirm (Q1/Q3 *m/z* 196.2/163.9) % Recovery
190-0001	ground water	20-Jan-2006	0.000	ND[c]	--	ND	--
193-0001	ground water	23-Jan-2006	0.000	ND	--	ND	--
190-0001	ground water	20-Jan-2006	0.015	0.0099[d]	NA[e]	0.0127	NA
193-0001	ground water	23-Jan-2006	0.015	0.0117	NA	0.0124	NA
190-0001	ground water	20-Jan-2006	0.050	0.0455	91	0.0433	87
190-0001	ground water	20-Jan-2006	0.050	0.0473	95	0.0415	83
190-0001	ground water	20-Jan-2006	0.050	0.0450	90	0.0436	87
193-0001	ground water	23-Jan-2006	0.050	0.0473	95	0.0476	95
193-0001	ground water	23-Jan-2006	0.050	0.0475	95	0.0462	92
193-0001	ground water	23-Jan-2006	0.050	0.0501	100	0.0487	97
190-0001	ground water	20-Jan-2006	5.00	4.720	94	4.709	94
190-0001	ground water	20-Jan-2006	5.00	4.880	98	4.808	96
190-0001	ground water	20-Jan-2006	5.00	4.846	97	4.786	96
193-0001	ground water	23-Jan-2006	5.00	4.991	100	5.004	100
193-0001	ground water	23-Jan-2006	5.00	4.908	98	5.001	100
193-0001	ground water	23-Jan-2006	5.00	4.965	99	4.958	99

[a] The 'Date of Analysis' indicates the date that the samples were prepared for analysis.
[b] All calculations were performed using Microsoft Excel 2002 with full precision.
[c] ND = not detected. The residue was below the 0.015-µg/L limit of detection and no background subtraction was performed.
[d] Samples fortified at the method's limit of detection of 0.015 µg/L are reported with a lower degree of confidence than values above the limit of quantitation.
[e] NA = not applicable. Samples were fortified below the method's limit of quantitation of 0.050 µg/L.

Table 10. Recovery of the ADTP Metabolite of XDE-742 from Surface Water

Sample Number	Sample Matrix	Date of Analysis[a]	Analyte Added (µg/L)	ADTP Quant (Q1/Q3 *m/z* 196.2/115.1) Found (µg/L)[b]	ADTP Quant (Q1/Q3 *m/z* 196.2/115.1) % Recovery	ADTP Confirm (Q1/Q3 *m/z* 196.2/163.9) Found (µg/L)[b]	ADTP Confirm (Q1/Q3 *m/z* 196.2/163.9) % Recovery
191-0001	surface water	20-Jan-2006	0.000	ND[c]	--	ND	--
192-0001	surface water	23-Jan-2006	0.000	ND	--	ND	--
191-0001	surface water	20-Jan-2006	0.015	0.0115[d]	NA[e]	0.0078	NA
192-0001	surface water	23-Jan-2006	0.015	0.0129	NA	0.0123	NA
191-0001	surface water	20-Jan-2006	0.050	0.0486	97	0.0452	90
191-0001	surface water	20-Jan-2006	0.050	0.0440	88	0.0472	94
191-0001	surface water	20-Jan-2006	0.050	0.0450	90	0.0484	97
192-0001	surface water	23-Jan-2006	0.050	0.0511	102	0.0525	105
192-0001	surface water	23-Jan-2006	0.050	0.0490	98	0.0516	103
192-0001	surface water	23-Jan-2006	0.050	0.0508	102	0.0513	103
191-0001	surface water	20-Jan-2006	5.00	4.918	98	4.905	98
191-0001	surface water	20-Jan-2006	5.00	5.068	101	5.038	101
191-0001	surface water	20-Jan-2006	5.00	5.100	102	5.044	101
192-0001	surface water	23-Jan-2006	5.00	4.844	97	4.754	95
192-0001	surface water	23-Jan-2006	5.00	5.247	105	5.109	102
192-0001	surface water	23-Jan-2006	5.00	5.251	105	5.134	103

[a] The 'Date of Analysis' indicates the date that the samples were prepared for analysis.
[b] All calculations were performed using Microsoft Excel 2002 with full precision.
[c] ND = not detected. The residue was below the 0.015-µg/L limit of detection and no background subtraction was performed.
[d] Samples fortified at the method's limit of detection of 0.015 µg/L are reported with a lower degree of confidence than values above the limit of quantitation.
[e] NA = not applicable. Samples were fortified below the method's limit of quantitation of 0.050 µg/L.

Table 11. Recovery of the ATSA Metabolite of XDE-742 from Drinking Water

Sample Number	Sample Matrix	Date of Analysis[a]	Analyte Added (µg/L)	ATSA Quant (Q1/Q3 *m/z* 339.0/99.1) Found (µg/L)[b]	ATSA Quant (Q1/Q3 *m/z* 339.0/99.1) % Recovery	ATSA Confirm (Q1/Q3 *m/z* 339.0/57.2) Found (µg/L)[b]	ATSA Confirm (Q1/Q3 *m/z* 339.0/57.2) % Recovery
189-0001	drinking water	20-Jan-2006	0.000	ND[c]	--	ND	--
194-0001	drinking water	23-Jan-2006	0.000	ND	--	ND	--
189-0001	drinking water	20-Jan-2006	0.015	0.0249[d]	NA[e]	0.0163	NA
194-0001	drinking water	23-Jan-2006	0.015	0.0272	NA	0.0209	NA
189-0001	drinking water	20-Jan-2006	0.050	0.0549	110	0.0452	90
189-0001	drinking water	20-Jan-2006	0.050	0.0557	111	0.0461	92
189-0001	drinking water	20-Jan-2006	0.050	0.0546	109	0.0520	104
194-0001	drinking water	23-Jan-2006	0.050	0.0561	112	0.0546	109
194-0001	drinking water	23-Jan-2006	0.050	0.0567	113	0.0593	119
194-0001	drinking water	23-Jan-2006	0.050	0.0570	114	0.0564	113
189-0001	drinking water	20-Jan-2006	5.00	4.890	98	4.796	96
189-0001	drinking water	20-Jan-2006	5.00	4.824	96	4.677	94
189-0001	drinking water	20-Jan-2006	5.00	4.861	97	4.726	95
194-0001	drinking water	23-Jan-2006	5.00	4.954	99	4.965	99
194-0001	drinking water	23-Jan-2006	5.00	5.004	100	4.915	98
194-0001	drinking water	23-Jan-2006	5.00	5.024	100	4.990	100

[a] The 'Date of Analysis' indicates the date that the samples were prepared for analysis.
[b] All calculations were performed using Microsoft Excel 2002 with full precision.
[c] ND = not detected. The residue was below the 0.015-µg/L limit of detection and no background subtraction was performed.
[d] Samples fortified at the method's limit of detection of 0.015 µg/L are reported with a lower degree of confidence than values above the limit of quantitation.
[e] NA = not applicable. Samples were fortified below the method's limit of quantitation of 0.050 µg/L.

Table 12. Recovery of the ATSA Metabolite of XDE-742 from Ground Water

Sample Number	Sample Matrix	Date of Analysis[a]	Analyte Added (µg/L)	ATSA Quant (Q1/Q3 *m/z* 339.0/99.1) Found (µg/L)[b]	ATSA Quant (Q1/Q3 *m/z* 339.0/99.1) % Recovery	ATSA Confirm (Q1/Q3 *m/z* 339.0/57.2) Found (µg/L)[b]	ATSA Confirm (Q1/Q3 *m/z* 339.0/57.2) % Recovery
190-0001	ground water	20-Jan-2006	0.000	ND[c]	--	ND	--
193-0001	ground water	23-Jan-2006	0.000	ND	--	ND	--
190-0001	ground water	20-Jan-2006	0.015	0.0253[d]	NA[e]	0.0180	NA
193-0001	ground water	23-Jan-2006	0.015	0.0266	NA	0.0292	NA
190-0001	ground water	20-Jan-2006	0.050	0.0543	109	0.0462	92
190-0001	ground water	20-Jan-2006	0.050	0.0550	110	0.0541	108
190-0001	ground water	20-Jan-2006	0.050	0.0546	109	0.0508	102
193-0001	ground water	23-Jan-2006	0.050	0.0577	115	0.0580	116
193-0001	ground water	23-Jan-2006	0.050	0.0591	118	0.0589	118
193-0001	ground water	23-Jan-2006	0.050	0.0583	117	0.0575	115
190-0001	ground water	20-Jan-2006	5.00	4.821	96	4.775	96
190-0001	ground water	20-Jan-2006	5.00	4.877	98	4.766	95
190-0001	ground water	20-Jan-2006	5.00	4.865	97	4.850	97
193-0001	ground water	23-Jan-2006	5.00	4.934	99	4.910	98
193-0001	ground water	23-Jan-2006	5.00	4.979	100	4.837	97
193-0001	ground water	23-Jan-2006	5.00	4.877	98	4.859	97

[a] The 'Date of Analysis' indicates the date that the samples were prepared for analysis.
[b] All calculations were performed using Microsoft Excel 2002 with full precision.
[c] ND = not detected. The residue was below the 0.015-µg/L limit of detection and no background subtraction was performed.
[d] Samples fortified at the method's limit of detection of 0.015 µg/L are reported with a lower degree of confidence than values above the limit of quantitation.
[e] NA = not applicable. Samples were fortified below the method's limit of quantitation of 0.050 µg/L.

Table 13. Recovery of the ATSA Metabolite of XDE-742 from Surface Water

Sample Number	Sample Matrix	Date of Analysis[a]	Analyte Added (µg/L)	ATSA Quant (Q1/Q3 *m/z* 339.0/99.1) Found (µg/L)[b]	ATSA Quant (Q1/Q3 *m/z* 339.0/99.1) % Recovery	ATSA Confirm (Q1/Q3 *m/z* 339.0/57.2) Found (µg/L)[b]	ATSA Confirm (Q1/Q3 *m/z* 339.0/57.2) % Recovery
191-0001	surface water	20-Jan-2006	0.000	ND[c]	--	ND	--
192-0001	surface water	23-Jan-2006	0.000	ND	--	ND	--
191-0001	surface water	20-Jan-2006	0.015	0.0249[d]	NA[e]	0.0165	NA
192-0001	surface water	23-Jan-2006	0.015	0.0275	NA	0.0284	NA
191-0001	surface water	20-Jan-2006	0.050	0.0542	108	0.0522	104
191-0001	surface water	20-Jan-2006	0.050	0.0532	106	0.0444	89
191-0001	surface water	20-Jan-2006	0.050	0.0533	107	0.0468	94
192-0001	surface water	23-Jan-2006	0.050	0.0597	119	0.0583	117
192-0001	surface water	23-Jan-2006	0.050	0.0559	112	0.0544	109
192-0001	surface water	23-Jan-2006	0.050	0.0590	118	0.0533	107
191-0001	surface water	20-Jan-2006	5.00	4.881	98	4.723	94
191-0001	surface water	20-Jan-2006	5.00	4.851	97	4.839	97
191-0001	surface water	20-Jan-2006	5.00	4.978	100	4.841	97
192-0001	surface water	23-Jan-2006	5.00	4.629	93	4.572	91
192-0001	surface water	23-Jan-2006	5.00	5.026	101	4.980	100
192-0001	surface water	23-Jan-2006	5.00	4.991	100	4.937	99

[a] The 'Date of Analysis' indicates the date that the samples were prepared for analysis.
[b] All calculations were performed using Microsoft Excel 2002 with full precision.
[c] ND = not detected. The residue was below the 0.015-µg/L limit of detection and no background subtraction was performed.
[d] Samples fortified at the method's limit of detection of 0.015 µg/L are reported with a lower degree of confidence than values above the limit of quantitation.
[e] NA = not applicable. Samples were fortified below the method's limit of quantitation of 0.050 µg/L.

Table 14. Recovery of the Sulfinic Acid Metabolite of XDE-742 from Drinking Water

Sample Number	Sample Matrix	Date of Analysis[a]	Analyte Added (µg/L)	Sulfinic Quant (Q1/Q3 *m/z* 239.9/175.8) Found (µg/L)[b]	Sulfinic Quant (Q1/Q3 *m/z* 239.9/175.8) % Recovery	Sulfinic Confirm (Q1/Q3 *m/z* 239.9/155.7) Found (µg/L)[b]	Sulfinic Confirm (Q1/Q3 *m/z* 239.9/155.7) % Recovery
189-0001	drinking water	20-Jan-2006	0.000	ND[c]	--	ND	--
194-0001	drinking water	23-Jan-2006	0.000	ND	--	ND	--
189-0001	drinking water	20-Jan-2006	0.015	0.0108[d]	NA[e]	0.0111	NA
194-0001	drinking water	23-Jan-2006	0.015	0.0144	NA	0.0119	NA
189-0001	drinking water	20-Jan-2006	0.050	0.0464	93	0.0394	79
189-0001	drinking water	20-Jan-2006	0.050	0.0417	83	0.0402	80
189-0001	drinking water	20-Jan-2006	0.050	0.0467	93	0.0406	81
194-0001	drinking water	23-Jan-2006	0.050	0.0504	101	0.0505	101
194-0001	drinking water	23-Jan-2006	0.050	0.0502	100	0.0464	93
194-0001	drinking water	23-Jan-2006	0.050	0.0500	100	0.0439	88
189-0001	drinking water	20-Jan-2006	5.00	4.510	90	4.611	92
189-0001	drinking water	20-Jan-2006	5.00	4.713	94	4.628	93
189-0001	drinking water	20-Jan-2006	5.00	4.640	93	4.482	90
194-0001	drinking water	23-Jan-2006	5.00	4.699	94	4.605	92
194-0001	drinking water	23-Jan-2006	5.00	4.999	100	4.409	88
194-0001	drinking water	23-Jan-2006	5.00	4.880	98	4.613	92

[a] The 'Date of Analysis' indicates the date that the samples were prepared for analysis.
[b] All calculations were performed using Microsoft Excel 2002 with full precision.
[c] ND = not detected. The residue was below the 0.015-µg/L limit of detection and no background subtraction was performed.
[d] Samples fortified at the method's limit of detection of 0.015 µg/L are reported with a lower degree of confidence than values above the limit of quantitation.
[e] NA = not applicable. Samples were fortified below the method's limit of quantitation of 0.050 µg/L.

Table 15. Recovery of the Sulfinic Acid Metabolite of XDE-742 from Ground Water

Sample Number	Sample Matrix	Date of Analysis[a]	Analyte Added (µg/L)	Sulfinic Quant (Q1/Q3 *m/z* 239.9/175.8) Found (µg/L)[b]	Sulfinic Quant (Q1/Q3 *m/z* 239.9/175.8) % Recovery	Sulfinic Confirm (Q1/Q3 *m/z* 239.9/155.7) Found (µg/L)[b]	Sulfinic Confirm (Q1/Q3 *m/z* 239.9/155.7) % Recovery[c]
190-0001	ground water	20-Jan-2006	0.000	ND[d]	--	ND	--
193-0001	ground water	23-Jan-2006	0.000	ND	--	0.0019	--
190-0001	ground water	20-Jan-2006	0.015	0.0134[e]	NA[f]	0.0103	NA
193-0001	ground water	23-Jan-2006	0.015	0.0138	NA	0.0103	NA
190-0001	ground water	20-Jan-2006	0.050	0.0449	90	0.0411	82
190-0001	ground water	20-Jan-2006	0.050	0.0471	94	0.0357	71
190-0001	ground water	20-Jan-2006	0.050	0.0420	84	0.0420	84
193-0001	ground water	23-Jan-2006	0.050	0.0491	98	0.0385	77
193-0001	ground water	23-Jan-2006	0.050	0.0508	102	0.0403	81
193-0001	ground water	23-Jan-2006	0.050	0.0438	88	0.0412	82
190-0001	ground water	20-Jan-2006	5.00	4.421	88	4.519	90
190-0001	ground water	20-Jan-2006	5.00	4.584	92	4.415	88
190-0001	ground water	20-Jan-2006	5.00	4.316	86	4.864	97
193-0001	ground water	23-Jan-2006	5.00	5.032	101	4.742	95
193-0001	ground water	23-Jan-2006	5.00	4.755	95	4.591	92
193-0001	ground water	23-Jan-2006	5.00	4.577	92	4.752	95

[a] The 'Date of Analysis' indicates the date that the samples were prepared for analysis.
[b] All calculations were performed using Microsoft Excel 2002 with full precision.
[c] Corrected for contribution from the control sample where appropriate.
[d] ND = not detected. The residue was below the 0.015-µg/L limit of detection and no background subtraction was performed.
[e] Samples fortified at the method's limit of detection of 0.015 µg/L are reported with a lower degree of confidence than values above the limit of quantitation.
[f] NA = not applicable. Samples were fortified below the method's limit of quantitation of 0.050 µg/L.

Table 16. Recovery of the Sulfinic Acid Metabolite of XDE-742 from Surface Water

Sample Number	Sample Matrix	Date of Analysis[a]	Analyte Added (µg/L)	Sulfinic Quant (Q1/Q3 *m/z* 239.9/175.8) Found (µg/L)[b]	Sulfinic Quant (Q1/Q3 *m/z* 239.9/175.8) % Recovery	Sulfinic Confirm (Q1/Q3 *m/z* 239.9/155.7) Found (µg/L)[b]	Sulfinic Confirm (Q1/Q3 *m/z* 239.9/155.7) % Recovery[c]
191-0001	surface water	20-Jan-2006	0.000	ND[d]	--	ND	--
192-0001	surface water	23-Jan-2006	0.000	ND	--	0.0034	--
191-0001	surface water	20-Jan-2006	0.015	0.0151[e]	NA[f]	0.0154	NA
192-0001	surface water	23-Jan-2006	0.015	0.0178	NA	0.0090	NA
191-0001	surface water	20-Jan-2006	0.050	0.0455	91	0.0443	89
191-0001	surface water	20-Jan-2006	0.050	0.0418	84	0.0437	87
191-0001	surface water	20-Jan-2006	0.050	0.0482	96	0.0529	106
192-0001	surface water	23-Jan-2006	0.050	0.0501	100	0.0420	84
192-0001	surface water	23-Jan-2006	0.050	0.0510	102	0.0475	95
192-0001	surface water	23-Jan-2006	0.050	0.0463	93	0.0481	96
191-0001	surface water	20-Jan-2006	5.00	4.336	87	4.381	88
191-0001	surface water	20-Jan-2006	5.00	4.771	95	4.587	92
191-0001	surface water	20-Jan-2006	5.00	4.675	94	4.415	88
192-0001	surface water	23-Jan-2006	5.00	4.350	87	4.065	81
192-0001	surface water	23-Jan-2006	5.00	5.033	101	4.733	95
192-0001	surface water	23-Jan-2006	5.00	5.032	101	4.491	90

[a] The 'Date of Analysis' indicates the date that the samples were prepared for analysis.
[b] All calculations were performed using Microsoft Excel 2002 with full precision.
[c] Corrected for contribution from the control sample where appropriate.
[d] ND = not detected. The residue was below the 0.015-µg/L limit of detection and no background subtraction was performed.
[e] Samples fortified at the method's limit of detection of 0.015 µg/L are reported with a lower degree of confidence than values above the limit of quantitation.
[f] NA = not applicable. Samples were fortified below the method's limit of quantitation of 0.050 µg/L.

Table 17. Recovery of the Sulfonic Acid Metabolite of XDE-742 from Drinking Water

Sample Number	Sample Matrix	Date of Analysis[a]	Analyte Added (µg/L)	Sulfonic Quant (Q1/Q3 *m/z* 255.7/149.0) Found (µg/L)[b]	% Recovery	Sulfonic Confirm (Q1/Q3 *m/z* 255.7/79.7) Found (µg/L)[b]	% Recovery
189-0001	drinking water	20-Jan-2006	0.000	ND[c]	--	ND	--
194-0001	drinking water	23-Jan-2006	0.000	ND	--	ND	--
189-0001	drinking water	20-Jan-2006	0.015	0.0159[d]	NA[e]	0.0178	NA
194-0001	drinking water	23-Jan-2006	0.015	0.0059	NA	0.0116	NA
189-0001	drinking water	20-Jan-2006	0.050	0.0536	107	0.0480	96
189-0001	drinking water	20-Jan-2006	0.050	0.0469	94	0.0580	116
189-0001	drinking water	20-Jan-2006	0.050	0.0417	83	0.0563	113
194-0001	drinking water	23-Jan-2006	0.050	0.0442	88	0.0541	108
194-0001	drinking water	23-Jan-2006	0.050	0.0384	77	0.0472	94
194-0001	drinking water	23-Jan-2006	0.050	0.0414	83	0.0548	110
189-0001	drinking water	20-Jan-2006	5.00	4.621	92	4.795	96
189-0001	drinking water	20-Jan-2006	5.00	4.529	91	4.569	91
189-0001	drinking water	20-Jan-2006	5.00	4.431	89	4.603	92
194-0001	drinking water	23-Jan-2006	5.00	4.777	96	4.810	96
194-0001	drinking water	23-Jan-2006	5.00	4.885	98	4.823	96
194-0001	drinking water	23-Jan-2006	5.00	4.778	96	4.919	98

[a] The 'Date of Analysis' indicates the date that the samples were prepared for analysis.
[b] All calculations were performed using Microsoft Excel 2002 with full precision.
[c] ND = not detected. The residue was below the 0.015-µg/L limit of detection and no background subtraction was performed.
[d] Samples fortified at the method's limit of detection of 0.015 µg/L are reported with a lower degree of confidence than values above the limit of quantitation.
[e] NA = not applicable. Samples were fortified below the method's limit of quantitation of 0.050 µg/L.

Table 18. Recovery of the Sulfonic Acid Metabolite of XDE-742 from Ground Water

Sample Number	Sample Matrix	Date of Analysis[a]	Analyte Added (µg/L)	Sulfonic Quant (Q1/Q3 *m/z* 255.7/149.0) Found (µg/L)[b]	Sulfonic Quant (Q1/Q3 *m/z* 255.7/149.0) % Recovery	Sulfonic Confirm (Q1/Q3 *m/z* 255.7/79.7) Found (µg/L)[b]	Sulfonic Confirm (Q1/Q3 *m/z* 255.7/79.7) % Recovery
190-0001	ground water	20-Jan-2006	0.000	ND	--	ND[c]	--
193-0001	ground water	23-Jan-2006	0.000	ND	--	ND	--
190-0001	ground water	20-Jan-2006	0.015	0.0165[d]	NA[e]	0.0163	NA
193-0001	ground water	23-Jan-2006	0.015	0.0112	NA	0.0088	NA
190-0001	ground water	20-Jan-2006	0.050	0.0502	100	0.0431	86
190-0001	ground water	20-Jan-2006	0.050	0.0425	85	0.0466	93
190-0001	ground water	20-Jan-2006	0.050	0.0450	90	0.0359	72
193-0001	ground water	23-Jan-2006	0.050	0.0438	88	0.0490	98
193-0001	ground water	23-Jan-2006	0.050	0.0423	85	0.0564	113
193-0001	ground water	23-Jan-2006	0.050	0.0362	72	0.0492	98
190-0001	ground water	20-Jan-2006	5.00	4.452	89	4.549	91
190-0001	ground water	20-Jan-2006	5.00	4.649	93	4.632	93
190-0001	ground water	20-Jan-2006	5.00	4.550	91	4.643	93
193-0001	ground water	23-Jan-2006	5.00	4.880	98	4.766	95
193-0001	ground water	23-Jan-2006	5.00	4.778	96	4.739	95
193-0001	ground water	23-Jan-2006	5.00	4.762	95	4.716	94

[a] The 'Date of Analysis' indicates the date that the samples were prepared for analysis.
[b] All calculations were performed using Microsoft Excel 2002 with full precision.
[c] ND = not detected. The residue was below the 0.015-µg/L limit of detection and no background subtraction was performed.
[d] Samples fortified at the method's limit of detection of 0.015 µg/L are reported with a lower degree of confidence than values above the limit of quantitation.
[e] NA = not applicable. Samples were fortified below the method's limit of quantitation of 0.050 µg/L.

Table 19. Recovery of the Sulfonic Acid Metabolite of XDE-742 from Surface Water

Sample Number	Sample Matrix	Date of Analysis[a]	Analyte Added (µg/L)	Sulfonic Quant (Q1/Q3 *m/z* 255.7/149.0) Found (µg/L)[b]	Sulfonic Quant (Q1/Q3 *m/z* 255.7/149.0) % Recovery	Sulfonic Confirm (Q1/Q3 *m/z* 255.7/79.7) Found (µg/L)[b]	Sulfonic Confirm (Q1/Q3 *m/z* 255.7/79.7) % Recovery
191-0001	surface water	20-Jan-2006	0.000	ND[c]	--	ND	--
192-0001	surface water	23-Jan-2006	0.000	ND	--	ND	--
191-0001	surface water	20-Jan-2006	0.015	0.0133[d]	NA[e]	0.0055	NA
192-0001	surface water	23-Jan-2006	0.015	0.0094	NA	0.0051	NA
191-0001	surface water	20-Jan-2006	0.050	0.0494	99	0.0380	76
191-0001	surface water	20-Jan-2006	0.050	0.0434	87	0.0380	76
191-0001	surface water	20-Jan-2006	0.050	0.0497	99	0.0502	100
192-0001	surface water	23-Jan-2006	0.050	0.0438	88	0.0572	114
192-0001	surface water	23-Jan-2006	0.050	0.0415	83	0.0474	95
192-0001	surface water	23-Jan-2006	0.050	0.0488	98	0.0496	99
191-0001	surface water	20-Jan-2006	5.00	4.474	89	4.568	91
191-0001	surface water	20-Jan-2006	5.00	4.524	90	4.786	96
191-0001	surface water	20-Jan-2006	5.00	4.444	89	4.731	95
192-0001	surface water	23-Jan-2006	5.00	4.339	87	4.589	92
192-0001	surface water	23-Jan-2006	5.00	4.902	98	5.016	100
192-0001	surface water	23-Jan-2006	5.00	4.812	96	4.935	99

[a] The 'Date of Analysis' indicates the date that the samples were prepared for analysis.
[b] All calculations were performed using Microsoft Excel 2002 with full precision.
[c] ND = not detected. The residue was below the 0.015-µg/L limit of detection and no background subtraction was performed.
[d] Samples fortified at the method's limit of detection of 0.015 µg/L are reported with a lower degree of confidence than values above the limit of quantitation.
[e] NA = not applicable. Samples were fortified below the method's limit of quantitation of 0.050 µg/L.

Table 20. Recovery Summary of the XDE-742 (Quantitation Ion, Q1/Q3 *m/z* 435.1/195.1) from Water

Sample Matrix	Fortification Level (µg/L)	Average Recovery (Percent)	Recovery Range (Percent)	Standard Deviation (Percent)	Relative Std. Dev. (Percent)	Number of Samples (n)
drinking water	0.050	92	89 - 94	2.1	2.3	6
	5.00	100	98 - 103	1.9	1.9	6
	0.050 - 5.00	96	89 - 103	4.3	4.5	12
ground water	0.050	93	89 - 96	2.2	2.4	6
	5.00	99	97 - 101	1.4	1.4	6
	0.050 - 5.00	96	89 - 101	3.8	4.0	12
surface water	0.050	96	90 - 102	4.8	5.0	6
	5.00	100	95 - 104	3.1	3.1	6
	0.050 - 5.00	98	90 - 104	4.5	4.6	12

Table 21. Recovery Summary of the XDE-742 (Confirmation Ion, Q1/Q3 *m/z* 435.1/82.0) from Water

Sample Matrix	Fortification Level (µg/L)	Average Recovery (Percent)	Recovery Range (Percent)	Standard Deviation (Percent)	Relative Std. Dev. (Percent)	Number of Samples (n)
drinking water	0.050	96	88 - 105	5.7	5.9	6
	5.00	96	94 - 98	1.7	1.7	6
	0.050 - 5.00	96	88 - 105	4.0	4.2	12
ground water	0.050	96	92 - 101	3.8	4.0	6
	5.00	95	94 - 97	1.2	1.3	6
	0.050 - 5.00	96	92 - 101	2.7	2.8	12
surface water	0.050	98	94 - 102	3.4	3.5	6
	5.00	96	92 - 100	3.2	3.3	6
	0.050 - 5.00	97	92 - 102	3.4	3.5	12

Table 22. Recovery Summary of the 7-OH-XDE-742 (Quantitation Ion, Q1/Q3 *m/z* 420.9/181.0) from Water

Sample Matrix	Fortification Level (µg/L)	Average Recovery (Percent)	Recovery Range (Percent)	Standard Deviation (Percent)	Relative Std. Dev. (Percent)	Number of Samples (n)
drinking water	0.050	105	100 - 110	3.3	3.1	6
	5.00	101	99 - 104	2.1	2.1	6
	0.050 - 5.00	103	99 - 110	3.3	3.2	12
ground water	0.050	92	87 - 96	3.5	3.8	6
	5.00	99	97 - 101	1.7	1.8	6
	0.050 - 5.00	95	87 - 101	4.7	4.9	12
surface water	0.050	100	92 - 108	7.7	7.7	6
	5.00	105	99 - 113	6.1	5.8	6
	0.050 - 5.00	103	92 - 113	7.1	6.9	12

Table 23. Recovery Summary of the 7-OH-XDE-742 (Confirmation Ion, Q1/Q3 *m/z* 420.9/148.1) from Water

Sample Matrix	Fortification Level (µg/L)	Average Recovery (Percent)	Recovery Range (Percent)	Standard Deviation (Percent)	Relative Std. Dev. (Percent)	Number of Samples (n)
drinking water	0.050	99	91 - 108	7.1	7.2	6
	5.00	101	98 - 104	2.2	2.2	6
	0.050 - 5.00	100	91 - 108	5.1	5.1	12
ground water	0.050	97	88 - 106	6.7	6.9	6
	5.00	100	97 - 101	1.6	1.6	6
	0.050 - 5.00	98	88 - 106	4.8	4.9	12
surface water	0.050	104	98 - 110	4.7	4.5	6
	5.00	105	99 - 113	6.0	5.7	6
	0.050 - 5.00	104	98 - 113	5.2	4.9	12

Table 24. Recovery Summary of the ADTP (Quantitation Ion, Q1/Q3 *m/z* 196.2/115.1) from Water

Sample Matrix	Fortification Level (µg/L)	Average Recovery (Percent)	Recovery Range (Percent)	Standard Deviation (Percent)	Relative Std. Dev. (Percent)	Number of Samples (n)
drinking water	0.050	97	88 - 101	4.6	4.8	6
	5.00	100	96 - 104	3.4	3.4	6
	0.050 - 5.00	99	88 - 104	4.1	4.2	12
ground water	0.050	94	90 - 100	3.6	3.8	6
	5.00	98	94 - 100	1.9	2.0	6
	0.050 - 5.00	96	90 - 100	3.3	3.4	12
surface water	0.050	96	88 - 102	6.0	6.2	6
	5.00	101	97 - 105	3.3	3.3	6
	0.050 - 5.00	99	88 - 105	5.4	5.4	12

Table 25. Recovery Summary of the ADTP (Confirmation Ion, Q1/Q3 *m/z* 196.2/163.9) from Water

Sample Matrix	Fortification Level (µg/L)	Average Recovery (Percent)	Recovery Range (Percent)	Standard Deviation (Percent)	Relative Std. Dev. (Percent)	Number of Samples (n)
drinking water	0.050	95	85 - 103	5.9	6.2	6
	5.00	100	97 - 104	3.4	3.4	6
	0.050 - 5.00	97	85 - 104	5.4	5.6	12
ground water	0.050	90	83 - 97	5.6	6.2	6
	5.00	98	94 - 100	2.5	2.6	6
	0.050 - 5.00	94	83 - 100	5.6	6.0	12
surface water	0.050	99	90 - 105	5.8	5.9	6
	5.00	100	95 - 103	2.9	2.9	6
	0.050 - 5.00	99	90 - 105	4.4	4.4	12

Table 26. Recovery Summary of the ATSA (Quantitation Ion., Q1/Q3 *m/z* 339.0/99.1) from Water

Sample Matrix	Fortification Level (µg/L)	Average Recovery (Percent)	Recovery Range (Percent)	Standard Deviation (Percent)	Relative Std. Dev. (Percent)	Number of Samples (n)
drinking water	0.050	112	109 - 114	1.9	1.7	6
	5.00	99	96 - 100	1.6	1.6	6
	0.050 - 5.00	105	96 - 114	7.0	6.7	12
ground water	0.050	113	109 - 118	4.2	3.7	6
	5.00	98	96 - 100	1.1	1.1	6
	0.050 - 5.00	105	96 - 118	8.5	8.0	12
surface water	0.050	112	106 - 119	5.8	5.1	6
	5.00	98	93 - 101	2.9	3.0	6
	0.050 - 5.00	105	93 - 119	8.5	8.1	12

Table 27. Recovery Summary of the ATSA (Confirmation Ion, Q1/Q3 *m/z* 339.0/57.2) from Water

Sample Matrix	Fortification Level (µg/L)	Average Recovery (Percent)	Recovery Range (Percent)	Standard Deviation (Percent)	Relative Std. Dev. (Percent)	Number of Samples (n)
drinking water	0.050	105	90 - 119	11.3	10.8	6
	5.00	97	94 - 100	2.6	2.7	6
	0.050 - 5.00	101	90 - 119	8.8	8.7	12
ground water	0.050	108	92 - 118	9.9	9.1	6
	5.00	97	95 - 98	1.1	1.1	6
	0.050 - 5.00	103	92 - 118	9.1	8.9	12
surface water	0.050	103	89 - 117	10.3	10.0	6
	5.00	96	91 - 100	3.0	3.1	6
	0.050 - 5.00	100	89 - 117	8.1	8.1	12

Table 28. Recovery Summary of the Sulfinic Acid (Quantitation Ion, Q1/Q3 *m/z* 239.9/175.8) from Water

Sample Matrix	Fortification Level (µg/L)	Average Recovery (Percent)	Recovery Range (Percent)	Standard Deviation (Percent)	Relative Std. Dev. (Percent)	Number of Samples (n)
drinking water	0.050	95	83 - 101	6.8	7.1	6
	5.00	95	90 - 100	3.5	3.7	6
	0.050 - 5.00	95	83 - 101	5.1	5.4	12
ground water	0.050	93	84 - 102	6.6	7.2	6
	5.00	92	86 - 101	5.1	5.5	6
	0.050 - 5.00	92	84 - 102	5.6	6.1	12
surface water	0.050	94	84 - 102	6.8	7.2	6
	5.00	94	87 - 101	6.2	6.6	6
	0.050 - 5.00	94	84 - 102	6.2	6.6	12

Table 29. Recovery Summary of the Sulfinic Acid (Confirmation Ion, Q1/Q3 *m/z* 239.9/155.7) from Water

Sample Matrix	Fortification Level (µg/L)	Average Recovery (Percent)	Recovery Range (Percent)	Standard Deviation (Percent)	Relative Std. Dev. (Percent)	Number of Samples (n)
drinking water	0.050	87	79 - 101	8.7	10.0	6
	5.00	91	88 - 93	1.8	2.0	6
	0.050 - 5.00	89	79 - 101	6.4	7.1	12
ground water	0.050	80	71 - 84	4.7	5.9	6
	5.00	93	88 - 97	3.3	3.6	6
	0.050 - 5.00	86	71 - 97	8.0	9.3	12
surface water	0.050	93	84 - 106	7.9	8.5	6
	5.00	89	81 - 95	4.5	5.1	6
	0.050 - 5.00	91	81 - 106	6.5	7.1	12

Table 30. Recovery Summary of the Sulfonic Acid (Quantitation Ion, Q1/Q3 *m/z* 255.7/149.0) from Water

Sample Matrix	Fortification Level (µg/L)	Average Recovery (Percent)	Recovery Range (Percent)	Standard Deviation (Percent)	Relative Std. Dev. (Percent)	Number of Samples (n)
drinking water	0.050	89	77 - 107	10.7	12.1	6
	5.00	93	89 - 98	3.5	3.7	6
	0.050 - 5.00	91	77 - 107	8.0	8.7	12
ground water	0.050	87	72 - 100	9.1	10.5	6
	5.00	94	89 - 98	3.2	3.4	6
	0.050 - 5.00	90	72 - 100	7.4	8.2	12
surface water	0.050	92	83 - 99	7.2	7.8	6
	5.00	92	87 - 98	4.5	4.9	6
	0.050 - 5.00	92	83 - 99	5.7	6.2	12

Table 31. Recovery Summary of the Sulfonic Acid Confirmation Ion, Q1/Q3 *m/z* 255.7/79.7) from Water

Sample Matrix	Fortification Level (µg/L)	Average Recovery (Percent)	Recovery Range (Percent)	Standard Deviation (Percent)	Relative Std. Dev. (Percent)	Number of Samples (n)
drinking water	0.050	106	94 - 116	8.9	8.4	6
	5.00	95	91 - 98	2.7	2.9	6
	0.050 - 5.00	101	91 - 116	8.5	8.5	12
ground water	0.050	93	72 - 113	13.7	14.7	6
	5.00	93	91 - 95	1.6	1.7	6
	0.050 - 5.00	93	72 - 113	9.3	10.0	12
surface water	0.050	93	76 - 114	15.0	16.1	6
	5.00	95	91 - 100	3.6	3.8	6
	0.050 - 5.00	94	76 - 114	10.5	11.1	12

Table 32. Calculated Limits of Detection and Quantitation for the Determination of XDE-742 (Quantitation Ion, Q1/Q3 *m/z* 435.1/195.1) in Water

Sample Matrix	Fortification Level (µg/L)	Average Recovery (µg/L)	Standard Deviation (*s*)	Limit of Detection (3*s*)	Limit of Quantitation (10*s*)	Number of Samples (n)
drinking water	0.050	0.0462	0.0010	0.0031	0.0105	6
ground water	0.050	0.0464	0.0011	0.0033	0.0110	6
surface water	0.050	0.0479	0.0024	0.0072	0.0240	6

Table 33. Calculated Limits of Detection and Quantitation for the Determination of XDE-742 (Confirmation Ion, Q1/Q3 *m/z* 435.1/82.0) in Water

Sample Matrix	Fortification Level (µg/L)	Average Recovery (µg/L)	Standard Deviation (*s*)	Limit of Detection (3*s*)	Limit of Quantitation (10*s*)	Number of Samples (n)
drinking water	0.050	0.0482	0.0029	0.0086	0.0285	6
ground water	0.050	0.0479	0.0019	0.0057	0.0189	6
surface water	0.050	0.0491	0.0017	0.0051	0.0171	6

Table 34. Calculated Limits of Detection and Quantitation for the Determination of 7-OH-XDE-742 (Quantitation Ion, Q1/Q3 *m/z* 420.9/181.0) in Water

Sample Matrix	Fortification Level (µg/L)	Average Recovery (µg/L)	Standard Deviation (*s*)	Limit of Detection (3*s*)	Limit of Quantitation (10*s*)	Number of Samples (n)
drinking water	0.050	0.0525	0.0016	0.0049	0.0164	6
ground water	0.050	0.0458	0.0018	0.0053	0.0176	6
surface water	0.050	0.0502	0.0039	0.0116	0.0385	6

Table 35. Calculated Limits of Detection and Quantitation for the Determination of 7-OH-XDE-742 (Confirmation Ion, Q1/Q3 *m/z* 420.9/148.1) in Water

Sample Matrix	Fortification Level (µg/L)	Average Recovery (µg/L)	Standard Deviation (*s*)	Limit of Detection (3*s*)	Limit of Quantitation (10*s*)	Number of Samples (n)
drinking water	0.050	0.0496	0.0036	0.0107	0.0356	6
ground water	0.050	0.0485	0.0033	0.0100	0.0333	6
surface water	0.050	0.0520	0.0024	0.0071	0.0236	6

Table 36. Calculated Limits of Detection and Quantitation for the Determination of ADTP (Quantitation Ion, Q1/Q3 *m/z* 196.2/115.1) in Water

Sample Matrix	Fortification Level (µg/L)	Average Recovery (µg/L)	Standard Deviation (*s*)	Limit of Detection (3*s*)	Limit of Quantitation (10*s*)	Number of Samples (n)
drinking water	0.050	0.0486	0.0023	0.0070	0.0232	6
ground water	0.050	0.0471	0.0018	0.0054	0.0180	6
surface water	0.050	0.0481	0.0030	0.0089	0.0298	6

Table 37. Calculated Limits of Detection and Quantitation for the Determination of ADTP (Confirmation Ion, Q1/Q3 *m/z* 196.2/163.9) in Water

Sample Matrix	Fortification Level (µg/L)	Average Recovery (µg/L)	Standard Deviation (*s*)	Limit of Detection (3*s*)	Limit of Quantitation (10*s*)	Number of Samples (n)
drinking water	0.050	0.0473	0.0030	0.0089	0.0296	6
ground water	0.050	0.0451	0.0028	0.0084	0.0279	6
surface water	0.050	0.0494	0.0029	0.0087	0.0289	6

Table 38. Calculated Limits of Detection and Quantitation for the Determination of ATSA (Quantitation Ion, Q1/Q3 *m/z* 339.0/99.1) in Water

Sample Matrix	Fortification Level (µg/L)	Average Recovery (µg/L)	Standard Deviation (*s*)	Limit of Detection (3*s*)	Limit of Quantitation (10*s*)	Number of Samples (n)
drinking water	0.050	0.0558	0.0010	0.0029	0.0095	6
ground water	0.050	0.0565	0.0021	0.0063	0.0211	6
surface water	0.050	0.0559	0.0029	0.0086	0.0288	6

Table 39. Calculated Limits of Detection and Quantitation for the Determination of ATSA (Confirmation Ion, Q1/Q3 *m/z* 339.0/57.2) in Water

Sample Matrix	Fortification Level (µg/L)	Average Recovery (µg/L)	Standard Deviation (*s*)	Limit of Detection (3*s*)	Limit of Quantitation (10*s*)	Number of Samples (n)
drinking water	0.050	0.0523	0.0056	0.0169	0.0565	6
ground water	0.050	0.0542	0.0049	0.0148	0.0495	6
surface water	0.050	0.0516	0.0052	0.0155	0.0515	6

Table 40. Calculated Limits of Detection and Quantitation for the Determination of Sulfinic Acid (Quantitation Ion, Q1/Q3 *m/z* 239.9/175.8) in Water

Sample Matrix	Fortification Level (µg/L)	Average Recovery (µg/L)	Standard Deviation (*s*)	Limit of Detection (3*s*)	Limit of Quantitation (10*s*)	Number of Samples (n)
drinking water	0.050	0.0475	0.0034	0.0102	0.0340	6
ground water	0.050	0.0463	0.0033	0.0100	0.0332	6
surface water	0.050	0.0471	0.0034	0.0101	0.0338	6

Table 41. Calculated Limits of Detection and Quantitation for the Determination of Sulfinic Acid (Confirmation Ion, Q1/Q3 *m/z* 239.9/155.7) in Water

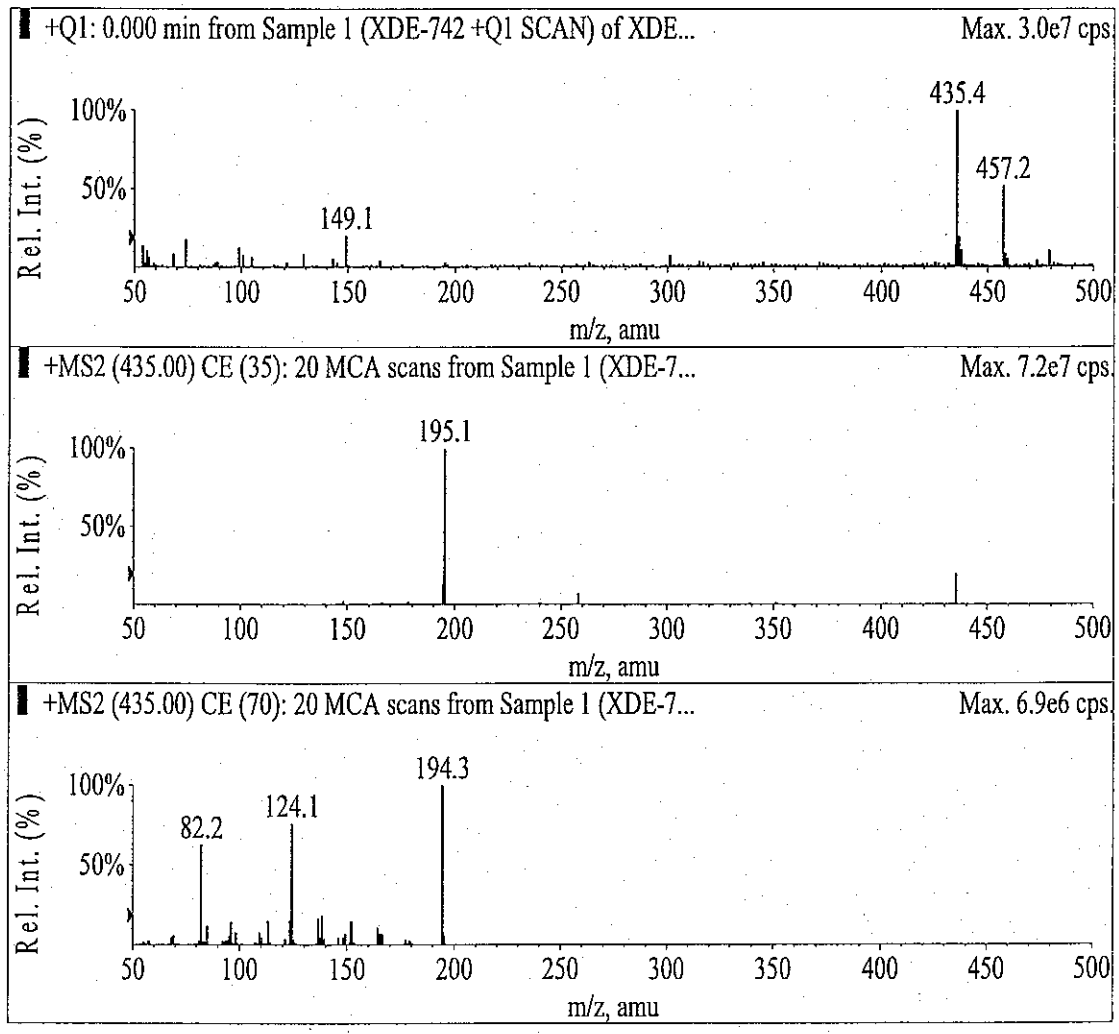
Sample Matrix	Fortification Level (µg/L)	Average Recovery (µg/L)	Standard Deviation (*s*)	Limit of Detection (3*s*)	Limit of Quantitation (10*s*)	Number of Samples (n)
drinking water	0.050	0.0435	0.0043	0.0130	0.0434	6
ground water	0.050	0.0398	0.0023	0.0070	0.0235	6
surface water	0.050	0.0464	0.0039	0.0118	0.0394	6

Table 42. Calculated Limits of Detection and Quantitation for the Determination of Sulfonic Acid (Quantitation Ion, Q1/Q3 *m/z* 255.7/149.0) in Water

Sample Matrix	Fortification Level (µg/L)	Average Recovery (µg/L)	Standard Deviation (*s*)	Limit of Detection (3*s*)	Limit of Quantitation (10*s*)	Number of Samples (n)
drinking water	0.050	0.0444	0.0054	0.0161	0.0535	6
ground water	0.050	0.0433	0.0046	0.0137	0.0455	6
surface water	0.050	0.0461	0.0036	0.0108	0.0361	6

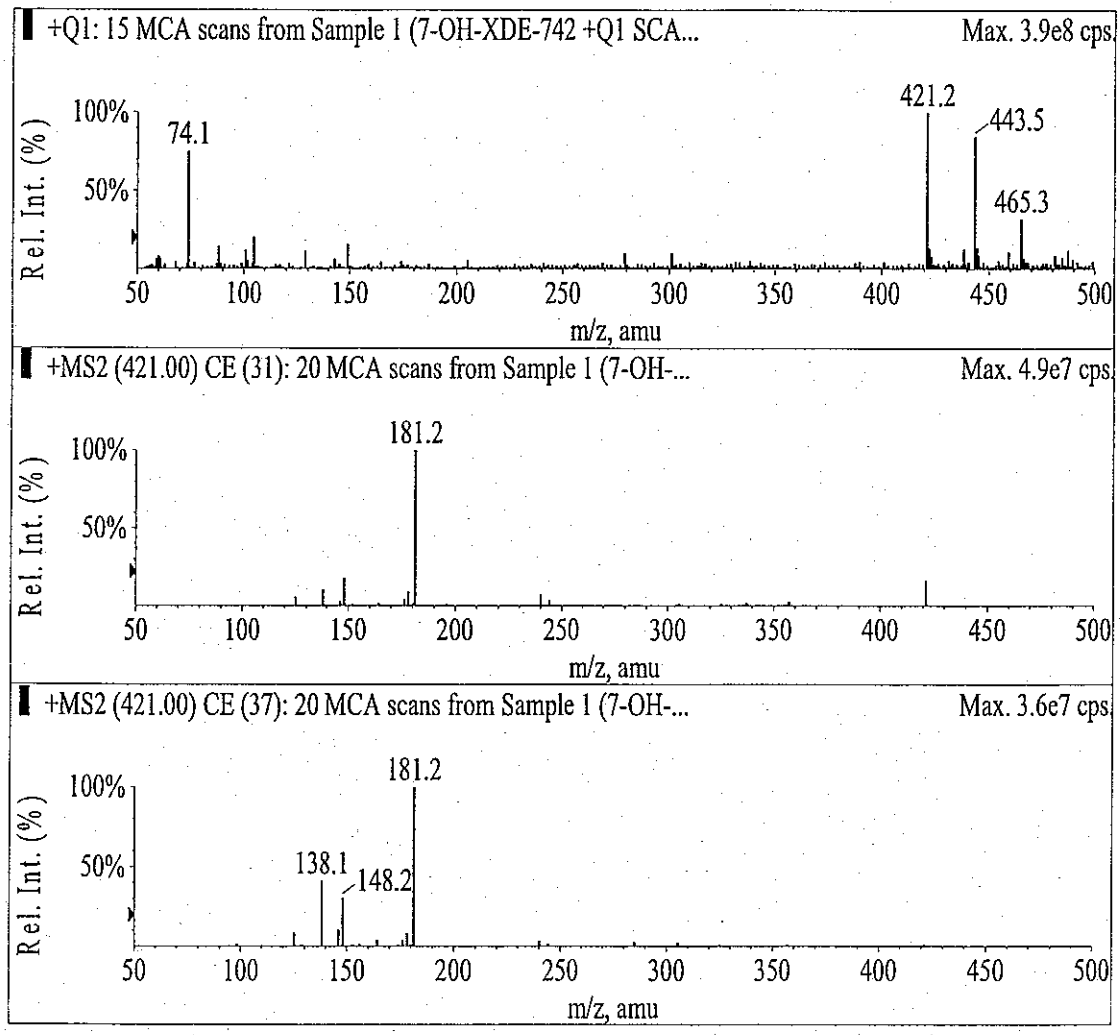
Table 43. Calculated Limits of Detection and Quantitation for the Determination of Sulfonic Acid (Confirmation Ion, Q1/Q3 *m/z* 255.7/79.7) in Water

Sample Matrix	Fortification Level (µg/L)	Average Recovery (µg/L)	Standard Deviation (*s*)	Limit of Detection (3*s*)	Limit of Quantitation (10*s*)	Number of Samples (n)
drinking water	0.050	0.0531	0.0045	0.0134	0.0445	6
ground water	0.050	0.0467	0.0069	0.0206	0.0687	6
surface water	0.050	0.0467	0.0075	0.0225	0.0751	6



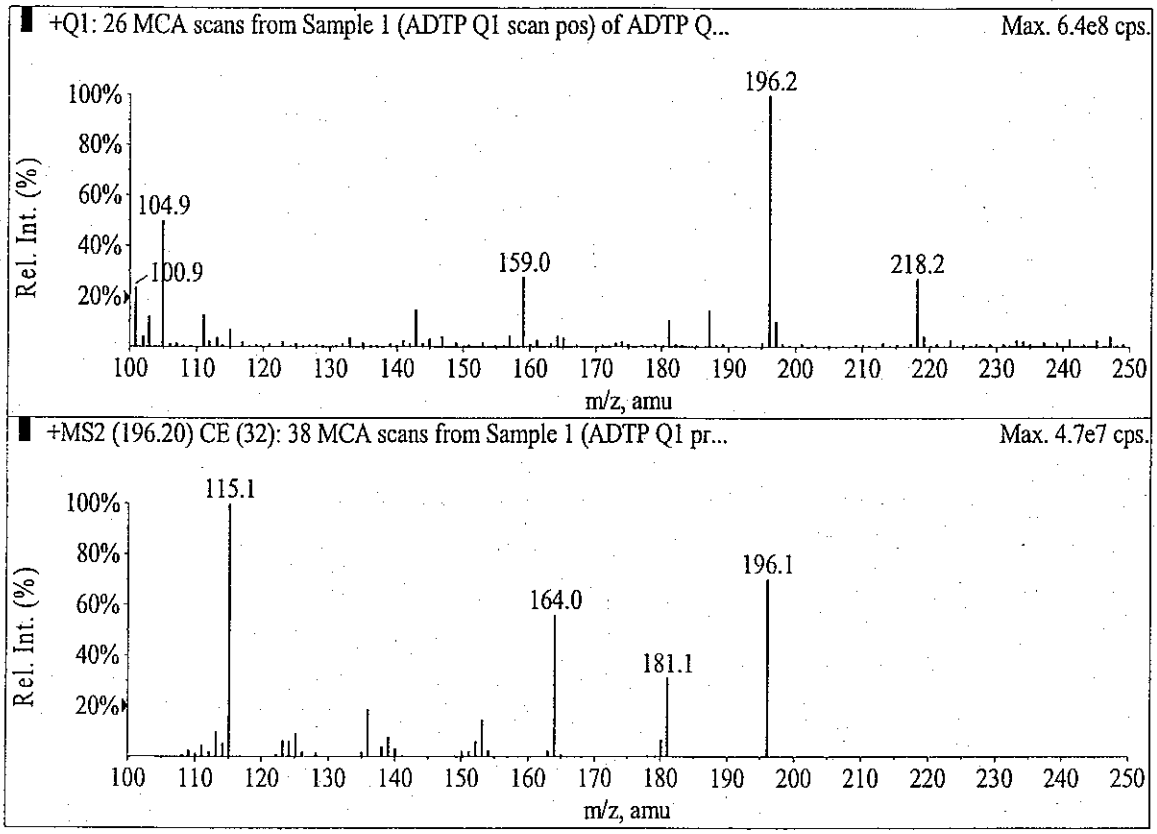
Top: Positive-ion scan showing $(M+H)^+$ at *m/z* 435
Middle: Product ion scan showing fragment used for quantitation at *m/z* 195
Bottom: Product ion scan showing fragment used for confirmation at *m/z* 82

Figure 1. Typical Positive-Ion Mass Spectra of XDE-742



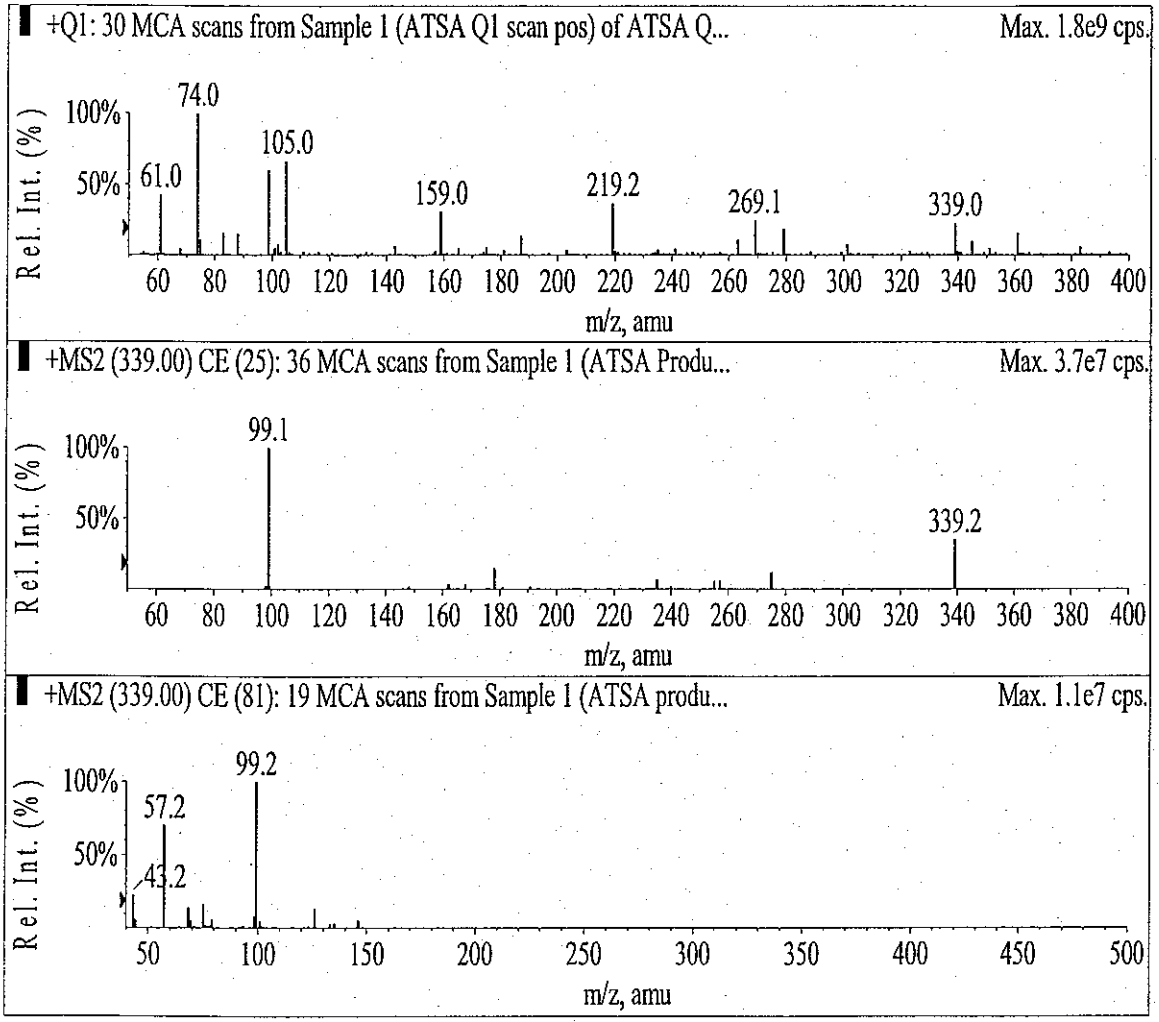
Top: Positive-ion scan showing $(M+H)^+$ at *m/z* 421
Middle: Product ion scan showing fragment used for quantitation at *m/z* 181
Bottom: Product ion scan showing fragment used for confirmation at *m/z* 148

Figure 2. Typical Positive-Ion Mass Spectra of 7-OH-XDE-742 Metabolite



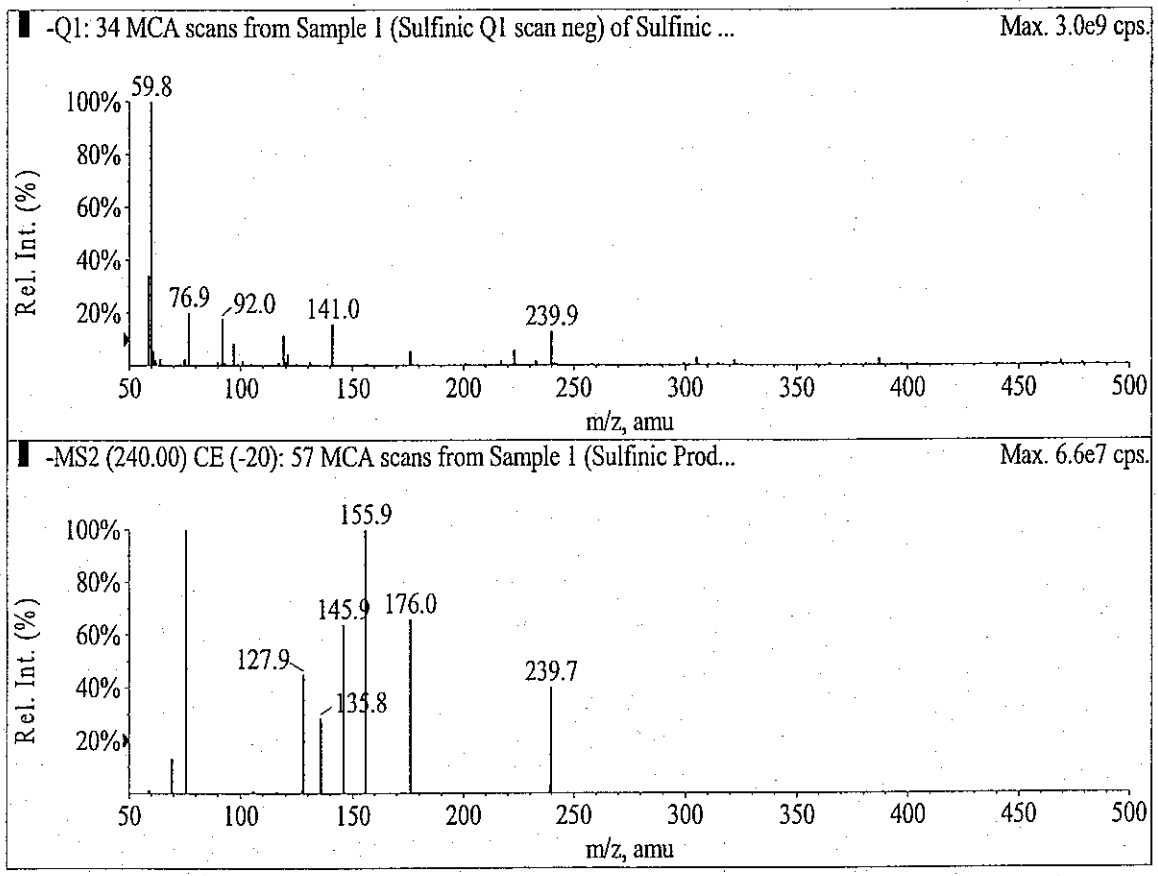
Top: Positive-ion scan showing $(M+H)^+$ at *m/z* 196
Bottom: Product ion scan showing fragment used for quantitation at *m/z* 115 and fragment used for confirmation at *m/z* 164

Figure 3. Typical Positive-Ion Mass Spectra of ADTP Metabolite of XDE-742



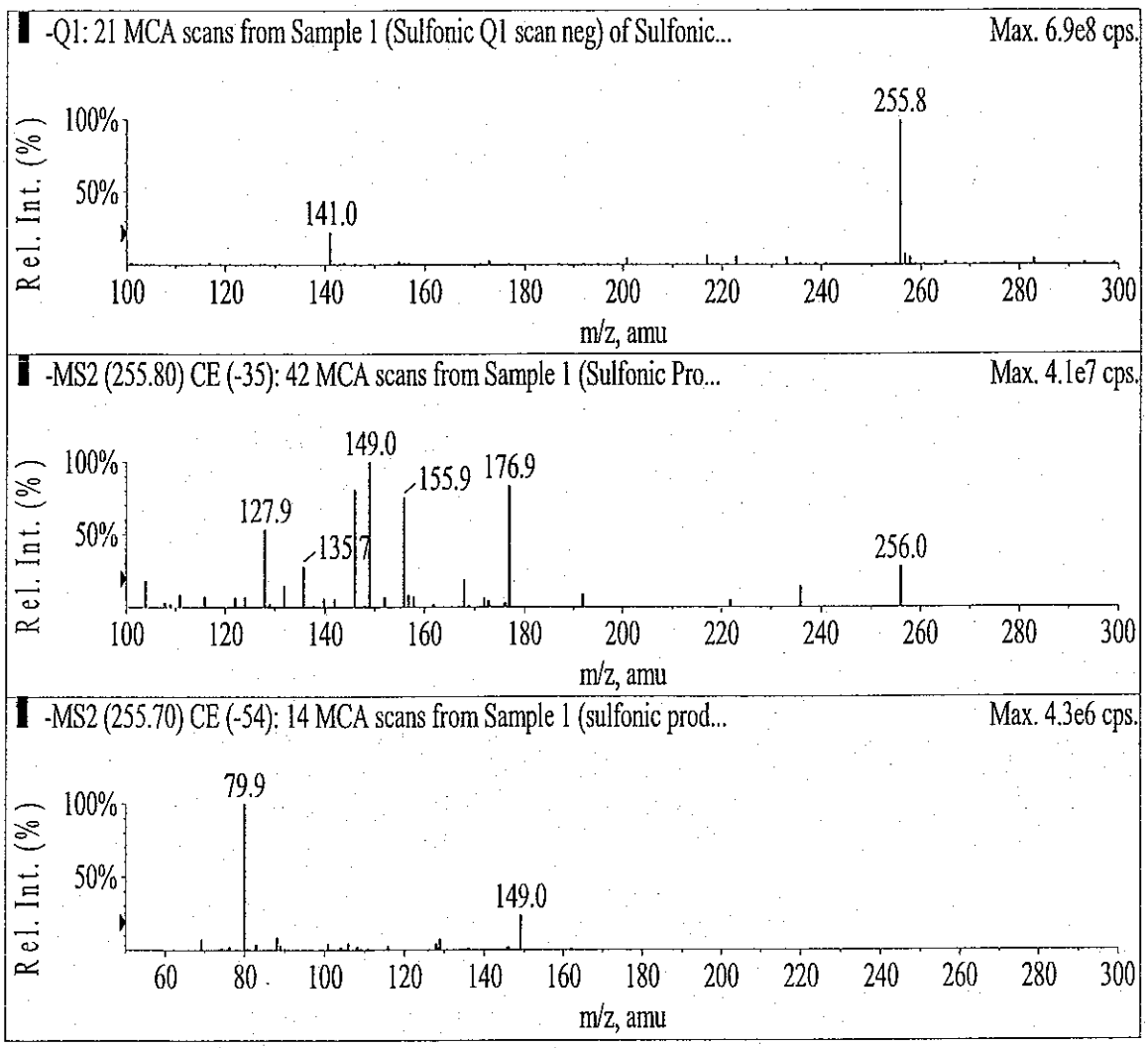
Top: Positive-ion scan showing $(M+H)^+$ at *m/z* 339
Middle: Product ion scan showing fragment used for quantitation at *m/z* 99
Bottom: Product ion scan showing fragment used for confirmation at *m/z* 57

Figure 4. Typical Positive-Ion Mass Spectra of ATSA Metabolite of XDE-742



Top: Negative-ion scan showing $(M-H)^-$ at *m/z* 240
Bottom: Product ion scan showing fragment used for quantitation at *m/z* 176 and fragment used for confirmation at *m/z* 156

Figure 5. Typical Negative-Ion Mass Spectra of Sulfinic Acid Metabolite of XDE-742



Top: Negative-ion scan showing $(M-H)^-$ at *m/z* 256
Middle: Product ion scan showing fragment used for quantitation at *m/z* 149
Bottom: Product ion scan showing fragment used for confirmation at *m/z* 80

Figure 6. Typical Negative-Ion Mass Spectra of Sulfonic Acid Metabolite of XDE-742

Analytical Set I.D.: 051039 S05 pos
Compound: XDE-742 Quant (Q1/Q3 *m/z* 435.1/195.1)

Calibration Data

Linear with 1/x Weighting

Slope =	2804644
Intercept =	30225.8087
r^2 =	0.9972

Standard Concentration (ng/mL)	Injection Number	Analyte Peak Area	Response Factor	Calculated Concentration	Percent of Theoretical
0.015	1	59380	3958667	0.01039	69
0.05	6	168747	3374940	0.04939	99
0.25	11	791836	3167344	0.27155	109
0.5	16	1558167	3116334	0.54479	109
1	21	3100259	3100259	1.09462	109
2.5	26	7531053	3012421	2.67443	107
5	31	14390892	2878178	5.12032	102
10	33	26813170	2681317	9.54950	95

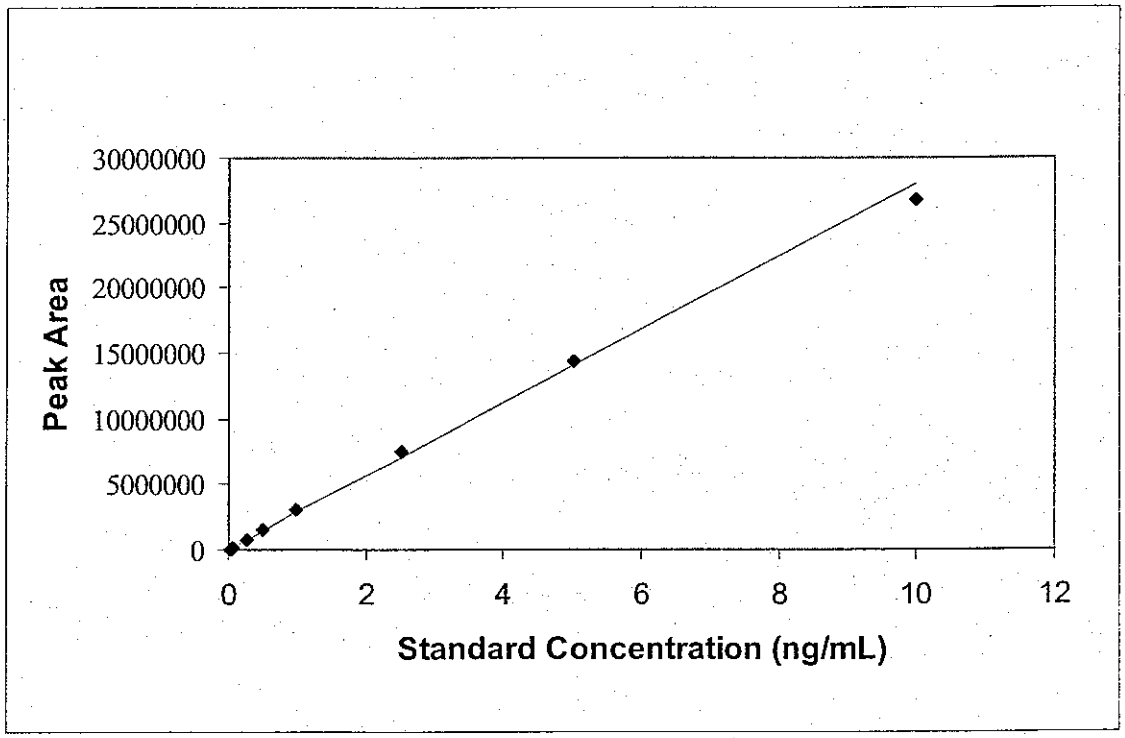


Figure 7. Typical Calibration Curve for the Determination of XDE-742 (Quantitation Ion, Q1/Q3 *m/z* 435.1/195.1) in Water

Analytical Set I.D.: 051039 S05 pos
Compound: XDE-742 Confirm (Q1/Q3 *m/z* 435.1/82.0)

Calibration Data

Linear with 1/x Weighting

Slope =	309835
Intercept =	1636.6990
r^2 =	0.9999

Standard Concentration (ng/mL)	Injection Number	Analyte Peak Area	Response Factor	Calculated Concentration	Percent of Theoretical
0.015	1	5858	390533	0.01362	91
0.05	6	17430	348600	0.05097	102
0.25	11	82753	331012	0.26180	105
0.5	16	162497	324994	0.51918	104
1	21	309982	309982	0.99519	100
2.5	26	776840	310736	2.50199	100
5	31	1530539	306108	4.93457	99
10	33	3111661	311166	10.03767	100

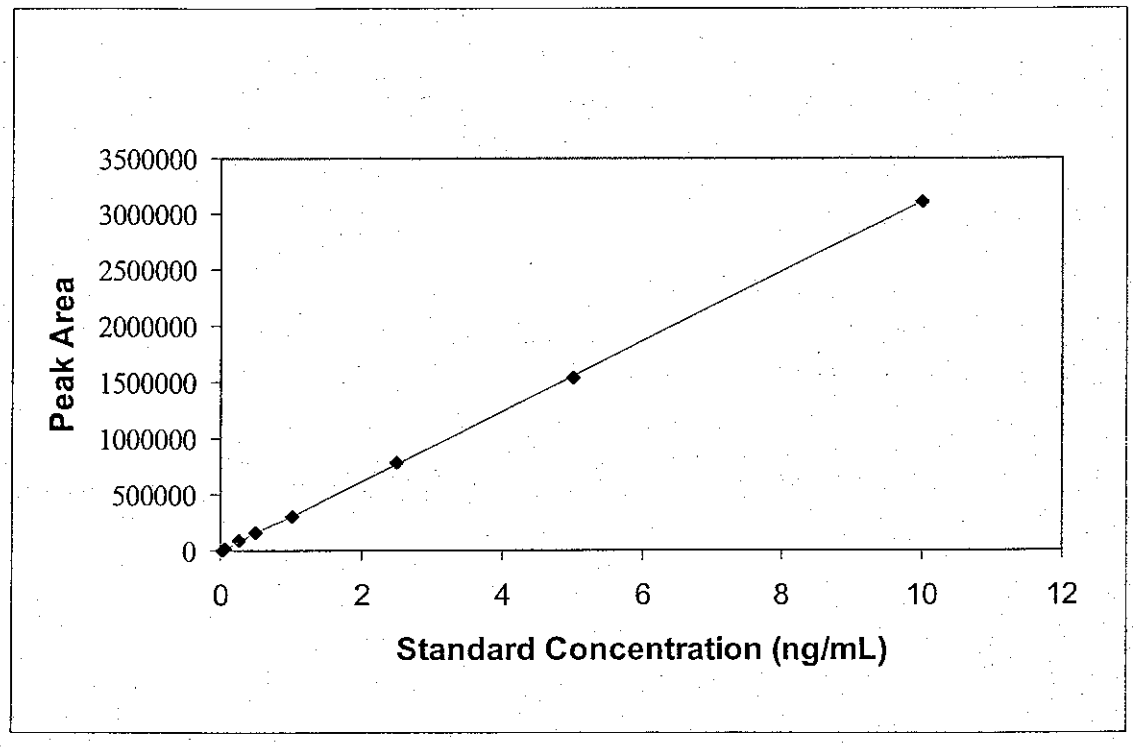


Figure 8. Typical Calibration Curve for the Determination of XDE-742 (Confirmation Ion, Q1/Q3 *m/z* 435.1/82.0) in Water

Analytical Set I.D.: 051039 S05 pos
Compound: 7-OH-XDE-742 Quant (Q1/Q3 *m/z* 420.9/181.0)

Calibration Data

Linear with 1/x Weighting

Slope =	710692
Intercept =	-2233.5549
r^2 =	0.9997

Standard Concentration (ng/mL)	Injection Number	Analyte Peak Area	Response Factor	Calculated Concentration	Percent of Theoretical
0.015	1	9440	629333	0.01643	110
0.05	6	33249	664980	0.04993	100
0.25	11	172948	691792	0.24649	99
0.5	16	343290	686580	0.48618	97
1	21	657571	657571	0.92840	93
2.5	26	1788116	715246	2.51916	101
5	31	3588085	717617	5.05186	101
10	33	7116454	711645	10.01655	100

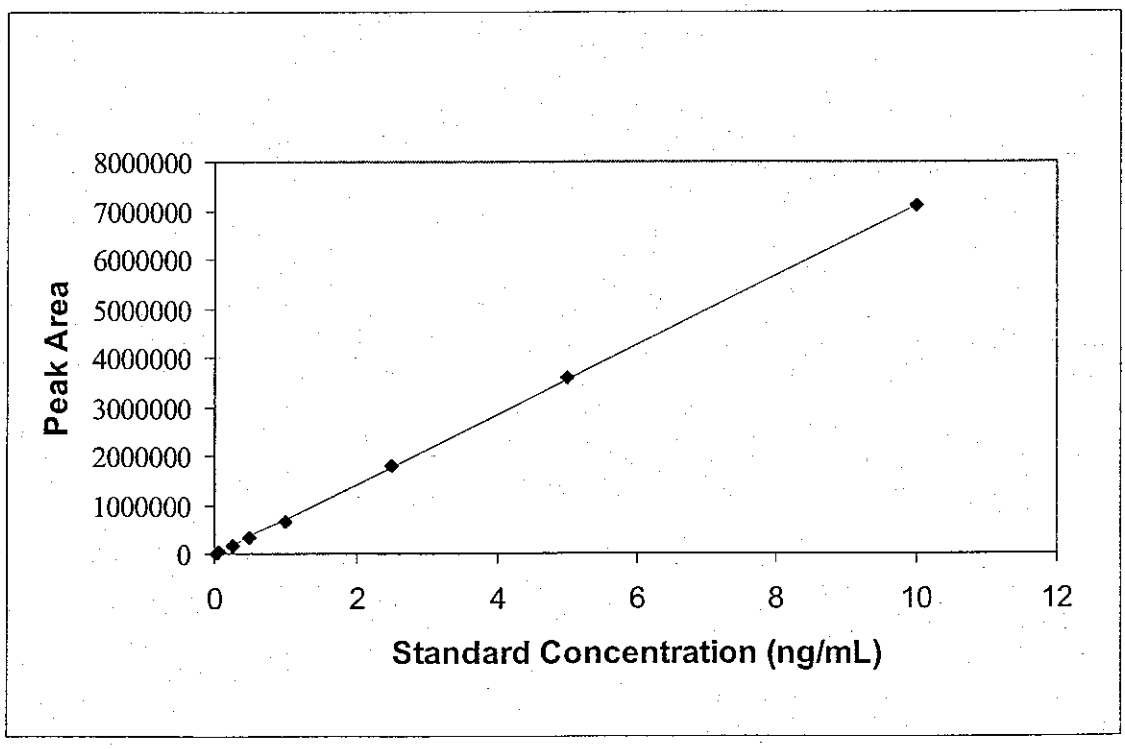


Figure 9. Typical Calibration Curve for the Determination of 7-OH-XDE-742 (Quantitation Ion, Q1/Q3 *m/z* 420.9/181.0) in Water

Analytical Set I.D.: 051039 S05 pos
Compound: 7-OH-XDE-742 Confirm (Q1/Q3 *m/z* 420.9/148.1)

Calibration Data

Linear with 1/x Weighting

Slope =	143443
Intercept =	-769.8530
r^2 =	0.9994

Standard Concentration (ng/mL)	Injection Number	Analyte Peak Area	Response Factor	Calculated Concentration	Percent of Theoretical
0.015	1	1806	120400	0.01796	120
0.05	6	6316	126320	0.04940	99
0.25	11	32443	129772	0.23154	93
0.5	16	68297	136594	0.48149	96
1	21	131171	131171	0.91982	92
2.5	26	351590	140636	2.45645	98
5	31	727194	145439	5.07494	101
10	33	1445622	144562	10.08340	101

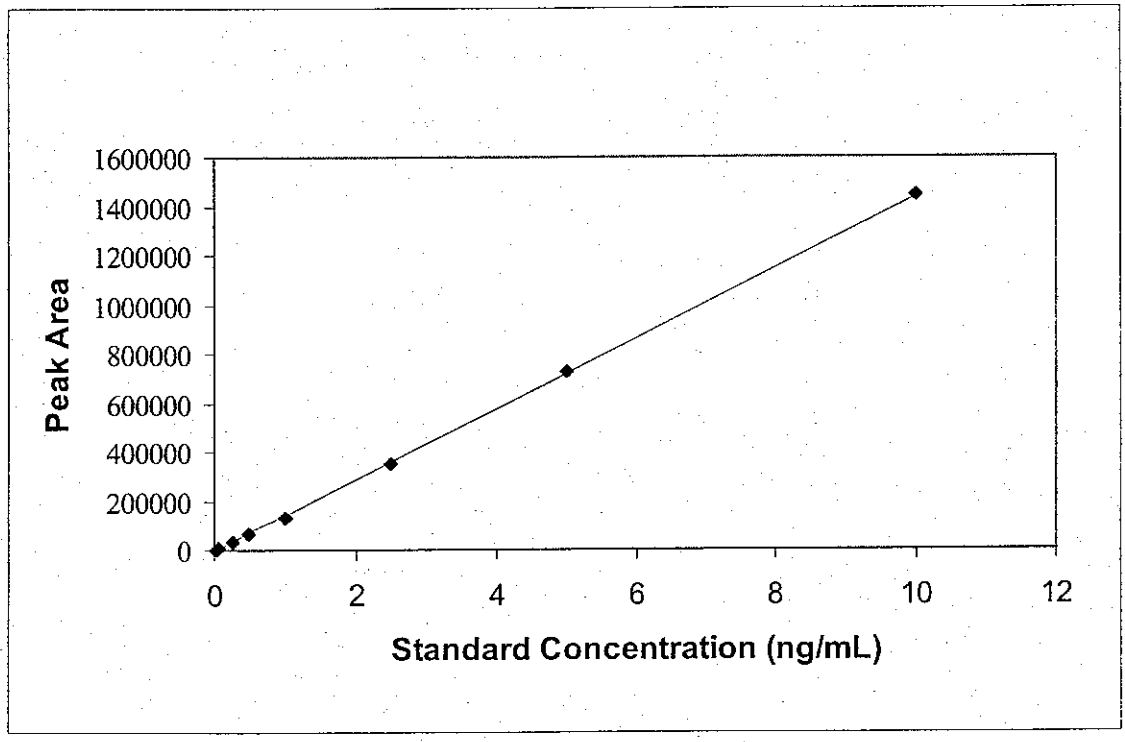


Figure 10. Typical Calibration Curve for the Determination of 7-OH-XDE-742 (Confirmation Ion, Q1/Q3 *m/z* 420.9/148.1) in Water

Analytical Set I.D.: 051039 S05 pos
Compound: ADTP Quant (Q1/Q3 m/z 196.2/115.1)

Calibration Data

Linear with 1/x Weighting

Slope =	688435
Intercept =	3158.7412
r^2 =	0.9998

Standard Concentration (ng/mL)	Injection Number	Analyte Peak Area	Response Factor	Calculated Concentration	Percent of Theoretical
0.015	1	11845	789667	0.01262	84
0.05	6	39197	783940	0.05235	105
0.25	11	187429	749716	0.26767	107
0.5	16	357079	714158	0.51409	103
1	21	699950	699950	1.01214	101
2.5	26	1732980	693192	2.51269	101
5	31	3455476	691095	5.01473	100
10	33	6838441	683844	9.92872	99

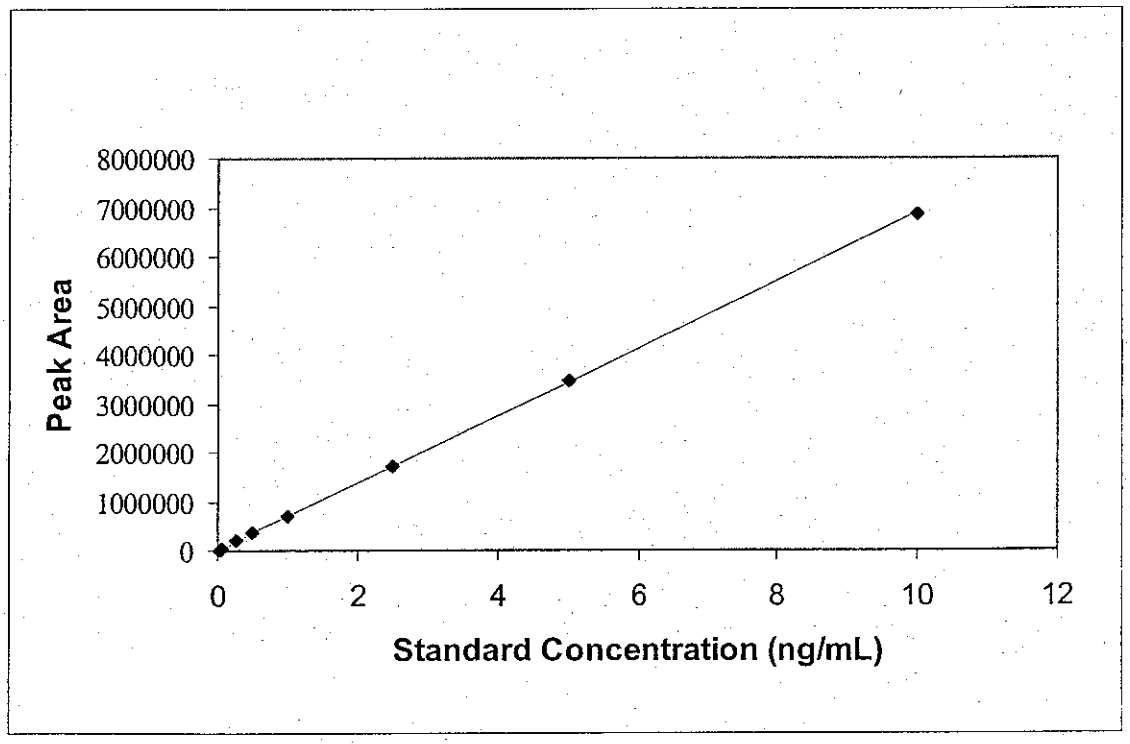


Figure 11. Typical Calibration Curve for the Determination of ADTP (Quantitation Ion, Q1/Q3 *m/z* 196.2/115.1) in Water

Analytical Set I.D.: 051039 S05 pos
Compound: ADTP Confirm (Q1/Q3 *m/z* 196.2/163.9)

Calibration Data

Linear with 1/x Weighting

Slope =	456660
Intercept =	2393.8647
r^2 =	0.9999

Standard Concentration (ng/mL)	Injection Number	Analyte Peak Area	Response Factor	Calculated Concentration	Percent of Theoretical
0.015	1	8489	565933	0.01335	89
0.05	6	25805	516100	0.05127	103
0.25	11	124103	496412	0.26652	107
0.5	16	234158	468316	0.50752	102
1	21	467111	467111	1.01764	102
2.5	26	1132851	453140	2.47549	99
5	31	2274668	454934	4.97585	100
10	33	4572356	457236	10.00736	100

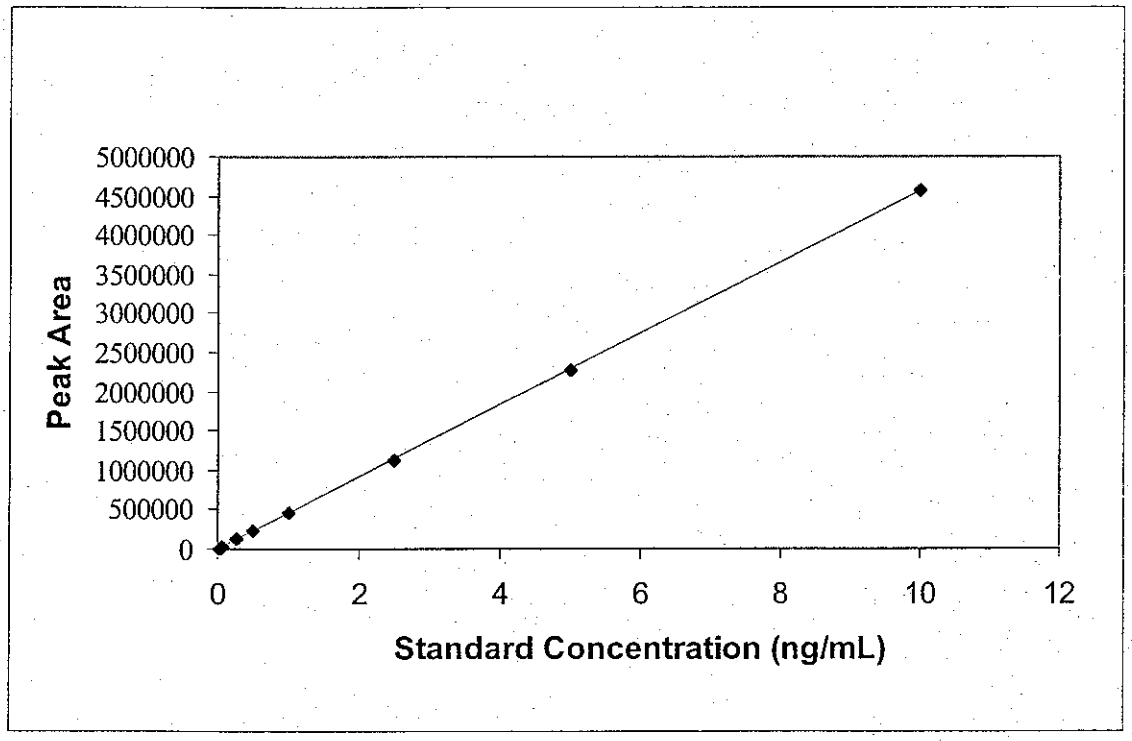


Figure 12. Typical Calibration Curve for the Determination of ADTP (Confirmation Ion, Q1/Q3 *m/z* 196.2/163.9) in Water

Analytical Set I.D.:051039 S05 pos
Compound:ATSA Quant (Q1/Q3 *m/z* 339.0/99.1)

Calibration Data

Linear with 1/x Weighting

Slope =	1159168
Intercept =	-16470.8391
r^2 =	0.9990

Standard Concentration (ng/mL)	Injection Number	Analyte Peak Area	Response Factor	Calculated Concentration	Percent of Theoretical
0.015	1	6861	457400	0.02013	134
0.05	6	29821	596420	0.03994	80
0.25	11	260282	1041128	0.23875	96
0.5	16	549135	1098270	0.48794	98
1	21	1023673	1023673	0.89732	90
2.5	26	2908422	1163369	2.52327	101
5	31	5908506	1181701	5.11140	102
10	33	11570868	1157087	9.99625	100

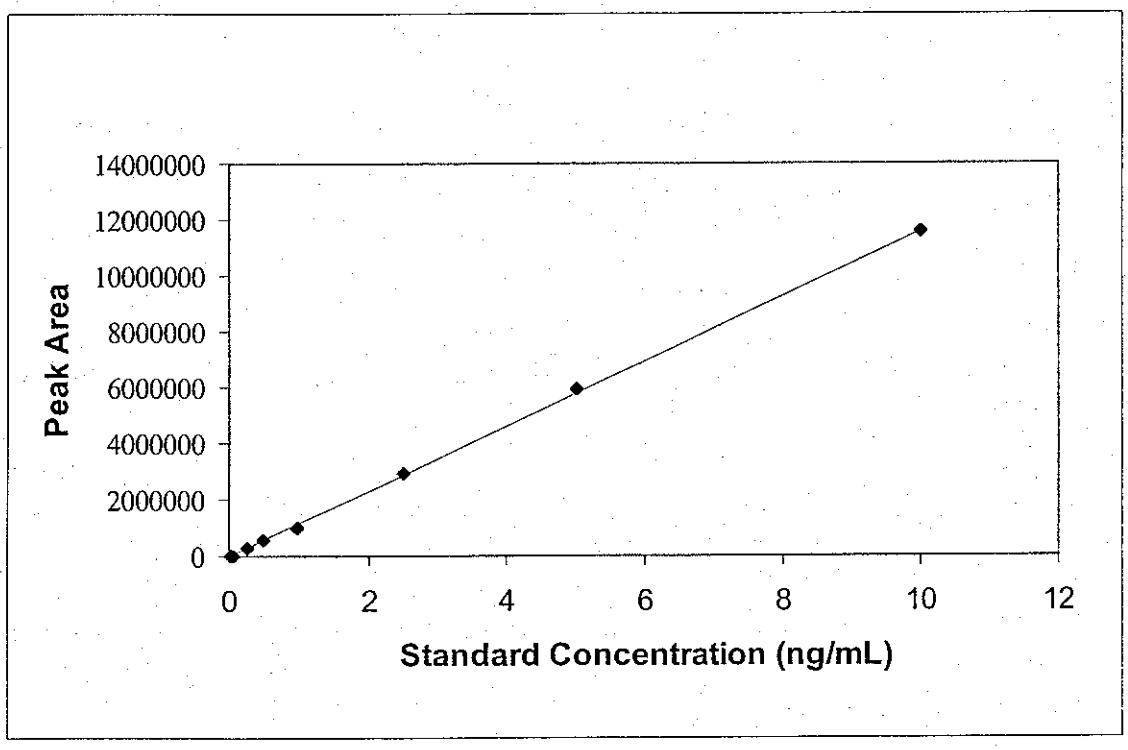


Figure 13. Typical Calibration Curve for the Determination of ATSA (Quantitation Ion, Q1/Q3 *m/z* 339.0/99.1) in Water

Analytical Set I.D.:051039 S05 pos
Compound:ATSA Confirm (Q1/Q3 *m/z* 339.0/57.2)

Calibration Data

Linear with 1/x Weighting

Slope =	145719
Intercept =	-1688.2017
r^2 =	0.9975

Standard Concentration (ng/mL)	Injection Number	Analyte Peak Area	Response Factor	Calculated Concentration	Percent of Theoretical
0.015	1	1593	106200	0.02252	150
0.05	6	4154	83080	0.04009	80
0.25	11	31828	127312	0.23001	92
0.5	16	66402	132804	0.46727	93
1	21	122252	122252	0.85054	85
2.5	26	352735	141094	2.43224	97
5	31	714911	142982	4.91768	98
10	33	1507183	150718	10.35466	104

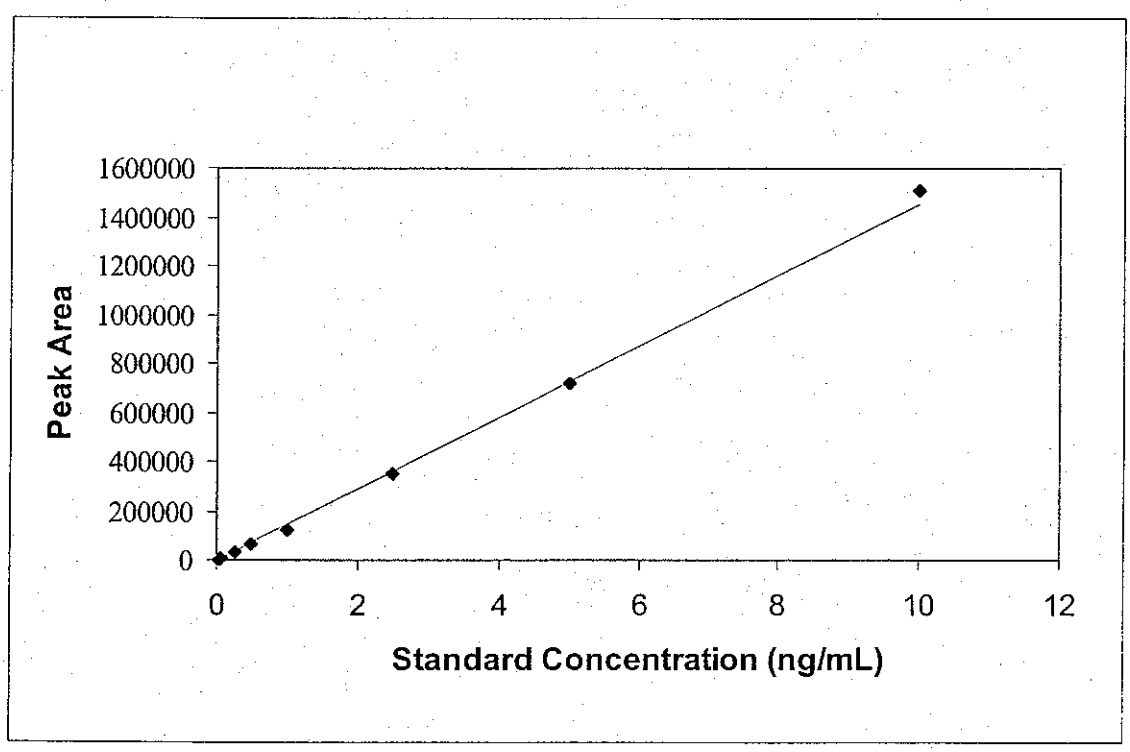


Figure 14. Typical Calibration Curve for the Determination of ATSA (Confirmation Ion, Q1/Q3 *m/z* 339.0/57.2) in Water

Analytical Set I.D.:051039 S05 neg
Compound:Sufinic Quant (Q1/Q3 *m/z* 239.9/175.8)

Calibration Data

Linear with 1/x Weighting

Slope =	99854
Intercept =	352.0435
r^2 =	0.9985

Standard Concentration (ng/mL)	Injection Number	Analyte Peak Area	Response Factor	Calculated Concentration	Percent of Theoretical
0.015	1	2153	143533	0.01804	120
0.05	6	4939	98780	0.04594	92
0.25	11	24432	97728	0.24115	96
0.5	16	49253	98506	0.48972	98
1	21	100366	100366	1.00160	100
2.5	26	227961	91184	2.27942	91
5	31	497164	99433	4.97538	100
10	33	1025229	102523	10.26375	103

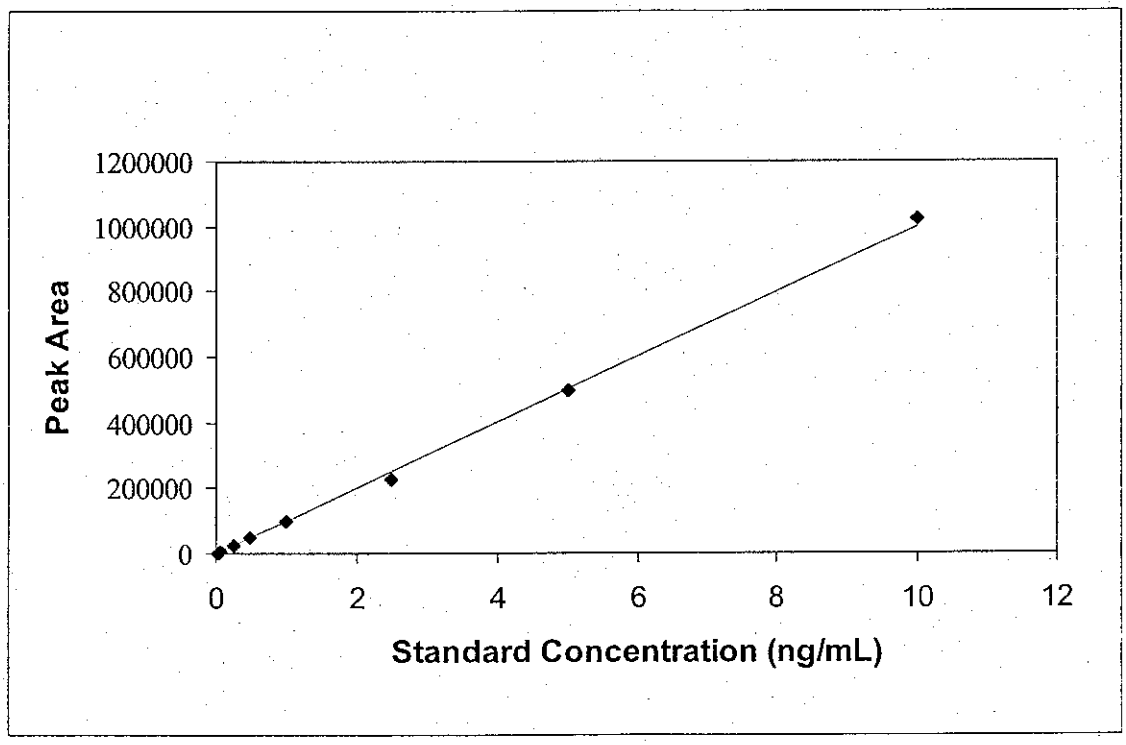


Figure 15. Typical Calibration Curve for the Determination of Sulfinic Acid (Quantitation Ion, Q1/Q3 *m/z* 239.9/175.8) in Water

Analytical Set I.D.:051039 S05 neg
Compound:Sulfinic Confirm (Q1/Q3 *m/z* 239.9/155.7)

Calibration Data

Linear with 1/x Weighting

Slope =	31213
Intercept =	87.2307
r^2 =	0.9974

Standard Concentration (ng/mL)	Injection Number	Analyte Peak Area	Response Factor	Calculated Concentration	Percent of Theoretical
0.015	1	638	42533	0.01765	118
0.05	6	1657	33140	0.05029	101
0.25	11	8158	32632	0.25857	103
0.5	16	14642	29284	0.46630	93
1	21	28703	28703	0.91679	92
2.5	26	71019	28408	2.27250	91
5	31	153607	30721	4.91843	98
10	33	325156	32516	10.41448	104

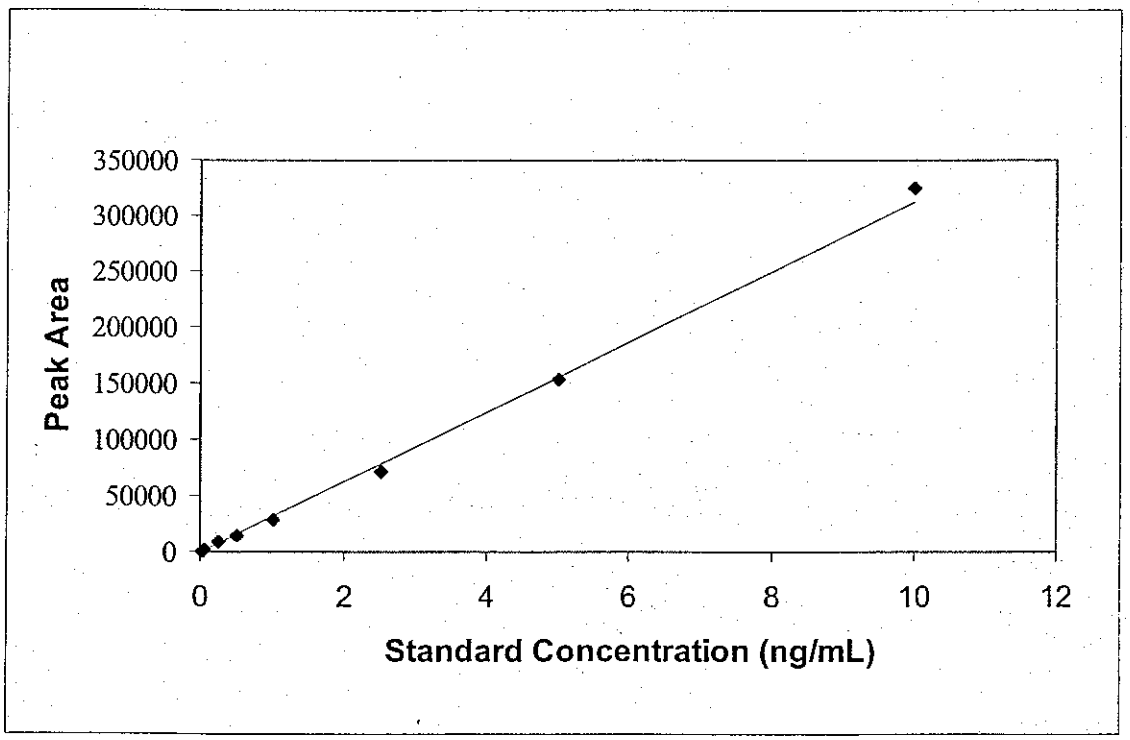


Figure 16. Typical Calibration Curve for the Determination of Sulfinic Acid (Confirmation Ion, Q1/Q3 *m/z* 239.9/155.7) in Water

Analytical Set I.D.: 051039 S05 neg
Compound: Sulfonic Quant (Q1/Q3 *m/z* 255.7/149.0)

Calibration Data

Linear with 1/x Weighting

Slope =	46822
Intercept =	136.4006
r^2 =	0.9997

Standard Concentration (ng/mL)	Injection Number	Analyte Peak Area	Response Factor	Calculated Concentration	Percent of Theoretical
0.015	1	891	59400	0.01612	107
0.05	6	2319	46380	0.04662	93
0.25	11	11581	46324	0.24443	98
0.5	16	24146	48292	0.51279	103
1	21	47594	47594	1.01358	101
2.5	26	115583	46233	2.46567	99
5	31	228875	45775	4.88532	98
10	33	474461	47446	10.13047	101

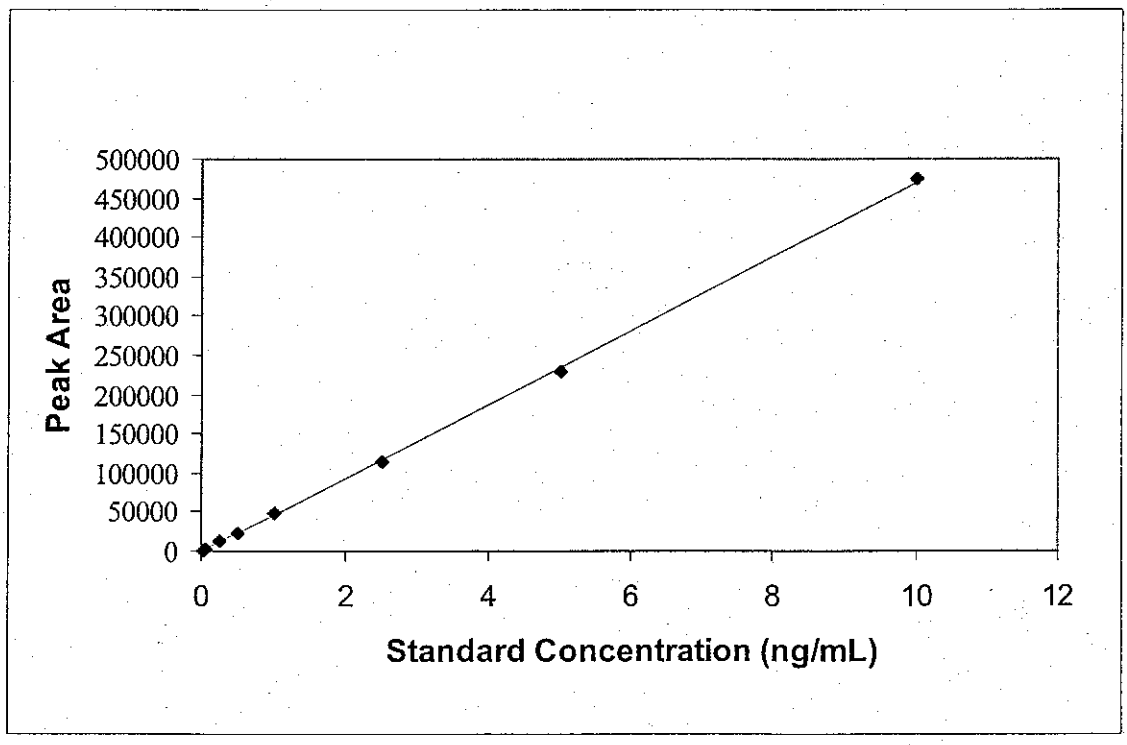


Figure 17. Typical Calibration Curve for the Determination of Sulfonic Acid (Quantitation Ion, Q1/Q3 *m/z* 255.7/149.0) in Water

Analytical Set I.D.: 051039 S05 neg
Compound: Sulfonic Confirm (Q1/Q3 *m/z* 255.7/79.7)

Calibration Data

Linear with 1/x Weighting

Slope =	42104
Intercept =	416.0580
r^2 =	0.9995

Standard Concentration (ng/mL)	Injection Number	Analyte Peak Area	Response Factor	Calculated Concentration	Percent of Theoretical
0.015	1	1089	72600	0.01598	107
0.05	6	2569	51380	0.05113	102
0.25	11	10122	40488	0.23052	92
0.5	16	21240	42480	0.49458	99
1	21	43531	43531	1.02400	102
2.5	26	104466	41786	2.47124	99
5	31	204754	40951	4.85314	97
10	33	428802	42880	10.17440	102

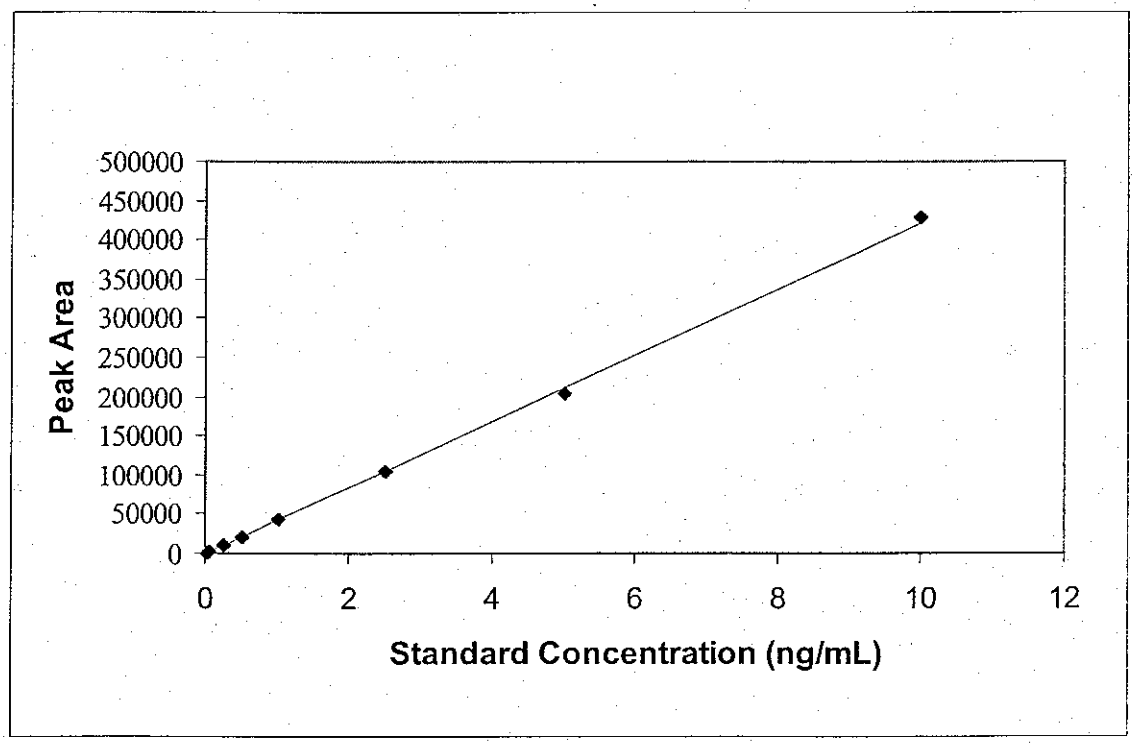
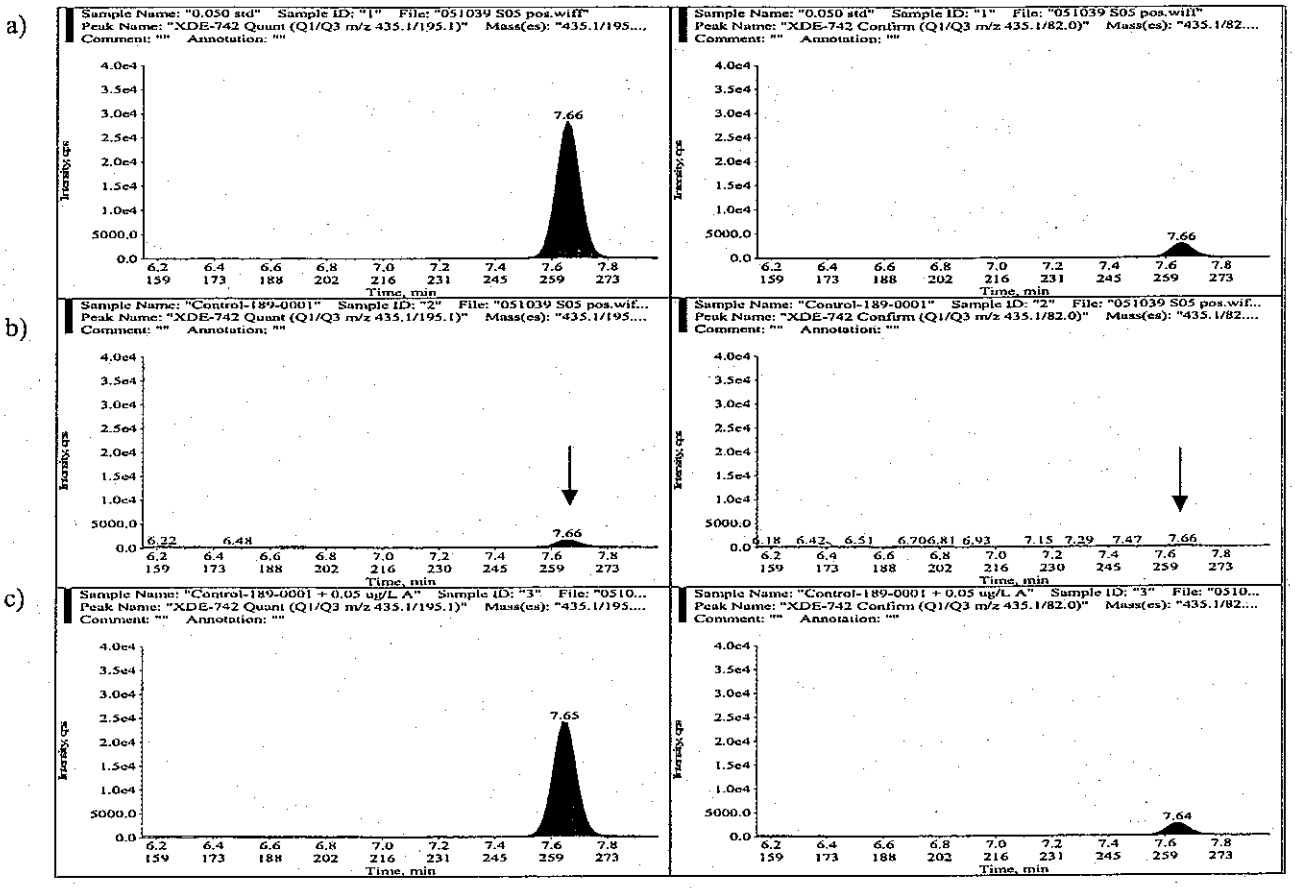


Figure 18. Typical Calibration Curve for the Determination of Sulfonic Acid (Confirmation Ion, Q1/Q3 *m/z* 255.7/79.7) in Water



a) XDE-742 Standard – 0.050 ng/mL	
Peak Area (*m/z* 435.1/195.1)	168747
Equivalent Concentration (µg/L)	0.0500
Concentration Found (µg/L)	0.0494
Theoretical Percent Recovery	99%

Confirmation	
Peak Area (*m/z* 435.1/82.0)	17430
Peak Area Ratio (*m/z* 435.1/82.0 / *m/z* 435.1/195.1)	0.1033
Average of Standards	0.1045
Absolute Percent Difference	1.2%

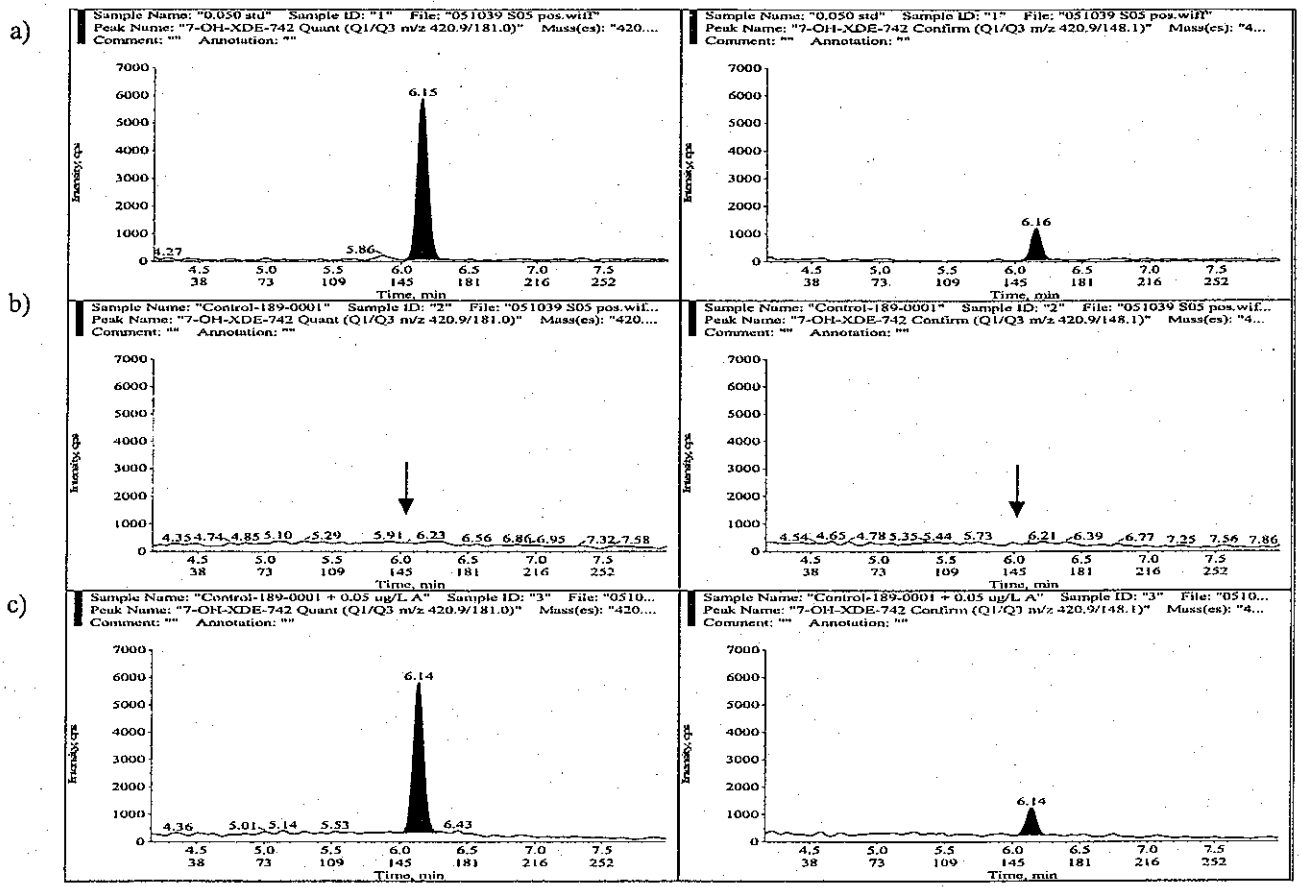
b) Drinking Water Sample – Control	
Peak Area (*m/z* 435.1/195.1)	9950
Concentration Added (µg/L)	0.0000
Concentration Found (µg/L)	0.0000
Percent Recovery	- - - -

Confirmation	
Peak Area (*m/z* 435.1/82.0)	719
Peak Area Ratio (*m/z* 435.1/82.0 / *m/z* 435.1/195.1)	0.0723
Average of Standards	0.1045
Absolute Percent Difference	- - - -

c) Drinking Water Sample – 0.050 µg/L	
Peak Area (*m/z* 435.1/195.1)	146523
Concentration Added (µg/L)	0.0500
Concentration Found (µg/L)	0.0460
Percent Recovery	92%

Confirmation	
Peal Area (*m/z* 435.1/82.0)	16240
Peak Area Ratio (*m/z* 435.1/82.0 / *m/z* 435.1/195.1)	0.1108
Average of Standards	0.1045
Absolute Percent Difference	6.0%

Figure 19. Typical Chromatograms for the Determination of XDE-742 in Drinking Water



a) 7-OH-XDE-742 Standard – 0.050 ng/mL	
Peak Area (*m/z* 420.9/181.0)	33249
Equivalent Concentration (µg/L)	0.0500
Concentration Found (µg/L)	0.0499
Theoretical Percent Recovery	100%

Confirmation	
Peak Area (*m/z* 420.9/148.1)	6316
Peak Area Ratio (*m/z* 420.9/148.1/ *m/z* 420.9/181.0)	0.1900
Average of Standards	0.1962
Absolute Percent Difference	3.2%

b) Drinking Water Sample – Control	
Peak Area (*m/z* 420.9/181.0)	0
Concentration Added (µg/L)	0.0000
Concentration Found (µg/L)	0.0000
Percent Recovery	- - - -

Confirmation	
Peak Area (*m/z* 420.9/148.1)	0
Peak Area Ratio (*m/z* 420.9/148.1/ *m/z* 420.9/181.0)	0.0000
Average of Standards	0.1962
Absolute Percent Difference	- - - -

c) Drinking Water Sample – 0.050 µg/L	
Peak Area (*m/z* 420.9/181.0)	30793
Concentration Added (µg/L)	0.0500
Concentration Found (µg/L)	0.0516
Percent Recovery	103%

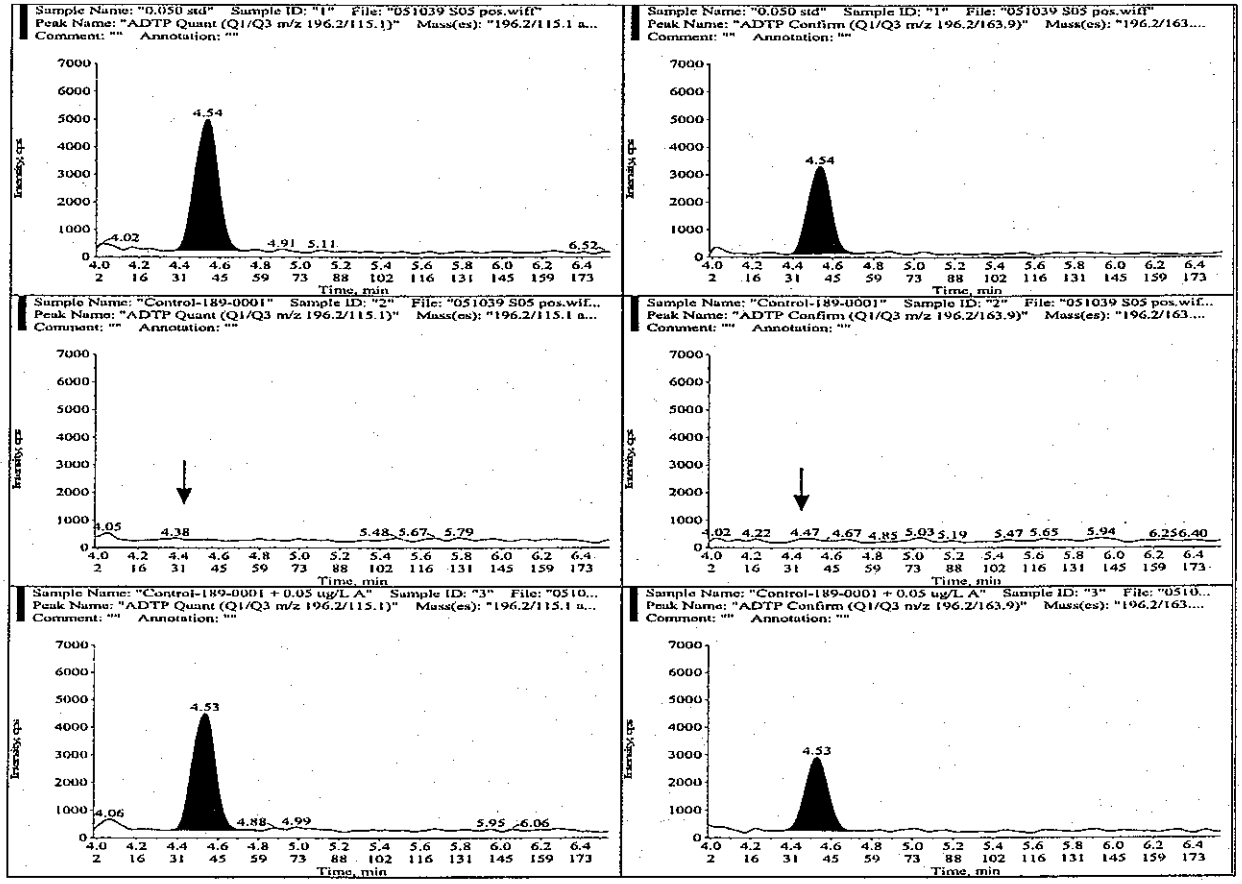
Confirmation	
Peak Area (*m/z* 420.9/148.1)	5718
Peak Area Ratio (*m/z* 420.9/148.1/ *m/z* 420.9/181.0)	0.1857
Average of Standards	0.1962
Absolute Percent Difference	5.4%

Figure 20. Typical Chromatograms for the Determination of 7-OH-XDE-742 in Drinking Water

a)

b)

c)

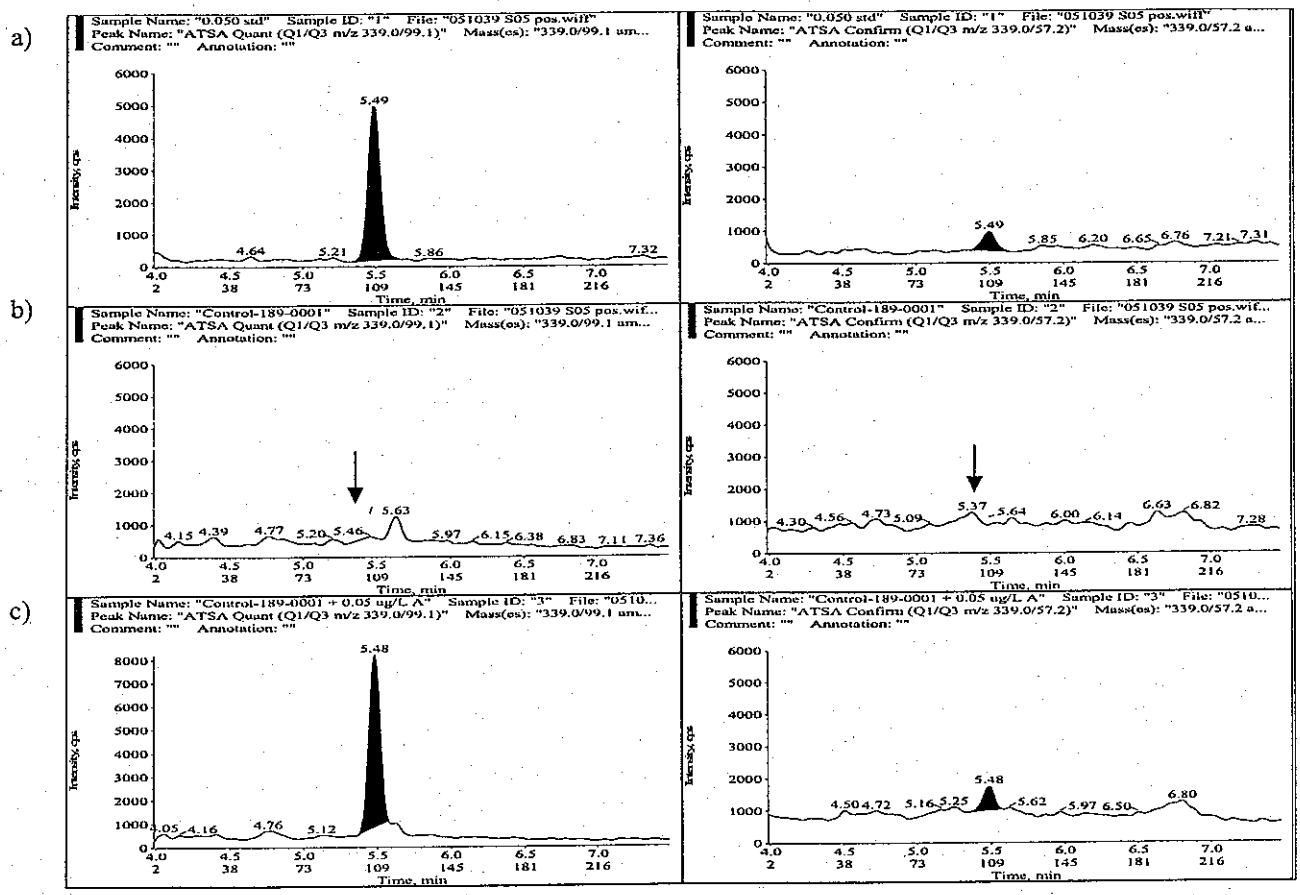


a) ADTP Standard – 0.050 ng/mL		Confirmation	
Peak Area (*m/z* 196.2/115.1)	39197	Peak Area (*m/z* 196.2/163.9)	25805
		Peak Area Ratio	
Equivalent Concentration (µg/L)	0.0500	(*m/z* 196.2/163.9/ *m/z* 196.2/115.1)	0.6583
Concentration Found (µg/L)	0.0524	Average of Standards	0.6676
Theoretical Percent Recovery	105%	Absolute Percent Difference	1.4%

b) Drinking Water Sample – Control		Confirmation	
Peak Area (*m/z* 196.2/115.1)	74	Peak Area (*m/z* 196.2/163.9)	0
		Peak Area Ratio	
Concentration Added (µg/L)	0.0000	(*m/z* 196.2/163.9/ *m/z* 196.2/115.1)	0.0000
Concentration Found (µg/L)	0.0000	Average of Standards	0.6676
Percent Recovery	- - - -	Absolute Percent Difference	- - - -

c) Drinking Water Sample – 0.050 µg/L		Confirmation	
Peak Area (*m/z* 196.2/115.1)	34459	Peak Area (*m/z* 196.2/163.9)	21931
Concentration Added (µg/L)	0.0500	Peak Area Ratio (*m/z* 196.2/163.9/ *m/z* 196.2/115.1)	0.6364
Concentration Found (µg/L)	0.0505	Average of Standards	0.6676
Percent Recovery	101%	Absolute Percent Difference	4.7%

Figure 21. Typical Chromatograms for the Determination of ADTP in Drinking Water



a) ATSA Standard – 0.050 ng/mL	
Peak Area (*m/z* 339.0/99.1)	29821
Equivalent Concentration (µg/L)	0.0500
Concentration Found (µg/L)	0.0399
Theoretical Percent Recovery	80%

Confirmation	
Peak Area (*m/z* 339.0/57.2)	4154
Peak Area Ratio (*m/z* 339.0/57.2/ *m/z* 339.0/99.1)	0.1393
Average of Standards	0.1383
Absolute Percent Difference	0.7%

b) Drinking Water Sample – Control	
Peak Area (*m/z* 339.0/99.1)	0
Concentration Added (µg/L)	0.0000
Concentration Found (µg/L)	0.0000
Percent Recovery	- - - -

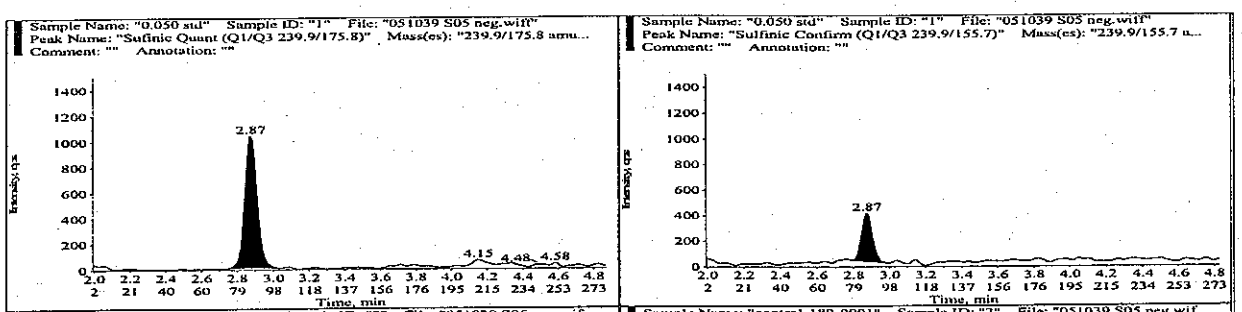
Confirmation	
Peak Area (*m/z* 339.0/57.2)	0
Peak Area Ratio (*m/z* 339.0/57.2/ *m/z* 339.0/99.1)	0.0000
Average of Standards	0.1383
Absolute Percent Difference	- - - -

c) Drinking Water Sample – 0.050 µg/L	
Peak Area (*m/z* 339.0/99.1)	40849
Concentration Added (µg/L)	0.0500
Concentration Found (µg/L)	0.0549
Percent Recovery	110%

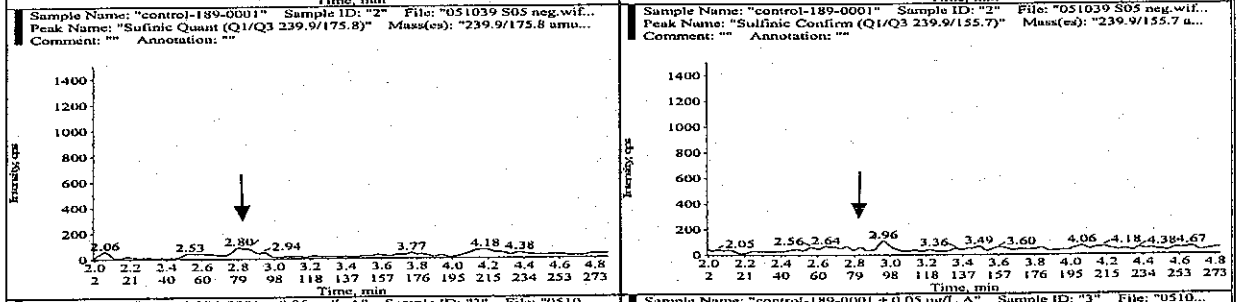
Confirmation	
Peak Area (*m/z* 339.0/57.2)	4250
Peak Area Ratio (*m/z* 339.0/57.2/ *m/z* 339.0/99.1)	0.1040
Average of Standards	0.1383
Absolute Percent Difference	24.8%

Figure 22. Typical Chromatograms for the Determination of ATSA in Drinking Water

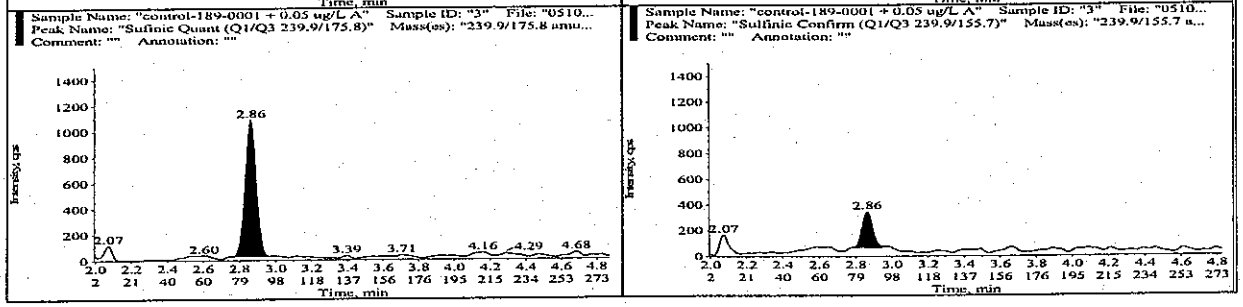
a)



b)



c)



a) Sulfinic Acid Standard – 0.050 ng/mL	
Peak Area (*m/z* 239.9/175.8)	4939
Equivalent Concentration (µg/L)	0.0500
Concentration Found (µg/L)	0.0459
Theoretical Percent Recovery	92%

Confirmation	
Peak Area (*m/z* 239.9/155.7)	1657
Peak Area Ratio (*m/z* 239.9/155.7/ *m/z* 239.9/175.8)	0.3355
Average of Standards	0.3108
Absolute Percent Difference	7.9%

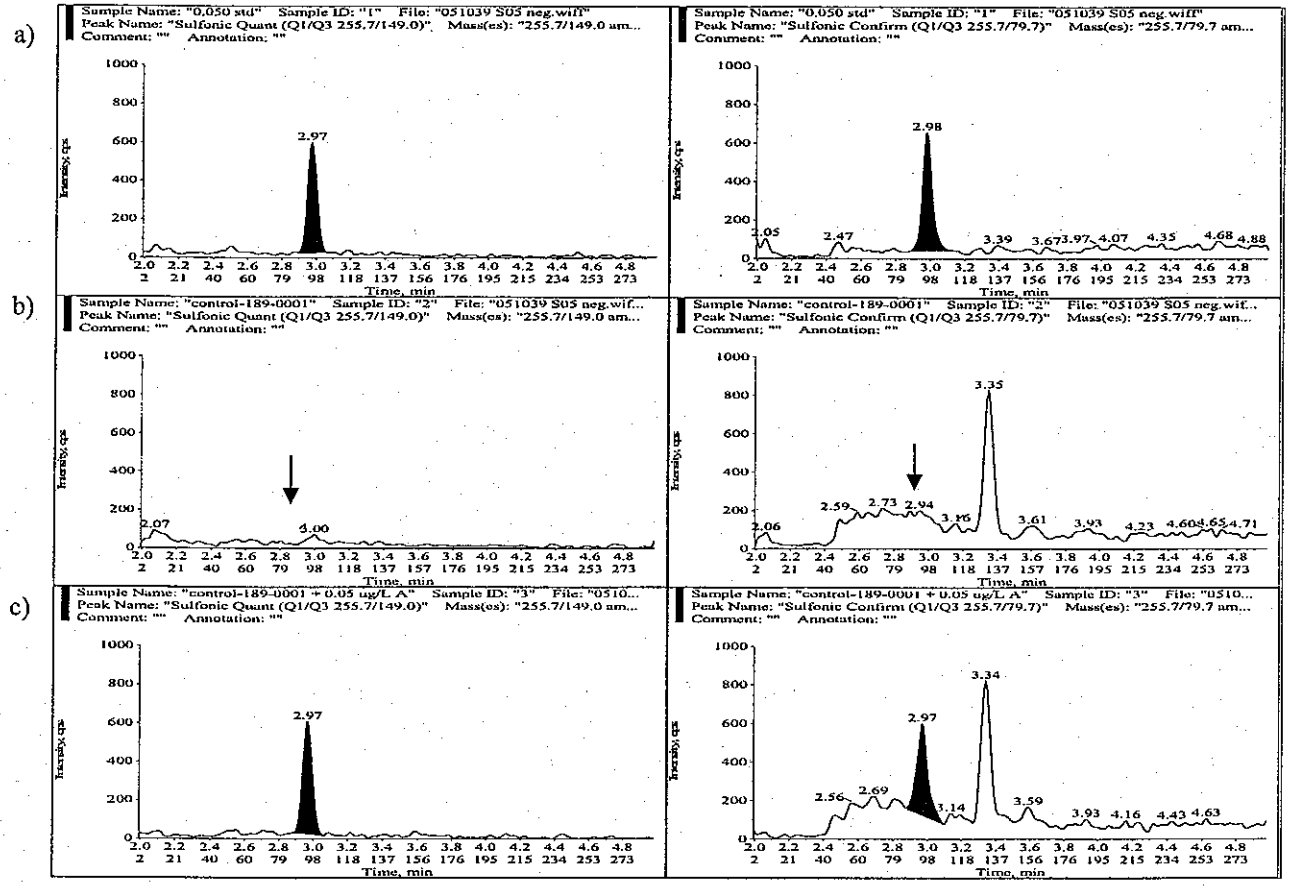
b) Drinking Water Sample – Control	
Peak Area (*m/z* 239.9/175.8)	0
Concentration Added (µg/L)	0.0000
Concentration Found (µg/L)	0.0000
Percent Recovery	- - - -

Confirmation	
Peak Area (*m/z* 239.9/155.7)	0
Peak Area Ratio (*m/z* 239.9/155.7/ *m/z* 239.9/175.8)	0.0000
Average of Standards	0.3108
Absolute Percent Difference	- - - -

c) Drinking Water Sample – 0.050 µg/L	
Peak Area (*m/z* 239.9/175.8)	4522
Concentration Added (µg/L)	0.0500
Concentration Found (µg/L)	0.0464
Percent Recovery	93%

Confirmation	
Peak Area (*m/z* 239.9/155.7)	1194
Peak Area Ratio (*m/z* 239.9/155.7/ *m/z* 239.9/175.8)	0.2640
Average of Standards	0.3108
Absolute Percent Difference	15.1%

Figure 23. Typical Chromatograms for the Determination of Sulfinic Acid in Drinking Water



a) Sulfonic Acid Standard – 0.050 ng/mL	
Peak Area (*m/z* 255.7/149.0)	2319
Equivalent Concentration (µg/L)	0.0500
Concentration Found (µg/L)	0.0466
Theoretical Percent Recovery	93%

Confirmation	
Peak Area (*m/z* 255.7/79.7)	2569
Peak Area Ratio (*m/z* 255.7/79.7/ *m/z* 255.7/149.0)	1.1078
Average of Standards	0.9626
Absolute Percent Difference	15.1%

b) Drinking Water Sample – Control	
Peak Area (*m/z* 255.7/149.0)	0
Concentration Added (µg/L)	0.0000
Concentration Found (µg/L)	0.0000
Percent Recovery	- - - -

Confirmation	
Peak Area (*m/z* 255.7/79.7)	0
Peak Area Ratio (*m/z* 255.7/79.7/ *m/z* 255.7/149.0)	0.0000
Average of Standards	0.9626
Absolute Percent Difference	- - - -

c) Drinking Water Sample – 0.050 µg/L	
Peak Area (*m/z* 255.7/149.0)	2396
Concentration Added (µg/L)	0.0500
Concentration Found (µg/L)	0.0536
Percent Recovery	107%

Confirmation	
Peak Area (*m/z* 255.7/79.7)	2235
Peak Area Ratio (*m/z* 255.7/79.7/ *m/z* 255.7/149.0)	0.9328
Average of Standards	0.9626
Absolute Percent Difference	3.1%

Figure 24. Typical Chromatograms for the Determination of Sulfonic Acid in Drinking Water