Dow AgroSciences LLC
9330 Zionsville Road
Indianapolis, Indiana 46268-1054

GRM: 05.04
EFFECTIVE: 06-Jul-2005
SUPERSEDES: New

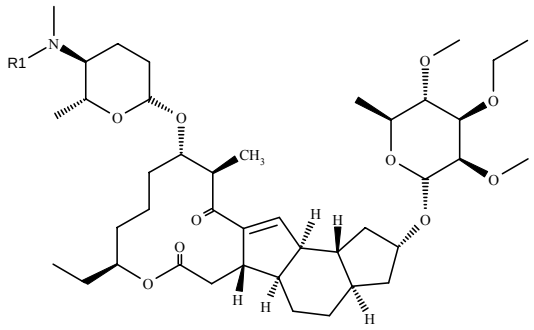
Dow AgroSciences™

Determination of Residues of XDE-175 and its Metabolites in Agricultural Commodities by On-Line Solid Phase Extraction and Liquid Chromatography with Tandem Mass Spectrometry

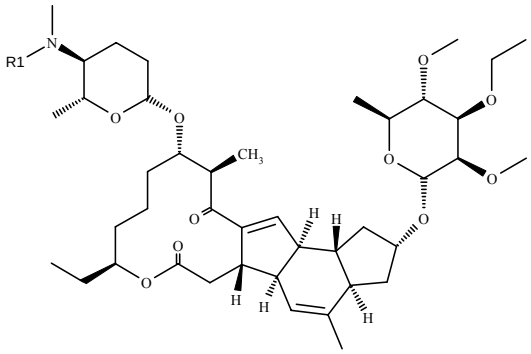
M. J. Hastings, B. M. Wendelburg

1. SCOPE

This method is applicable for the quantitative determination of XDE-175-J and XDE-175-L and their metabolites XDE-175-*N*-demethyl-J, XDE-175-*N*-demethyl-L, XDE-175-*N*-formyl-J and XDE-175-*N*-formyl-L in agricultural commodities (dry crops, wet crops, oily crops and acidic crops). The method was validated over the concentration range of 0.01-1.0 µg/g for all crops except lettuce which was validated over the concentration range of 0.01-10.0 µg/g. The validated limit of quantitation for all crops was 0.01 µg/g.



XDE-175-J, R1 = CH_3
N-demethyl-XDE-175-J, R1 = H
N-formyl-XDE-175-J, R1 = C(O)H



XDE-175-L, R1 = CH_3
N-demethyl-XDE-175-L, R1 = H
N-formyl-XDE-175-L, R1 = C(O)H

Common and chemical names along with other identifying information are given in Table 1.

2. PRINCIPLE

Residues of XDE-175 and its metabolites are extracted from the crop sample by homogenizing and by shaking with an acetonitrile/water solution (80:20). A mixed XDE-175 and metabolites stable isotope internal standard solution is added to each sample and the final solution is purified by on-line solid phase extraction using a C18 cartridge. The extract is loaded onto the SPE cartridge with an

acetonitrile/methanol/water (15:15:70) solution containing 10 mM ammonium acetate. The SPE cartridge is washed with an acetonitrile/methanol/water solution (25:25:50) containing 10 mM ammonium acetate and eluted onto the analytical column with a gradient elution technique using the HPLC mobile phase. XDE-175 and its metabolites are analyzed by liquid chromatography with positive ion electrospray ionization (ESI) tandem mass spectrometry (LC/MS/MS).

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 volatile and should be used in well-ventilated areas away from ignition sources.

3.3. Formic acid is corrosive and can 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., Hightstown, NJ 08520.

4.1.2. Balance, pan, Model PG2002, Mettler-Toledo, Inc.

4.1.3. Centrifuge, with rotor to accommodate 8-oz bottles, Model Centra-GP8, International Equipment Company, Needham Heights, MA 02494.

4.1.4. Dispenser, Bottle-Top, adjustable, Brinkmann, 20-100 mL, catalog number 13-688-136, Fisher Scientific, Pittsburgh, PA 15219.

4.1.5. Hammer mill, with 1/8 and 3/16-inch screen, Model 2001, AGVISE Laboratories, Inc., Northwood, ND 58267.

4.1.6. Homogenizer, Omni-mixer, Model ES, Omni International, Inc., Warrenton, VA 20187.

4.1.7. Homogenizer generator, 20-mm probe, catalog number 15020W, Omni International, Inc.

4.1.8. Pipettor, adjustable, Gilson Microman M250, 50-250 µL, catalog number F148505, Gilson Inc., Middleton, WI 53562.

4.1.9. Pipettor, adjustable, Gilson Microman M1000, 100-1000 µL, catalog number F148506, Gilson Inc.

4.1.10. Shaker, variable speed reciprocating with box carrier, Model 6000, Eberbach Corporation, Ann Arbor, MI 48106.

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

4.2. Chromatographic System

4.2.1. Column, analytical, YMC ODS-AM, 50 x 4.6 mm, 5-µm, catalog number AM12S05-0546WT, Waters, Milford, MA 01757.

4.2.2. Column, confirmatory, Synergi Polar RP, 75 x 4.6 mm, 4-µm, catalog number 00C-4336-E0, Phenomenex, Torrance, CA 90501.

4.2.3. Mass spectrometer, Model API 4000, MDS/Sciex, Foster City, CA 94404.

4.2.4. Mass spectrometer data system, Analyst 1.4, MDS/Sciex.

4.2.5. On-line SPE/Liquid chromatograph, Symbiosis Pharma, Spark Holland Inc., Plainsboro, NJ 08536.

5. GLASSWARE AND MATERIALS (Note 12.1.)

5.1. Bottle, 250-mL, HDPE, catalog number 03-313-4D, Fisher Scientific.

5.2. Bottle, 1.0-L, media bottle, catalog number 06-423-3D, Fisher Scientific.

5.3. Bottle, 2.0-L, media bottle, catalog number 06-423-3E, Fisher Scientific.

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

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

5.6. Cylinder, graduated, 100-mL, catalog number C7000-100, National Scientific Company, Lawrenceville, GA 30243.

5.7. Cylinder, graduated, 500-mL, catalog number C7000-500, National Scientific Company.

5.8. Cylinder, graduated, 1000-mL, catalog number C7000-1L, National Scientific Company.

5.9. Cylinder, graduated, 2000-mL, catalog number C7000-2L, National Scientific Company.

5.10. Flask, volumetric, 100-mL, catalog number 161-8987, National Scientific Company.

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

5.12. Pipet, volumetric, 0.5-mL, catalog number 261-6010, National Scientific Company.

5.13. Pipet, volumetric, 1.0-mL, catalog number 261-6011, National Scientific Company.

5.14. Pipet, volumetric, 2.0-mL, catalog number 261-6012, National Scientific Company.

5.15. Pipet, volumetric, 3.0-mL, catalog number 261-6013, National Scientific Company.

5.16. Pipet, volumetric, 5.0-mL, catalog number 261-6015, National Scientific Company.

5.17. Pipet, volumetric, 10.0-mL, catalog number 261-6020, National Scientific Company.

5.18. Pipetter tips, Gilson Microman CP250, catalog number F148114, Gilson Inc.

5.19. Pipetter tips, Gilson Microman CP1000, catalog number F148560, Gilson Inc.

5.20. SPE cartridges, Hysphere C18HD, 7-µm, catalog number 0722.609, Spark Holland Inc.

5.21. Vial, 40-mL, with PTFE-lined screw cap, catalog number B7800-6, 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. Ammonium acetate, HPLC grade, catalog number A639-500, Fisher Scientific.

6.1.3. Formic acid, 96%, ACS grade, catalog number 251364, Sigma-Aldrich, Milwaukee, WI 53201.

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

6.1.5. Nitrogen, refrigerated liquid, BOC Group Inc., Murray Hill, NJ 07974.

6.1.6. Water, HPLC grade, catalog number WX0004-1, EM Science, Gibbstown, NJ 08027.

6.2. Standards

6.2.1. Analytical standard information for XDE-175-J, XDE-175-L, XDE-175-*N*-demethyl-J, XDE-175-*N*-demethyl-L, XDE-175-*N*-formyl-J and XDE-175-*N*-formyl-L are listed in Table 1.

Compounds can be obtained from Test Substance Coordinator, Dow AgroSciences LLC, 9330 Zionsville Road, Building 304, Indianapolis, IN 46268-1054.

6.2.2. Stable isotope labeled internal standards information for XDE-175-J, XDE-175-L, XDE-175-*N*-demethyl-J, XDE-175-*N*-demethyl-L are listed in Table 1.

Obtain from Specialty Synthesis Group, Dow AgroSciences LLC, 9330 Zionsville Road, Building 306, Indianapolis, IN 46268-1054.

6.3. Prepared Solutions

6.3.1. acetonitrile/methanol (1:1) containing 2 mM ammonium acetate

Weigh 0.15 g of ammonium acetate into a 40-mL vial and quantitatively transfer with 100 mL of methanol into a 1-L bottle. Add a further 400 mL of methanol to the bottle. Measure 500 mL of acetonitrile using a 500-mL graduated cylinder and then transfer to the 1.0-L bottle. Cap the bottle and mix. Allow the solution to equilibrate to room temperature before use.

6.3.2. acetonitrile/methanol/water (15:15:70) containing 10 mM ammonium acetate

Weigh 0.77 g of ammonium acetate into a 40-mL vial and quantitatively transfer with 100 mL of HPLC water into a 1-L bottle. Measure 600 mL of water, 150 mL of methanol and 150 mL of acetonitrile using a graduated cylinder and transfer to the 1.0-L bottle. Cap the bottle and mix. Allow the solution to equilibrate to room temperature before use.

6.3.3. acetonitrile/methanol/water (25:25:50) containing 10 mM ammonium acetate

Weigh 0.77 g of ammonium acetate into a 40-mL vial and quantitatively transfer with 100 mL of HPLC water into a 1-L bottle. Measure 400 mL of water, 250 mL of methanol and 250 mL of acetonitrile using a graduated cylinder and transfer to the 1.0-L bottle. Cap the bottle and mix. Allow the solution to equilibrate to room temperature before use.

6.3.4. acetonitrile/water (80:20)

Measure 1600 mL of acetonitrile using a 2-L graduated cylinder and then transfer into a 2.0-L bottle. Measure 400 mL of water using a 500-mL graduated cylinder and then transfer into the 2.0-L bottle. Cap the bottle and mix. Allow the solution to equilibrate to room temperature before use.

6.3.5. acetonitrile/water (80:20) containing 0.1% formic acid

Measure 800 mL of acetonitrile using a 1-L graduated cylinder and then transfer into a 1.0-L bottle. Measure 200 mL of water using a 500-mL graduated cylinder and then transfer into the 1.0-L bottle. Pipet 1.0 mL of formic acid into the bottle. Cap the bottle and mix. Allow the solution to equilibrate to room temperature before use.

6.3.6. water containing 2 mM ammonium acetate

Weigh 0.15 g of ammonium acetate into a 40-mL vial and quantitatively transfer with 100 mL of HPLC water into a 1-L bottle. Add a further 900 mL of HPLC water to the bottle. Cap the bottle and mix. Allow the solution to equilibrate to room temperature before use.

7. PREPARATION OF STANDARD SOLUTIONS

7.1. Preparation of XDE-175 and Metabolite Spiking Solutions

7.1.1. Weigh 0.0100 g of each XDE-175 analytical standard (XDE-175-J, XDE-175-L, XDE-175-*N*-demethyl-J, XDE-175-*N*-demethyl-L, XDE-175-*N*-formyl-J, XDE-175-*N*-formyl-L) and quantitatively transfer each standard to separate 100-mL volumetric flasks with acetonitrile. Dilute to volume with acetonitrile to obtain a 100-µg/mL stock solution of each analyte.

7.1.2. Pipet 10.0 mL of each 100-µg/mL solution (Section 7.1.1.) into a 100-mL volumetric flask. Dilute to volume with acetonitrile to obtain a 10.0-µg/mL mixed XDE-175 and metabolite spiking solution. Further dilute the 10.0-µg/mL mixed XDE-175 and metabolite spiking solution with acetonitrile according to the following suggested scheme:

Concentration of Initial Stock Solution	Aliquot of Stock Solution	Final Soln. Volume	Spiking Soln. Final Conc.	Equivalent Sample Conc.[a]	Volume of Spiking Soln.
µg/mL	mL	mL	µg/mL	µg/g	µL
- -	- -	- -	100.0	10.0	500[b]
100.0	10.0	100	10.0	1.0	500
- -	- -	- -	10.0	0.1	50
10.0	10.0	100	1.0	0.01	50
1.0	10.0	100	0.1	0.003	150
0.1	10.0	100	0.01	- -	- -

[a] The equivalent sample concentration is based on fortifying a 5-g crop sample.

[b] 500 µL of each of the XDE-175 and metabolite 100 µg/mL spiking solutions (Section 7.1.1.).

7.2. Preparation of XDE-175 and Metabolite Stable Isotope Internal Standard Solutions

7.2.1. Weigh 0.0100 g of each XDE-175 stable isotope standard (XDE-175-J IS, XDE-175-L IS, XDE-175-*N*-demethyl-J IS and XDE-175-*N*-demethyl-L IS) and quantitatively transfer each standard to separate 100-mL volumetric flasks with acetonitrile. Dilute to volume with acetonitrile to obtain a 100-µg/mL stock solution of stable isotope standard.

7.2.2. Pipet 10.0 mL of each 100-µg/mL solution (Section 7.2.1.) into a 100-mL volumetric flask. Dilute to volume with acetonitrile to obtain a 10.0-µg/mL mixed XDE-175 and metabolite stable isotope internal standard solution.

7.2.3. Pipet 1.0 mL of the 10.0-µg/mL mixed XDE-175 stable isotope internal standard solution (Section 7.2.2.) into a 100-mL volumetric flask. Dilute to volume with acetonitrile to obtain a 0.1-µg/mL mixed XDE-175 and metabolite stable isotope internal standard solution.

7.2.4. Pipet 5.0 mL of the 0.1-µg/mL mixed XDE-175 stable isotope internal standard solution (Section 7.2.3.) into a 100-mL volumetric flask. Add 75 mL of acetonitrile to the flask. Dilute to volume with water to obtain an acetonitrile/water solution (80:20) containing 5-ng/mL mixed XDE-175 and metabolite stable isotope internal standard solution.

7.3. Preparation of Mixed XDE-175 and Metabolite Calibration Solutions

7.3.1. Prepare calibration standard solutions by pipeting 5.0 mL of the 1.0-µg/mL mixed XDE-175 and metabolites stable isotope solution, prepared in Section 7.2.3, into each volumetric flask and diluting the 1.0-, 0.1- and 0.01-µg/mL mixed XDE-175 spiking solutions (Section 7.1.2.) with acetonitrile/water (80:20) to give calibration standards

over the range 0.15–50 ng/mL. Calibration standards may be prepared following the suggested scheme:

Concentration of Stock Solution	Aliquot of Spiking Solution	Final Soln. Volume	Calibration Soln. Final Conc.	Equivalent Sample Conc.[a]
µg/mL	mL	mL	ng/mL	µg/g
1.0	5.0	100	50	1.0
1.0	3.5	100	35	0.7
1.0	2.0	100	20	0.4
1.0	1.0	100	10	0.2
0.1	5.0	100	5.0	0.1
0.1	1.0	100	1.0	0.02
0.01	5.0	100	0.5	0.01
0.01	1.5	100	0.15	0.003

[a] The equivalent sample concentration is based on extracting a 5-g crop sample.

8. ON-LINE SPE/LIQUID CHROMATOGRAPHY/TANDEM MASS SPECTROMETRY

8.1. Typical Liquid Chromatography Operating Conditions (Note 12.2.)

Instrumentation:	Spark Holland Symbiosis Pharma MDS/Sciex API 4000 LC/MS/MS System MDS/Sciex Analyst 1.4 data system
Column:	YMC ODS-AM, 50 x 4.6 mm, 5-µm (Quantitation) Synergi Polar RP, 75 x 4.6 mm, 4-µm (Confirmation)
Column Temperature:	Ambient
Injection Volume:	20 µL
Autosampler Wash Program:	Autosampler loop and needle washed with: 1) 500 µL of acetonitrile/water (80:20) containing 0.1% formic acid 2) 4 x 300 µL of acetonitrile/water (80:20) containing 0.1% formic acid with valve wash 3) 500 µL of acetonitrile/water (80:20) containing 0.1% formic acid
Run Time:	Approx 5 mins 30 secs
Mobile Phase:	A –acetonitrile/methanol (1:1) containing 2 mM ammonium acetate B –water containing 2 mM ammonium acetate
Flow:	1.0 mL/min (approx 200 µL/min split to source)

Gradient:	Time, (min:secs)	A, %	B, %
	00:01	70	30
	02:00	100	0
	04:00	100	0
	04:15	70	30
	05:30	70	30

Flow Diverter Program: 1) 0.0→2.0 min: flow to waste
2) 2.0→4.0 min: flow to source
3) 4.0→end of run: flow to waste

8.2. Typical On-Line Solid Phase Extraction Operating Conditions

SPE Cartridge: Hysphere C18HD, 7-µm

SPE Solvation: acetonitrile, 1 mL at 5 mL/min (SSM A)

SPE Equilibration: water, 2 mL at 5 mL/min (SSM B)

Sample Extraction: acetonitrile/methanol/water (15:15:70) containing 10 mM ammonium acetate, 750 µL at 2 mL/min (HPD1)

SPE Wash 1: acetonitrile/methanol/water (25:25:50) containing 10 mM ammonium acetate, 750 µL at 2 mL/min (HPD1)

SPE Elution: gradient mode, HPLC mobile phase at 1.0 mL/min for 3 min

Clamp Flush 1: acetonitrile/water (80:20) containing 0.1 % formic acid, 1 mL at 5.0 mL/min (HPD2)

Clamp Flush 2: water, 2 mL at 5 mL/min (HPD2)

8.3. Typical Mass Spectrometry Operating Conditions (Note 12.2.)

Ionization Mode: ESI
Polarity: Positive
Scan Type: MRM
Resolution: Q1 – unit, Q3 – unit
Curtain Gas (CUR): 12 psi
Collision Gas (CAD): 4 psi
Temperature (TEM): 425 °C
Ion Source Gas 1 (GS1): 40 psi
Ion Source Gas 2 (GS2): 60 psi

Period 1
Acquisition Time Delay: 2.0 mins

Period Duration: 2.0 mins
Ion Spray Voltage (IS): 5500 V

Compound:	Ion, *m/z*		Time, ms	Collision Energy, V
	Q1	Q3		
XDE-175-J	748.6	142.2	50	37
XDE-175-L	760.9	142.2	50	37
XDE-175-*N*-demethyl-J	734.9	128.2	50	31
XDE-175-*N*-demethyl-L	746.7	128.2	50	33
XDE-175-*N*-formyl-J	762.8	156.2	50	29
XDE-175-*N*-formyl-L	774.6	156.2	50	23
XDE-175-J IS	757.9	146.2	50	37
XDE-175-L IS	769.9	146.2	50	37
XDE-175-*N*-Demethyl-J IS	739.9	128.2	50	33
XDE-175-*N*-Demethyl-L IS	751.7	128.2	50	33

8.4. Typical Mass Spectra

Typical mass spectra and product ion spectra of XDE-175, its metabolites and stable isotope internal standards are presented in Figures 1-20.

8.5. Typical Calibration Curve

Typical calibration curves for the determination of XDE-175 and its metabolites in wet crops are shown in Figures 21-26.

8.6. Typical Chromatograms

Typical chromatograms of a 0.5-ng/mL calibration standard, a control lettuce sample, a control lettuce sample fortified at 0.01 µg/g (limit of quantitation), and a control lettuce sample fortified at 10 µg/g (1000 times the limit of quantitation) are presented in Figures 27-32. Typical chromatograms generated using the confirmatory HPLC column are presented in Figures 33-38.

9. DETERMINATION OF RECOVERY OF XDE-175 AND ITS METABOLITES IN AGRICULTURAL COMMODITIES

9.1. Method Validation Prior to Field Sample Analysis

Unless otherwise specified, a sample set should contain, at the minimum, the following samples:

At least one reagent blank
At least one 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 a higher concentration

9.2. Sample Preparation

Prepare samples for analysis by freezing the crop with dry ice or liquid nitrogen and then grinding or chopping with a hammer mill equipped with a 1/8 or 3/16-inch screen size.

9.3. Sample Analysis for XDE-175 and Metabolites in Agricultural Commodities

9.3.1. Weigh 5 ± 0.05 g portions of sample into 250-mL HDPE bottles.

9.3.2. Add the required volume of the appropriate fortification solution to the recovery samples (Section 7.1.2.) using a positive displacement pipet.

9.3.3. Add 100 mL of acetonitrile/water (80:20).

9.3.4. Homogenize the samples with a 20-mm homogenizer probe for 1 minute. Cap the sample and shake for 30 minutes on a flat bed shaker at approximately 180 excursions per minute.

9.3.5. Centrifuge the sample for 5 minutes at 2000 rpm.

9.3.6. Pipet 500 µL of the extraction solution into a 96-well plate.

9.3.7. Add 25 µL of the 0.1-µg/mL mixed XDE-175 and metabolite stable isotope standard (Section 7.2.3.).

9.3.8. Add approximately 500 µL of each calibration standard (Section 7.3.1.) to empty wells of the 96-well plate, cap and vortex mix for approximately 30 seconds.

9.3.9. Chromatograph the samples and standard using the conditions given in Section 8, injecting the calibration standards evenly spaced throughout the run.

9.3.10. For sample extracts which contain XDE-175 and metabolite concentrations > 50 ng/mL (equivalent to >1 µg/g), dilute with acetonitrile:water (80:20) containing 5-ng/mL mixed XDE-175 and metabolite stable isotope standard. Determine the suitability of the chromatographic system using the following criteria:

a. Standard curve linearity: Determine that the correlation coefficient 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: Determine visually that sufficient resolution has been achieved for the analyte relative to any background interferences.

c. Appearance of chromatograms: Visually determine that the chromatograms resemble those shown in Figures 27-32 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 the 0.5-ng/mL calibration standard (equivalent to 0.01 µg/g of XDE-175 and or metabolites in the crop sample).

10. CALCULATIONS

10.1. Determination of Isotopic Crossover

In this assay, the analyte and internal standard are quantitated using MS/MS transitions characteristic of each compound. When using stable-isotope labeled internal standards, there is a possibility that isotopic contributions will occur between the transitions used for quantitation of the unlabeled and labeled compounds. This isotopic overlap between the analyte and the internal standard can be determined empirically by analyzing standard solutions of each compound and should be addressed for accurate determination of concentrations.

10.1.1. To determine the isotopic crossover for XDE-175 and its metabolites and their respective stable isotopes, inject a 5-ng/mL mixed XDE-175 and metabolite standard and a 5-ng/mL mixed XDE-175 stable isotope standard and determine the peak areas for the analyte and internal standard as indicated below. For example, to determine the contribution of the unlabeled XDE-175-J to the stable isotope labeled XDE-175-J internal standard:

XDE-175-J	*m/z* Q1/Q3 748.6/142.2
XDE-175-J IS	*m/z* Q1/Q3 757.9/146.2

To determine the contribution of the unlabeled XDE-175-J to the labeled XDE-175-J internal standard:

$$\text{Crossover Factor (analyte}\rightarrow\text{ISTD)} = \frac{\text{peak area of internal standard transition}}{\text{peak area of analyte transition}}$$

$$\text{Crossover Factor (analyte}\rightarrow\text{ISTD)} = \frac{\text{peak area at } m/z \text{ 757.9/146.2}}{\text{peak area at } m/z \text{ 748.6/142.2}}$$

In a similar manner, to determine the contribution of the labeled XDE-175-J stable isotope to the unlabeled XDE-175-J:

$$\text{Crossover Factor (ISTD}\rightarrow\text{ analyte)} = \frac{\text{peak area of analyte transition}}{\text{peak area of internal standard transition}}$$

$$\text{Crossover Factor (ISTD} \rightarrow \text{analyte)} = \frac{\text{peak area at } m/z \text{ 748.6/142.2}}{\text{peak area at } m/z \text{ 757.9/146.2}}$$

During method development, no mass spectral isotopic crossover was observed and therefore no correction of the measured quantitation ratio was performed. If isotopic crossover is encountered it should be assessed and the respective quantitation ratios corrected for accurate determination of concentrations (13.1, 13.2).

10.2. Calculation of Standard Calibration Curve for XDE-175 and its Metabolites

10.2.1. Inject a series of calibration standards (Section 7.3.) using the conditions described in Section 8 and determine the peak areas for XDE-175, its metabolites and internal standards as indicated below:

XDE-175-J	*m/z* Q1/Q3 748.6/142.2
XDE-175-L	*m/z* Q1/Q3 760.9/142.2
XDE-175-*N*-demethyl-J	*m/z* Q1/Q3 734.9/128.2
XDE-175-*N*-demethyl-L	*m/z* Q1/Q3 746.7/128.2
XDE-175-*N*-formyl-J	*m/z* Q1/Q3 762.8/156.2
XDE-175-*N*-formyl-L	*m/z* Q1/Q3 774.6/156.2
XDE-175-J IS	*m/z* Q1/Q3 757.9/146.2
XDE-175-L IS	*m/z* Q1/Q3 769.9/146.2
XDE-175-*N*-demethyl-J IS	*m/z* Q1/Q3 739.9/128.2
XDE-175-*N*-demethyl-L IS	*m/z* Q1/Q3 751.7/128.2

Quantitation of XDE-175-*N*-formyl-J and XDE-175-*N*-formyl-L metabolites are performed using the XDE-175-*N*-demethyl-J IS and XDE-175-*N*-demethyl-L IS respectively.

10.2.2. For each standard, calculate the XDE-175 quantitation ratio.

For example, using the data for XDE-175-J from injection no. 9, Figure 21:

$$\text{Quantitation Ratio} = \frac{\text{peak area of quantitation ion}}{\text{peak area of internal standard ion}}$$

$$\text{Quantitation Ratio} = \frac{\text{XDE-175-J peak area}}{\text{XDE-175-J IS stable isotope internal standard peak area}}$$

$$\text{Quantitation Ratio} = \frac{51007}{379980}$$

$$\text{Quantitation Ratio} = 0.1342$$

10.2.3. Prepare a standard curve by plotting the concentration of the analytes on the abscissa (x-axis) and the respective quantitation ratio on the ordinate (y-axis), as shown in Figures 21-26. Using linear regression analysis (13.3.) with a 1/x weighting (13.4.), determine the equation for the curve with respect to the abscissa.

For example, using the XDE-175-J data from Figure 21:

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

$$\text{XDE-175-J conc. (ng/mL)} = \left(\frac{\text{XDE-175-J quantitation ratio} - \text{intercept}}{\text{slope}}\right)$$

$$\text{XDE-175-J conc. (ng/mL)} = \left(\frac{\text{XDE-175-J quantitation ratio} - (-0.0015)}{0.2571}\right)$$

10.3. Calculation of Percent Recovery for XDE-175 and its Metabolites

10.3.1. Determine the gross concentration in each recovery sample by substituting the quantitation ratio obtained into the above equation and solving for the concentration.

For example, using the data for XDE-175-J data from injection no. 22, Figure 21:

$$\text{XDE-175-J conc. (ng/mL)} = \left(\frac{\text{XDE-175-J quantitation ratio} - (-0.0015)}{0.2571}\right)$$

$$\text{XDE-175-J conc. (gross ng/mL)} = \left(\frac{0.12128 - (-0.0015)}{0.2571}\right)$$

$$\text{XDE-175-J conc. (gross)} = 0.47756 \text{ ng/mL}$$

Convert the concentration of ng/mL of XDE-175-J found in the final sample extract prepared for analysis to µg/g of XDE-175-J in the original crop sample as follows:

$$\text{XDE-175-J conc. (gross ng/g)} = 0.47756 \text{ ng/mL} \times \frac{(100 \text{ mL})}{5 \text{ g}} \times \text{DF}$$

Where DF = Dilution Factor where applicable

$$\text{XDE-175-J conc. (gross)} = 9.55 \text{ ng/g or } 0.0096 \text{ µg/g}$$

10.3.2. Determine the net concentration in each recovery sample by subtracting the concentration found at the retention time of each analyte in the untreated control sample from that of the gross analyte concentration in the recovery sample.

For example, using the data for XDE-175-J from Figure 21:

$$\text{XDE-175-J conc. (net µg/g)} = \text{XDE-175-J conc. (gross µg/g)} - \text{XDE-175-J conc. (control µg/g)}$$

$$\text{XDE-175-J conc. (net µg/g)} = 0.0096 \text{ µg/g} - 0.0000 \text{ µg/g}$$

$$\text{XDE-175-J conc. (net)} = 0.0096 \text{ µg/g}$$

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

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

$$\text{Recovery} = \frac{0.0096 \text{ µg/g}}{0.01 \text{ µg/g}} \text{ x } 100\%$$

$$\text{Recovery} = 96\%$$

10.4. Determination of XDE-175 and its Metabolites in Agricultural Commodities

10.4.1. Determine the gross concentration of XDE-175 and its metabolites in each sample by substituting the respective quantitation ratio into the equation for the calibration curve and calculating the uncorrected residue result as described in Section 10.3.1.

10.4.2. For those samples that require correction for the method procedural recovery, use the average recovery of all the recovery samples at or above the limit of quantitation, as described in Section 9.1, from a given sample set to correct for method efficiency. For example, continuing with the data from Figure 21 and the average recovery from Table 2 for the samples analyzed on 03-Dec-2004:

$$\text{XDE-175-J conc. (corrected µg/g)} = \text{XDE-175-J conc. (gross µg/g)} \times \left(\frac{100}{\text{Average \% Recovery}} \right)$$

$$\text{XDE-175-J conc. (corrected µg/g)} = 0.0096 \text{ µg/g} \times \frac{100}{101}$$

$$\text{XDE-175-J conc. (corrected)} = 0.0095 \text{ µg/g}$$

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-175 and its metabolites in agricultural commodities. Individual results are outlined in Tables 2-5 and are summarized in Tables 6-9.

For all of the analytes, the individual recoveries for all samples in the validation study (no outliers were rejected) were between 63 and 140% with standard deviations less than or equal to 20.7%. For all analyses, the average recoveries for each analyte at each fortification level were between 70 and 110% except for the 1.0 µg/g XDE-175-*N*-formyl-J and XDE-175-*N*-formyl-L average recoveries which were 115 and 111% respectively.

11.1.2. Standard Curve Linearity

For the linear regression analysis, the coefficients of determination (r^2) were greater or equal to 0.997 for all of the calibration curve determinations during the method validation. 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 (13.5.), the limits of quantitation (LOQ) and detection (LOD) were calculated for XDE-175 and its metabolites using the standard deviation for the 0.01-µg/g (LOQ) 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 LOQ results. The results are summarized in Tables 10-13.

The calculated method LOQ supports the validated LOQ of 0.01 µg/g for each analyte in each crop group with one exception. The calculated LOQ for XDE-175-*N*-formyl-L

in oily crops was 0.02 µg/g. Since the lowest level of fortification for recovery samples was 0.01 µg/g, the method LOQ is considered to be 0.01 µg/g. The calculated LOD's for XDE-175-J, XDE-175-L, XDE-175-*N*-demethyl-J and XDE-175-*N*-demethyl-L were all <0.003 µg/g for each crop group which supports a method LOD of 0.003 µg/g. The calculated LOD's for XDE-175-*N*-formyl-J were in the range 0.0020-0.0040. The calculated LOD's for XDE-175-*N*-formyl-L were in the range 0.0021-0.0062. In actual residue samples, numerical results should be reported as less than the LOQ (<0.01 µg/g) for residues that are greater than or equal to the LOD but less than the validated LOQ. For results less than the LOD, numerical results should be reported as not detected (ND).

11.2. Confirmation of Residue Identity

The presence of XDE-175 and its metabolites is confirmed by comparing the liquid chromatography retention times of the analyte in the calibration standards with those found in the samples while monitoring analyte specific MS/MS transitions. According to recently published guidelines (13.6.), when detection is performed by tandem mass spectrometry methods, confirmation of the presence of the analyte requires the observation of a precursor ion plus one structurally significant product ion observed at the same retention time. Due to the lack of confirmatory MS/MS transitions, further confirmation of residue identity can be achieved, if necessary, by re-injecting the sample on the different selectivity column described in Section 8.

11.3. Assay Time and Stopping Points

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 15 samples. This typical analytical set can be prepared in approximately 3 hours followed by the chromatographic analysis.

There are four acceptable "stopping points" in the method, where sample preparation (Section 9) may be suspended, upon completion of a step, without deleterious effects on the sample analysis. These are indicated below:

Step 9.3.1. (store frozen)
Step 9.3.4. (store refrigerated)
Step 9.3.7. (store refrigerated)
Step 9.3.8. (store refrigerated)

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. Operating conditions may be modified to obtain optimal chromatographic separation and performance, if necessary.

13. REFERENCES

13.1. Jenden, D. J.; Roch, M.; Booth, R. A. *Anal. Biochem.* **1973**, 55, 438-448.

13.2. Barbalas, M. P.; Garland, W. A., *J. Pharm. Sci.* **1991**, *80*(10), 922-927.

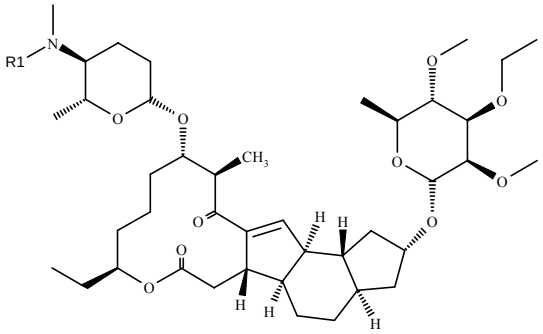
13.3. Freund, J. E.; Williams, F. J. *Dictionary/Outline of Basic Statistics*; Dover: New York, 1991; p 170.

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

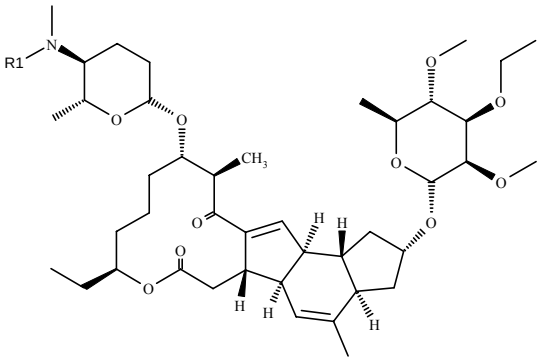
13.5. Keith, L. H.; Crummett, W.; Deegan, J., Jr.; Libby, R. A.; Taylor, J. K.; Wentler, G. *Anal. Chem.* **1983**, *55*, 2210-2218.

13.6. Baldwin, R.; Bethem, R. A.; Boyd, R. K.; Budde, W. L.; Cairns, T.; Gibbons, R. D.; Henion, J. D.; Kaiser, M. A.; Lewis, D. L.; Matusik, J. E.; Sphon, J. A.; Stephany, R. W.; Trubey, R. K.; *J. Am. Soc. Mass Spectrom.* **1997**, *8*, 1180-1190.

Table 1. Identity and Structure of XDE-175, its Metabolites and Stable Isotope Internal Standards



XDE-175-J IS, R1 = CH_3
N-demethyl-XDE-175-J IS, R1 = H
N-formyl-XDE-175-J IS, R1 = C(O)H



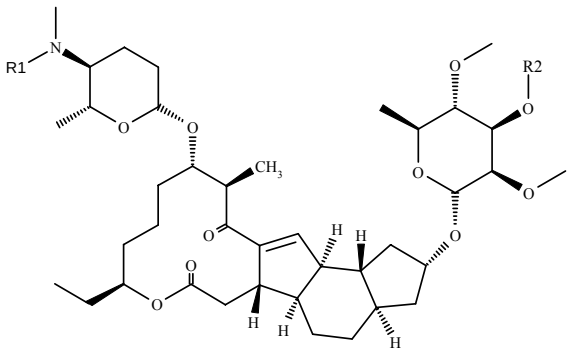
XDE-175-L IS, R1 = CH_3
N-demethyl-XDE-175-L IS, R1 = H
N-formyl-XDE-175-L IS, R1 = C(O)H

Common Name of Compound
XDE-175-J Molecular Formula: $C_{42}H_{69}NO_{10}$ Formula Weight: 748.010 Nominal Mass: 747.5 CAS Registry Number: 187166-40-1 CAS Name: 1H-as-Indaceno[3,2-d]oxacyclododecin-7,15-dione, 2-[(6-deoxy-3-O-ethyl-2,4-di-O methyl-a-L-mannopyranosyl)oxy]-13-[[(2R,5S,6R)-5-(dimethylamino)tetrahydro-6-methyl 2H-pyran-2-yl]oxy]-9-ethyl-2,3,3a,4,5,5a,5b,6,9,10,11,12,13,14,16a,16b-hexadecahydro 14-methyl-, (2R,3aR,5aR,5bS,9S,13S,14R,16aS,16bR)
XDE-175-L Molecular Formula: $C_{43}H_{69}NO_{10}$ Formula Weight: 760.022 Nominal Mass: 759.5 CAS Registry Number: 187166-15-0 CAS Name: 1H-as-Indaceno[3,2-d]oxacyclododecin-7,15-dione, 2-[(6-deoxy-3-O-ethyl-2,4-di-O-methyl-a-L-mannopyranosyl)oxy]-13-[[(2R,5S,6R)-5-(dimethylamino)tetrahydro-6-methyl-2H-pyran-2-yl]oxy]-9-ethyl-2,3,3a,5a,5b,6,9,10,11,12,13,14,16a,16b-tetradecahydro-4,14-dimethyl-, (2S,3aR,5aS,5bS,9S,13S,14R,16aS,16bS)

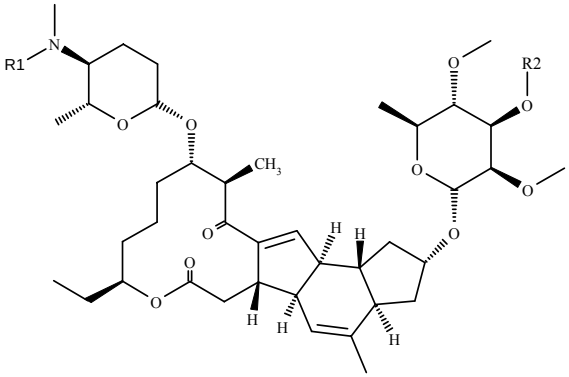
Table 1. (Cont.) Identity and Structure of XDE-175, its Metabolites and Stable Isotope Internal Standards

XDE-175-*N*-demethyl-J Molecular Formula: $C_{41}H_{67}NO_{10}$ Formula Weight: 733.984 Nominal Mass: 733.5 CAS Registry Number: N/A IUPAC Name: (2R,3aR,5aR,5bS,9S,13S,14R,16aS,16bR)-9-ethyl-14-methyl-13-{[(2S,5S,6R)-6-methyl-5-(methylamino)tetrahydro-2H-pyran-2-yl]oxy}-7,15-dioxo-2,3,3a,4,5,5a,5b,6,7,9,10,11,12,13,14,15,16a,16b-octadecahydro-1H-as-indaceno[3,2-d]oxacyclododecin-2-yl 6-deoxy-3-O-ethyl-2,4-di-O-methyl-beta-L-mannopyranoside
XDE-175-*N*-demethyl-L Molecular Formula: $C_{42}H_{67}NO_{10}$ Formula Weight: 745.995 Nominal Mass: 745.5 CAS Registry Number: N/A IUPAC Name: (2S,3aR,5aS,5bS,9S,13S,14R,16aS,16bS)-9-ethyl-4,14-dimethyl-13-{[(2S,5S,6R)-6-methyl-5-(methylamino)tetrahydro-2H-pyran-2-yl]oxy}-7,15-dioxo-2,3,3a,5a,5b,6,7,9,10,11,12,13,14,15,16a,16b-hexadecahydro-1H-as-indaceno[3,2-d]oxacyclododecin-2-yl 6-deoxy-3-O-ethyl-2,4-di-O-methyl-beta-L-mannopyranoside
XDE-175-*N*-formyl-J Molecular Formula: $C_{42}H_{67}NO_{11}$ Formula Weight: 761.994 Nominal Mass: 761.5 CAS Registry Number: N/A IUPAC Name: (2R,3S,6S)-6-({(2R,3aR,5aR,5bS,9S,13S,14R,16aS,16bR)-2-[(6-deoxy-3-O-ethyl-2,4-di-O-methyl-beta-L-mannopyranosyl)oxy]-9-ethyl-14-methyl-7,15-dioxo-2,3,3a,4,5,5a,5b,6,7,9,10,11,12,13,14,15,16a,16b-octadecahydro-1H-as-indaceno[3,2-d]oxacyclododecin-13-yl}oxy)-2-methyltetrahydro-2H-pyran-3-yl(methyl)formamide
XDE-175-*N*-formyl-L Molecular Formula: $C_{43}H_{67}NO_{11}$ Formula Weight: 774.005 Nominal Mass: 773.5 CAS Registry Number: N/A IUPAC Name: (2R,3S,6S)-6-({(2S,3aR,5aS,5bS,9S,13S,14R,16aS,16bS)-2-[(6-deoxy-3-O-ethyl-2,4-di-O-methyl-beta-L-mannopyranosyl)oxy]-9-ethyl-4,14-dimethyl-7,15-dioxo-2,3,3a,5a,5b,6,7,9,10,11,12,13,14,15,16a,16b-hexadecahydro-1H-as-indaceno[3,2-d]oxacyclododecin-13-yl}oxy)-2-methyltetrahydro-2H-pyran-3-yl(methyl)formamide

Table 1. (Cont.) Identity and Structure of XDE-175, its Metabolites and Stable Isotope Internal Standards



XDE-175-J, R1 = $^{13}CD_3$, R2 = C_2D_5
XDE-175-*N*-Demethyl-J, R1 = H, R2 = C_2D_5



XDE-175-L, R1 = $^{13}CD_3$, R2 = C_2D_5
XDE-175-*N*-Demethyl-L, R1 = H, R2 = C_2D_5

Common Name of Internal Standard	
XDE-175-J IS	
Molecular Formula:	$C_{41}{}^{13}CH_{61}D_8NO_{10}$
Formula Weight:	757.051
Nominal Mass:	756.5
CAS Registry Number:	N/A
XDE-175-L IS	
Molecular Formula:	$C_{42}{}^{13}CH_{61}D_8NO_{10}$
Formula Weight:	769.062
Nominal Mass:	768.5
CAS Registry Number:	N/A
XDE-175-N-Demethyl-J IS	
Molecular Formula:	$C_{41}H_{62}D_5NO_{10}$
Formula Weight:	739.014
Nominal Mass:	738.5
CAS Registry Number:	N/A
XDE-175-N-Demethyl-L	
Molecular Formula:	$C_{42}H_{62}D_5NO_{10}$
Formula Weight:	751.025
Nominal Mass:	750.5
CAS Registry Number:	N/A

Table 2. Recovery of XDE-175-J from Wet Crops

Sample Name	Matrix	Date of Analysis[a]	XDE-175-J Added	XDE-175-J Found	XDE-175-J % Recovery[b]
10505	Broccoli Flower Head and Stem	3-Dec-2004	NA[c]	ND[d]	NA
13155	Grape Whole Fruit	3-Dec-2004	NA	ND	NA
24801	Lettuce Whole Leaf	3-Dec-2004	NA	ND	NA
4301	Onion sweet yellow	3-Dec-2004	NA	ND	NA
16401	Peppers	3-Dec-2004	NA	ND	NA
99301	Cabbage Whole Head	3-Dec-2004	NA	ND	NA
13210	Tomato Whole Fruit	3-Dec-2004	NA	ND	NA
15812	Succulent Green Bean	7-Dec-2004	NA	ND	NA
15822	Leek Whole Plant	7-Dec-2004	NA	ND	NA
24801	Lettuce Whole Leaf	25-Apr-2005	NA	ND	NA
10505	Broccoli Flower Head and Stem	3-Dec-2004	0.003	0.0028[e]	NA
13155	Grape Whole Fruit	3-Dec-2004	0.003	0.0033	NA
24801	Lettuce Whole Leaf	3-Dec-2004	0.003	0.0031	NA
4301	Onion sweet yellow	3-Dec-2004	0.003	0.0031	NA
16401	Peppers	3-Dec-2004	0.003	0.0030	NA
13210	Tomato Whole Fruit	3-Dec-2004	0.003	0.0032	NA
15812	Succulent Green Bean	7-Dec-2004	0.003	0.0031	NA
15822	Leek Whole Plant	7-Dec-2004	0.003	0.0030	NA
10505	Broccoli Flower Head and Stem	3-Dec-2004	0.01	0.0097	97
10505	Broccoli Flower Head and Stem	3-Dec-2004	0.01	0.0096	96
13155	Grape Whole Fruit	3-Dec-2004	0.01	0.0103	103
13155	Grape Whole Fruit	3-Dec-2004	0.01	0.0101	101
24801	Lettuce Whole Leaf	3-Dec-2004	0.01	0.0095	95
24801	Lettuce Whole Leaf	3-Dec-2004	0.01	0.0104	104
4301	Onion sweet yellow	3-Dec-2004	0.01	0.0103	103
4301	Onion sweet yellow	3-Dec-2004	0.01	0.0108	108
16401	Peppers	3-Dec-2004	0.01	0.0103	103
16401	Peppers	3-Dec-2004	0.01	0.0101	101
99301	Cabbage Whole Head	3-Dec-2004	0.01	0.0110	110
99301	Cabbage Whole Head	3-Dec-2004	0.01	0.0095	95
13210	Tomato Whole Fruit	3-Dec-2004	0.01	0.0102	102
13210	Tomato Whole Fruit	3-Dec-2004	0.01	0.0105	105
15812	Succulent Green Bean	7-Dec-2004	0.01	0.0098	98
15812	Succulent Green Bean	7-Dec-2004	0.01	0.0104	104
15822	Leek Whole Plant	7-Dec-2004	0.01	0.0107	107
15822	Leek Whole Plant	7-Dec-2004	0.01	0.0118	118

Table 2. (Cont.) Recovery of XDE-175-J from Wet Crops

Sample Name	Matrix	Date of Analysis	XDE-175-J Added	XDE-175-J Found	% Recovery
10505	Broccoli Flower Head and Stem	3-Dec-2004	1.0	0.98	98
10505	Broccoli Flower Head and Stem	3-Dec-2004	1.0	0.92	92
13155	Grape Whole Fruit	3-Dec-2004	1.0	1.02	102
13155	Grape Whole Fruit	3-Dec-2004	1.0	1.00	100
4301	Onion sweet yellow	3-Dec-2004	1.0	1.02	102
4301	Onion sweet yellow	3-Dec-2004	1.0	1.03	103
16401	Peppers	3-Dec-2004	1.0	0.96	96
16401	Peppers	3-Dec-2004	1.0	1.03	103
99301	Cabbage Whole Head	3-Dec-2004	1.0	0.95	95
99301	Cabbage Whole Head	3-Dec-2004	1.0	1.00	100
13210	Tomato Whole Fruit	3-Dec-2004	1.0	1.01	101
13210	Tomato Whole Fruit	3-Dec-2004	1.0	1.02	102
15812	Succulent Green Bean	7-Dec-2004	1.0	0.98	98
15812	Succulent Green Bean	7-Dec-2004	1.0	0.95	95
15822	Leek Whole Plant	7-Dec-2004	1.0	0.96	96
15822	Leek Whole Plant	7-Dec-2004	1.0	0.96	96
24801	Lettuce Whole Leaf	3-Dec-2004	10.0	9.3	93
24801	Lettuce Whole Leaf	3-Dec-2004	10.0	9.4	94
24801	Lettuce Whole Leaf	25-Apr-2005	10.0	9.3	93
24801	Lettuce Whole Leaf	25-Apr-2005	10.0	9.2	92
24801	Lettuce Whole Leaf	25-Apr-2005	10.0	9.0	90
24801	Lettuce Whole Leaf	25-Apr-2005	10.0	9.2	92

Table 2. (Cont.) Recovery of XDE-175-L from Wet Crops

Name	Matrix	Date of Analysis	XDE-175-L Added	XDE-175-L Found	% Recovery
10505	Broccoli Flower Head and Stem	3-Dec-2004	NA	ND	NA
13155	Grape Whole Fruit	3-Dec-2004	NA	ND	NA
24801	Lettuce Whole Leaf	3-Dec-2004	NA	ND	NA
4301	Onion sweet yellow	3-Dec-2004	NA	ND	NA
16401	Peppers	3-Dec-2004	NA	ND	NA
99301	Cabbage Whole Head	3-Dec-2004	NA	ND	NA
13210	Tomato Whole Fruit	3-Dec-2004	NA	ND	NA
15812	Succulent Green Bean	7-Dec-2004	NA	ND	NA
15822	Leek Whole Plant	7-Dec-2004	NA	ND	NA
24801	Lettuce Whole Leaf	25-Apr-2005	NA	ND	NA
10505	Broccoli Flower Head and Stem	3-Dec-2004	0.003	0.0032	NA
13155	Grape Whole Fruit	3-Dec-2004	0.003	0.0033	NA
24801	Lettuce Whole Leaf	3-Dec-2004	0.003	0.0032	NA
4301	Onion sweet yellow	3-Dec-2004	0.003	0.0030	NA
16401	Peppers	3-Dec-2004	0.003	0.0030	NA
13210	Tomato Whole Fruit	3-Dec-2004	0.003	0.0033	NA
15812	Succulent Green Bean	7-Dec-2004	0.003	0.0032	NA
15822	Leek Whole Plant	7-Dec-2004	0.003	0.0031	NA
10505	Broccoli Flower Head and Stem	3-Dec-2004	0.01	0.0102	102
10505	Broccoli Flower Head and Stem	3-Dec-2004	0.01	0.0107	107
13155	Grape Whole Fruit	3-Dec-2004	0.01	0.0101	101
13155	Grape Whole Fruit	3-Dec-2004	0.01	0.0096	96
24801	Lettuce Whole Leaf	3-Dec-2004	0.01	0.0100	100
24801	Lettuce Whole Leaf	3-Dec-2004	0.01	0.0108	108
4301	Onion sweet yellow	3-Dec-2004	0.01	0.0102	102
4301	Onion sweet yellow	3-Dec-2004	0.01	0.0112	112
16401	Peppers	3-Dec-2004	0.01	0.0100	100
16401	Peppers	3-Dec-2004	0.01	0.0098	98
99301	Cabbage Whole Head	3-Dec-2004	0.01	0.0114	114
99301	Cabbage Whole Head	3-Dec-2004	0.01	0.0097	97
13210	Tomato Whole Fruit	3-Dec-2004	0.01	0.0102	102
13210	Tomato Whole Fruit	3-Dec-2004	0.01	0.0107	107
15812	Succulent Green Bean	7-Dec-2004	0.01	0.0106	106
15812	Succulent Green Bean	7-Dec-2004	0.01	0.0105	105
15822	Leek Whole Plant	7-Dec-2004	0.01	0.0108	108
15822	Leek Whole Plant	7-Dec-2004	0.01	0.0114	114

Table 2. (Cont.) Recovery of XDE-175-L from Wet Crops

Name	Matrix	Date of Analysis	XDE-175-L Added	XDE-175-L Found	% Recovery
10505	Broccoli Flower Head and Stem	3-Dec-2004	1.0	1.07	107
10505	Broccoli Flower Head and Stem	3-Dec-2004	1.0	1.04	104
13155	Grape Whole Fruit	3-Dec-2004	1.0	0.97	97
13155	Grape Whole Fruit	3-Dec-2004	1.0	0.97	97
4301	Onion sweet yellow	3-Dec-2004	1.0	1.00	100
4301	Onion sweet yellow	3-Dec-2004	1.0	1.04	104
16401	Peppers	3-Dec-2004	1.0	1.00	100
16401	Peppers	3-Dec-2004	1.0	1.00	100
99301	Cabbage Whole Head	3-Dec-2004	1.0	1.03	103
99301	Cabbage Whole Head	3-Dec-2004	1.0	1.00	100
13210	Tomato Whole Fruit	3-Dec-2004	1.0	1.01	101
13210	Tomato Whole Fruit	3-Dec-2004	1.0	1.00	100
15812	Succulent Green Bean	7-Dec-2004	1.0	1.00	100
15812	Succulent Green Bean	7-Dec-2004	1.0	0.95	95
15822	Leek Whole Plant	7-Dec-2004	1.0	1.01	101
15822	Leek Whole Plant	7-Dec-2004	1.0	1.00	100
24801	Lettuce Whole Leaf	3-Dec-2004	10.0	9.1	91
24801	Lettuce Whole Leaf	3-Dec-2004	10.0	9.8	98
24801	Lettuce Whole Leaf	25-Apr-2005	10.0	9.1	91
24801	Lettuce Whole Leaf	25-Apr-2005	10.0	9.1	91
24801	Lettuce Whole Leaf	25-Apr-2005	10.0	8.9	89
24801	Lettuce Whole Leaf	25-Apr-2005	10.0	9.1	91

Table 2. (Cont.) Recovery of XDE-175-*N*-demethyl-J from Wet Crops

Sample Name	Matrix	Date of Analysis	XDE-175-*N*-demethyl-J Added	Found	% Recovery
10505	Broccoli Flower Head and Stem	3-Dec-2004	NA	ND	NA
13155	Grape Whole Fruit	3-Dec-2004	NA	ND	NA
24801	Lettuce Whole Leaf	3-Dec-2004	NA	ND	NA
4301	Onion sweet yellow	3-Dec-2004	NA	ND	NA
16401	Peppers	3-Dec-2004	NA	ND	NA
99301	Cabbage Whole Head	3-Dec-2004	NA	ND	NA
13210	Tomato Whole Fruit	3-Dec-2004	NA	0.0000	NA
15812	Succulent Green Bean	7-Dec-2004	NA	ND	NA
15822	Leek Whole Plant	7-Dec-2004	NA	0.0001	NA
24801	Lettuce Whole Leaf	25-Apr-2005	NA	ND	NA
10505	Broccoli Flower Head and Stem	3-Dec-2004	0.003	0.0030	NA
13155	Grape Whole Fruit	3-Dec-2004	0.003	0.0029	NA
24801	Lettuce Whole Leaf	3-Dec-2004	0.003	0.0027	NA
4301	Onion sweet yellow	3-Dec-2004	0.003	0.0027	NA
16401	Peppers	3-Dec-2004	0.003	0.0027	NA
13210	Tomato Whole Fruit	3-Dec-2004	0.003	0.0027	NA
15812	Succulent Green Bean	7-Dec-2004	0.003	0.0033	NA
15822	Leek Whole Plant	7-Dec-2004	0.003	0.0031	NA
10505	Broccoli Flower Head and Stem	3-Dec-2004	0.01	0.0100	100
10505	Broccoli Flower Head and Stem	3-Dec-2004	0.01	0.0101	101
13155	Grape Whole Fruit	3-Dec-2004	0.01	0.0102	102
13155	Grape Whole Fruit	3-Dec-2004	0.01	0.0096	96
24801	Lettuce Whole Leaf	3-Dec-2004	0.01	0.0094	94
24801	Lettuce Whole Leaf	3-Dec-2004	0.01	0.0101	101
4301	Onion sweet yellow	3-Dec-2004	0.01	0.0099	99
4301	Onion sweet yellow	3-Dec-2004	0.01	0.0100	100
16401	Peppers	3-Dec-2004	0.01	0.0094	94
16401	Peppers	3-Dec-2004	0.01	0.0097	97
99301	Cabbage Whole Head	3-Dec-2004	0.01	0.0100	100
99301	Cabbage Whole Head	3-Dec-2004	0.01	0.0094	94
13210	Tomato Whole Fruit	3-Dec-2004	0.01	0.0097	97
13210	Tomato Whole Fruit	3-Dec-2004	0.01	0.0098	98
15812	Succulent Green Bean	7-Dec-2004	0.01	0.0103	103
15812	Succulent Green Bean	7-Dec-2004	0.01	0.0110	110
15822	Leek Whole Plant	7-Dec-2004	0.01	0.0103	103
15822	Leek Whole Plant	7-Dec-2004	0.01	0.0117	117

Table 2. (Cont.) Recovery of XDE-175-*N*-demethyl-J from Wet Crops

Sample Name	Matrix	Date of Analysis	XDE-175-*N*-demethyl-J Added	Found	% Recovery
10505	Broccoli Flower Head and Stem	3-Dec-2004	1.0	1.01	101
10505	Broccoli Flower Head and Stem	3-Dec-2004	1.0	0.95	95
13155	Grape Whole Fruit	3-Dec-2004	1.0	0.99	99
13155	Grape Whole Fruit	3-Dec-2004	1.0	0.94	94
4301	Onion sweet yellow	3-Dec-2004	1.0	1.00	100
4301	Onion sweet yellow	3-Dec-2004	1.0	1.00	100
16401	Peppers	3-Dec-2004	1.0	1.02	102
16401	Peppers	3-Dec-2004	1.0	0.99	99
99301	Cabbage Whole Head	3-Dec-2004	1.0	0.99	99
99301	Cabbage Whole Head	3-Dec-2004	1.0	1.00	100
13210	Tomato Whole Fruit	3-Dec-2004	1.0	0.99	99
13210	Tomato Whole Fruit	3-Dec-2004	1.0	0.99	99
15812	Succulent Green Bean	7-Dec-2004	1.0	1.05	105
15812	Succulent Green Bean	7-Dec-2004	1.0	0.98	98
15822	Leek Whole Plant	7-Dec-2004	1.0	1.02	102
15822	Leek Whole Plant	7-Dec-2004	1.0	1.04	104
24801	Lettuce Whole Leaf	3-Dec-2004	10.0	10.1	101
24801	Lettuce Whole Leaf	3-Dec-2004	10.0	10.1	101
24801	Lettuce Whole Leaf	25-Apr-2005	10.0	9.1	91
24801	Lettuce Whole Leaf	25-Apr-2005	10.0	9.0	90
24801	Lettuce Whole Leaf	25-Apr-2005	10.0	8.9	89
24801	Lettuce Whole Leaf	25-Apr-2005	10.0	9.1	91

Table 2. (Cont.) Recovery of XDE-175-*N*-demethyl-L from Wet Crops

Sample Name	Matrix	Date of Analysis	XDE-175-*N*-demethyl-L Added	Found	% Recovery
10505	Broccoli Flower Head and Stem	3-Dec-2004	NA	0.0000	NA
13155	Grape Whole Fruit	3-Dec-2004	NA	ND	NA
24801	Lettuce Whole Leaf	3-Dec-2004	NA	ND	NA
4301	Onion sweet yellow	3-Dec-2004	NA	ND	NA
16401	Peppers	3-Dec-2004	NA	0.0000	NA
99301	Cabbage Whole Head	3-Dec-2004	NA	ND	NA
13210	Tomato Whole Fruit	3-Dec-2004	NA	0.0000	NA
15812	Succulent Green Bean	7-Dec-2004	NA	ND	NA
15822	Leek Whole Plant	7-Dec-2004	NA	ND	NA
24801	Lettuce Whole Leaf	25-Apr-2005	NA	0.0002	NA
10505	Broccoli Flower Head and Stem	3-Dec-2004	0.003	0.0027	NA
13155	Grape Whole Fruit	3-Dec-2004	0.003	0.0030	NA
24801	Lettuce Whole Leaf	3-Dec-2004	0.003	0.0029	NA
4301	Onion sweet yellow	3-Dec-2004	0.003	0.0029	NA
16401	Peppers	3-Dec-2004	0.003	0.0028	NA
13210	Tomato Whole Fruit	3-Dec-2004	0.003	0.0030	NA
15812	Succulent Green Bean	7-Dec-2004	0.003	0.0033	NA
15822	Leek Whole Plant	7-Dec-2004	0.003	0.0029	NA
10505	Broccoli Flower Head and Stem	3-Dec-2004	0.01	0.0100	100
10505	Broccoli Flower Head and Stem	3-Dec-2004	0.01	0.0101	101
13155	Grape Whole Fruit	3-Dec-2004	0.01	0.0102	102
13155	Grape Whole Fruit	3-Dec-2004	0.01	0.0094	94
24801	Lettuce Whole Leaf	3-Dec-2004	0.01	0.0095	95
24801	Lettuce Whole Leaf	3-Dec-2004	0.01	0.0101	101
4301	Onion sweet yellow	3-Dec-2004	0.01	0.0109	109
4301	Onion sweet yellow	3-Dec-2004	0.01	0.0106	106
16401	Peppers	3-Dec-2004	0.01	0.0098	98
16401	Peppers	3-Dec-2004	0.01	0.0098	98
99301	Cabbage Whole Head	3-Dec-2004	0.01	0.0110	110
99301	Cabbage Whole Head	3-Dec-2004	0.01	0.0092	92
13210	Tomato Whole Fruit	3-Dec-2004	0.01	0.0101	101
13210	Tomato Whole Fruit	3-Dec-2004	0.01	0.0096	96
15812	Succulent Green Bean	7-Dec-2004	0.01	0.0102	102
15812	Succulent Green Bean	7-Dec-2004	0.01	0.0104	104
15822	Leek Whole Plant	7-Dec-2004	0.01	0.0109	109
15822	Leek Whole Plant	7-Dec-2004	0.01	0.0120	120

Table 2. (Cont.) Recovery of XDE-175-*N*-demethyl-L from Wet Crops

Sample Name	Matrix	Date of Analysis	XDE-175-*N*-demethyl-L Added	Found	% Recovery
10505	Broccoli Flower Head and Stem	3-Dec-2004	1.0	1.03	103
10505	Broccoli Flower Head and Stem	3-Dec-2004	1.0	0.95	95
13155	Grape Whole Fruit	3-Dec-2004	1.0	0.99	99
13155	Grape Whole Fruit	3-Dec-2004	1.0	0.98	98
4301	Onion sweet yellow	3-Dec-2004	1.0	1.01	101
4301	Onion sweet yellow	3-Dec-2004	1.0	1.00	100
16401	Peppers	3-Dec-2004	1.0	1.04	104
16401	Peppers	3-Dec-2004	1.0	1.01	101
99301	Cabbage Whole Head	3-Dec-2004	1.0	0.98	98
99301	Cabbage Whole Head	3-Dec-2004	1.0	1.02	102
13210	Tomato Whole Fruit	3-Dec-2004	1.0	1.03	103
13210	Tomato Whole Fruit	3-Dec-2004	1.0	1.01	101
15812	Succulent Green Bean	7-Dec-2004	1.0	1.01	101
15812	Succulent Green Bean	7-Dec-2004	1.0	1.00	100
15822	Leek Whole Plant	7-Dec-2004	1.0	1.02	102
15822	Leek Whole Plant	7-Dec-2004	1.0	1.01	101
24801	Lettuce Whole Leaf	3-Dec-2004	10.0	9.9	99
24801	Lettuce Whole Leaf	3-Dec-2004	10.0	9.8	98
24801	Lettuce Whole Leaf	25-Apr-2005	10.0	9.0	90
24801	Lettuce Whole Leaf	25-Apr-2005	10.0	8.7	87
24801	Lettuce Whole Leaf	25-Apr-2005	10.0	8.7	87
24801	Lettuce Whole Leaf	25-Apr-2005	10.0	8.8	88

Table 2. (Cont.) Recovery of XDE-175-*N*-formyl-J from Wet Crops

Sample Name	Matrix	Date of Analysis	XDE-175-*N*-formyl-J Added	Found	% Recovery
10505	Broccoli Flower Head and Stem	3-Dec-2004	NA	ND	NA
13155	Grape Whole Fruit	3-Dec-2004	NA	ND	NA
24801	Lettuce Whole Leaf	3-Dec-2004	NA	ND	NA
4301	Onion sweet yellow	3-Dec-2004	NA	ND	NA
16401	Peppers	3-Dec-2004	NA	ND	NA
99301	Cabbage Whole Head	3-Dec-2004	NA	ND	NA
13210	Tomato Whole Fruit	3-Dec-2004	NA	ND	NA
15812	Succulent Green Bean	7-Dec-2004	NA	ND	NA
15822	Leek Whole Plant	7-Dec-2004	NA	ND	NA
24801	Lettuce Whole Leaf	25-Apr-2005	NA	ND	NA
10505	Broccoli Flower Head and Stem	3-Dec-2004	0.003	0.0033	NA
13155	Grape Whole Fruit	3-Dec-2004	0.003	0.0023	NA
24801	Lettuce Whole Leaf	3-Dec-2004	0.003	0.0026	NA
4301	Onion sweet yellow	3-Dec-2004	0.003	0.0028	NA
16401	Peppers	3-Dec-2004	0.003	0.0021	NA
13210	Tomato Whole Fruit	3-Dec-2004	0.003	0.0022	NA
15812	Succulent Green Bean	7-Dec-2004	0.003	0.0030	NA
15822	Leek Whole Plant	7-Dec-2004	0.003	0.0020	NA
10505	Broccoli Flower Head and Stem	3-Dec-2004	0.01	0.0106	106
10505	Broccoli Flower Head and Stem	3-Dec-2004	0.01	0.0087	87
13155	Grape Whole Fruit	3-Dec-2004	0.01	0.0091	91
13155	Grape Whole Fruit	3-Dec-2004	0.01	0.0088	88
24801	Lettuce Whole Leaf	3-Dec-2004	0.01	0.0088	88
24801	Lettuce Whole Leaf	3-Dec-2004	0.01	0.0092	92
4301	Onion sweet yellow	3-Dec-2004	0.01	0.0093	93
4301	Onion sweet yellow	3-Dec-2004	0.01	0.0098	98
16401	Peppers	3-Dec-2004	0.01	0.0088	88
16401	Peppers	3-Dec-2004	0.01	0.0089	89
99301	Cabbage Whole Head	3-Dec-2004	0.01	0.0094	94
99301	Cabbage Whole Head	3-Dec-2004	0.01	0.0105	105
13210	Tomato Whole Fruit	3-Dec-2004	0.01	0.0086	86
13210	Tomato Whole Fruit	3-Dec-2004	0.01	0.0086	86
15812	Succulent Green Bean	7-Dec-2004	0.01	0.0093	93
15812	Succulent Green Bean	7-Dec-2004	0.01	0.0103	103
15822	Leek Whole Plant	7-Dec-2004	0.01	0.0086	86
15822	Leek Whole Plant	7-Dec-2004	0.01	0.0100	100

Table 2. (Cont.) Recovery of XDE-175-*N*-formyl-J from Wet Crops

Sample Name	Matrix	Date of Analysis	XDE-175-*N*-formyl-J Added	Found	% Recovery
10505	Broccoli Flower Head and Stem	3-Dec-2004	1.0	1.16	116
10505	Broccoli Flower Head and Stem	3-Dec-2004	1.0	1.10	110
13155	Grape Whole Fruit	3-Dec-2004	1.0	0.93	93
13155	Grape Whole Fruit	3-Dec-2004	1.0	0.93	93
4301	Onion sweet yellow	3-Dec-2004	1.0	1.01	101
4301	Onion sweet yellow	3-Dec-2004	1.0	1.02	102
16401	Peppers	3-Dec-2004	1.0	1.08	108
16401	Peppers	3-Dec-2004	1.0	1.04	104
99301	Cabbage Whole Head	3-Dec-2004	1.0	1.02	102
99301	Cabbage Whole Head	3-Dec-2004	1.0	1.00	100
13210	Tomato Whole Fruit	3-Dec-2004	1.0	1.03	103
13210	Tomato Whole Fruit	3-Dec-2004	1.0	1.04	104
15812	Succulent Green Bean	7-Dec-2004	1.0	1.02	102
15812	Succulent Green Bean	7-Dec-2004	1.0	0.97	97
15822	Leek Whole Plant	7-Dec-2004	1.0	0.91	91
15822	Leek Whole Plant	7-Dec-2004	1.0	0.91	91
24801	Lettuce Whole Leaf	3-Dec-2004	10.0	10.1	101
24801	Lettuce Whole Leaf	3-Dec-2004	10.0	9.9	99
24801	Lettuce Whole Leaf	25-Apr-2005	10.0	9.3	93
24801	Lettuce Whole Leaf	25-Apr-2005	10.0	8.4	84
24801	Lettuce Whole Leaf	25-Apr-2005	10.0	8.6	86
24801	Lettuce Whole Leaf	25-Apr-2005	10.0	8.6	86

Table 2. (Cont.) Recovery of XDE-175-*N*-formyl-L from Wet Crops

Sample Name	Matrix	Date of Analysis	XDE-175-*N*-formyl-L Added	Found	% Recovery
10505	Broccoli Flower Head and Stem	3-Dec-2004	NA	0.0006	NA
13155	Grape Whole Fruit	3-Dec-2004	NA	ND	NA
24801	Lettuce Whole Leaf	3-Dec-2004	NA	ND	NA
4301	Onion sweet yellow	3-Dec-2004	NA	ND	NA
16401	Peppers	3-Dec-2004	NA	0.0013	NA
99301	Cabbage Whole Head	3-Dec-2004	NA	ND	NA
13210	Tomato Whole Fruit	3-Dec-2004	NA	ND	NA
15812	Succulent Green Bean	7-Dec-2004	NA	ND	NA
15822	Leek Whole Plant	7-Dec-2004	NA	ND	NA
24801	Lettuce Whole Leaf	25-Apr-2005	NA	ND	NA
10505	Broccoli Flower Head and Stem	3-Dec-2004	0.003	0.0028	NA
13155	Grape Whole Fruit	3-Dec-2004	0.003	0.0033	NA
24801	Lettuce Whole Leaf	3-Dec-2004	0.003	0.0024	NA
4301	Onion sweet yellow	3-Dec-2004	0.003	0.0034	NA
16401	Peppers	3-Dec-2004	0.003	0.0017	NA
13210	Tomato Whole Fruit	3-Dec-2004	0.003	0.0030	NA
15812	Succulent Green Bean	7-Dec-2004	0.003	0.0022	NA
15822	Leek Whole Plant	7-Dec-2004	0.003	0.0019	NA
10505	Broccoli Flower Head and Stem	3-Dec-2004	0.01	0.0109	109
10505	Broccoli Flower Head and Stem	3-Dec-2004	0.01	0.0116	116
13155	Grape Whole Fruit	3-Dec-2004	0.01	0.0092	92
13155	Grape Whole Fruit	3-Dec-2004	0.01	0.0094	94
24801	Lettuce Whole Leaf	3-Dec-2004	0.01	0.0102	102
24801	Lettuce Whole Leaf	3-Dec-2004	0.01	0.0107	107
4301	Onion sweet yellow	3-Dec-2004	0.01	0.0115	115
4301	Onion sweet yellow	3-Dec-2004	0.01	0.0119	119
16401	Peppers	3-Dec-2004	0.01	0.0079	79
16401	Peppers	3-Dec-2004	0.01	0.0086	86
99301	Cabbage Whole Head	3-Dec-2004	0.01	0.0113	113
99301	Cabbage Whole Head	3-Dec-2004	0.01	0.0097	97
13210	Tomato Whole Fruit	3-Dec-2004	0.01	0.0097	97
13210	Tomato Whole Fruit	3-Dec-2004	0.01	0.0099	99
15812	Succulent Green Bean	7-Dec-2004	0.01	0.0096	96
15812	Succulent Green Bean	7-Dec-2004	0.01	0.0096	96
15822	Leek Whole Plant	7-Dec-2004	0.01	0.0091	91
15822	Leek Whole Plant	7-Dec-2004	0.01	0.0089	89

Table 2. (Cont.) Recovery of XDE-175-*N*-formyl-L from Wet Crops

Sample Name	Matrix	Date of Analysis	XDE-175-*N*-formyl-L Added	Found	% Recovery
10505	Broccoli Flower Head and Stem	3-Dec-2004	1.0	1.11	111
10505	Broccoli Flower Head and Stem	3-Dec-2004	1.0	1.04	104
13155	Grape Whole Fruit	3-Dec-2004	1.0	0.91	91
13155	Grape Whole Fruit	3-Dec-2004	1.0	0.92	92
4301	Onion sweet yellow	3-Dec-2004	1.0	0.99	99
4301	Onion sweet yellow	3-Dec-2004	1.0	1.00	100
16401	Peppers	3-Dec-2004	1.0	1.02	102
16401	Peppers	3-Dec-2004	1.0	0.99	99
99301	Cabbage Whole Head	3-Dec-2004	1.0	0.98	98
99301	Cabbage Whole Head	3-Dec-2004	1.0	0.96	96
13210	Tomato Whole Fruit	3-Dec-2004	1.0	1.01	101
13210	Tomato Whole Fruit	3-Dec-2004	1.0	1.02	102
15812	Succulent Green Bean	7-Dec-2004	1.0	0.98	98
15812	Succulent Green Bean	7-Dec-2004	1.0	0.96	96
15822	Leek Whole Plant	7-Dec-2004	1.0	0.97	97
15822	Leek Whole Plant	7-Dec-2004	1.0	0.96	96
24801	Lettuce Whole Leaf	3-Dec-2004	10.0	9.6	96
24801	Lettuce Whole Leaf	3-Dec-2004	10.0	9.5	95
24801	Lettuce Whole Leaf	25-Apr-2005	10.0	9.9	99
24801	Lettuce Whole Leaf	25-Apr-2005	10.0	8.2	82
24801	Lettuce Whole Leaf	25-Apr-2005	10.0	8.1	81
24801	Lettuce Whole Leaf	25-Apr-2005	10.0	8.6	86

[a] The date of analysis represents the date the samples were extracted.
[b] All calculations were performed using Microsoft Excel 2002 with full precision. Recovery values corrected for control value where appropriate.
[c] Not Applicable.
[d] Not Detected.
[e] Samples were fortified at the method's proposed limit of detection (0.003 µg/g). Values are reported with a lower degree of confidence than values above the limit of quantitation.

Table 3. Recovery of XDE-175-J from Dry Crops

Sample Name	Matrix	Date of Analysis[a]	XDE-175-J Added	XDE-175-J Found	% Recovery[b]
943	Barley Forage	2-Dec-2004	NA[c]	ND[d]	NA
923	Barley Grain	2-Dec-2004	NA	ND	NA
19302	Barley Straw	2-Dec-2004	NA	ND	NA
834	Corn, Field Forage	2-Dec-2004	NA	ND	NA
844	Corn, Field Grain	2-Dec-2004	NA	ND	NA
824	Corn, Field Stover	2-Dec-2004	NA	ND	NA
14250	Grass Forage	2-Dec-2004	NA	ND	NA
782	Wheat Forage	2-Dec-2004	NA	ND	NA
762	Wheat Grain	2-Dec-2004	NA	ND	NA
777	Wheat Straw	2-Dec-2004	NA	ND	NA
943	Barley Forage	2-Dec-2004	0.003	0.0027[e]	NA
824	Corn, Field Stover	2-Dec-2004	0.003	0.0024	NA
14250	Grass Forage	2-Dec-2004	0.003	0.0025	NA
762	Wheat Grain	2-Dec-2004	0.003	0.0026	NA
943	Barley Forage	2-Dec-2004	0.010	0.0083	83
943	Barley Forage	2-Dec-2004	0.010	0.0090	90
923	Barley Grain	2-Dec-2004	0.010	0.0085	85
923	Barley Grain	2-Dec-2004	0.010	0.0083	83
19302	Barley Straw	2-Dec-2004	0.01	0.0082	82
19302	Barley Straw	2-Dec-2004	0.01	0.0089	89
834	Corn, Field Forage	2-Dec-2004	0.01	0.0081	81
834	Corn, Field Forage	2-Dec-2004	0.01	0.0089	89
844	Corn, Field Grain	2-Dec-2004	0.01	0.0084	84
844	Corn, Field Grain	2-Dec-2004	0.01	0.0088	88
824	Corn, Field Stover	2-Dec-2004	0.01	0.0089	89
824	Corn, Field Stover	2-Dec-2004	0.01	0.0082	82
14250	Grass Forage	2-Dec-2004	0.01	0.0084	84
14250	Grass Forage	2-Dec-2004	0.01	0.0086	86
782	Wheat Forage	2-Dec-2004	0.01	0.0087	87
782	Wheat Forage	2-Dec-2004	0.01	0.0092	92
762	Wheat Grain	2-Dec-2004	0.01	0.0105	105
762	Wheat Grain	2-Dec-2004	0.01	0.0090	90
777	Wheat Straw	2-Dec-2004	0.01	0.0091	91
777	Wheat Straw	2-Dec-2004	0.01	0.0089	89

Table 3. (Cont.) Recovery of XDE-175-J from Dry Crops

Sample Name	Matrix	Date of Analysis	XDE-175-J Added	XDE-175-J Found	% Recovery
943	Barley Forage	2-Dec-2004	1.0	0.91	91
943	Barley Forage	2-Dec-2004	1.0	0.89	89
923	Barley Grain	2-Dec-2004	1.0	0.89	89
923	Barley Grain	2-Dec-2004	1.0	0.91	91
19302	Barley Straw	2-Dec-2004	1.0	0.85	85
19302	Barley Straw	2-Dec-2004	1.0	0.86	86
834	Corn, Field Forage	2-Dec-2004	1.0	0.82	82
834	Corn, Field Forage	2-Dec-2004	1.0	0.82	82
844	Corn, Field Grain	2-Dec-2004	1.0	0.82	82
844	Corn, Field Grain	2-Dec-2004	1.0	0.83	83
824	Corn, Field Stover	2-Dec-2004	1.0	0.85	85
824	Corn, Field Stover	2-Dec-2004	1.0	0.81	81
14250	Grass Forage	2-Dec-2004	1.0	0.86	86
14250	Grass Forage	2-Dec-2004	1.0	0.96	96
782	Wheat Forage	2-Dec-2004	1.0	0.87	87
782	Wheat Forage	2-Dec-2004	1.0	0.90	90
762	Wheat Grain	2-Dec-2004	1.0	0.98	98
762	Wheat Grain	2-Dec-2004	1.0	0.94	94
777	Wheat Straw	2-Dec-2004	1.0	0.88	88
777	Wheat Straw	2-Dec-2004	1.0	0.88	88

Table 3. (Cont.) Recovery of XDE-175-L from Dry Crops

Sample Name	Matrix	Date of Analysis	XDE-175-L Added	XDE-175-L Found	% Recovery
943	Barley Forage	2-Dec-2004	NA	ND	NA
923	Barley Grain	2-Dec-2004	NA	ND	NA
19302	Barley Straw	2-Dec-2004	NA	ND	NA
834	Corn, Field Forage	2-Dec-2004	NA	ND	NA
844	Corn, Field Grain	2-Dec-2004	NA	ND	NA
824	Corn, Field Stover	2-Dec-2004	NA	ND	NA
14250	Grass Forage	2-Dec-2004	NA	ND	NA
782	Wheat Forage	2-Dec-2004	NA	ND	NA
762	Wheat Grain	2-Dec-2004	NA	ND	NA
777	Wheat Straw	2-Dec-2004	NA	ND	NA
943	Barley Forage	2-Dec-2004	0.003	0.0029	NA
824	Corn, Field Stover	2-Dec-2004	0.003	0.0029	NA
14250	Grass Forage	2-Dec-2004	0.003	0.0031	NA
762	Wheat Grain	2-Dec-2004	0.003	0.0026	NA
943	Barley Forage	2-Dec-2004	0.01	0.0095	95
943	Barley Forage	2-Dec-2004	0.01	0.0108	108
923	Barley Grain	2-Dec-2004	0.01	0.0105	105
923	Barley Grain	2-Dec-2004	0.01	0.0107	107
19302	Barley Straw	2-Dec-2004	0.01	0.0103	103
19302	Barley Straw	2-Dec-2004	0.01	0.0110	110
834	Corn, Field Forage	2-Dec-2004	0.01	0.0106	106
834	Corn, Field Forage	2-Dec-2004	0.01	0.0105	105
844	Corn, Field Grain	2-Dec-2004	0.01	0.0094	94
844	Corn, Field Grain	2-Dec-2004	0.01	0.0106	106
824	Corn, Field Stover	2-Dec-2004	0.01	0.0099	99
824	Corn, Field Stover	2-Dec-2004	0.01	0.0114	114
14250	Grass Forage	2-Dec-2004	0.01	0.0099	99
14250	Grass Forage	2-Dec-2004	0.01	0.0094	94
782	Wheat Forage	2-Dec-2004	0.01	0.0099	99
782	Wheat Forage	2-Dec-2004	0.01	0.0105	105
762	Wheat Grain	2-Dec-2004	0.01	0.0097	97
762	Wheat Grain	2-Dec-2004	0.01	0.0096	96
777	Wheat Straw	2-Dec-2004	0.01	0.0099	99
777	Wheat Straw	2-Dec-2004	0.01	0.0104	104

Table 3. (Cont.) Recovery of XDE-175-L from Dry Crops

Sample Name	Matrix	Date of Analysis	XDE-175-L Added	XDE-175-L Found	% Recovery
943	Barley Forage	2-Dec-2004	1.0	1.00	100
943	Barley Forage	2-Dec-2004	1.0	0.93	93
923	Barley Grain	2-Dec-2004	1.0	1.13	113
923	Barley Grain	2-Dec-2004	1.0	1.14	114
19302	Barley Straw	2-Dec-2004	1.0	0.98	98
19302	Barley Straw	2-Dec-2004	1.0	1.02	102
834	Corn, Field Forage	2-Dec-2004	1.0	1.01	101
834	Corn, Field Forage	2-Dec-2004	1.0	1.01	101
844	Corn, Field Grain	2-Dec-2004	1.0	1.08	108
844	Corn, Field Grain	2-Dec-2004	1.0	1.08	108
824	Corn, Field Stover	2-Dec-2004	1.0	1.04	104
824	Corn, Field Stover	2-Dec-2004	1.0	1.04	104
14250	Grass Forage	2-Dec-2004	1.0	0.95	95
14250	Grass Forage	2-Dec-2004	1.0	0.95	95
782	Wheat Forage	2-Dec-2004	1.0	0.92	92
782	Wheat Forage	2-Dec-2004	1.0	0.95	95
762	Wheat Grain	2-Dec-2004	1.0	1.00	100
762	Wheat Grain	2-Dec-2004	1.0	1.03	103
777	Wheat Straw	2-Dec-2004	1.0	1.01	101
777	Wheat Straw	2-Dec-2004	1.0	1.11	111

Table 3. (Cont.) Recovery of XDE-175-*N*-demethyl-J from Dry Crops

Sample Name	Matrix	Date of Analysis	XDE-175-*N*-demethyl-J Added	Found	% Recovery
943	Barley Forage	2-Dec-2004	NA	ND	NA
923	Barley Grain	2-Dec-2004	NA	ND	NA
19302	Barley Straw	2-Dec-2004	NA	ND	NA
834	Corn, Field Forage	2-Dec-2004	NA	ND	NA
844	Corn, Field Grain	2-Dec-2004	NA	ND	NA
824	Corn, Field Stover	2-Dec-2004	NA	ND	NA
14250	Grass Forage	2-Dec-2004	NA	ND	NA
782	Wheat Forage	2-Dec-2004	NA	ND	NA
762	Wheat Grain	2-Dec-2004	NA	ND	NA
777	Wheat Straw	2-Dec-2004	NA	ND	NA
943	Barley Forage	2-Dec-2004	0.003	0.0031	NA
824	Corn, Field Stover	2-Dec-2004	0.003	0.0029	NA
14250	Grass Forage	2-Dec-2004	0.003	0.0031	NA
762	Wheat Grain	2-Dec-2004	0.003	0.0028	NA
943	Barley Forage	2-Dec-2004	0.01	0.0096	96
943	Barley Forage	2-Dec-2004	0.01	0.0099	99
923	Barley Grain	2-Dec-2004	0.01	0.0092	92
923	Barley Grain	2-Dec-2004	0.01	0.0104	104
19302	Barley Straw	2-Dec-2004	0.01	0.0096	96
19302	Barley Straw	2-Dec-2004	0.01	0.0105	105
834	Corn, Field Forage	2-Dec-2004	0.01	0.0098	98
834	Corn, Field Forage	2-Dec-2004	0.01	0.0104	104
844	Corn, Field Grain	2-Dec-2004	0.01	0.0105	105
844	Corn, Field Grain	2-Dec-2004	0.01	0.0114	114
824	Corn, Field Stover	2-Dec-2004	0.01	0.0105	105
824	Corn, Field Stover	2-Dec-2004	0.01	0.0110	110
14250	Grass Forage	2-Dec-2004	0.01	0.0103	103
14250	Grass Forage	2-Dec-2004	0.01	0.0098	98
782	Wheat Forage	2-Dec-2004	0.01	0.0096	96
782	Wheat Forage	2-Dec-2004	0.01	0.0102	102
762	Wheat Grain	2-Dec-2004	0.01	0.0099	99
762	Wheat Grain	2-Dec-2004	0.01	0.0104	104
777	Wheat Straw	2-Dec-2004	0.01	0.0104	104
777	Wheat Straw	2-Dec-2004	0.01	0.0103	103

Table 3. (Cont.) Recovery of XDE-175-*N*-demethyl-J from Dry Crops

Sample Name	Matrix	Date of Analysis	XDE-175-*N*-demethyl-J Added	Found	% Recovery
943	Barley Forage	2-Dec-2004	1.0	1.06	106
943	Barley Forage	2-Dec-2004	1.0	1.04	104
923	Barley Grain	2-Dec-2004	1.0	1.04	104
923	Barley Grain	2-Dec-2004	1.0	1.02	102
19302	Barley Straw	2-Dec-2004	1.0	0.99	99
19302	Barley Straw	2-Dec-2004	1.0	1.02	102
834	Corn, Field Forage	2-Dec-2004	1.0	0.95	95
834	Corn, Field Forage	2-Dec-2004	1.0	1.00	100
844	Corn, Field Grain	2-Dec-2004	1.0	1.07	107
844	Corn, Field Grain	2-Dec-2004	1.0	1.14	114
824	Corn, Field Stover	2-Dec-2004	1.0	1.02	102
824	Corn, Field Stover	2-Dec-2004	1.0	1.03	103
14250	Grass Forage	2-Dec-2004	1.0	1.01	101
14250	Grass Forage	2-Dec-2004	1.0	1.04	104
782	Wheat Forage	2-Dec-2004	1.0	0.97	97
782	Wheat Forage	2-Dec-2004	1.0	0.99	99
762	Wheat Grain	2-Dec-2004	1.0	1.01	101
762	Wheat Grain	2-Dec-2004	1.0	1.02	102
777	Wheat Straw	2-Dec-2004	1.0	1.01	101
777	Wheat Straw	2-Dec-2004	1.0	1.03	103

Table 3. (Cont.) Recovery of XDE-175-*N*-demethyl-L from Dry Crops

Sample Name	Matrix	Date of Analysis	XDE-175-*N*-demethyl-L Added	Found	% Recovery
943	Barley Forage	2-Dec-2004	NA	0.0001	NA
923	Barley Grain	2-Dec-2004	NA	0.0001	NA
19302	Barley Straw	2-Dec-2004	NA	0.0002	NA
834	Corn, Field Forage	2-Dec-2004	NA	ND	NA
844	Corn, Field Grain	2-Dec-2004	NA	ND	NA
824	Corn, Field Stover	2-Dec-2004	NA	ND	NA
14250	Grass Forage	2-Dec-2004	NA	ND	NA
782	Wheat Forage	2-Dec-2004	NA	0.0000	NA
762	Wheat Grain	2-Dec-2004	NA	0.0001	NA
777	Wheat Straw	2-Dec-2004	NA	ND	NA
943	Barley Forage	2-Dec-2004	0.003	0.0032	NA
824	Corn, Field Stover	2-Dec-2004	0.003	0.0031	NA
14250	Grass Forage	2-Dec-2004	0.003	0.0032	NA
762	Wheat Grain	2-Dec-2004	0.003	0.0032	NA
943	Barley Forage	2-Dec-2004	0.01	0.0102	102
943	Barley Forage	2-Dec-2004	0.01	0.0103	103
923	Barley Grain	2-Dec-2004	0.01	0.0107	107
923	Barley Grain	2-Dec-2004	0.01	0.0100	100
19302	Barley Straw	2-Dec-2004	0.01	0.0102	102
19302	Barley Straw	2-Dec-2004	0.01	0.0110	110
834	Corn, Field Forage	2-Dec-2004	0.01	0.0110	110
834	Corn, Field Forage	2-Dec-2004	0.01	0.0101	101
844	Corn, Field Grain	2-Dec-2004	0.01	0.0100	100
844	Corn, Field Grain	2-Dec-2004	0.01	0.0114	114
824	Corn, Field Stover	2-Dec-2004	0.01	0.0111	111
824	Corn, Field Stover	2-Dec-2004	0.01	0.0102	102
14250	Grass Forage	2-Dec-2004	0.01	0.0100	100
14250	Grass Forage	2-Dec-2004	0.01	0.0102	102
782	Wheat Forage	2-Dec-2004	0.01	0.0099	99
782	Wheat Forage	2-Dec-2004	0.01	0.0102	102
762	Wheat Grain	2-Dec-2004	0.01	0.0107	107
762	Wheat Grain	2-Dec-2004	0.01	0.0109	109
777	Wheat Straw	2-Dec-2004	0.01	0.0111	111
777	Wheat Straw	2-Dec-2004	0.01	0.0105	105

Table 3. (Cont.) Recovery of XDE-175-*N*-demethyl-L from Dry Crops

Sample Name	Matrix	Date of Analysis	XDE-175-*N*-demethyl-L Added	Found	% Recovery
943	Barley Forage	2-Dec-2004	1.0	1.05	105
943	Barley Forage	2-Dec-2004	1.0	1.07	107
923	Barley Grain	2-Dec-2004	1.0	1.09	109
923	Barley Grain	2-Dec-2004	1.0	1.09	109
19302	Barley Straw	2-Dec-2004	1.0	1.03	103
19302	Barley Straw	2-Dec-2004	1.0	1.00	100
834	Corn, Field Forage	2-Dec-2004	1.0	1.01	101
834	Corn, Field Forage	2-Dec-2004	1.0	1.02	102
844	Corn, Field Grain	2-Dec-2004	1.0	1.04	104
844	Corn, Field Grain	2-Dec-2004	1.0	1.10	110
824	Corn, Field Stover	2-Dec-2004	1.0	1.03	103
824	Corn, Field Stover	2-Dec-2004	1.0	1.06	106
14250	Grass Forage	2-Dec-2004	1.0	1.00	100
14250	Grass Forage	2-Dec-2004	1.0	1.06	106
782	Wheat Forage	2-Dec-2004	1.0	0.96	96
782	Wheat Forage	2-Dec-2004	1.0	1.03	103
762	Wheat Grain	2-Dec-2004	1.0	1.02	102
762	Wheat Grain	2-Dec-2004	1.0	1.09	109
777	Wheat Straw	2-Dec-2004	1.0	1.00	100
777	Wheat Straw	2-Dec-2004	1.0	1.08	108

Table 3. (Cont.) Recovery of XDE-175-*N*-formyl-J from Dry Crops

Sample Name	Matrix	Date of Analysis	XDE-175-*N*-formyl-J Added	Found	% Recovery
943	Barley Forage	2-Dec-2004	NA	0.0004	NA
923	Barley Grain	2-Dec-2004	NA	ND	NA
19302	Barley Straw	2-Dec-2004	NA	ND	NA
834	Corn, Field Forage	2-Dec-2004	NA	ND	NA
844	Corn, Field Grain	2-Dec-2004	NA	ND	NA
824	Corn, Field Stover	2-Dec-2004	NA	ND	NA
14250	Grass Forage	2-Dec-2004	NA	ND	NA
782	Wheat Forage	2-Dec-2004	NA	ND	NA
762	Wheat Grain	2-Dec-2004	NA	ND	NA
777	Wheat Straw	2-Dec-2004	NA	ND	NA
943	Barley Forage	2-Dec-2004	0.003	0.0025	NA
824	Corn, Field Stover	2-Dec-2004	0.003	0.0035	NA
14250	Grass Forage	2-Dec-2004	0.003	0.0030	NA
762	Wheat Grain	2-Dec-2004	0.003	0.0035	NA
943	Barley Forage	2-Dec-2004	0.01	0.0100	100
943	Barley Forage	2-Dec-2004	0.01	0.0099	99
923	Barley Grain	2-Dec-2004	0.01	0.0107	107
923	Barley Grain	2-Dec-2004	0.01	0.0113	113
19302	Barley Straw	2-Dec-2004	0.01	0.0108	108
19302	Barley Straw	2-Dec-2004	0.01	0.0101	101
834	Corn, Field Forage	2-Dec-2004	0.01	0.0106	106
834	Corn, Field Forage	2-Dec-2004	0.01	0.0094	94
844	Corn, Field Grain	2-Dec-2004	0.01	0.0077	77
844	Corn, Field Grain	2-Dec-2004	0.01	0.0108	108
824	Corn, Field Stover	2-Dec-2004	0.01	0.0118	118
824	Corn, Field Stover	2-Dec-2004	0.01	0.0118	118
14250	Grass Forage	2-Dec-2004	0.01	0.0107	107
14250	Grass Forage	2-Dec-2004	0.01	0.0088	88
782	Wheat Forage	2-Dec-2004	0.01	0.0088	88
782	Wheat Forage	2-Dec-2004	0.01	0.0110	110
762	Wheat Grain	2-Dec-2004	0.01	0.0122	122
762	Wheat Grain	2-Dec-2004	0.01	0.0131	131
777	Wheat Straw	2-Dec-2004	0.01	0.0117	117
777	Wheat Straw	2-Dec-2004	0.01	0.0093	93

Table 3. (Cont.) Recovery of XDE-175-*N*-formyl-J from Dry Crops

Sample Name	Matrix	Date of Analysis	XDE-175-*N*-formyl-J Added	Found	% Recovery
943	Barley Forage	2-Dec-2004	1.0	1.13	113
943	Barley Forage	2-Dec-2004	1.0	1.10	110
923	Barley Grain	2-Dec-2004	1.0	1.16	116
923	Barley Grain	2-Dec-2004	1.0	1.15	115
19302	Barley Straw	2-Dec-2004	1.0	1.03	103
19302	Barley Straw	2-Dec-2004	1.0	1.04	104
834	Corn, Field Forage	2-Dec-2004	1.0	1.11	111
834	Corn, Field Forage	2-Dec-2004	1.0	1.07	107
844	Corn, Field Grain	2-Dec-2004	1.0	1.09	109
844	Corn, Field Grain	2-Dec-2004	1.0	1.13	113
824	Corn, Field Stover	2-Dec-2004	1.0	1.13	113
824	Corn, Field Stover	2-Dec-2004	1.0	1.10	110
14250	Grass Forage	2-Dec-2004	1.0	1.07	107
14250	Grass Forage	2-Dec-2004	1.0	1.10	110
782	Wheat Forage	2-Dec-2004	1.0	0.97	97
782	Wheat Forage	2-Dec-2004	1.0	1.06	106
762	Wheat Grain	2-Dec-2004	1.0	1.21	121
762	Wheat Grain	2-Dec-2004	1.0	1.21	121
777	Wheat Straw	2-Dec-2004	1.0	1.10	110
777	Wheat Straw	2-Dec-2004	1.0	1.11	111

Table 3. (Cont.) Recovery of XDE-175-*N*-formyl-L from Dry Crops

Sample Name	Matrix	Date of Analysis	XDE-175-*N*-formyl-L Added	Found	% Recovery
943	Barley Forage	2-Dec-2004	NA	ND	NA
923	Barley Grain	2-Dec-2004	NA	ND	NA
19302	Barley Straw	2-Dec-2004	NA	ND	NA
834	Corn, Field Forage	2-Dec-2004	NA	ND	NA
844	Corn, Field Grain	2-Dec-2004	NA	ND	NA
824	Corn, Field Stover	2-Dec-2004	NA	ND	NA
14250	Grass Forage	2-Dec-2004	NA	ND	NA
782	Wheat Forage	2-Dec-2004	NA	ND	NA
762	Wheat Grain	2-Dec-2004	NA	ND	NA
777	Wheat Straw	2-Dec-2004	NA	ND	NA
943	Barley Forage	2-Dec-2004	0.003	0.0030	NA
824	Corn, Field Stover	2-Dec-2004	0.003	0.0041	NA
14250	Grass Forage	2-Dec-2004	0.003	0.0029	NA
762	Wheat Grain	2-Dec-2004	0.003	0.0034	NA
943	Barley Forage	2-Dec-2004	0.01	0.0087	87
943	Barley Forage	2-Dec-2004	0.01	0.0115	115
923	Barley Grain	2-Dec-2004	0.01	0.0088	88
923	Barley Grain	2-Dec-2004	0.01	0.0079	79
19302	Barley Straw	2-Dec-2004	0.01	0.0108	108
19302	Barley Straw	2-Dec-2004	0.01	0.0102	102
834	Corn, Field Forage	2-Dec-2004	0.01	0.0082	82
834	Corn, Field Forage	2-Dec-2004	0.01	0.0090	90
844	Corn, Field Grain	2-Dec-2004	0.01	0.0090	90
844	Corn, Field Grain	2-Dec-2004	0.01	0.0088	88
824	Corn, Field Stover	2-Dec-2004	0.01	0.0092	92
824	Corn, Field Stover	2-Dec-2004	0.01	0.0104	104
14250	Grass Forage	2-Dec-2004	0.01	0.0095	95
14250	Grass Forage	2-Dec-2004	0.01	0.0098	98
782	Wheat Forage	2-Dec-2004	0.01	0.0095	95
782	Wheat Forage	2-Dec-2004	0.01	0.0119	119
762	Wheat Grain	2-Dec-2004	0.01	0.0095	95
762	Wheat Grain	2-Dec-2004	0.01	0.0078	78
777	Wheat Straw	2-Dec-2004	0.01	0.0107	107
777	Wheat Straw	2-Dec-2004	0.01	0.0084	84

Table 3. (Cont.) Recovery of XDE-175-*N*-formyl-L from Dry Crops

Sample Name	Matrix	Date of Analysis	XDE-175-*N*-formyl-L Added	Found	% Recovery
943	Barley Forage	2-Dec-2004	1.0	1.00	100
943	Barley Forage	2-Dec-2004	1.0	1.03	103
923	Barley Grain	2-Dec-2004	1.0	0.80	80
923	Barley Grain	2-Dec-2004	1.0	0.90	90
19302	Barley Straw	2-Dec-2004	1.0	0.90	90
19302	Barley Straw	2-Dec-2004	1.0	0.94	94
834	Corn, Field Forage	2-Dec-2004	1.0	0.89	89
834	Corn, Field Forage	2-Dec-2004	1.0	0.85	85
844	Corn, Field Grain	2-Dec-2004	1.0	0.78	78
844	Corn, Field Grain	2-Dec-2004	1.0	0.83	83
824	Corn, Field Stover	2-Dec-2004	1.0	0.91	91
824	Corn, Field Stover	2-Dec-2004	1.0	0.90	90
14250	Grass Forage	2-Dec-2004	1.0	0.98	98
14250	Grass Forage	2-Dec-2004	1.0	0.97	97
782	Wheat Forage	2-Dec-2004	1.0	1.09	109
782	Wheat Forage	2-Dec-2004	1.0	1.12	112
762	Wheat Grain	2-Dec-2004	1.0	0.77	77
762	Wheat Grain	2-Dec-2004	1.0	0.78	78
777	Wheat Straw	2-Dec-2004	1.0	0.99	99
777	Wheat Straw	2-Dec-2004	1.0	1.17	117

[a] The date of analysis represents the date the samples were extracted.
[b] All calculations were performed using Microsoft Excel 2002 with full precision. Recovery values corrected for control value where appropriate.
[c] Not Applicable.
[d] Not Detected.
[e] Samples were fortified at the method's proposed limit of detection (0.003 µg/g). Values are reported with a lower degree of confidence than values above the limit of quantitation.

Table 4. Recovery of XDE-175-J from Acidic Crops

Sample Name	Matrix	Date of Analysis[a]	XDE-175-J Added	XDE-175-J Found	% Recovery[b]
1345	Orange Whole Fruit	6-Dec-2004	NA[c]	ND[d]	NA
1355	Orange Peel	6-Dec-2004	NA	ND	NA
1363	Orange Pulp	6-Dec-2004	NA	ND	NA
1373	Apple Whole Fruit	7-Dec-2004	NA	ND	NA
20024	Lemon Whole Fruit	7-Dec-2004	NA	ND	NA
15832	Peach Fruit Without Seed	7-Dec-2004	NA	ND	NA
15852	Plum Fruit Without Seed	7-Dec-2004	NA	ND	NA
15842	Cherry Without Seed	7-Dec-2004	NA	0.0003	NA
15862	Pear Whole Fruit	7-Dec-2004	NA	ND	NA
1345	Orange Whole Fruit	6-Dec-2004	0.003	0.0031[e]	NA
15832	Peach Fruit Without Seed	7-Dec-2004	0.003	0.0028	NA
15842	Cherry Without Seed	7-Dec-2004	0.003	0.0031	NA
1345	Orange Whole Fruit	6-Dec-2004	0.01	0.0099	99
1345	Orange Whole Fruit	6-Dec-2004	0.01	0.0103	103
1355	Orange Peel	6-Dec-2004	0.01	0.0093	93
1355	Orange Peel	6-Dec-2004	0.01	0.0098	98
1363	Orange Pulp	6-Dec-2004	0.01	0.0098	98
1363	Orange Pulp	6-Dec-2004	0.01	0.0096	96
1373	Apple Whole Fruit	7-Dec-2004	0.01	0.0094	94
1373	Apple Whole Fruit	7-Dec-2004	0.01	0.0099	99
20024	Lemon Whole Fruit	7-Dec-2004	0.01	0.0109	109
20024	Lemon Whole Fruit	7-Dec-2004	0.01	0.0115	115
15832	Peach Fruit Without Seed	7-Dec-2004	0.01	0.0101	101
15832	Peach Fruit Without Seed	7-Dec-2004	0.01	0.0106	106
15852	Plum Fruit Without Seed	7-Dec-2004	0.01	0.0110	110
15852	Plum Fruit Without Seed	7-Dec-2004	0.01	0.0105	105
15842	Cherry Without Seed	7-Dec-2004	0.01	0.0106	106
15842	Cherry Without Seed	7-Dec-2004	0.01	0.0107	107
15862	Pear Whole Fruit	7-Dec-2004	0.01	0.0101	101

Table 4. (Cont.) Recovery of XDE-175-J from Acidic Crops

Sample Name	Matrix	Date of Analysis	XDE-175-J Added	XDE-175-J Found	% Recovery
1345	Orange Whole Fruit	6-Dec-2004	1.0	0.95	95
1345	Orange Whole Fruit	6-Dec-2004	1.0	1.01	101
1355	Orange Peel	6-Dec-2004	1.0	0.97	97
1355	Orange Peel	6-Dec-2004	1.0	0.94	94
1363	Orange Pulp	6-Dec-2004	1.0	0.98	98
1363	Orange Pulp	6-Dec-2004	1.0	1.04	104
1373	Apple Whole Fruit	7-Dec-2004	1.0	1.03	103
1373	Apple Whole Fruit	7-Dec-2004	1.0	1.04	104
20024	Lemon Whole Fruit	7-Dec-2004	1.0	1.02	102
20024	Lemon Whole Fruit	7-Dec-2004	1.0	0.98	98
15832	Peach Fruit Without Seed	7-Dec-2004	1.0	1.01	101
15832	Peach Fruit Without Seed	7-Dec-2004	1.0	1.00	100
15852	Plum Fruit Without Seed	7-Dec-2004	1.0	1.05	105
15852	Plum Fruit Without Seed	7-Dec-2004	1.0	1.05	105
15842	Cherry Without Seed	7-Dec-2004	1.0	1.05	105
15842	Cherry Without Seed	7-Dec-2004	1.0	1.02	102
15862	Pear Whole Fruit	7-Dec-2004	1.0	1.02	102
15862	Pear Whole Fruit	7-Dec-2004	1.0	1.00	100

Table 4. (Cont.) Recovery of XDE-175-L from Acidic Crops

Sample Name	Matrix	Date of Analysis	XDE-175-L Added	XDE-175-L Found	% Recovery
1345	Orange Whole Fruit	6-Dec-2004	NA	ND	NA
1355	Orange Peel	6-Dec-2004	NA	ND	NA
1363	Orange Pulp	6-Dec-2004	NA	ND	NA
1373	Apple Whole Fruit	7-Dec-2004	NA	ND	NA
20024	Lemon Whole Fruit	7-Dec-2004	NA	ND	NA
15832	Peach Fruit Without Seed	7-Dec-2004	NA	ND	NA
15852	Plum Fruit Without Seed	7-Dec-2004	NA	ND	NA
15842	Cherry Without Seed	7-Dec-2004	NA	ND	NA
15862	Pear Whole Fruit	7-Dec-2004	NA	ND	NA
1345	Orange Whole Fruit	6-Dec-2004	0.003	0.0032	NA
15832	Peach Fruit Without Seed	7-Dec-2004	0.003	0.0031	NA
15842	Cherry Without Seed	7-Dec-2004	0.003	0.0033	NA
1345	Orange Whole Fruit	6-Dec-2004	0.01	0.0106	106
1345	Orange Whole Fruit	6-Dec-2004	0.01	0.0105	105
1355	Orange Peel	6-Dec-2004	0.01	0.0101	101
1355	Orange Peel	6-Dec-2004	0.01	0.0104	104
1363	Orange Pulp	6-Dec-2004	0.01	0.0100	100
1363	Orange Pulp	6-Dec-2004	0.01	0.0105	105
1373	Apple Whole Fruit	7-Dec-2004	0.01	0.0101	101
1373	Apple Whole Fruit	7-Dec-2004	0.01	0.0098	98
20024	Lemon Whole Fruit	7-Dec-2004	0.01	0.0111	111
20024	Lemon Whole Fruit	7-Dec-2004	0.01	0.0114	114
15832	Peach Fruit Without Seed	7-Dec-2004	0.01	0.0107	107
15832	Peach Fruit Without Seed	7-Dec-2004	0.01	0.0112	112
15852	Plum Fruit Without Seed	7-Dec-2004	0.01	0.0113	113
15852	Plum Fruit Without Seed	7-Dec-2004	0.01	0.0107	107
15842	Cherry Without Seed	7-Dec-2004	0.01	0.0111	111
15842	Cherry Without Seed	7-Dec-2004	0.01	0.0109	109
15862	Pear Whole Fruit	7-Dec-2004	0.01	0.0101	101

Table 4. (Cont.) Recovery of XDE-175-L from Acidic Crops

Sample Name	Matrix	Date of Analysis	XDE-175-L Added	XDE-175-L Found	% Recovery
1345	Orange Whole Fruit	6-Dec-2004	1.0	0.98	98
1345	Orange Whole Fruit	6-Dec-2004	1.0	1.02	102
1355	Orange Peel	6-Dec-2004	1.0	1.02	102
1355	Orange Peel	6-Dec-2004	1.0	0.99	99
1363	Orange Pulp	6-Dec-2004	1.0	1.00	100
1363	Orange Pulp	6-Dec-2004	1.0	1.03	103
1373	Apple Whole Fruit	7-Dec-2004	1.0	1.00	100
1373	Apple Whole Fruit	7-Dec-2004	1.0	1.04	104
20024	Lemon Whole Fruit	7-Dec-2004	1.0	0.99	99
20024	Lemon Whole Fruit	7-Dec-2004	1.0	0.96	96
15832	Peach Fruit Without Seed	7-Dec-2004	1.0	0.97	97
15832	Peach Fruit Without Seed	7-Dec-2004	1.0	0.97	97
15852	Plum Fruit Without Seed	7-Dec-2004	1.0	1.01	101
15852	Plum Fruit Without Seed	7-Dec-2004	1.0	0.97	97
15842	Cherry Without Seed	7-Dec-2004	1.0	1.00	100
15842	Cherry Without Seed	7-Dec-2004	1.0	1.03	103
15862	Pear Whole Fruit	7-Dec-2004	1.0	1.03	103
15862	Pear Whole Fruit	7-Dec-2004	1.0	0.92	92

Table 4. (Cont.) Recovery of XDE-175-*N*-demethyl-J from Acidic Crops

Sample Name	Matrix	Date of Analysis	XDE-175-*N*-demethyl-J Added	Found	% Recovery
1345	Orange Whole Fruit	6-Dec-2004	NA	0.0000	NA
1355	Orange Peel	6-Dec-2004	NA	ND	NA
1363	Orange Pulp	6-Dec-2004	NA	ND	NA
1373	Apple Whole Fruit	7-Dec-2004	NA	ND	NA
20024	Lemon Whole Fruit	7-Dec-2004	NA	0.0001	NA
15832	Peach Fruit Without Seed	7-Dec-2004	NA	0.0002	NA
15852	Plum Fruit Without Seed	7-Dec-2004	NA	0.0003	NA
15842	Cherry Without Seed	7-Dec-2004	NA	0.0004	NA
15862	Pear Whole Fruit	7-Dec-2004	NA	ND	NA
1345	Orange Whole Fruit	6-Dec-2004	0.003	0.0029	NA
15832	Peach Fruit Without Seed	7-Dec-2004	0.003	0.0027	NA
15842	Cherry Without Seed	7-Dec-2004	0.003	0.0030	NA
1345	Orange Whole Fruit	6-Dec-2004	0.01	0.0104	104
1345	Orange Whole Fruit	6-Dec-2004	0.01	0.0102	102
1355	Orange Peel	6-Dec-2004	0.01	0.0098	98
1355	Orange Peel	6-Dec-2004	0.01	0.0097	97
1363	Orange Pulp	6-Dec-2004	0.01	0.0097	97
1363	Orange Pulp	6-Dec-2004	0.01	0.0096	96
1373	Apple Whole Fruit	7-Dec-2004	0.01	0.0096	96
1373	Apple Whole Fruit	7-Dec-2004	0.01	0.0102	102
20024	Lemon Whole Fruit	7-Dec-2004	0.01	0.0110	110
20024	Lemon Whole Fruit	7-Dec-2004	0.01	0.0108	108
15832	Peach Fruit Without Seed	7-Dec-2004	0.01	0.0102	102
15832	Peach Fruit Without Seed	7-Dec-2004	0.01	0.0113	113
15852	Plum Fruit Without Seed	7-Dec-2004	0.01	0.0114	114
15852	Plum Fruit Without Seed	7-Dec-2004	0.01	0.0108	108
15842	Cherry Without Seed	7-Dec-2004	0.01	0.0107	107
15842	Cherry Without Seed	7-Dec-2004	0.01	0.0106	106
15862	Pear Whole Fruit	7-Dec-2004	0.01	0.0103	103

Table 4. (Cont.) Recovery of XDE-175-*N*-demethyl-J from Acidic Crops

Sample Name	Matrix	Date of Analysis	XDE-175-*N*-demethyl-J Added	Found	% Recovery
1345	Orange Whole Fruit	6-Dec-2004	1.0	1.01	101
1345	Orange Whole Fruit	6-Dec-2004	1.0	1.04	104
1355	Orange Peel	6-Dec-2004	1.0	0.99	99
1355	Orange Peel	6-Dec-2004	1.0	1.03	103
1363	Orange Pulp	6-Dec-2004	1.0	1.03	103
1363	Orange Pulp	6-Dec-2004	1.0	1.03	103
1373	Apple Whole Fruit	7-Dec-2004	1.0	1.00	100
1373	Apple Whole Fruit	7-Dec-2004	1.0	1.07	107
20024	Lemon Whole Fruit	7-Dec-2004	1.0	0.99	99
20024	Lemon Whole Fruit	7-Dec-2004	1.0	0.97	97
15832	Peach Fruit Without Seed	7-Dec-2004	1.0	1.06	106
15832	Peach Fruit Without Seed	7-Dec-2004	1.0	1.01	101
15852	Plum Fruit Without Seed	7-Dec-2004	1.0	1.07	107
15852	Plum Fruit Without Seed	7-Dec-2004	1.0	0.97	97
15842	Cherry Without Seed	7-Dec-2004	1.0	1.00	100
15842	Cherry Without Seed	7-Dec-2004	1.0	1.01	101
15862	Pear Whole Fruit	7-Dec-2004	1.0	1.05	105
15862	Pear Whole Fruit	7-Dec-2004	1.0	1.01	101

Table 4. (Cont.) Recovery of XDE-175-*N*-demethyl-L from Acidic Crops

Sample Name	Matrix	Date of Analysis	XDE-175-*N*-demethyl-L Added	Found	% Recovery
1345	Orange Whole Fruit	6-Dec-2004	NA	ND	NA
1355	Orange Peel	6-Dec-2004	NA	0.0004	NA
1363	Orange Pulp	6-Dec-2004	NA	0.0004	NA
1373	Apple Whole Fruit	7-Dec-2004	NA	0.0002	NA
20024	Lemon Whole Fruit	7-Dec-2004	NA	0.0002	NA
15832	Peach Fruit Without Seed	7-Dec-2004	NA	0.0003	NA
15852	Plum Fruit Without Seed	7-Dec-2004	NA	0.0003	NA
15842	Cherry Without Seed	7-Dec-2004	NA	0.0003	NA
15862	Pear Whole Fruit	7-Dec-2004	NA	ND	NA
1345	Orange Whole Fruit	6-Dec-2004	0.003	0.0031	NA
15832	Peach Fruit Without Seed	7-Dec-2004	0.003	0.0027	NA
15842	Cherry Without Seed	7-Dec-2004	0.003	0.0030	NA
1345	Orange Whole Fruit	6-Dec-2004	0.01	0.0108	108
1345	Orange Whole Fruit	6-Dec-2004	0.01	0.0105	105
1355	Orange Peel	6-Dec-2004	0.01	0.0092	92
1355	Orange Peel	6-Dec-2004	0.01	0.0095	95
1363	Orange Pulp	6-Dec-2004	0.01	0.0090	90
1363	Orange Pulp	6-Dec-2004	0.01	0.0094	94
1373	Apple Whole Fruit	7-Dec-2004	0.01	0.0087	87
1373	Apple Whole Fruit	7-Dec-2004	0.01	0.0097	97
20024	Lemon Whole Fruit	7-Dec-2004	0.01	0.0111	111
20024	Lemon Whole Fruit	7-Dec-2004	0.01	0.0110	110
15832	Peach Fruit Without Seed	7-Dec-2004	0.01	0.0096	96
15832	Peach Fruit Without Seed	7-Dec-2004	0.01	0.0100	100
15852	Plum Fruit Without Seed	7-Dec-2004	0.01	0.0113	113
15852	Plum Fruit Without Seed	7-Dec-2004	0.01	0.0102	102
15842	Cherry Without Seed	7-Dec-2004	0.01	0.0094	94
15842	Cherry Without Seed	7-Dec-2004	0.01	0.0106	106
15862	Pear Whole Fruit	7-Dec-2004	0.01	0.0102	102

Table 4. (Cont.) Recovery of XDE-175-*N*-demethyl-L from Acidic Crops

Sample Name	Matrix	Date of Analysis	XDE-175-*N*-demethyl-L Added	Found	% Recovery
1345	Orange Whole Fruit	6-Dec-2004	1.0	1.03	103
1345	Orange Whole Fruit	6-Dec-2004	1.0	1.01	101
1355	Orange Peel	6-Dec-2004	1.0	0.96	96
1355	Orange Peel	6-Dec-2004	1.0	1.00	100
1363	Orange Pulp	6-Dec-2004	1.0	1.00	100
1363	Orange Pulp	6-Dec-2004	1.0	1.02	102
1373	Apple Whole Fruit	7-Dec-2004	1.0	0.98	98
1373	Apple Whole Fruit	7-Dec-2004	1.0	1.06	106
20024	Lemon Whole Fruit	7-Dec-2004	1.0	1.07	107
20024	Lemon Whole Fruit	7-Dec-2004	1.0	1.01	101
15832	Peach Fruit Without Seed	7-Dec-2004	1.0	1.02	102
15832	Peach Fruit Without Seed	7-Dec-2004	1.0	0.98	98
15852	Plum Fruit Without Seed	7-Dec-2004	1.0	1.03	103
15852	Plum Fruit Without Seed	7-Dec-2004	1.0	1.00	100
15842	Cherry Without Seed	7-Dec-2004	1.0	0.98	98
15842	Cherry Without Seed	7-Dec-2004	1.0	1.02	102
15862	Pear Whole Fruit	7-Dec-2004	1.0	1.02	102
15862	Pear Whole Fruit	7-Dec-2004	1.0	0.96	96

Table 4. (Cont.) Recovery of XDE-175-*N*-formyl-J from Acidic Crops

Sample Name	Matrix	Date of Analysis	XDE-175-*N*-formyl-J Added	Found	% Recovery
1345	Orange Whole Fruit	6-Dec-2004	NA	ND	NA
1355	Orange Peel	6-Dec-2004	NA	ND	NA
1363	Orange Pulp	6-Dec-2004	NA	ND	NA
1373	Apple Whole Fruit	7-Dec-2004	NA	ND	NA
20024	Lemon Whole Fruit	7-Dec-2004	NA	ND	NA
15832	Peach Fruit Without Seed	7-Dec-2004	NA	ND	NA
15852	Plum Fruit Without Seed	7-Dec-2004	NA	ND	NA
15842	Cherry Without Seed	7-Dec-2004	NA	ND	NA
15862	Pear Whole Fruit	7-Dec-2004	NA	ND	NA
1345	Orange Whole Fruit	6-Dec-2004	0.003	0.0035	NA
15832	Peach Fruit Without Seed	7-Dec-2004	0.003	0.0025	NA
15842	Cherry Without Seed	7-Dec-2004	0.003	0.0023	NA
1345	Orange Whole Fruit	6-Dec-2004	0.01	0.0094	94
1345	Orange Whole Fruit	6-Dec-2004	0.01	0.0082	82
1355	Orange Peel	6-Dec-2004	0.01	0.0109	109
1355	Orange Peel	6-Dec-2004	0.01	0.0097	97
1363	Orange Pulp	6-Dec-2004	0.01	0.0093	93
1363	Orange Pulp	6-Dec-2004	0.01	0.0101	101
1373	Apple Whole Fruit	7-Dec-2004	0.01	0.0094	94
1373	Apple Whole Fruit	7-Dec-2004	0.01	0.0091	91
20024	Lemon Whole Fruit	7-Dec-2004	0.01	0.0118	118
20024	Lemon Whole Fruit	7-Dec-2004	0.01	0.0110	110
15832	Peach Fruit Without Seed	7-Dec-2004	0.01	0.0081	81
15832	Peach Fruit Without Seed	7-Dec-2004	0.01	0.0117	117
15852	Plum Fruit Without Seed	7-Dec-2004	0.01	0.0095	95
15852	Plum Fruit Without Seed	7-Dec-2004	0.01	0.0076	76
15842	Cherry Without Seed	7-Dec-2004	0.01	0.0092	92
15842	Cherry Without Seed	7-Dec-2004	0.01	0.0092	92
15862	Pear Whole Fruit	7-Dec-2004	0.01	0.0106	106

Table 4. (Cont.) Recovery of XDE-175-*N*-formyl-J from Acidic Crops

Sample Name	Matrix	Date of Analysis	XDE-175-*N*-formyl-J Added	XDE-175-*N*-formyl-J Found	% Recovery
1345	Orange Whole Fruit	6-Dec-2004	1.0	0.99	99
1345	Orange Whole Fruit	6-Dec-2004	1.0	1.02	102
1355	Orange Peel	6-Dec-2004	1.0	1.09	109
1355	Orange Peel	6-Dec-2004	1.0	0.99	99
1363	Orange Pulp	6-Dec-2004	1.0	1.02	102
1363	Orange Pulp	6-Dec-2004	1.0	1.01	101
1373	Apple Whole Fruit	7-Dec-2004	1.0	1.02	102
1373	Apple Whole Fruit	7-Dec-2004	1.0	1.06	106
20024	Lemon Whole Fruit	7-Dec-2004	1.0	1.15	115
20024	Lemon Whole Fruit	7-Dec-2004	1.0	1.18	118
15832	Peach Fruit Without Seed	7-Dec-2004	1.0	1.06	106
15832	Peach Fruit Without Seed	7-Dec-2004	1.0	1.05	105
15852	Plum Fruit Without Seed	7-Dec-2004	1.0	1.04	104
15852	Plum Fruit Without Seed	7-Dec-2004	1.0	0.94	94
15842	Cherry Without Seed	7-Dec-2004	1.0	0.95	95
15842	Cherry Without Seed	7-Dec-2004	1.0	0.98	98
15862	Pear Whole Fruit	7-Dec-2004	1.0	1.02	102
15862	Pear Whole Fruit	7-Dec-2004	1.0	1.00	100

Table 4. (Cont.) Recovery of XDE-175-*N*-formyl-L from Acidic Crops

Sample Name	Matrix	Date of Analysis	XDE-175-*N*-formyl-L Added	Found	% Recovery
1345	Orange Whole Fruit	6-Dec-2004	NA	ND	NA
1355	Orange Peel	6-Dec-2004	NA	ND	NA
1363	Orange Pulp	6-Dec-2004	NA	ND	NA
1373	Apple Whole Fruit	7-Dec-2004	NA	ND	NA
20024	Lemon Whole Fruit	7-Dec-2004	NA	ND	NA
15832	Peach Fruit Without Seed	7-Dec-2004	NA	ND	NA
15852	Plum Fruit Without Seed	7-Dec-2004	NA	ND	NA
15842	Cherry Without Seed	7-Dec-2004	NA	ND	NA
15862	Pear Whole Fruit	7-Dec-2004	NA	ND	NA
1345	Orange Whole Fruit	6-Dec-2004	0.003	0.0027	NA
15832	Peach Fruit Without Seed	7-Dec-2004	0.003	0.0021	NA
15842	Cherry Without Seed	7-Dec-2004	0.003	0.0022	NA
1345	Orange Whole Fruit	6-Dec-2004	0.01	0.0091	91
1345	Orange Whole Fruit	6-Dec-2004	0.01	0.0086	86
1355	Orange Peel	6-Dec-2004	0.01	0.0107	107
1355	Orange Peel	6-Dec-2004	0.01	0.0087	87
1363	Orange Pulp	6-Dec-2004	0.01	0.0088	88
1363	Orange Pulp	6-Dec-2004	0.01	0.0086	86
1373	Apple Whole Fruit	7-Dec-2004	0.01	0.0090	90
1373	Apple Whole Fruit	7-Dec-2004	0.01	0.0094	94
20024	Lemon Whole Fruit	7-Dec-2004	0.01	0.0096	96
20024	Lemon Whole Fruit	7-Dec-2004	0.01	0.0102	102
15832	Peach Fruit Without Seed	7-Dec-2004	0.01	0.0088	88
15832	Peach Fruit Without Seed	7-Dec-2004	0.01	0.0102	102
15852	Plum Fruit Without Seed	7-Dec-2004	0.01	0.0103	103
15852	Plum Fruit Without Seed	7-Dec-2004	0.01	0.0087	87
15842	Cherry Without Seed	7-Dec-2004	0.01	0.0098	98
15842	Cherry Without Seed	7-Dec-2004	0.01	0.0097	97
15862	Pear Whole Fruit	7-Dec-2004	0.01	0.0089	89

Table 4. (Cont.) Recovery of XDE-175-*N*-formyl-L from Acidic Crops

Sample Name	Matrix	Date of Analysis	XDE-175-*N*-formyl-L Added	Found	% Recovery
1345	Orange Whole Fruit	6-Dec-2004	1.0	0.97	97
1345	Orange Whole Fruit	6-Dec-2004	1.0	0.98	98
1355	Orange Peel	6-Dec-2004	1.0	1.07	107
1355	Orange Peel	6-Dec-2004	1.0	0.94	94
1363	Orange Pulp	6-Dec-2004	1.0	0.94	94
1363	Orange Pulp	6-Dec-2004	1.0	1.00	100
1373	Apple Whole Fruit	7-Dec-2004	1.0	0.91	91
1373	Apple Whole Fruit	7-Dec-2004	1.0	0.97	97
20024	Lemon Whole Fruit	7-Dec-2004	1.0	1.02	102
20024	Lemon Whole Fruit	7-Dec-2004	1.0	0.94	94
15832	Peach Fruit Without Seed	7-Dec-2004	1.0	0.97	97
15832	Peach Fruit Without Seed	7-Dec-2004	1.0	0.93	93
15852	Plum Fruit Without Seed	7-Dec-2004	1.0	1.00	100
15852	Plum Fruit Without Seed	7-Dec-2004	1.0	0.92	92
15842	Cherry Without Seed	7-Dec-2004	1.0	0.92	92
15842	Cherry Without Seed	7-Dec-2004	1.0	0.94	94
15862	Pear Whole Fruit	7-Dec-2004	1.0	1.00	100
15862	Pear Whole Fruit	7-Dec-2004	1.0	0.95	95

[a] The date of analysis represents the date the samples were extracted.
[b] All calculations were performed using Microsoft Excel 2002 with full precision. Recovery values corrected for control value where appropriate.
[c] Not Applicable.
[d] Not Detected.
[e] Samples were fortified at the method's proposed limit of detection (0.003 µg/g). Values are reported with a lower degree of confidence than values above the limit of quantitation.

Table 5. Recovery of XDE-175-J from Oily Crops

Sample Name	Matrix	Date of Analysis[a]	XDE-175-J Added	XDE-175-J Found	% Recovery[b]
13395	Canola Seed	6-Dec-2004	NA[c]	0.0004	NA
14794	Olive Fruit Without Seed	6-Dec-2004	NA	0.0004	NA
14188	Olive Oil	6-Dec-2004	NA	ND[d]	NA
13377	Soybean Grain	6-Dec-2004	NA	ND	NA
46001	Cotton Seed	6-Dec-2004	NA	0.0004	NA
13395	Canola Seed	6-Dec-2004	0.003	0.0026[e]	NA
14794	Olive Fruit Without Seed	6-Dec-2004	0.003	0.0023	NA
14188	Olive Oil	6-Dec-2004	0.003	0.0030	NA
13395	Canola Seed	6-Dec-2004	0.01	0.0089	89
13395	Canola Seed	6-Dec-2004	0.01	0.0087	87
14794	Olive Fruit Without Seed	6-Dec-2004	0.01	0.0084	84
14794	Olive Fruit Without Seed	6-Dec-2004	0.01	0.0098	98
14188	Olive Oil	6-Dec-2004	0.01	0.0098	98
14188	Olive Oil	6-Dec-2004	0.01	0.0099	99
13377	Soybean Grain	6-Dec-2004	0.01	0.0098	98
13377	Soybean Grain	6-Dec-2004	0.01	0.0105	105
46001	Cotton Seed	6-Dec-2004	0.01	0.0086	86
46001	Cotton Seed	6-Dec-2004	0.01	0.0093	93
13395	Canola Seed	6-Dec-2004	1.0	0.93	93
13395	Canola Seed	6-Dec-2004	1.0	0.89	89
14794	Olive Fruit Without Seed	6-Dec-2004	1.0	0.94	94
14794	Olive Fruit Without Seed	6-Dec-2004	1.0	0.99	99
14188	Olive Oil	6-Dec-2004	1.0	1.00	100
14188	Olive Oil	6-Dec-2004	1.0	1.02	102
13377	Soybean Grain	6-Dec-2004	1.0	1.01	101
13377	Soybean Grain	6-Dec-2004	1.0	1.00	100
46001	Cotton Seed	6-Dec-2004	1.0	0.93	93
46001	Cotton Seed	6-Dec-2004	1.0	0.92	92

Table 5. (Cont.) Recovery of XDE-175-L from Oily Crops

Sample Name	Matrix	Date of Analysis	XDE-175-L Added	XDE-175-L Found	XDE-175-L % Recovery
13395	Canola Seed	6-Dec-2004	NA	ND	NA
14794	Olive Fruit Without Seed	6-Dec-2004	NA	0.0008	NA
14188	Olive Oil	6-Dec-2004	NA	ND	NA
13377	Soybean Grain	6-Dec-2004	NA	ND	NA
46001	Cotton Seed	6-Dec-2004	NA	0.0006	NA
13395	Canola Seed	6-Dec-2004	0.003	0.0031	NA
14794	Olive Fruit Without Seed	6-Dec-2004	0.003	0.0025	NA
14188	Olive Oil	6-Dec-2004	0.003	0.0030	NA
13395	Canola Seed	6-Dec-2004	0.01	0.0103	103
13395	Canola Seed	6-Dec-2004	0.01	0.0105	105
14794	Olive Fruit Without Seed	6-Dec-2004	0.01	0.0093	93
14794	Olive Fruit Without Seed	6-Dec-2004	0.01	0.0097	97
14188	Olive Oil	6-Dec-2004	0.01	0.0097	97
14188	Olive Oil	6-Dec-2004	0.01	0.0099	99
13377	Soybean Grain	6-Dec-2004	0.01	0.0106	106
13377	Soybean Grain	6-Dec-2004	0.01	0.0110	110
46001	Cotton Seed	6-Dec-2004	0.01	0.0090	90
46001	Cotton Seed	6-Dec-2004	0.01	0.0094	94
13395	Canola Seed	6-Dec-2004	1.0	1.06	106
13395	Canola Seed	6-Dec-2004	1.0	1.05	105
14794	Olive Fruit Without Seed	6-Dec-2004	1.0	0.97	97
14794	Olive Fruit Without Seed	6-Dec-2004	1.0	0.96	96
14188	Olive Oil	6-Dec-2004	1.0	0.95	95
14188	Olive Oil	6-Dec-2004	1.0	0.95	95
13377	Soybean Grain	6-Dec-2004	1.0	1.03	103
13377	Soybean Grain	6-Dec-2004	1.0	1.07	107
46001	Cotton Seed	6-Dec-2004	1.0	1.03	103
46001	Cotton Seed	6-Dec-2004	1.0	1.03	103

Table 5. (Cont.) Recovery of XDE-175-*N*-demethyl-J from Oily Crops

Sample Name	Matrix	Date of Analysis	XDE-175-*N*-demethyl-J Added	Found	% Recovery
13395	Canola Seed	6-Dec-2004	NA	0.0002	NA
14794	Olive Fruit Without Seed	6-Dec-2004	NA	0.0005	NA
14188	Olive Oil	6-Dec-2004	NA	0.0001	NA
13377	Soybean Grain	6-Dec-2004	NA	0.0002	NA
46001	Cotton Seed	6-Dec-2004	NA	0.0002	NA
13395	Canola Seed	6-Dec-2004	0.003	0.0026	NA
14794	Olive Fruit Without Seed	6-Dec-2004	0.003	0.0022	NA
14188	Olive Oil	6-Dec-2004	0.003	0.0027	NA
13395	Canola Seed	6-Dec-2004	0.01	0.0100	100
13395	Canola Seed	6-Dec-2004	0.01	0.0100	100
14794	Olive Fruit Without Seed	6-Dec-2004	0.01	0.0086	86
14794	Olive Fruit Without Seed	6-Dec-2004	0.01	0.0090	90
14188	Olive Oil	6-Dec-2004	0.01	0.0091	91
14188	Olive Oil	6-Dec-2004	0.01	0.0098	98
13377	Soybean Grain	6-Dec-2004	0.01	0.0101	101
13377	Soybean Grain	6-Dec-2004	0.01	0.0101	101
46001	Cotton Seed	6-Dec-2004	0.01	0.0096	96
46001	Cotton Seed	6-Dec-2004	0.01	0.0105	105
13395	Canola Seed	6-Dec-2004	1.0	1.05	105
13395	Canola Seed	6-Dec-2004	1.0	1.05	105
14794	Olive Fruit Without Seed	6-Dec-2004	1.0	0.93	93
14794	Olive Fruit Without Seed	6-Dec-2004	1.0	1.01	101
14188	Olive Oil	6-Dec-2004	1.0	1.02	102
14188	Olive Oil	6-Dec-2004	1.0	0.99	99
13377	Soybean Grain	6-Dec-2004	1.0	1.01	101
13377	Soybean Grain	6-Dec-2004	1.0	1.00	100
46001	Cotton Seed	6-Dec-2004	1.0	1.10	110
46001	Cotton Seed	6-Dec-2004	1.0	1.11	111

Table 5. (Cont.) Recovery of XDE-175-*N*-demethyl-L from Oily Crops

Sample Name	Matrix	Date of Analysis	XDE-175-*N*-demethyl-L Added	Found	% Recovery
13395	Canola Seed	6-Dec-2004	NA	0.0008	NA
14794	Olive Fruit Without Seed	6-Dec-2004	NA	0.0008	NA
14188	Olive Oil	6-Dec-2004	NA	0.0004	NA
13377	Soybean Grain	6-Dec-2004	NA	0.0006	NA
46001	Cotton Seed	6-Dec-2004	NA	0.0007	NA
13395	Canola Seed	6-Dec-2004	0.003	0.0022	NA
14794	Olive Fruit Without Seed	6-Dec-2004	0.003	0.0025	NA
14188	Olive Oil	6-Dec-2004	0.003	0.0029	NA
13395	Canola Seed	6-Dec-2004	0.01	0.0090	90
13395	Canola Seed	6-Dec-2004	0.01	0.0092	92
14794	Olive Fruit Without Seed	6-Dec-2004	0.01	0.0084	84
14794	Olive Fruit Without Seed	6-Dec-2004	0.01	0.0092	92
14188	Olive Oil	6-Dec-2004	0.01	0.0094	94
14188	Olive Oil	6-Dec-2004	0.01	0.0094	94
13377	Soybean Grain	6-Dec-2004	0.01	0.0091	91
13377	Soybean Grain	6-Dec-2004	0.01	0.0101	101
46001	Cotton Seed	6-Dec-2004	0.01	0.0083	83
46001	Cotton Seed	6-Dec-2004	0.01	0.0083	83
13395	Canola Seed	6-Dec-2004	1.0	0.96	96
13395	Canola Seed	6-Dec-2004	1.0	0.99	99
14794	Olive Fruit Without Seed	6-Dec-2004	1.0	0.93	93
14794	Olive Fruit Without Seed	6-Dec-2004	1.0	0.99	99
14188	Olive Oil	6-Dec-2004	1.0	1.02	102
14188	Olive Oil	6-Dec-2004	1.0	1.00	100
13377	Soybean Grain	6-Dec-2004	1.0	1.00	100
13377	Soybean Grain	6-Dec-2004	1.0	0.99	99
46001	Cotton Seed	6-Dec-2004	1.0	0.92	92
46001	Cotton Seed	6-Dec-2004	1.0	0.96	96

Table 5. (Cont.) Recovery of XDE-175-*N*-formyl-J from Oily Crops

Sample Name	Matrix	Date of Analysis	XDE-175-*N*-formyl-J Added	Found	% Recovery
13395	Canola Seed	6-Dec-2004	NA	0.0005	NA
14794	Olive Fruit Without Seed	6-Dec-2004	NA	ND	NA
14188	Olive Oil	6-Dec-2004	NA	ND	NA
13377	Soybean Grain	6-Dec-2004	NA	ND	NA
46001	Cotton Seed	6-Dec-2004	NA	ND	NA
13395	Canola Seed	6-Dec-2004	0.003	0.0039	NA
14794	Olive Fruit Without Seed	6-Dec-2004	0.003	0.0028	NA
14188	Olive Oil	6-Dec-2004	0.003	0.0034	NA
13395	Canola Seed	6-Dec-2004	0.01	0.0093	93
13395	Canola Seed	6-Dec-2004	0.01	0.0082	82
14794	Olive Fruit Without Seed	6-Dec-2004	0.01	0.0098	98
14794	Olive Fruit Without Seed	6-Dec-2004	0.01	0.0096	96
14188	Olive Oil	6-Dec-2004	0.01	0.0112	112
14188	Olive Oil	6-Dec-2004	0.01	0.0103	103
13377	Soybean Grain	6-Dec-2004	0.01	0.0102	102
13377	Soybean Grain	6-Dec-2004	0.01	0.0117	117
46001	Cotton Seed	6-Dec-2004	0.01	0.0108	108
46001	Cotton Seed	6-Dec-2004	0.01	0.0120	120
13395	Canola Seed	6-Dec-2004	1.0	1.08	108
13395	Canola Seed	6-Dec-2004	1.0	1.13	113
14794	Olive Fruit Without Seed	6-Dec-2004	1.0	1.10	110
14794	Olive Fruit Without Seed	6-Dec-2004	1.0	1.02	102
14188	Olive Oil	6-Dec-2004	1.0	1.08	108
14188	Olive Oil	6-Dec-2004	1.0	1.08	108
13377	Soybean Grain	6-Dec-2004	1.0	1.24	124
13377	Soybean Grain	6-Dec-2004	1.0	1.25	125
46001	Cotton Seed	6-Dec-2004	1.0	1.16	116
46001	Cotton Seed	6-Dec-2004	1.0	1.40	140

Table 5. (Cont.) Recovery of XDE-175-*N*-formyl-L from Oily Crops

Sample Name	Matrix	Date of Analysis	XDE-175-*N*-formyl-L Added	Found	% Recovery
13395	Canola Seed	6-Dec-2004	NA	ND	NA
14794	Olive Fruit Without Seed	6-Dec-2004	NA	ND	NA
14188	Olive Oil	6-Dec-2004	NA	ND	NA
13377	Soybean Grain	6-Dec-2004	NA	ND	NA
46001	Cotton Seed	6-Dec-2004	NA	ND	NA
13395	Canola Seed	6-Dec-2004	0.003	0.0024	NA
14794	Olive Fruit Without Seed	6-Dec-2004	0.003	0.0028	NA
14188	Olive Oil	6-Dec-2004	0.003	0.0033	NA
13395	Canola Seed	6-Dec-2004	0.01	0.0091	91
13395	Canola Seed	6-Dec-2004	0.01	0.0115	115
14794	Olive Fruit Without Seed	6-Dec-2004	0.01	0.0114	114
14794	Olive Fruit Without Seed	6-Dec-2004	0.01	0.0095	95
14188	Olive Oil	6-Dec-2004	0.01	0.0115	115
14188	Olive Oil	6-Dec-2004	0.01	0.0104	104
13377	Soybean Grain	6-Dec-2004	0.01	0.0122	122
13377	Soybean Grain	6-Dec-2004	0.01	0.0138	138
46001	Cotton Seed	6-Dec-2004	0.01	0.0063	63
46001	Cotton Seed	6-Dec-2004	0.01	0.0091	91
13395	Canola Seed	6-Dec-2004	1.0	0.99	99
13395	Canola Seed	6-Dec-2004	1.0	1.16	116
14794	Olive Fruit Without Seed	6-Dec-2004	1.0	1.18	118
14794	Olive Fruit Without Seed	6-Dec-2004	1.0	1.09	109
14188	Olive Oil	6-Dec-2004	1.0	1.10	110
14188	Olive Oil	6-Dec-2004	1.0	1.09	109
13377	Soybean Grain	6-Dec-2004	1.0	1.28	128
13377	Soybean Grain	6-Dec-2004	1.0	1.39	139
46001	Cotton Seed	6-Dec-2004	1.0	0.76	76
46001	Cotton Seed	6-Dec-2004	1.0	1.02	102

[a] The date of analysis represents the date the samples were extracted.
[b] All calculations were performed using Microsoft Excel 2002 with full precision. Recovery values corrected for control value where appropriate.
[c] Not Applicable.
[d] Not Detected.
[e] Samples were fortified at the method's proposed limit of detection (0.003 µg/g). Values are reported with a lower degree of confidence than values above the limit of quantitation.

Table 6. Recovery Summary of XDE-175 and its Metabolites from Wet Crops

Compound	Fortification Level (µg/g)	Average Recovery (%)	Recovery Range (%)	SD (%)	RSD (%)	n
XDE-175-*N*-formyl-L	0.01	100	79 - 119	11.2	11.2	18
	1.0	99	91 - 111	4.8	4.9	16
	10.0	90	81 - 99	7.8	7.8	6
	0.01 – 10.0	98	79 - 119	9.2	9.3	40
XDE-175-*N*-formyl-J	0.01	93	86 - 106	6.7	7.2	18
	1.0	101	91 - 116	7.0	6.9	16
	10.0	91	84 - 101	7.1	7.6	6
	0.01 – 10.0	96	84 - 116	7.9	8.3	40
XDE-175-*N*-demethyl-L	0.01	102	92 - 120	6.8	6.6	18
	1.0	101	95 - 104	2.3	2.3	16
	10.0	91	87 - 99	5.5	5.4	6
	0.01 – 10.0	100	87 - 120	6.3	6.3	40
XDE-175-*N*-demethyl-J	0.01	100	94 - 117	5.9	5.8	18
	1.0	100	94 - 105	2.8	2.8	16
	10.0	94	89 - 101	5.5	5.5	6
	0.01 – 10.0	99	89 - 117	5.2	5.3	40
XDE-175-L	0.01	104	96 - 114	5.5	5.3	18
	1.0	101	95 - 107	3.0	3.0	16
	10.0	92	89 - 98	3.3	3.2	6
	0.01 – 10.0	101	89 - 114	6.0	6.0	40
XDE-175-J	0.01	103	95 - 118	5.8	5.6	18
	1.0	99	92 - 103	3.5	3.5	16
	10.0	92	90 - 94	1.3	1.3	6
	0.01 – 10.0	100	90 - 118	5.7	5.7	40

Table 7. Recovery Summary of XDE-175 and its Metabolites from Dry Crops

Compound	Fortification Level (µg/g)	Average Recovery (%)	Recovery Range (%)	SD (%)	RSD (%)	n
XDE-175-*N*-formyl-L	0.01	95	78 - 119	11.5	12.1	20
	1.0	93	77 - 117	11.6	12.5	20
	0.01 – 1.0	94	77 - 119	11.4	12.2	40
XDE-175-*N*-formyl-J	0.01	105	77 - 131	13.2	12.5	20
	1.0	110	97 - 121	5.9	5.3	20
	0.01 – 1.0	108	77 - 131	10.4	9.7	40
XDE-175-*N*-demethyl-L	0.01	105	99 - 114	4.5	4.3	20
	1.0	104	96 - 110	3.8	3.6	20
	0.01 – 1.0	105	96 - 114	4.1	4.0	40
XDE-175-*N*-demethyl-J	0.01	102	92 - 114	5.2	5.1	20
	1.0	102	95 - 114	3.9	3.8	20
	0.01 – 1.0	102	92 - 114	4.5	4.4	40
XDE-175-L	0.01	102	94 - 114	5.7	5.5	20
	1.0	102	92 - 114	6.3	6.2	20
	0.01 – 1.0	102	92 - 114	5.9	5.8	40
XDE-175-J	0.01	88	81 - 105	5.2	6.0	20
	1.0	88	81 - 98	4.9	5.6	20
	0.01 – 1.0	88	81 - 105	5.0	5.7	40

Table 8. Recovery Summary of XDE-175 and its Metabolites from Acidic Crops

Compound	Fortification Level (µg/g)	Average Recovery (%)	Recovery Range (%)	SD (%)	RSD (%)	n
XDE-175-*N*-formyl-L	0.01	94	86 - 107	7.0	7.4	17
	1.0	97	91 - 107	4.1	4.2	18
	0.01 – 1.0	95	86 - 107	5.8	6.1	35
XDE-175-*N*-formyl-J	0.01	97	76 - 118	12.1	12.4	17
	1.0	103	94 - 118	6.1	6.0	18
	0.01 – 1.0	100	76 - 118	9.8	9.8	35
XDE-175-*N*-demethyl-L	0.01	100	87 - 113	7.9	7.8	17
	1.0	101	96 - 107	2.9	2.9	18
	0.01 – 1.0	100	87 - 113	5.8	5.8	35
XDE-175-*N*-demethyl-J	0.01	104	96 - 114	5.8	5.6	17
	1.0	102	97 - 107	3.1	3.1	18
	0.01 – 1.0	103	96 - 114	4.6	4.5	35
XDE-175-L	0.01	106	98 - 114	4.9	4.6	17
	1.0	100	92 - 104	3.0	3.0	18
	0.01 – 1.0	103	92 - 114	5.2	5.0	35
XDE-175-J	0.01	102	93 - 115	6.1	6.0	17
	1.0	101	94 - 105	3.3	3.3	18
	0.01 – 1.0	102	93 - 115	4.9	4.8	35

Table 9. Recovery Summary of XDE-175 and its Metabolites from Oily Crops

Compound	Fortification Level (µg/g)	Average Recovery (%)	Recovery Range (%)	SD (%)	RSD (%)	n
XDE-175-*N*-formyl-L	0.01	105	63 - 138	20.7	19.7	10
	1.0	111	76 - 139	17.0	15.4	10
	0.01 – 1.0	108	63 - 139	18.7	17.3	20
XDE-175-*N*-formyl-J	0.01	103	82 - 120	11.6	11.2	10
	1.0	115	102 - 140	11.3	9.8	10
	0.01 – 1.0	109	82 - 140	12.8	11.7	20
XDE-175-*N*-demethyl-L	0.01	90	83 - 101	5.8	6.4	10
	1.0	98	92 - 102	3.3	3.4	10
	0.01 – 1.0	94	83 - 102	5.9	6.2	20
XDE-175-*N*-demethyl-J	0.01	97	86 - 105	6.0	6.2	10
	1.0	103	93 - 111	5.2	5.1	10
	0.01 – 1.0	100	86 - 111	6.3	6.3	20
XDE-175-L	0.01	99	90 - 110	6.3	6.3	10
	1.0	101	95 - 107	4.7	4.7	10
	0.01 – 1.0	100	90 - 110	5.5	5.5	20
XDE-175-J	0.01	94	84 - 105	6.9	7.4	10
	1.0	96	89 - 102	4.7	4.9	10
	0.01 – 1.0	95	84 - 105	5.9	6.2	20

Table 10. Calculated Limits of Detection and Quantitation for XDE-175 and its Metabolites in Wet Crops

Compound	Average Recovery (µg/g)	Standard Deviation (*s*)	Calculated Limit of Detection (3*s*)	Calculated Limit of Quantitation (10*s*)	Number of Samples (n)
XDE-175-*N*-formyl-L	0.00999	0.00112	0.00335	0.01117	18
XDE-175-*N*-formyl-J	0.00929	0.00067	0.00202	0.00673	18
XDE-175-*N*-demethyl-L	0.01022	0.00068	0.00203	0.00676	18
XDE-175-*N*-demethyl-J	0.01003	0.00059	0.00176	0.00585	18
XDE-175-L	0.01044	0.00055	0.00165	0.00549	18
XDE-175-J	0.01028	0.00058	0.00174	0.00579	18

Table 11. Calculated Limits of Detection and Quantitation for XDE-175 and its Metabolites in Dry Crops

Compound	Average Recovery (µg/g)	Standard Deviation (*s*)	Calculated Limit of Detection (3*s*)	Calculated Limit of Quantitation (10*s*)	Number of Samples (n)
XDE-175-*N*-formyl-L	0.00949	0.00115	0.00345	0.01149	20
XDE-175-*N*-formyl-J	0.01053	0.00132	0.00396	0.01320	20
XDE-175-*N*-demethl-L	0.01049	0.00045	0.00136	0.00455	20
XDE-175-*N*-demethl-J	0.01018	0.00052	0.00155	0.00515	20
XDE-175-L	0.01022	0.00057	0.00170	0.00567	20
XDE-175-J	0.00875	0.00052	0.00157	0.00525	20

Table 12. Calculated Limits of Detection and Quantitation for XDE-175 and its Metabolites in Acidic Crops

Compound	Average Recovery (µg/g)	Standard Deviation (*s*)	Calculated Limit of Detection (3*s*)	Calculated Limit of Quantitation (10*s*)	Number of Samples (n)
XDE-175-*N*-formyl-L	0.00936	0.00070	0.00209	0.00696	17
XDE-175-*N*-formyl-J	0.00970	0.00121	0.00362	0.01208	17
XDE-175-*N*-demethl-L	0.01001	0.00079	0.00236	0.00785	17
XDE-175-*N*-demethl-J	0.01038	0.00058	0.00173	0.00577	17
XDE-175-L	0.01062	0.00049	0.00148	0.00492	17
XDE-175-J	0.01024	0.00061	0.00183	0.00610	17

Table 13. Calculated Limits of Detection and Quantitation for XDE-175 and its Metabolites in Oily Crops

Compound	Average Recovery (µg/g)	Standard Deviation (*s*)	Calculated Limit of Detection (3*s*)	Calculated Limit of Quantitation (10*s*)	Number of Samples (n)
XDE-175-*N*-formyl-L	0.01049	0.00207	0.00621	0.02070	10
XDE-175-*N*-formyl-J	0.01030	0.00116	0.00347	0.01158	10
XDE-175-*N*-demethl-L	0.00903	0.00058	0.00173	0.00576	10
XDE-175-*N*-demethl-J	0.00967	0.00060	0.00179	0.00597	10
XDE-175-L	0.00994	0.00063	0.00189	0.00631	10
XDE-175-J	0.00936	0.00069	0.00207	0.00689	10

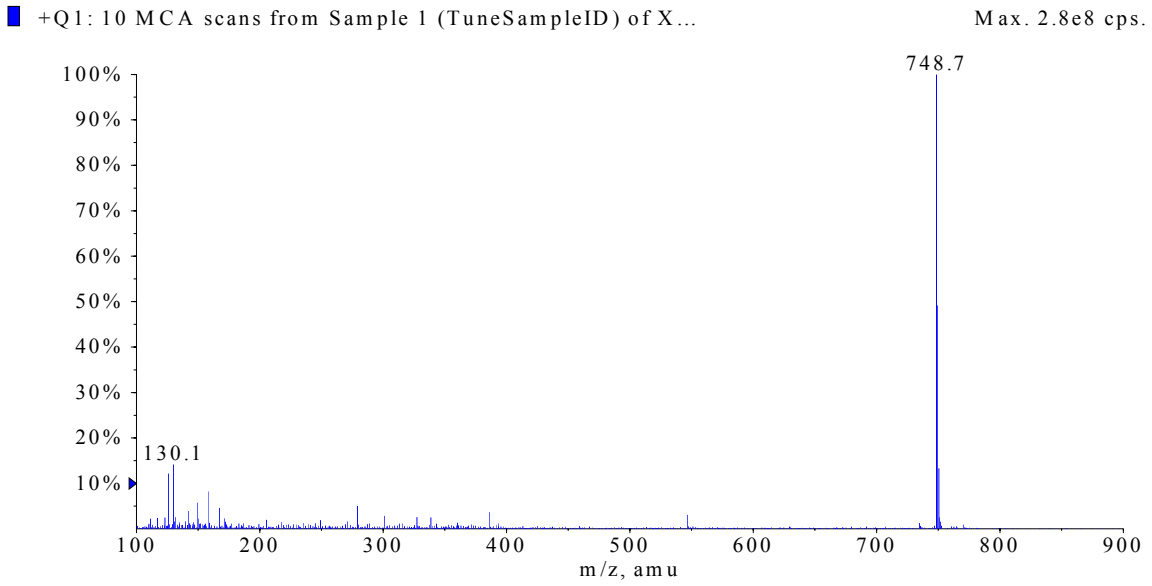


Figure 1. Mass Spectra of XDE-175-J Showing $(M+H)^+$ at *m/z* 748.7

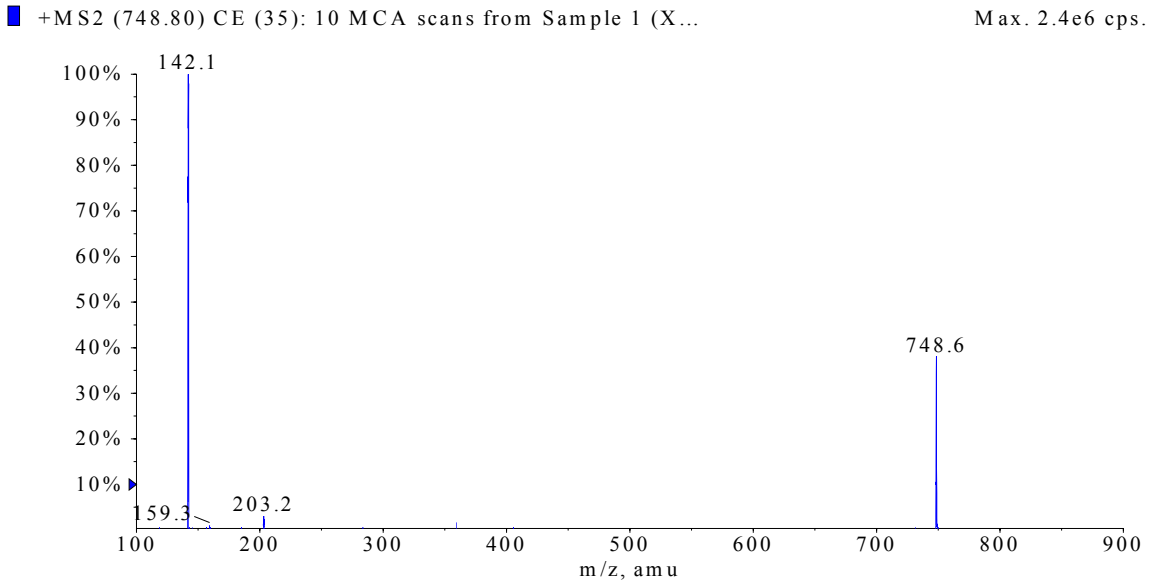


Figure 2. Product-Ion Mass Spectra of XDE-175-J Showing Fragment Ion at *m/z* 142.1

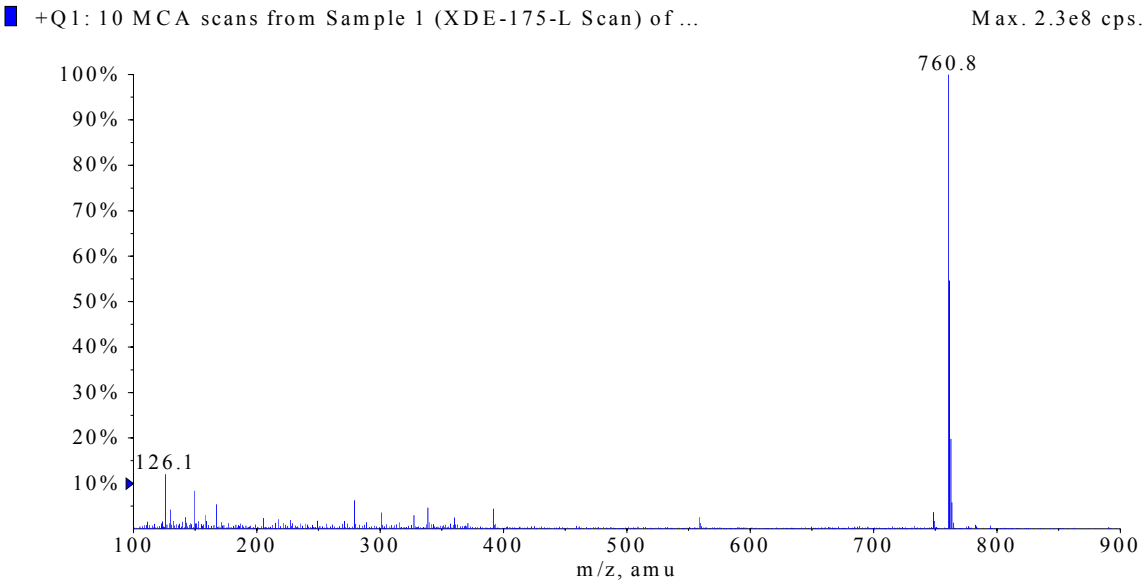


Figure 3. Mass Spectra of XDE-175-L Showing $(M+H)^+$ at *m/z* 760.8

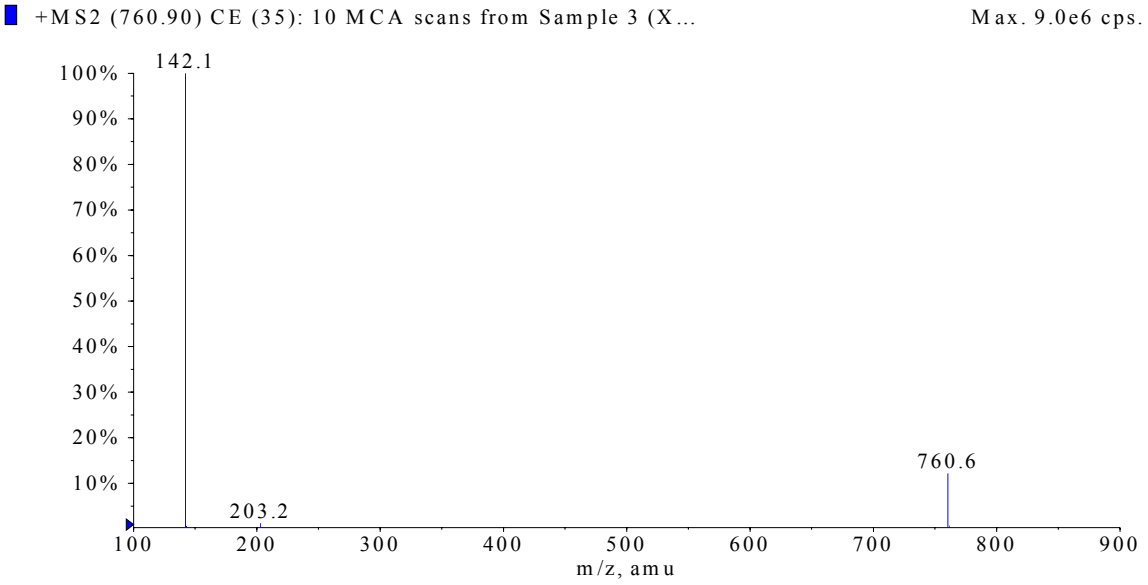


Figure 4. Product-Ion Mass Spectra of XDE-175-L Showing Fragment Ion at *m/z* 142.1

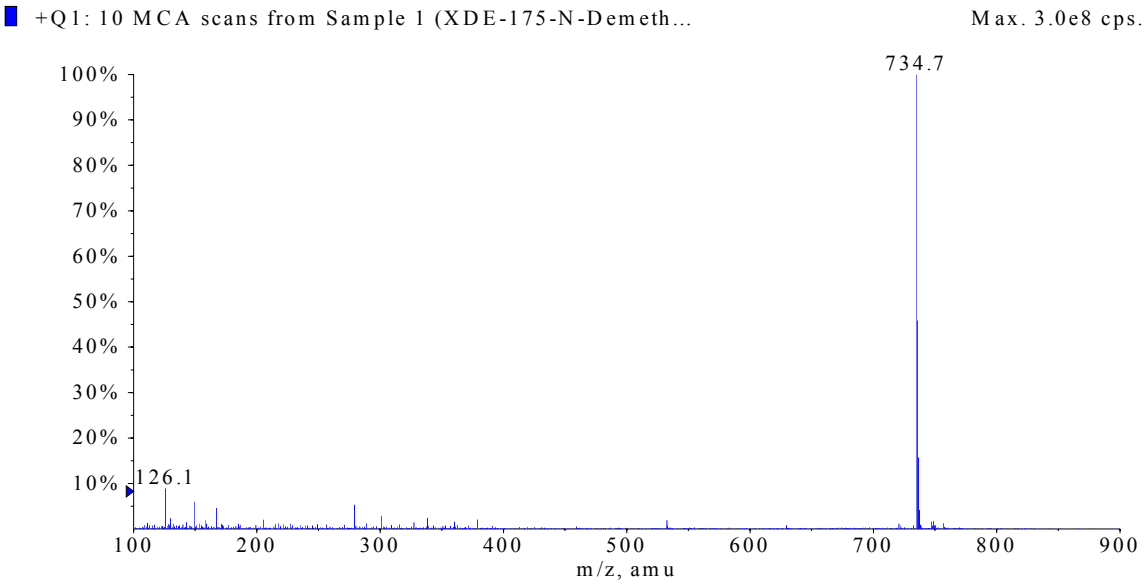


Figure 5. Mass Spectra of XDE-175-*N*-demethyl-J Showing $(M+H)^+$ at *m/z* 734.7

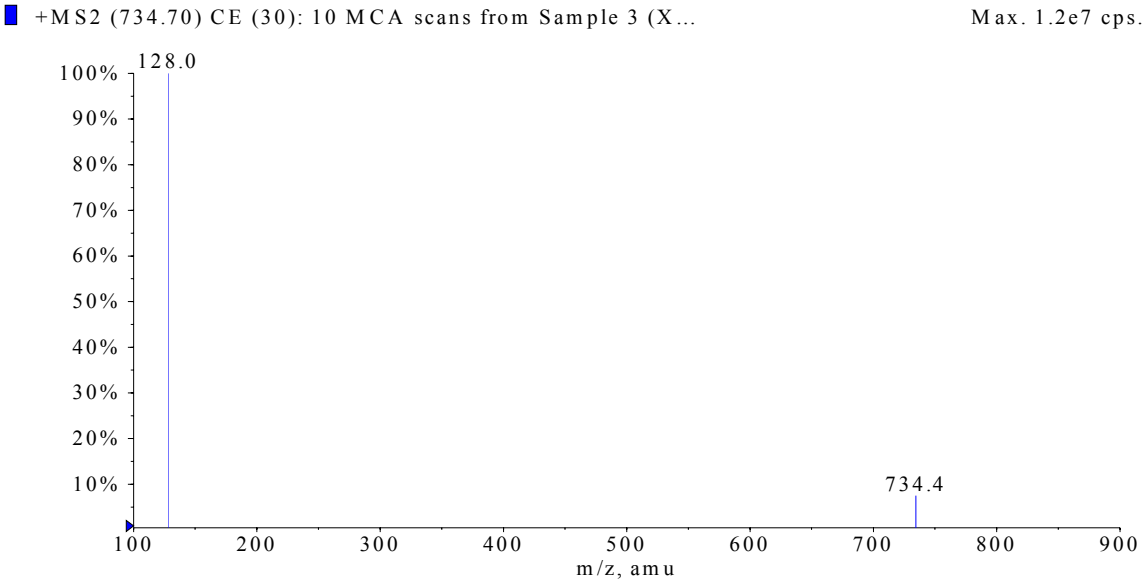


Figure 6. Product-Ion Mass Spectra of XDE-175-*N*-demethyl-J Showing Fragment Ion at *m/z* 128.0

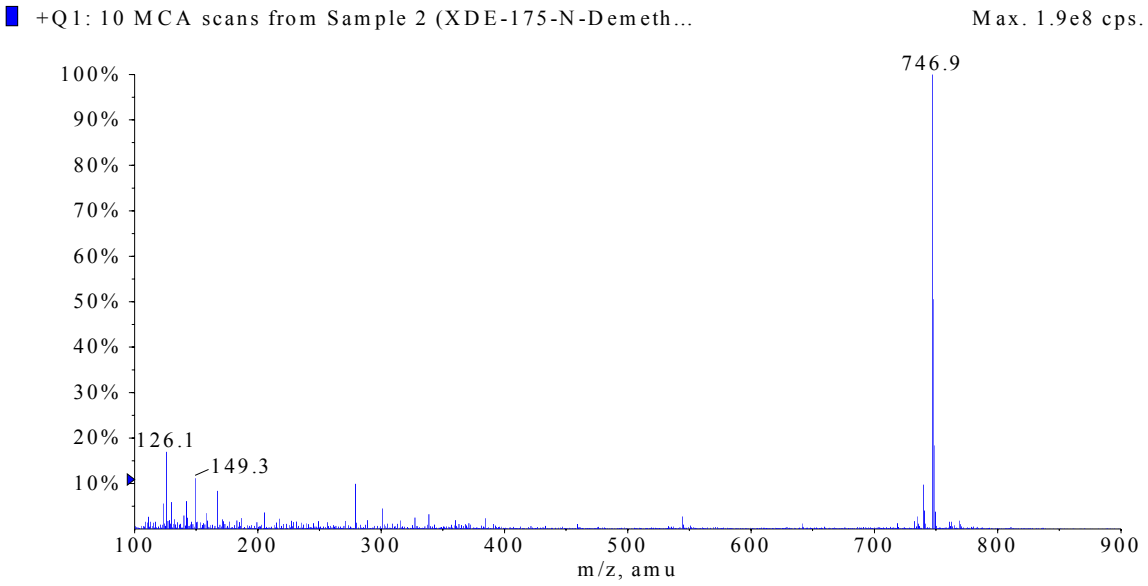


Figure 7. Mass Spectra of XDE-175-*N*-demethyl-L Showing $(M+H)^+$ at *m/z* 746.9

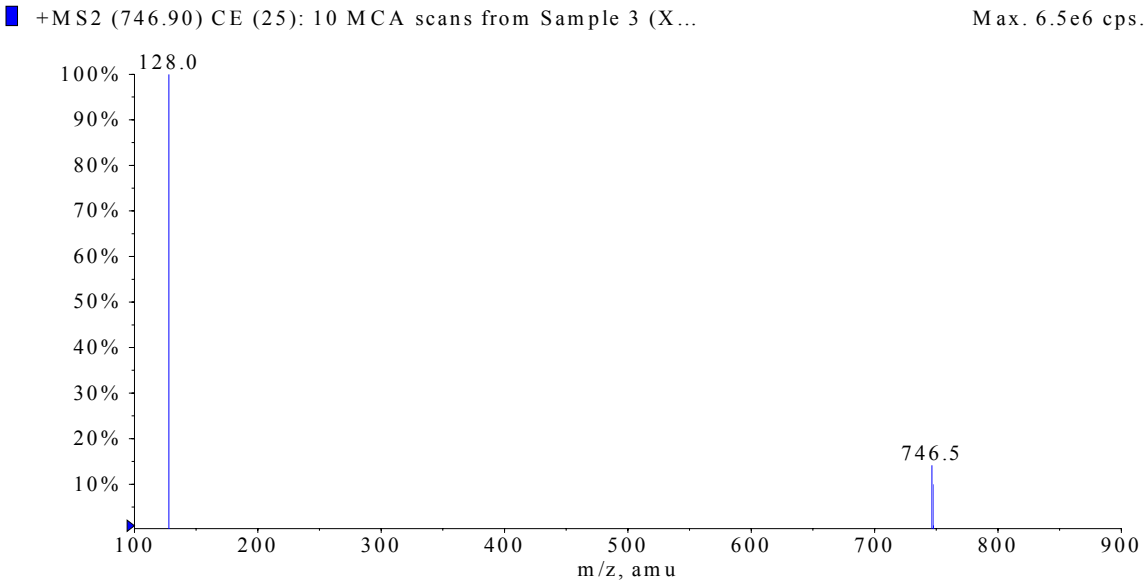


Figure 8. Product-Ion Mass Spectra of XDE-175-*N*-demethyl-L Showing Fragment Ion at *m/z* 128.0

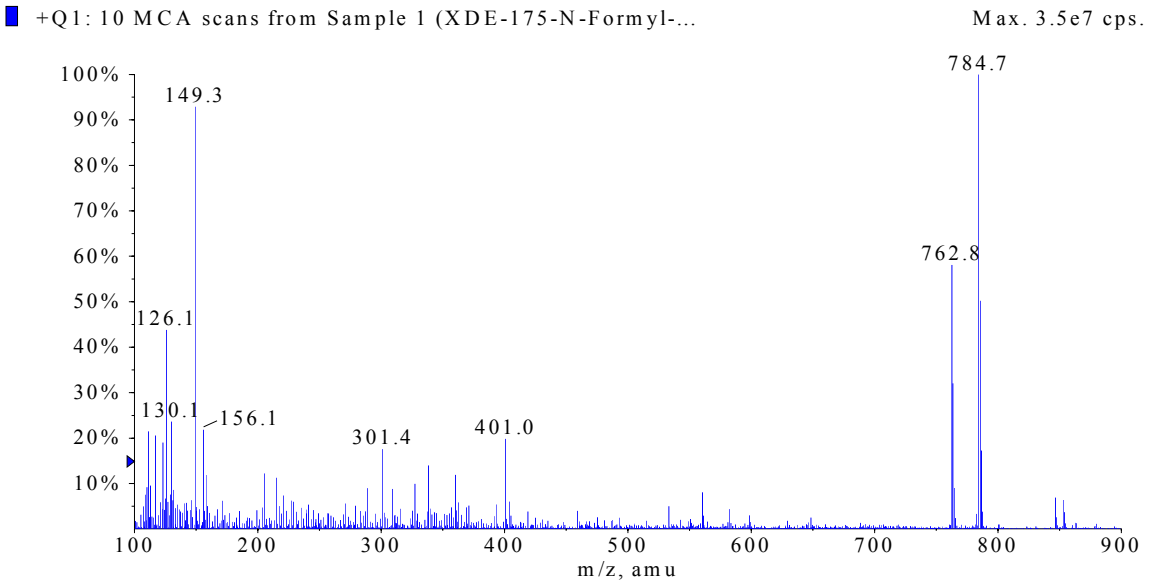


Figure 9. Mass Spectra of XDE-175-*N*-formyl-J Showing $(M+H)^+$ at *m/z* 762.8

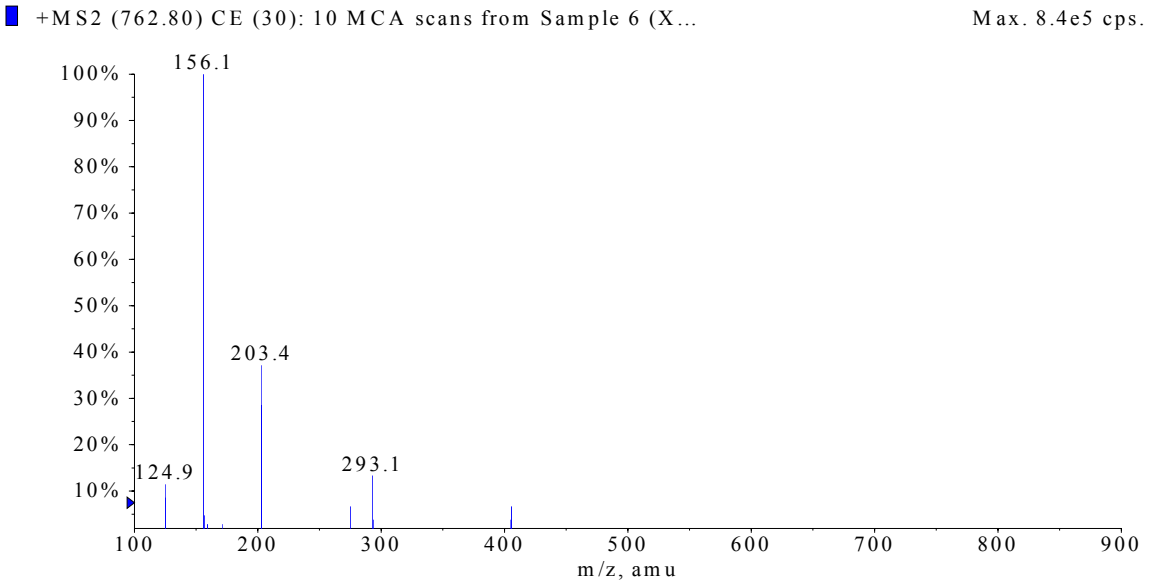


Figure 10. Product-Ion Mass Spectra of XDE-175-*N*-formyl-J Showing Fragment Ion at *m/z* 156.1

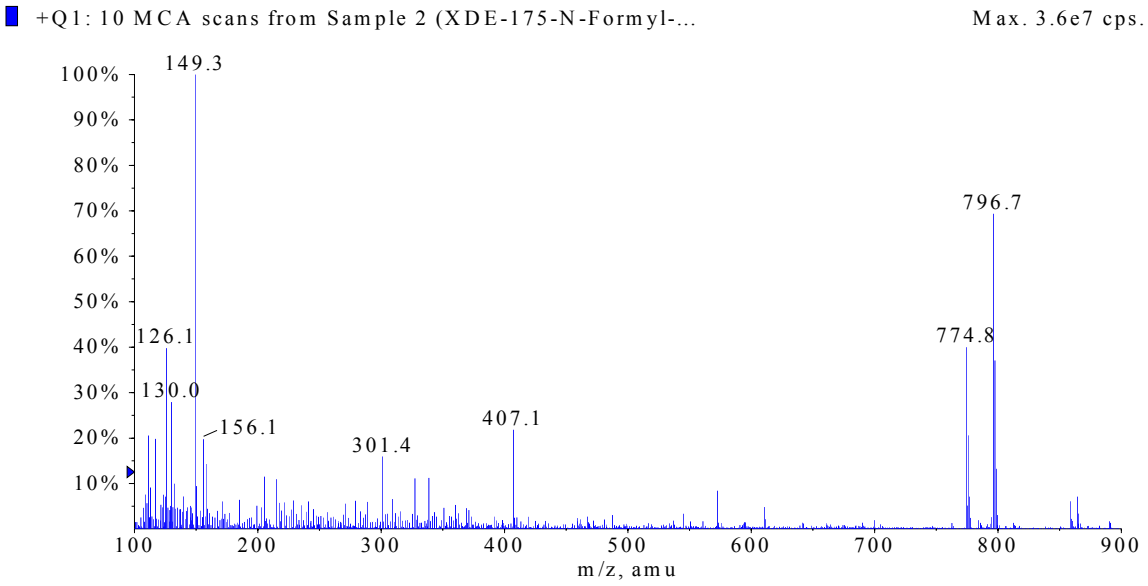


Figure 11. Mass Spectra of XDE-175-*N*-formyl-L Showing $(M+H)^+$ at *m/z* 774.8

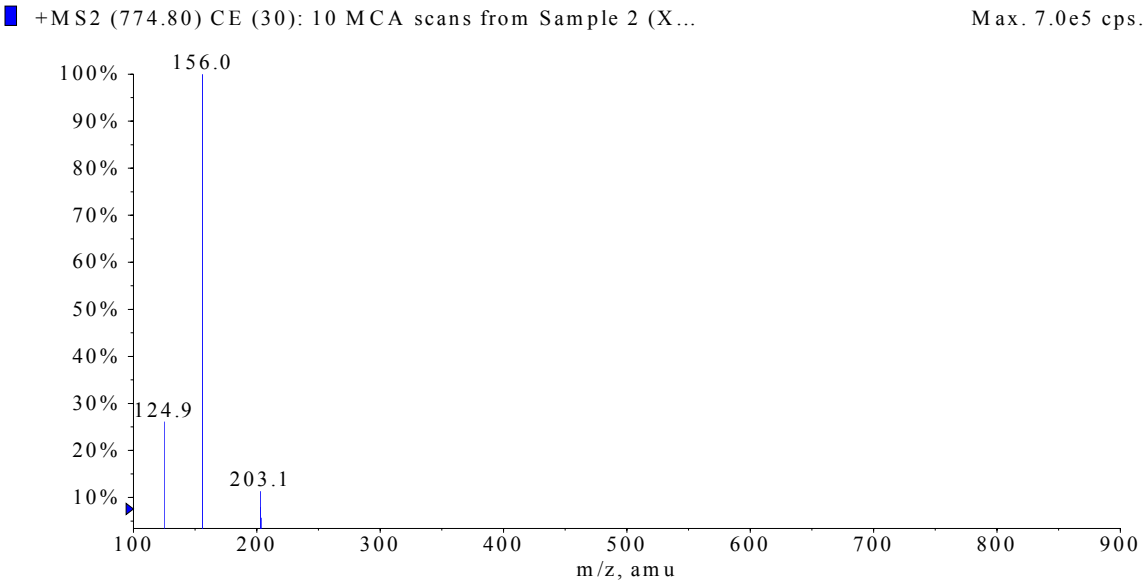


Figure 12. Product-Ion Mass Spectra of XDE-175-*N*-formyl-L Showing Fragment Ion at *m/z* 156.0

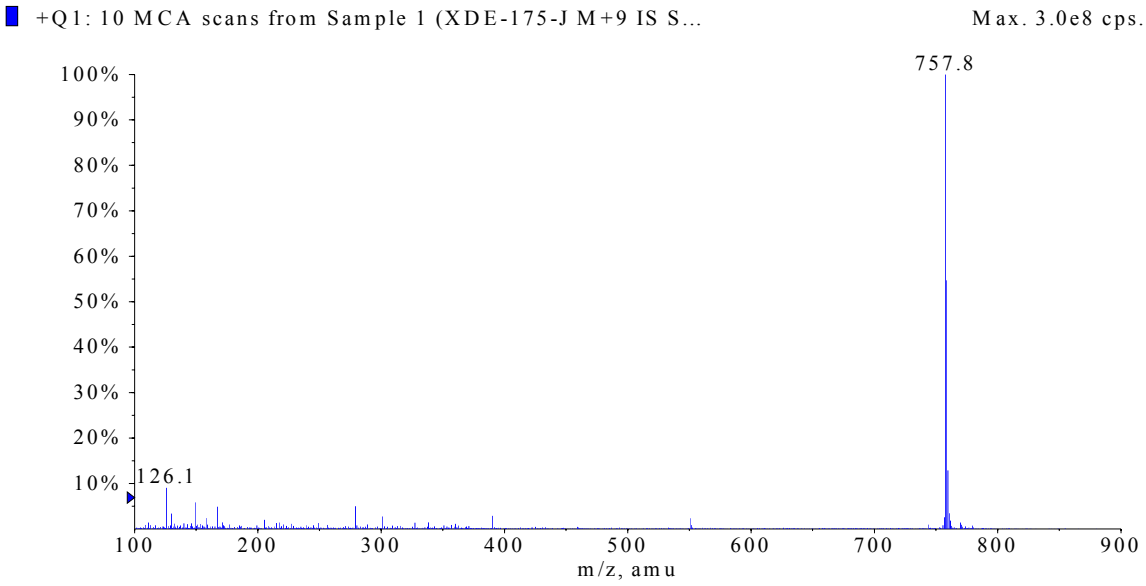


Figure 13. Mass Spectra of XDE-175-J Stable Isotope Internal Standard Showing $(M+H)^+$ at *m/z* 757.8

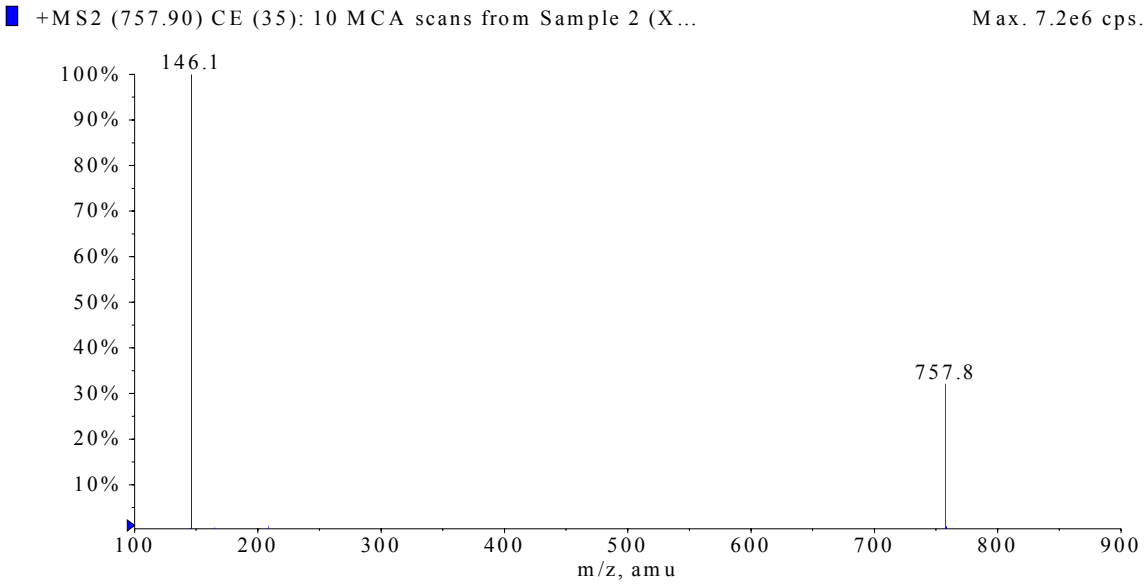


Figure 14. Product-Ion Mass Spectra of XDE-175-J Stable Isotope Internal Standard Showing Fragment Ion at *m/z* 146.1

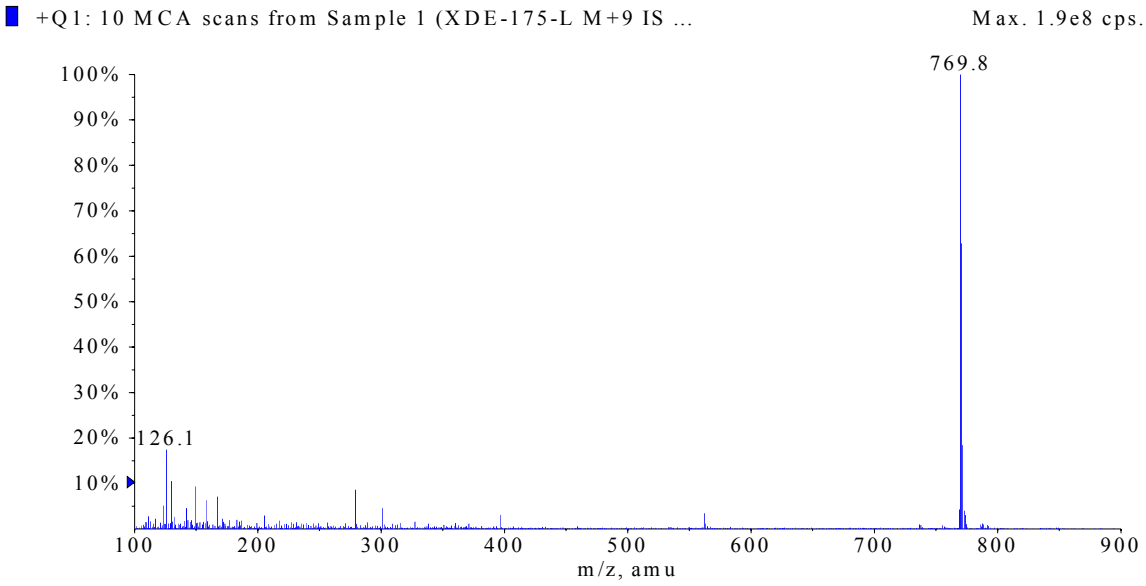


Figure 15. Mass Spectra of XDE-175-L Stable Isotope Internal Standard Showing $(M+H)^+$ at *m/z* 769.8

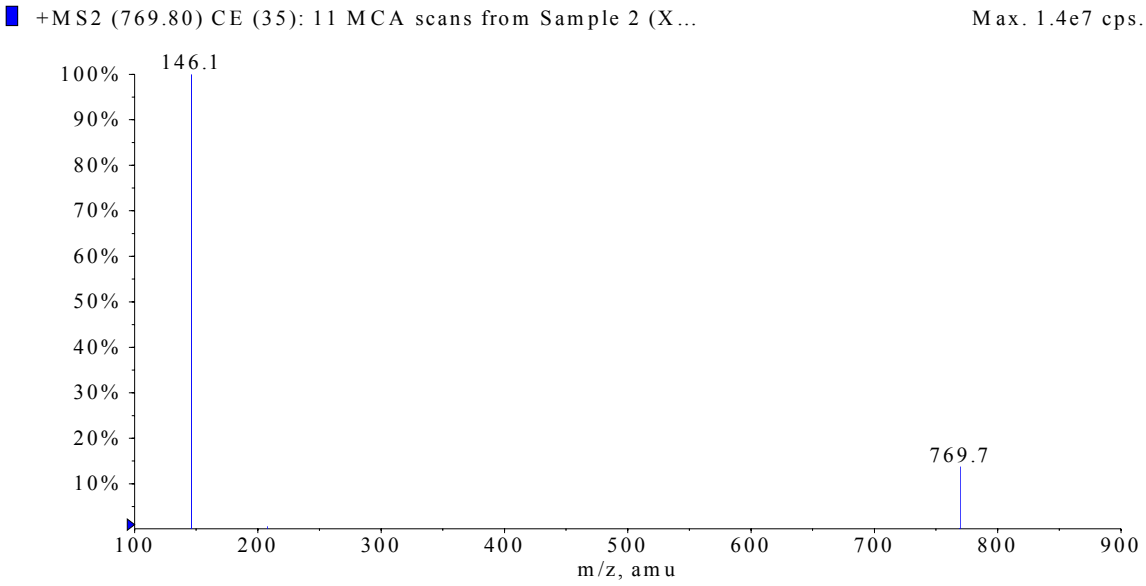


Figure 16. Product-Ion Mass Spectra of XDE-175-L Stable Isotope Internal Standard Showing Fragment Ion at *m/z* 146.1

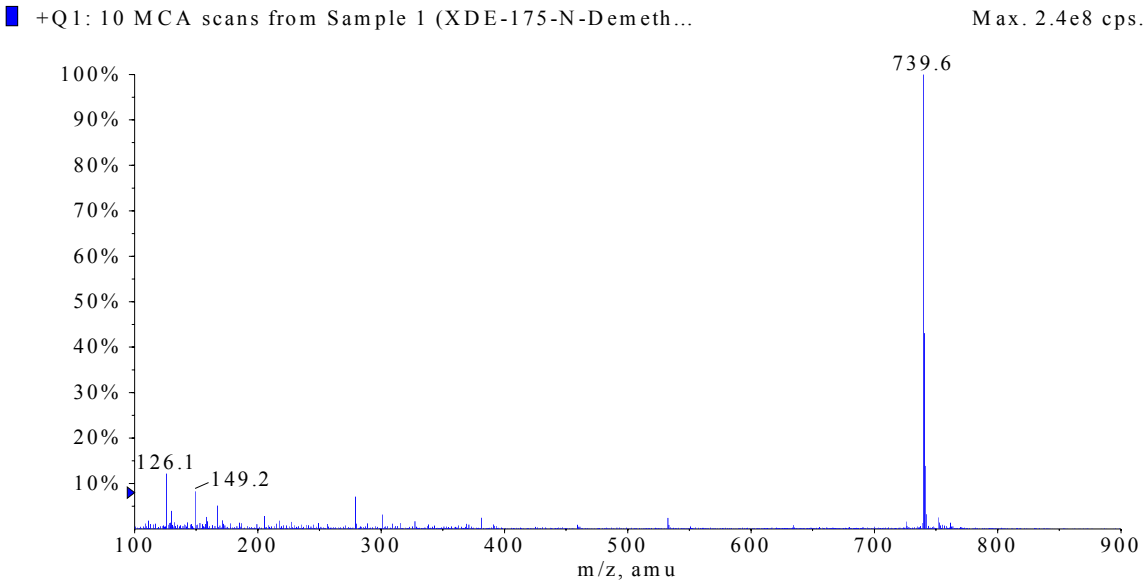


Figure 17. Mass Spectra of XDE-175-*N*-Demethyl-J Stable Isotope Internal Standard Showing $(M+H)^+$ at *m/z* 739.6

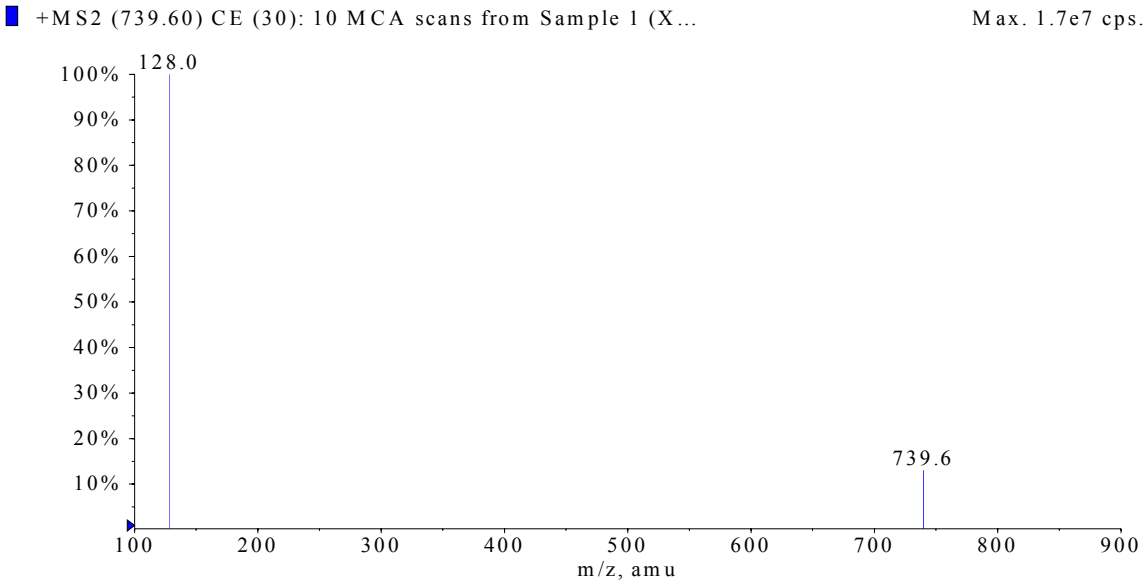


Figure 18. Product-Ion Mass Spectra of XDE-175-*N*-Demethyl-J Stable Isotope Internal Standard Showing Fragment Ion at *m/z* 128.0

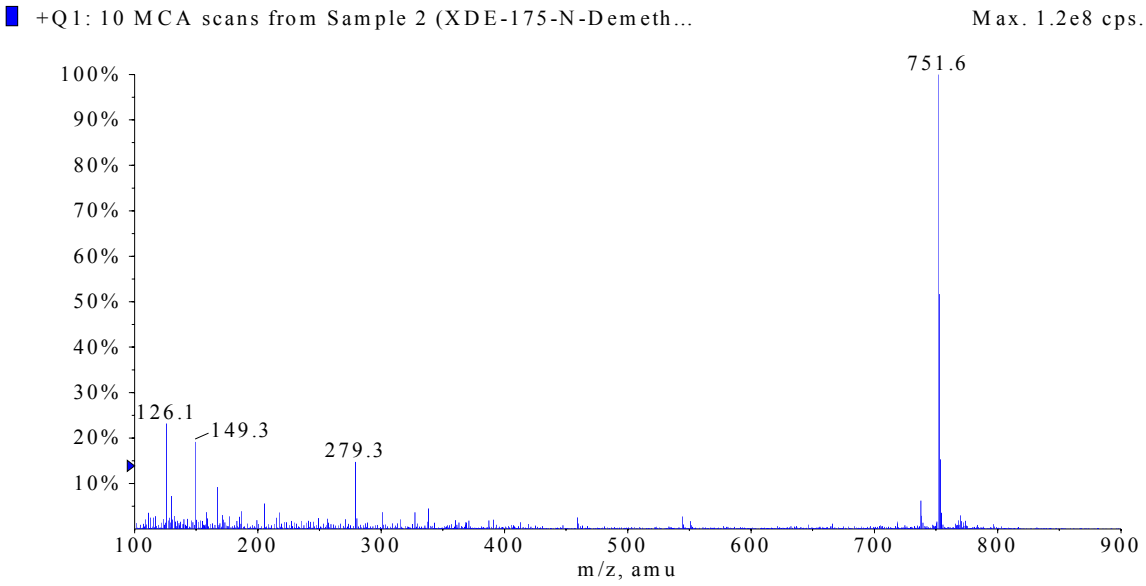


Figure 19. Mass Spectra of XDE-175-*N*-Demethyl-L Stable Isotope Internal Standard Showing $(M+H)^+$ at *m/z* 751.6

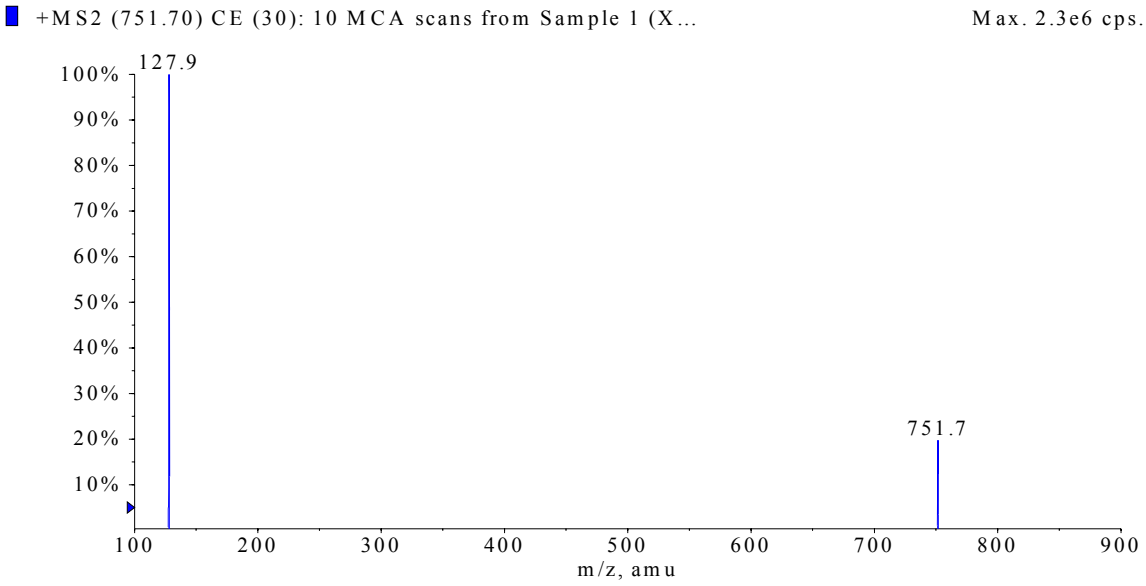


Figure 20. Product-Ion Mass Spectra of XDE-175-*N*-Demethyl-L Stable Isotope Internal Standard Showing Fragment Ion at *m/z* 127.9

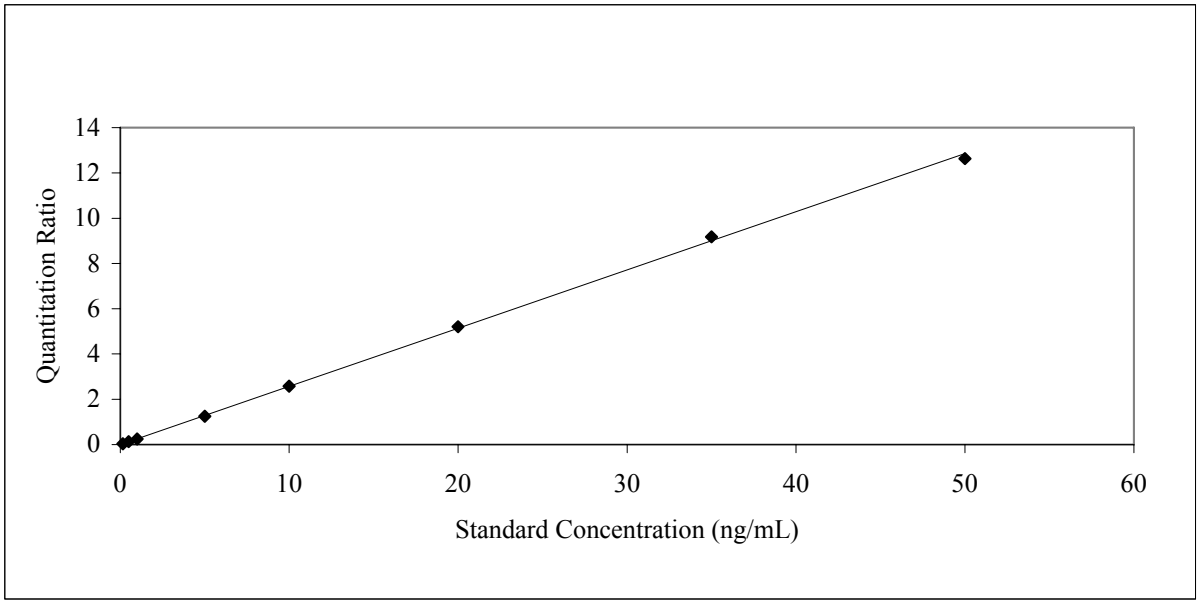
Analytical Set I.D.: 041021 set 2b
Compound: XDE-175 J

Calibration Data

Linear with 1/x Weighting

Slope =	0.2571
Intercept =	-0.0015
r^2 =	0.9997

Standard Concentration (ng/mL)	Injection Number	Analyte Peak Area	ISTD Peak Area	Quantitation Ratio	Response Factor	Calculated Concentration	Percent of Theoretical
0.15	1	19607	545738	0.036	0.2395	0.14543	97
0.5	9	51007	379980	0.134	0.2685	0.52776	106
1	17	104824	418307	0.251	0.2506	0.98027	98
5	25	527104	421216	1.251	0.2503	4.87245	97
10	33	1167023	451851	2.583	0.2583	10.05026	101
20	41	2170012	416761	5.207	0.2603	20.25555	101
35	49	3243716	353419	9.178	0.2622	35.70006	102
50	50	4550286	360324	12.628	0.2526	49.11821	98



Sample	Injection Number	Analyte Peak Area	ISTD Peak Area	Quantitation Ratio
24801	5	0	468460	0.00000
24801 + 0.003 µg/g	13	15927	416450	0.03824
24801 + 0.01 µg/g	22	54482	449216	0.12128
24801 + 0.01 µg/g	23	49945	377032	0.13247

Figure 21. Typical Calibration Curve for the Determination of XDE-175-J in Wet Crops

Analytical Set I.D.: 041021 set 2b
Compound: XDE-175 L

Calibration Data

Linear with 1/x Weighting

Slope =	0.2607
Intercept =	-0.0031
r^2 =	0.9995

Standard Concentration (ng/mL)	Injection Number	Analyte Peak Area	ISTD Peak Area	Quantitation Ratio	Response Factor	Calculated Concentration	Percent of Theoretical
0.15	1	16704	451049	0.037	0.2469	0.15397	103
0.5	9	46144	369983	0.125	0.2494	0.49027	98
1	17	95990	380881	0.252	0.2520	0.97852	98
5	25	463299	361670	1.281	0.2562	4.92502	99
10	33	1060375	400707	2.646	0.2646	10.16128	102
20	41	2010441	381074	5.276	0.2638	20.24621	101
35	49	2969918	317965	9.340	0.2669	35.83568	102
50	50	4180007	328204	12.736	0.2547	48.85905	98

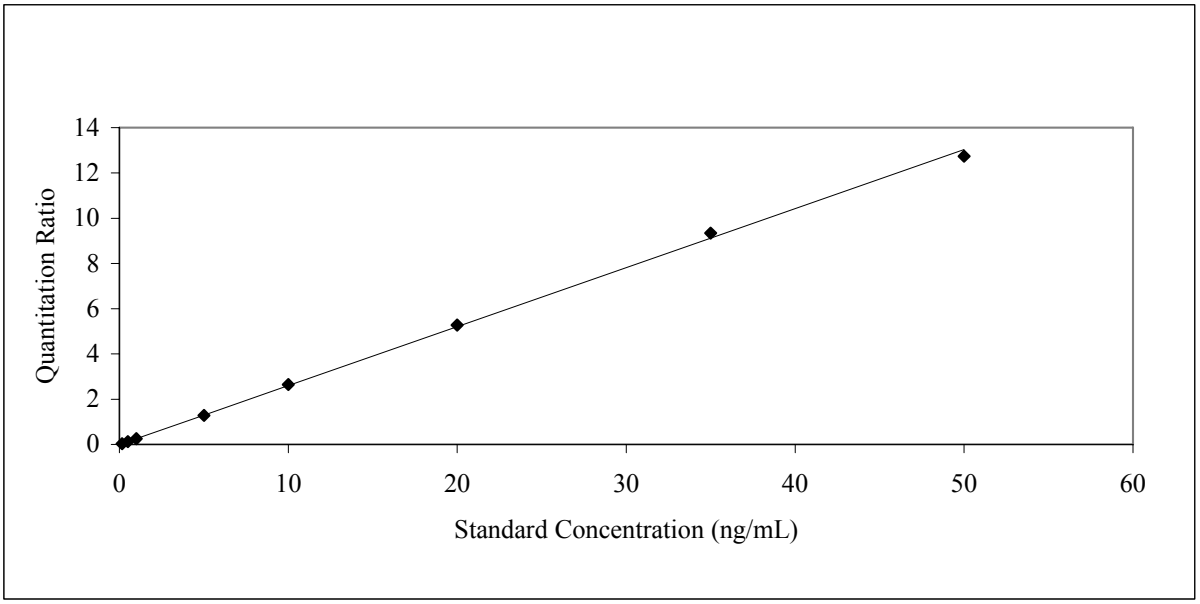


Figure 22. Typical Calibration Curve for the Determination of XDE-175-L in Wet Crops

Analytical Set I.D.: 041021 set 2b
Compound: XDE-175 N-demethl J

Calibration Data

Linear with 1/x Weighting

Slope =	0.2625
Intercept =	0.0030
r^2 =	0.9992

Standard Concentration (ng/mL)	Injection Number	Analyte Peak Area	ISTD Peak Area	Quantitation Ratio	Response Factor	Calculated Concentration	Percent of Theoretical
0.15	1	38180	823858	0.046	0.3090	0.16515	110
0.5	9	55457	426504	0.130	0.2601	0.48395	97
1	17	118038	451070	0.262	0.2617	0.98549	99
5	25	522115	394648	1.323	0.2646	5.02852	101
10	33	1123179	455449	2.466	0.2466	9.38315	94
20	41	2317491	444940	5.209	0.2604	19.83049	99
35	49	3545876	392903	9.025	0.2579	34.36850	98
50	50	4759780	352658	13.497	0.2699	51.40475	103

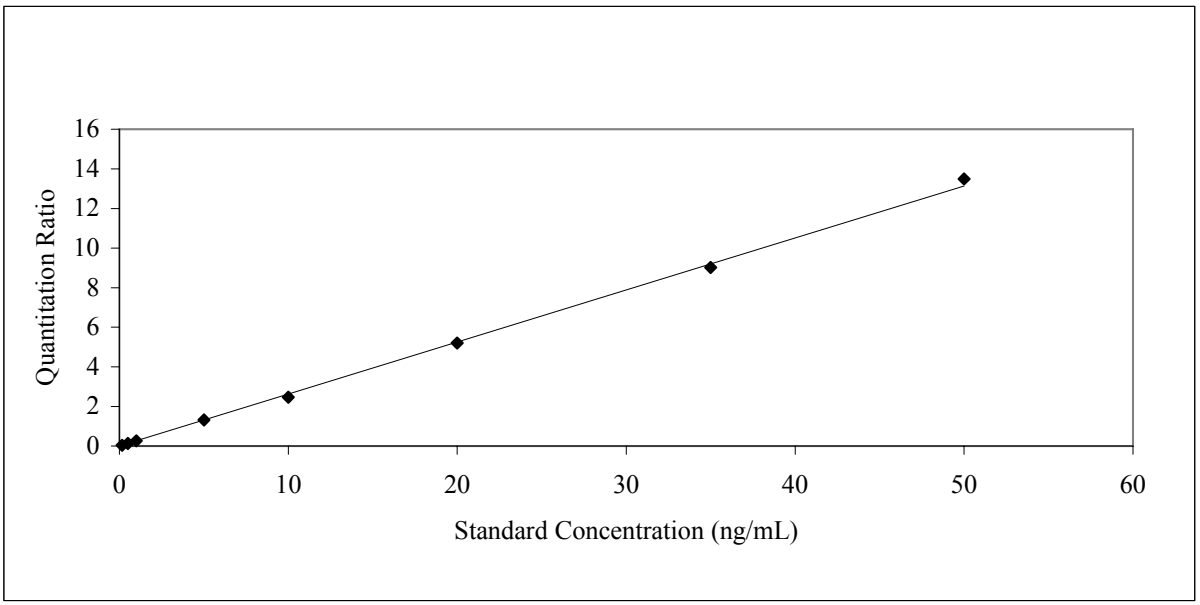


Figure 23. Typical Calibration Curve for the Determination of XDE-175-*N*-demethyl-J in Wet Crops

Analytical Set I.D.: 041021 set 2b
Compound: XDE-175 N-demethl L

Calibration Data

Linear with 1/x Weighting

Slope =	0.2709
Intercept =	0.0023
r^2 =	0.9998

Standard Concentration (ng/mL)	Injection Number	Analyte Peak Area	ISTD Peak Area	Quantitation Ratio	Response Factor	Calculated Concentration	Percent of Theoretical
0.15	1	20358	472565	0.043	0.2872	0.15065	100
0.5	9	45360	345467	0.131	0.2626	0.47634	95
1	17	99872	360985	0.277	0.2767	1.01300	101
5	25	468072	338900	1.381	0.2762	5.09053	102
10	33	989000	358467	2.759	0.2759	10.17717	102
20	41	1912725	356179	5.370	0.2685	19.81701	99
35	49	3017151	313131	9.635	0.2753	35.56365	102
50	50	4160993	311151	13.373	0.2675	49.36166	99

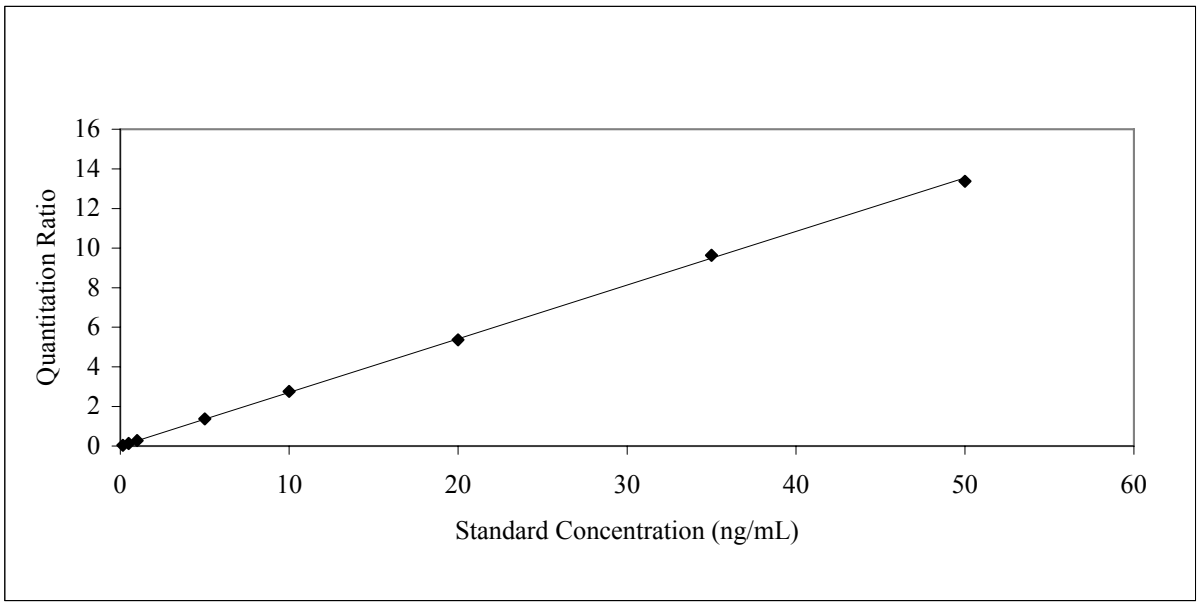


Figure 24. Typical Calibration Curve for the Determination of XDE-175-*N*-demethyl-L in Wet Crops

Analytical Set I.D.: 041021 set 2b
Compound: XDE-175 N-formyl J

Calibration Data

Linear with 1/x Weighting

Slope =	0.0281
Intercept =	0.0009
r^2 =	0.9990

Standard Concentration (ng/mL)	Injection Number	Analyte Peak Area	ISTD Peak Area	Quantitation Ratio	Response Factor	Calculated Concentration	Percent of Theoretical
0.15	1	2655	472565	0.006	0.0375	0.16921	113
0.5	9	4587	345467	0.013	0.0266	0.44146	88
1	17	10089	360985	0.028	0.0279	0.96293	96
5	25	47510	338900	0.140	0.0280	4.95247	99
10	33	103206	358467	0.288	0.0288	10.20314	102
20	41	202557	356179	0.569	0.0284	20.18354	101
35	49	320254	313131	1.023	0.0292	36.32269	104
50	50	424079	311151	1.363	0.0273	48.41456	97

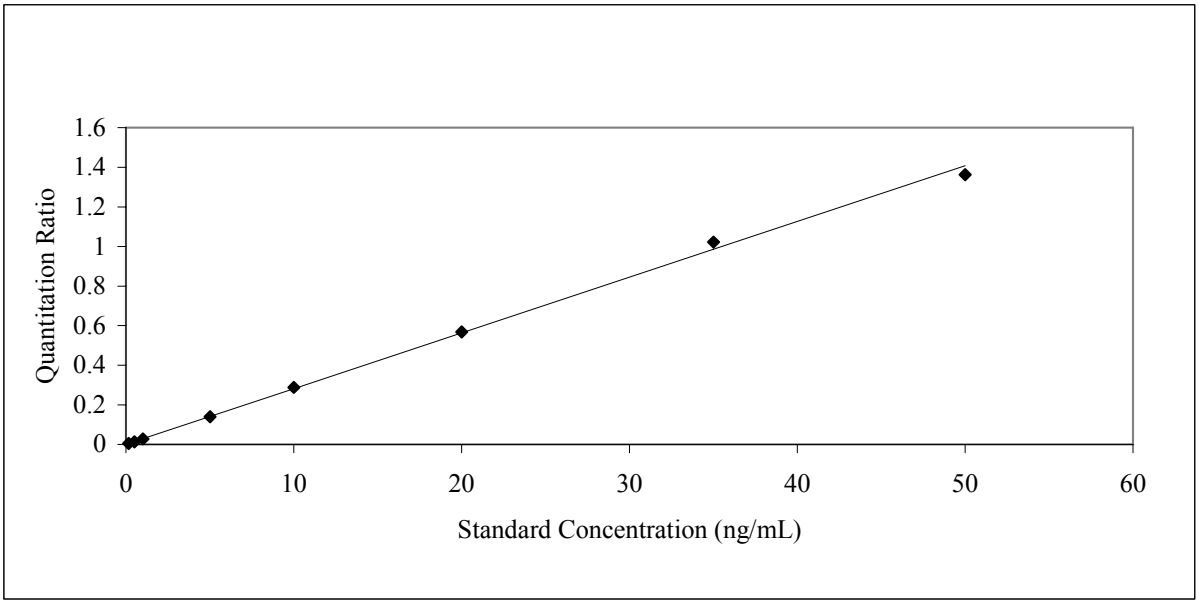


Figure 25. Typical Calibration Curve for the Determination of XDE-175-*N*-formyl-J in Wet Crops

Analytical Set I.D.: 041021 set 2b
Compound: XDE-175 N-formyl L

Calibration Data

Linear with 1/x Weighting

Slope =	0.0268
Intercept =	-0.0004
r^2 =	0.9997

Standard Concentration (ng/mL)	Injection Number	Analyte Peak Area	ISTD Peak Area	Quantitation Ratio	Response Factor	Calculated Concentration	Percent of Theoretical
0.15	1	1868	472565	0.004	0.0264	0.16307	109
0.5	9	4463	345467	0.013	0.0258	0.49780	100
1	17	8952	360985	0.025	0.0248	0.94133	94
5	25	45115	338900	0.133	0.0266	4.98547	100
10	33	94451	358467	0.263	0.0263	9.85248	99
20	41	185769	356179	0.522	0.0261	19.48746	97
35	49	298082	313131	0.952	0.0272	35.55526	102
50	50	417976	311151	1.343	0.0269	50.16712	100

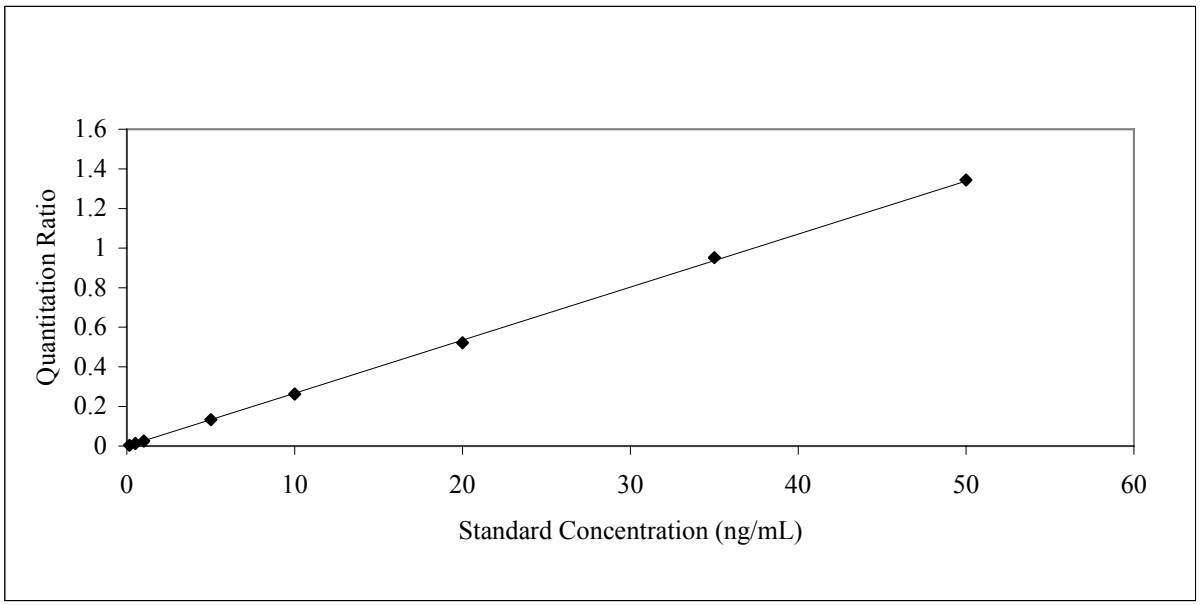


Figure 26. Typical Calibration Curve for the Determination of XDE-175-*N*-formyl-L in Wet Crops

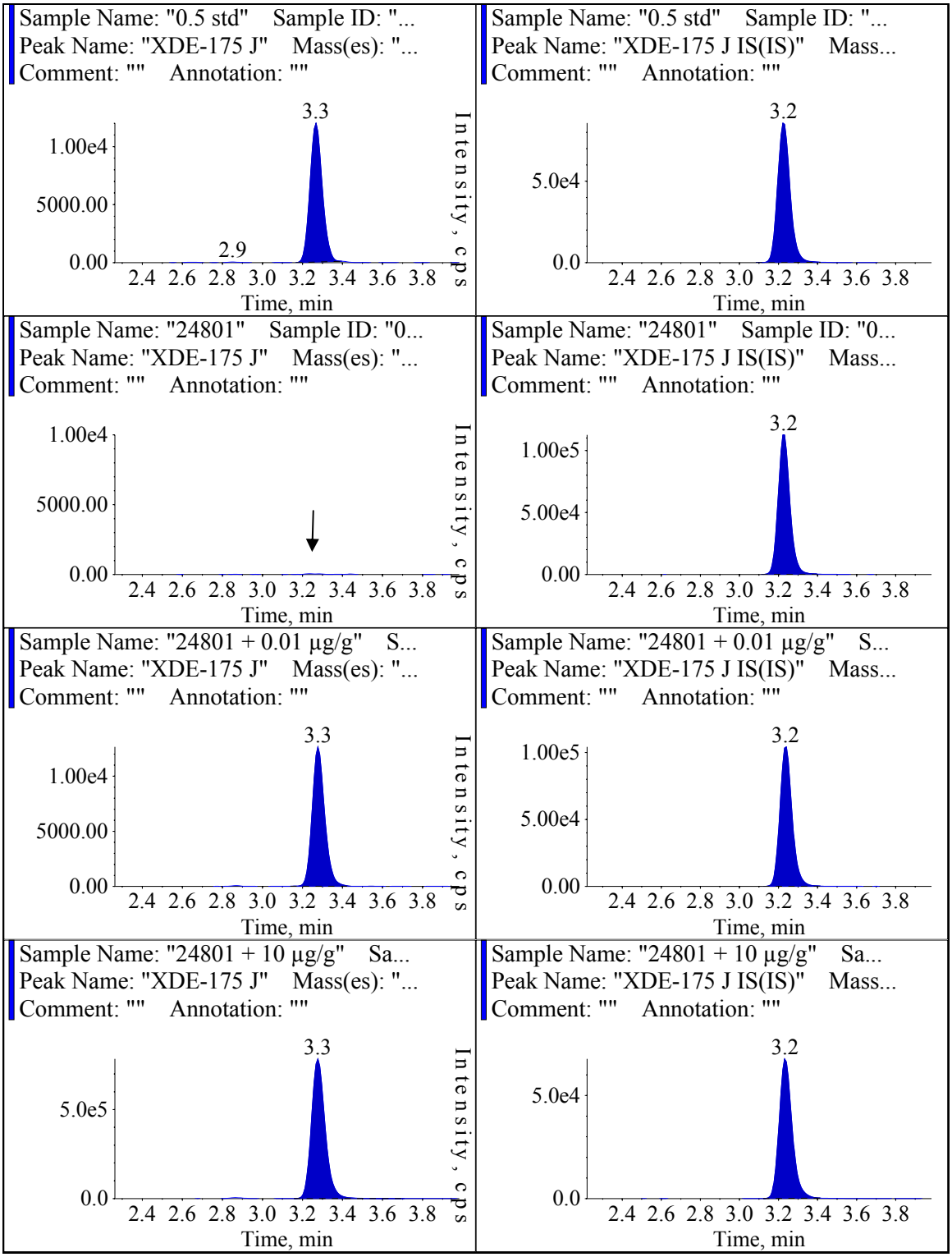


Figure 27. Typical MRM Chromatograms for the Determination of XDE-175-J in Lettuce

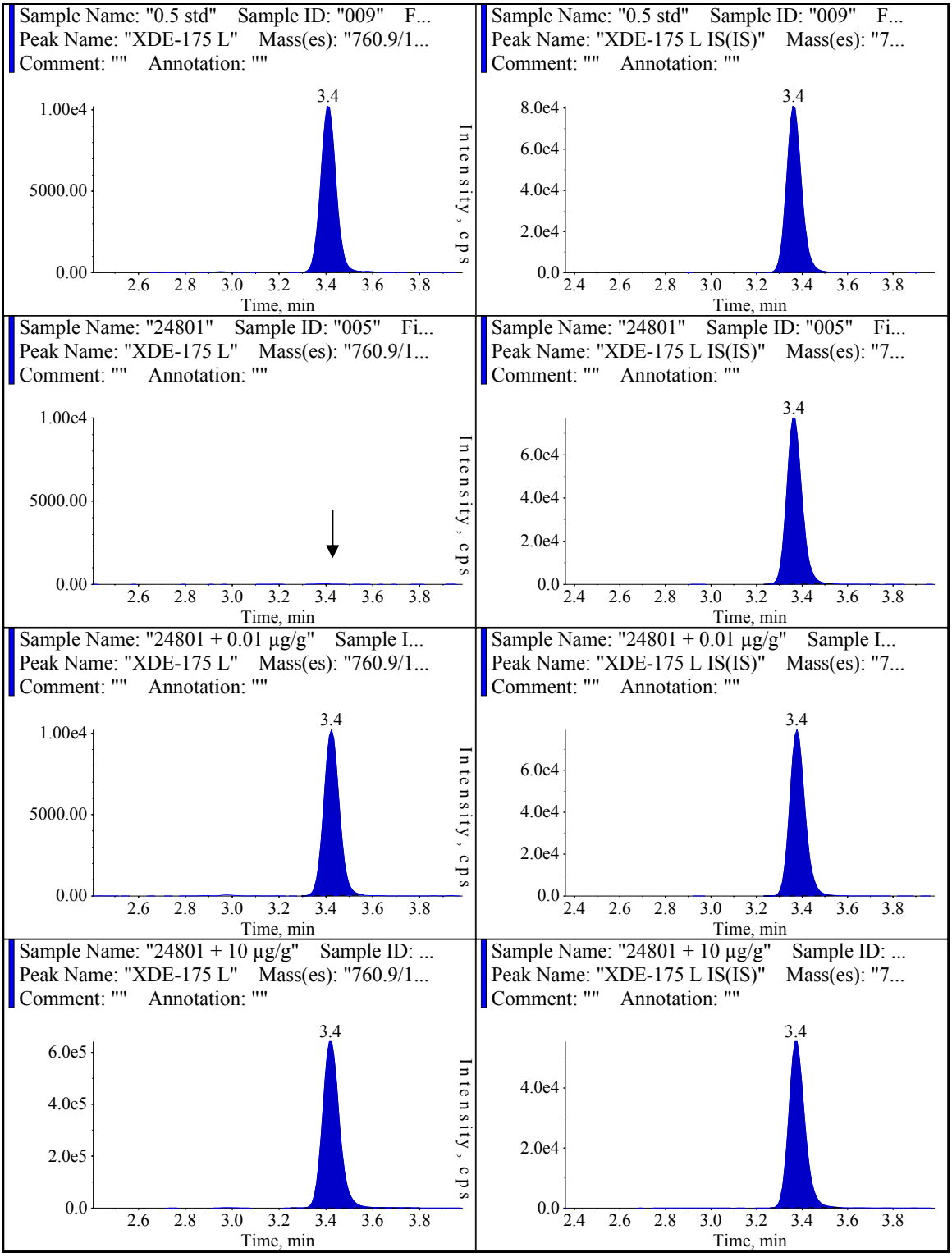


Figure 28. Typical MRM Chromatograms for the Determination of XDE-175-L in Lettuce

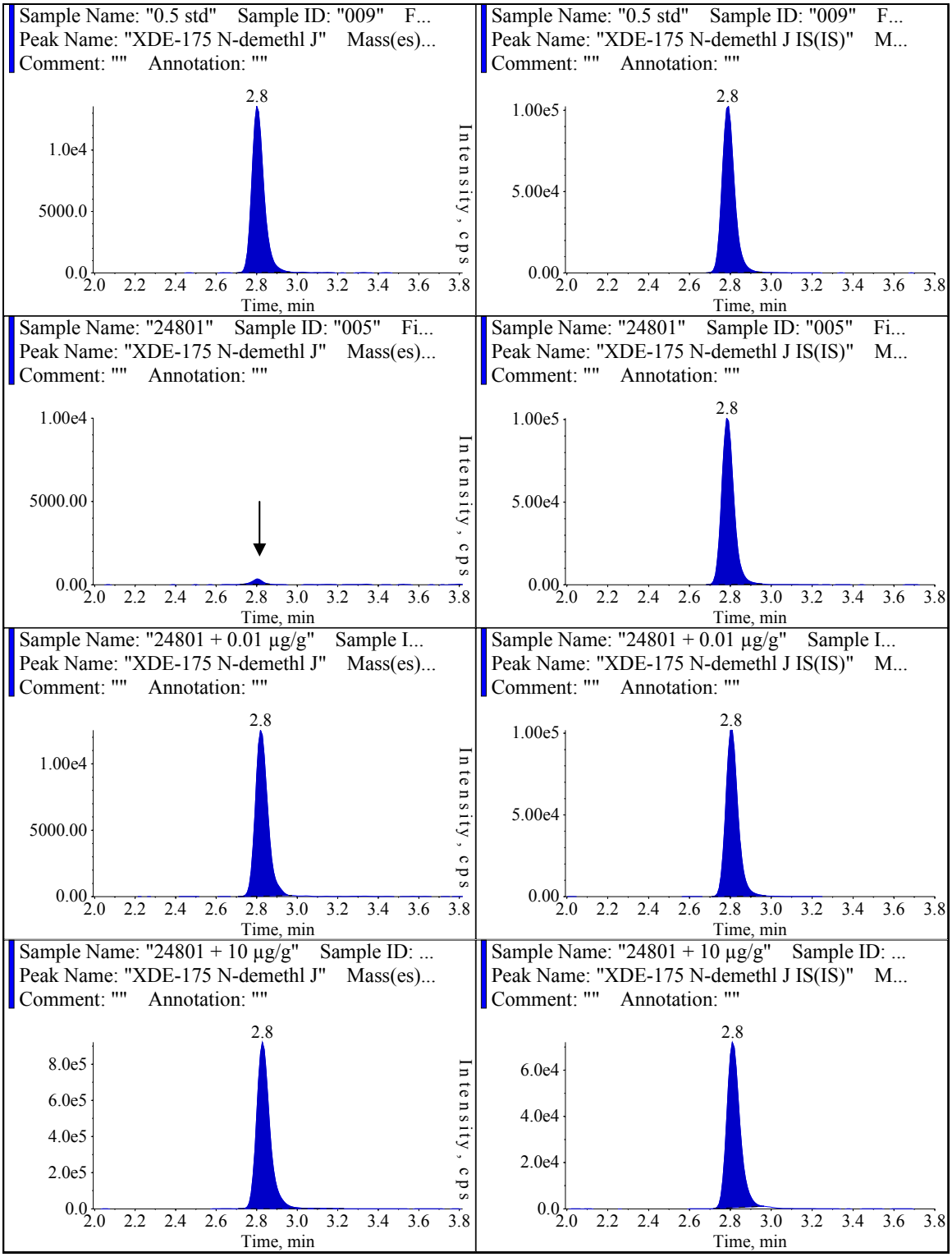


Figure 29. Typical MRM Chromatograms for the Determination of XDE-175-*N*-demethyl-J in Lettuce

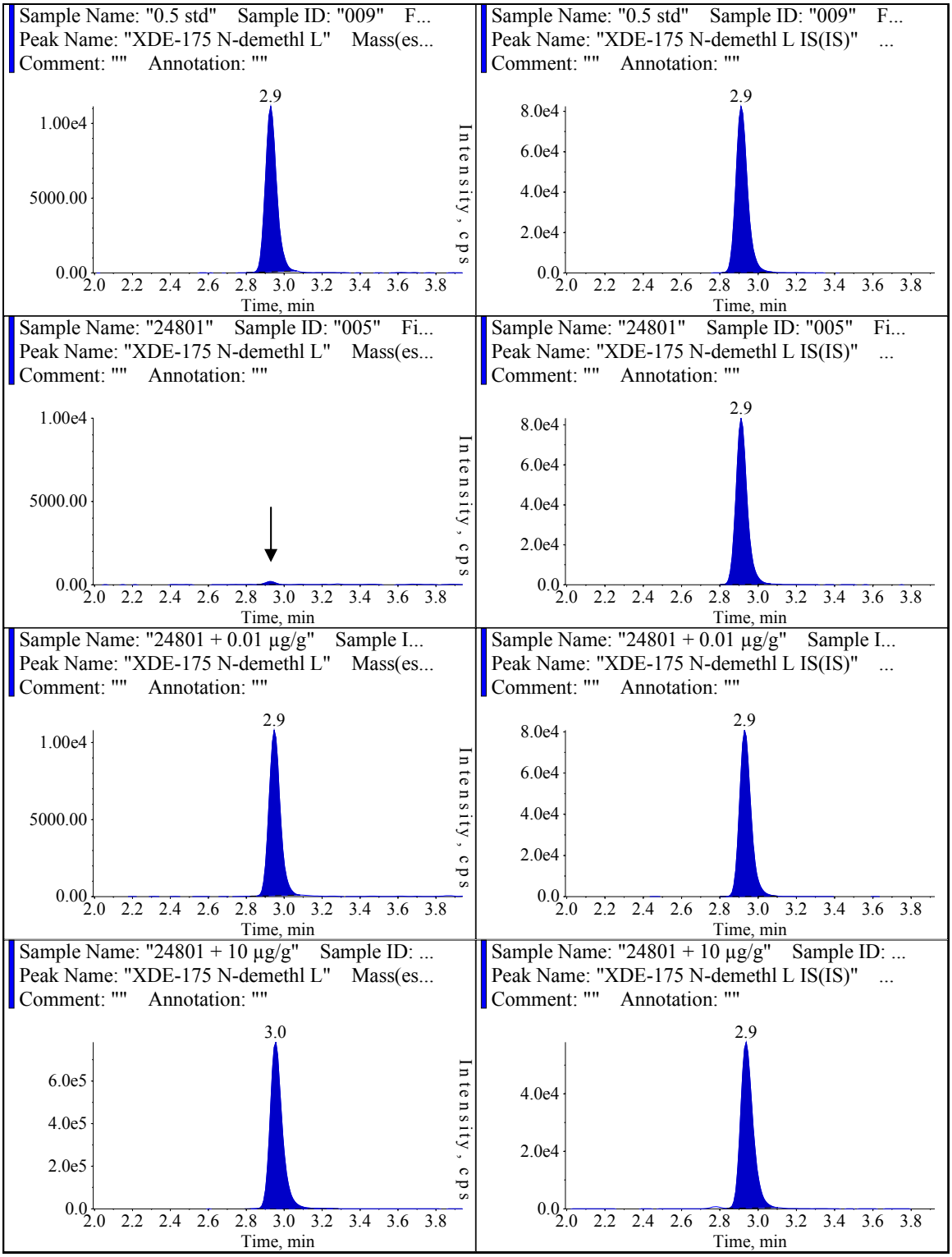


Figure 30. Typical MRM Chromatograms for the Determination of XDE-175-*N*-demethyl-L in Lettuce

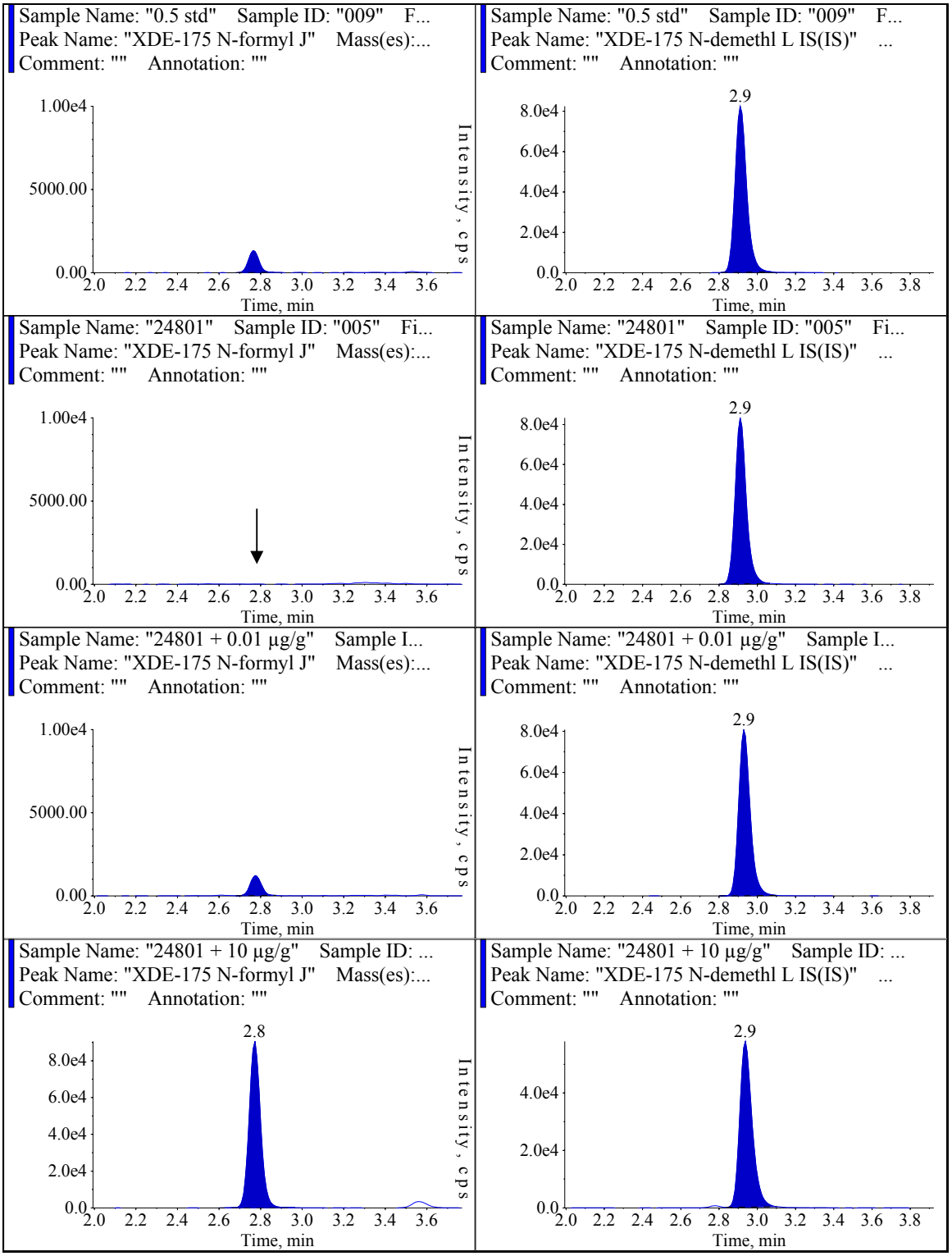


Figure 31. Typical MRM Chromatograms for the Determination of XDE-175-*N*-formyl-J in Lettuce

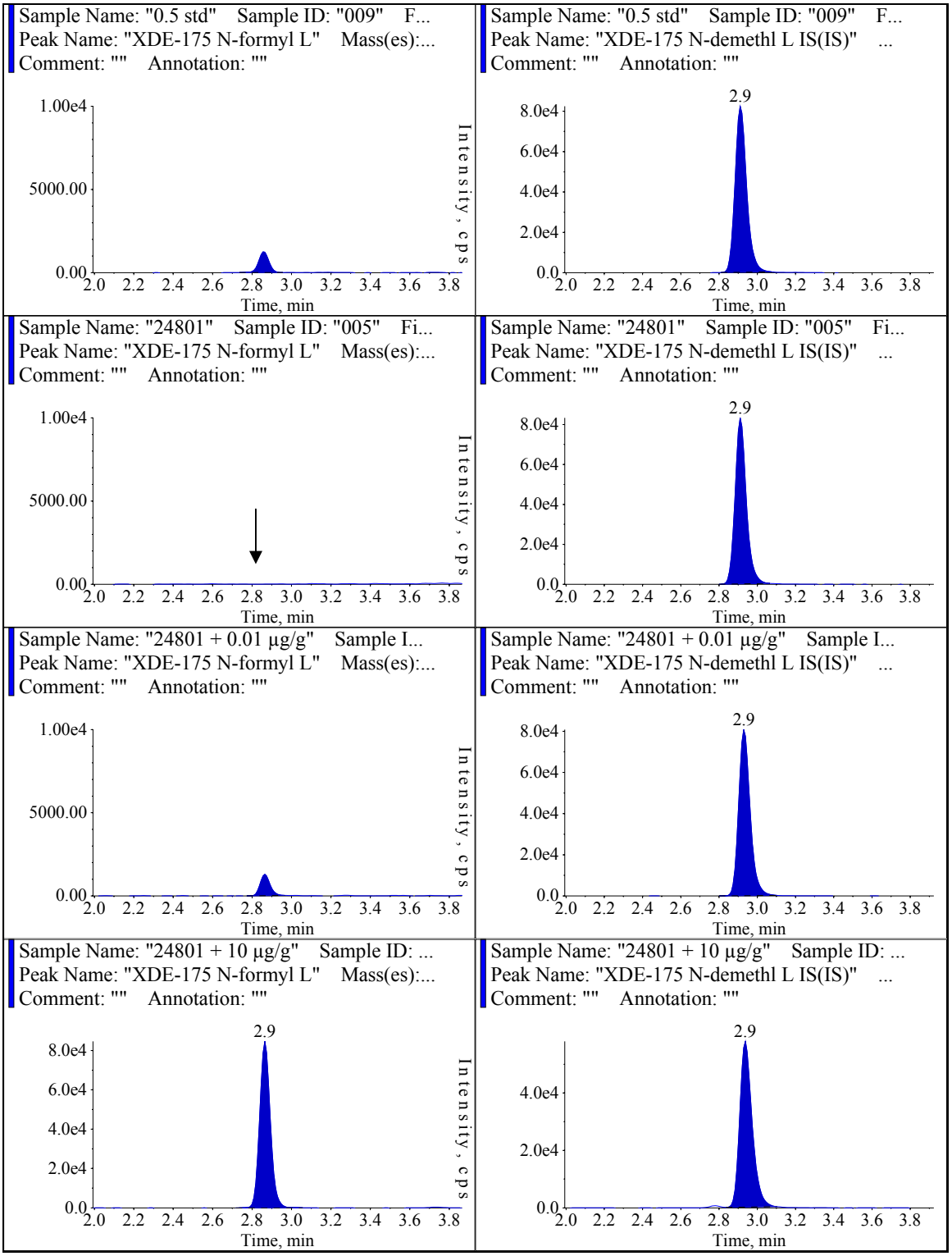


Figure 32. Typical MRM Chromatograms for the Determination of XDE-175-*N*-formyl-L in Lettuce

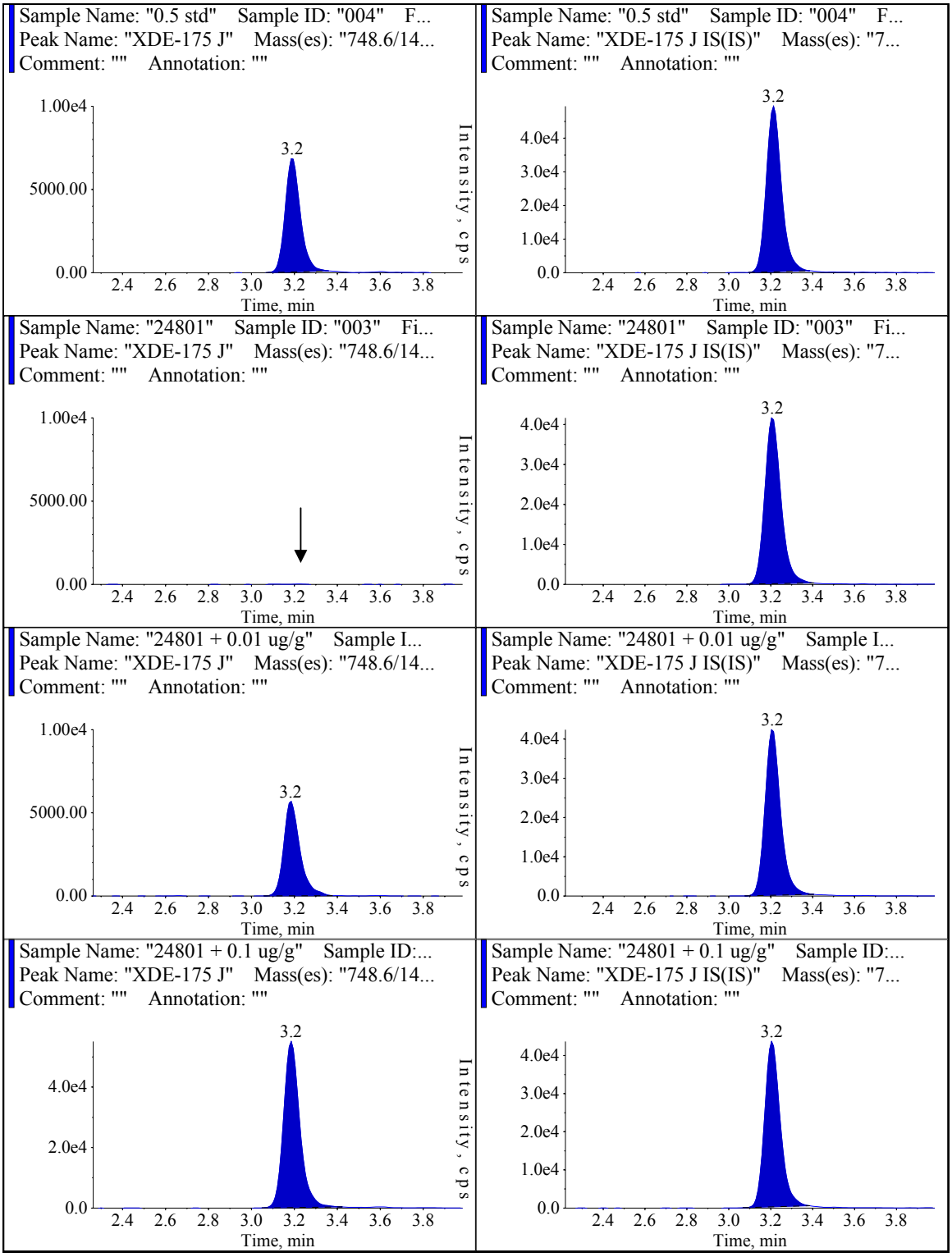


Figure 33. Typical MRM Chromatograms for the Confirmation of XDE-175-J Residues in Lettuce

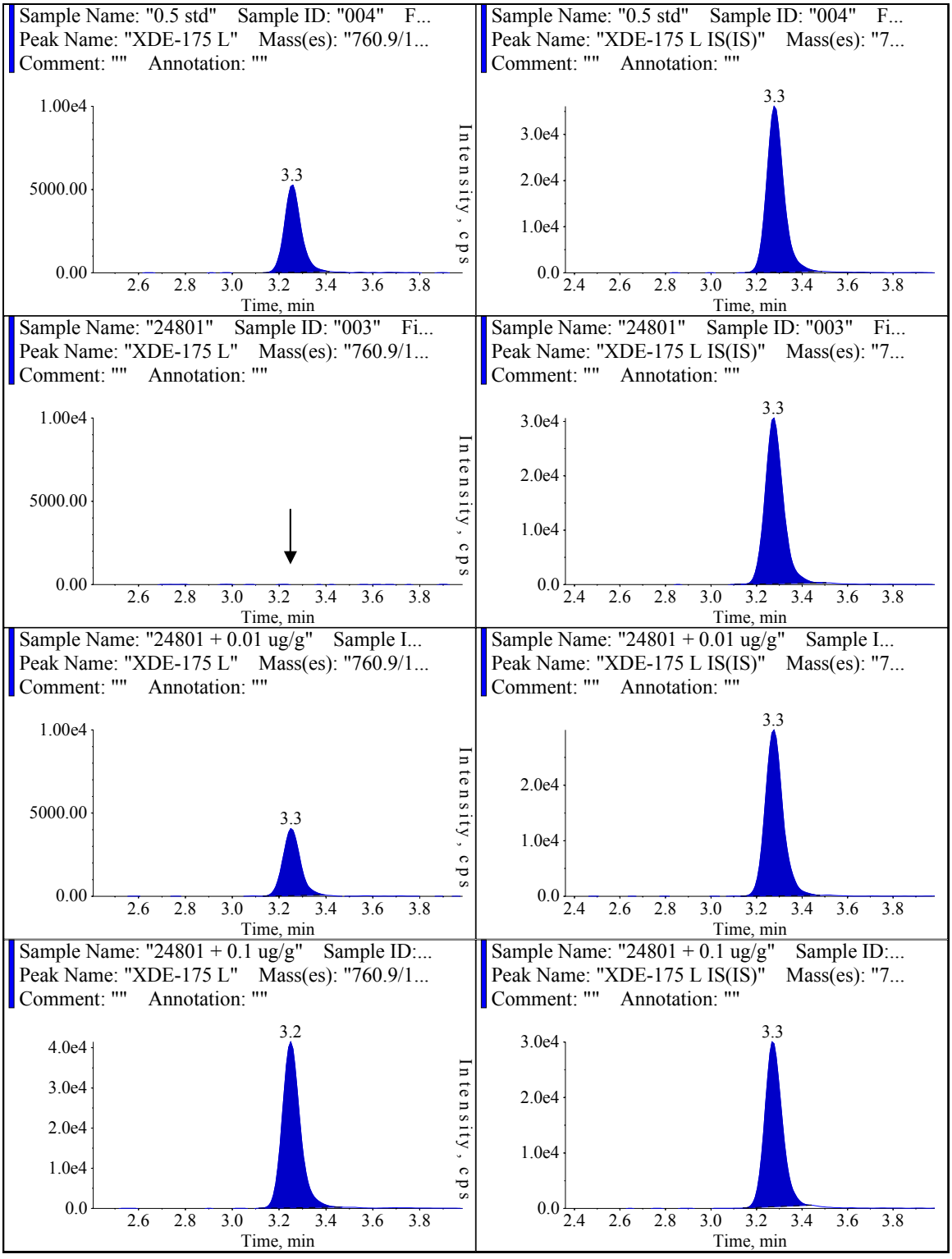


Figure 34. Typical MRM Chromatograms for the Confirmation of XDE-175-L Residues in Lettuce

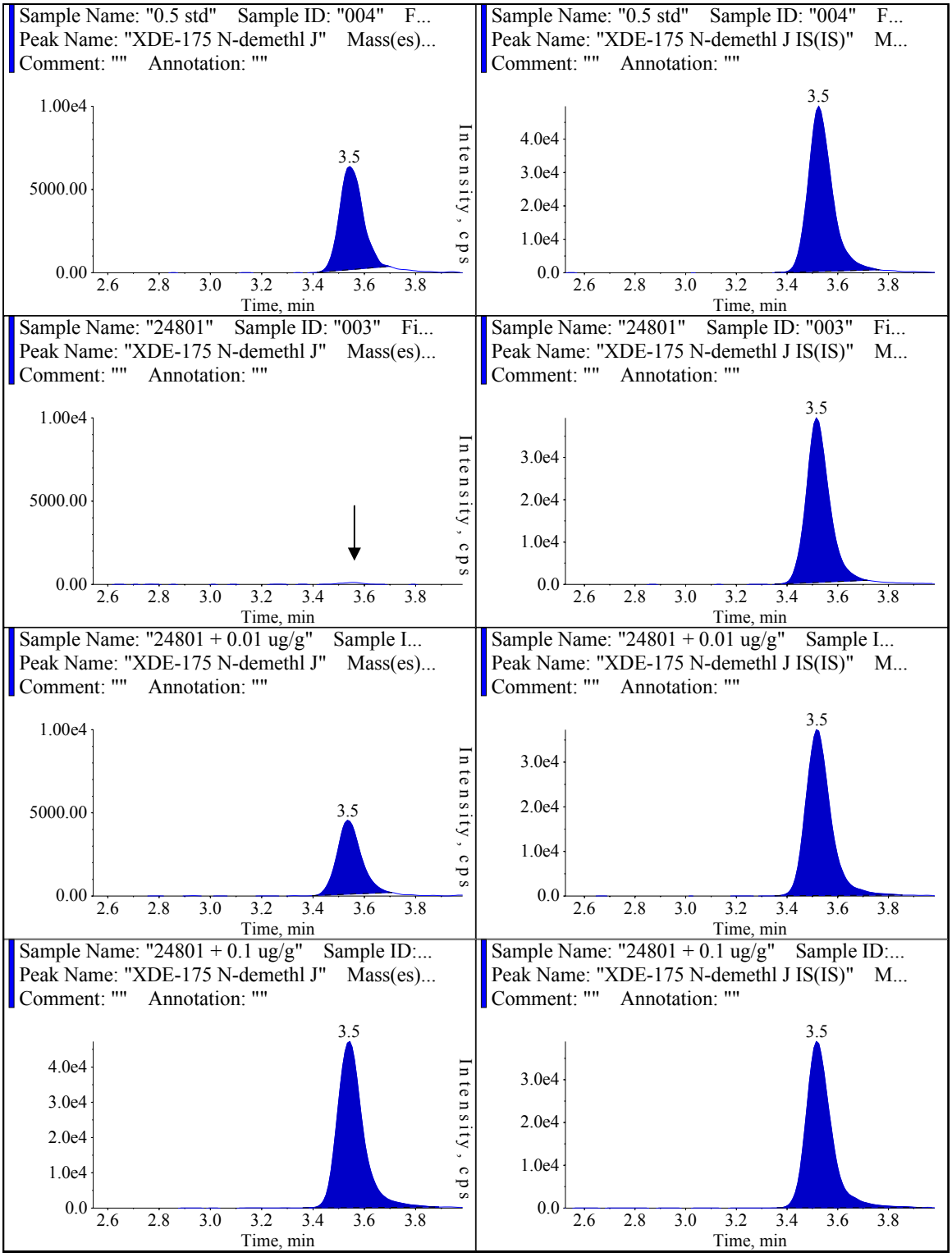


Figure 35. Typical MRM Chromatograms for the Confirmation of XDE-175-*N*-demethyl-J Residues in Lettuce

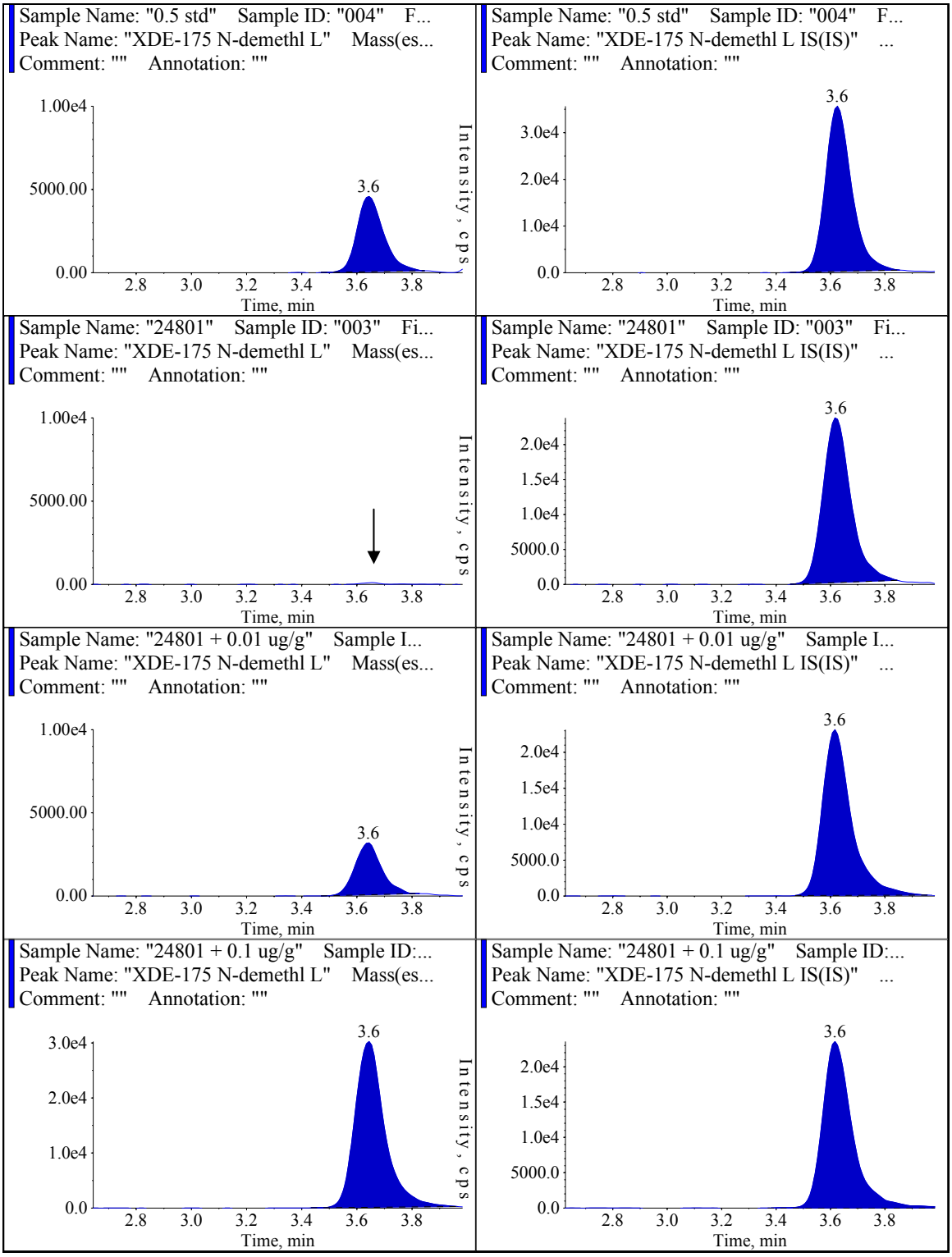


Figure 36. Typical MRM Chromatograms for the Confirmation of XDE-175-*N*-demethyl-L Residues in Lettuce

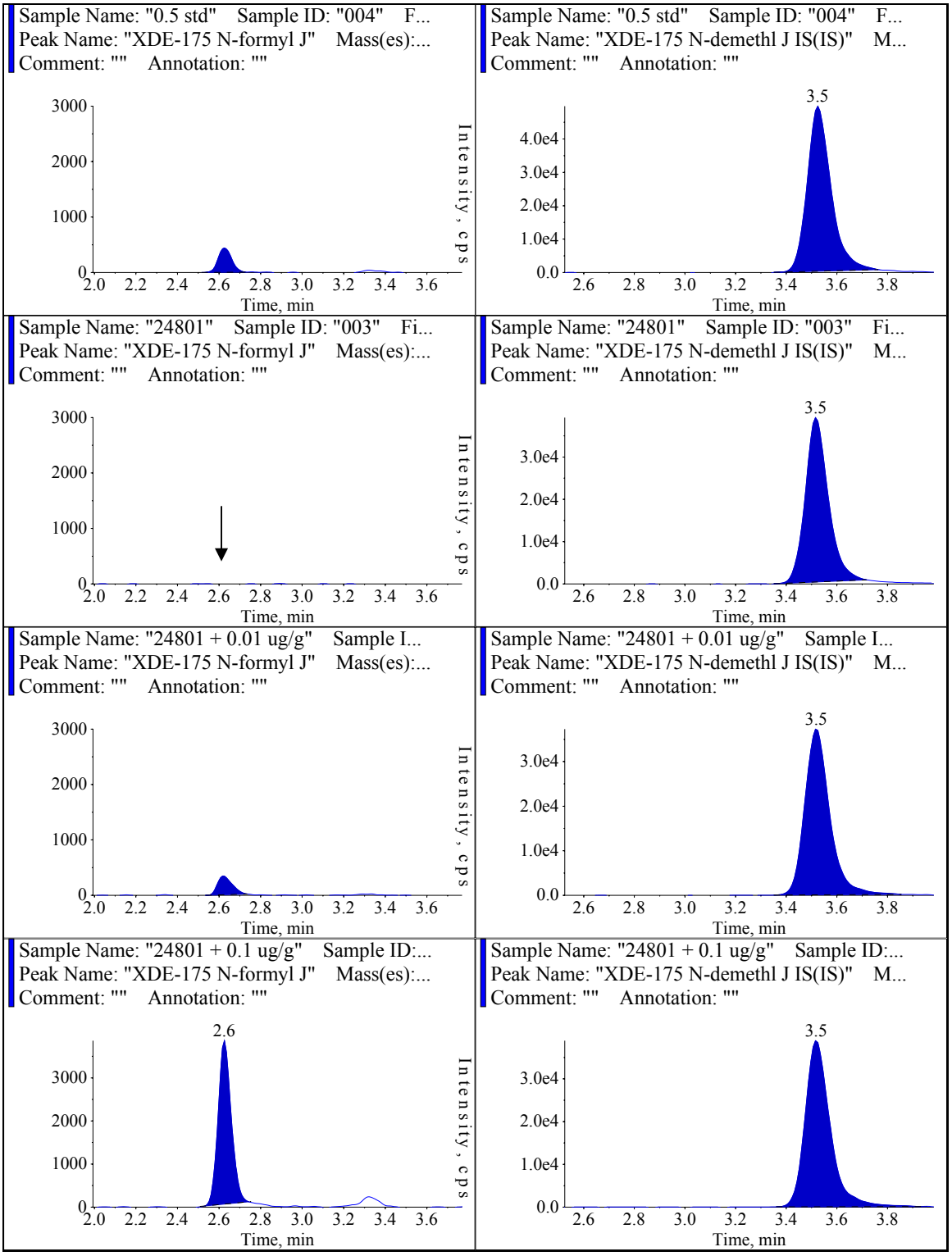


Figure 37. Typical MRM Chromatograms for the Confirmation of XDE-175-*N*-formyl-J Residues in Lettuce

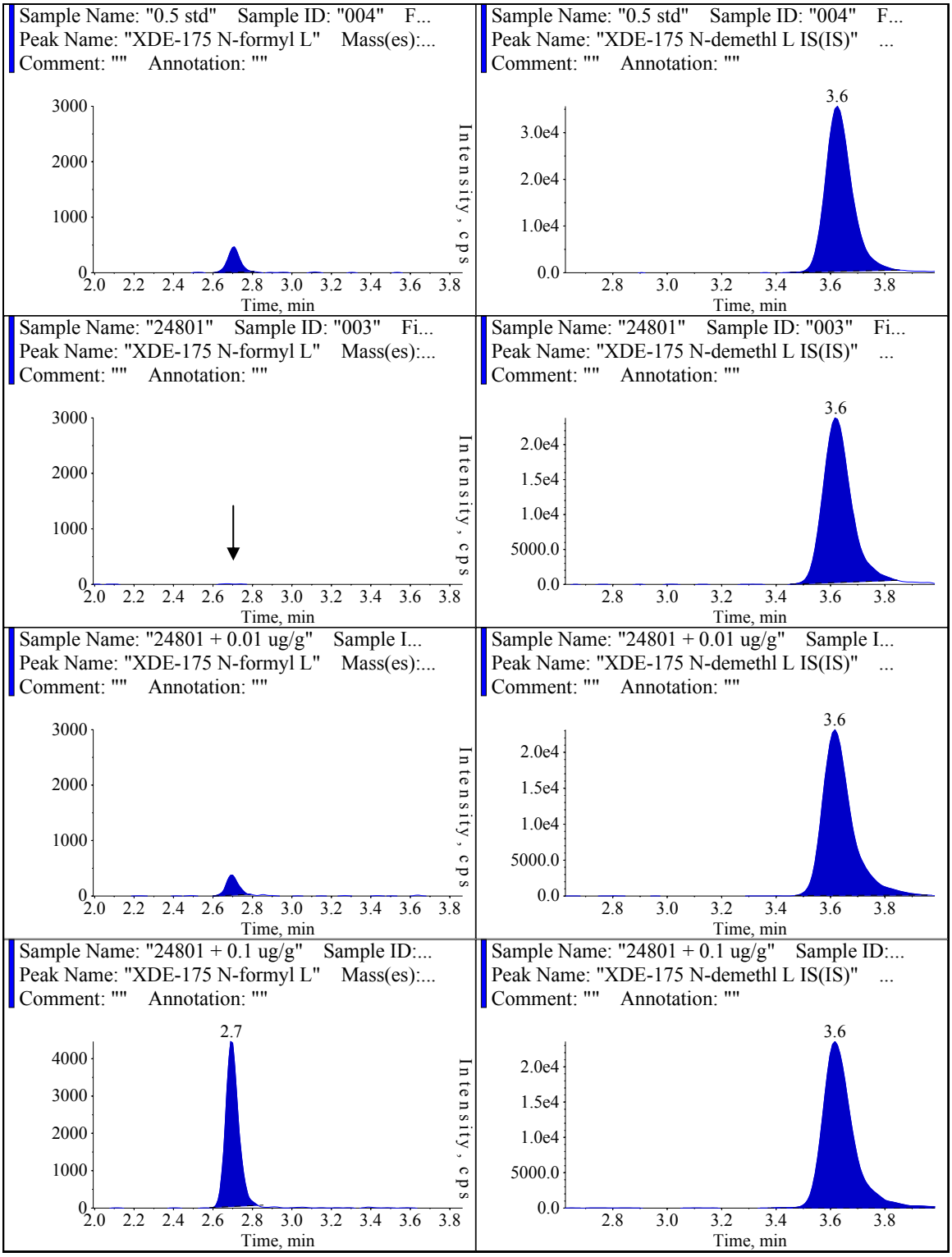


Figure 38. Typical MRM Chromatograms for the Confirmation of XDE-175-*N*-formyl-L Residues in Lettuce