BASF Corporation
BASF Crop Protection ▪ Research Triangle Park, North Carolina, USA

Final Report:

Validation of BASF Analytical Method D1302: "Determination of Topramezone (BAS 670 H) and Its Metabolite (M670H05) in Plant Matrices by LC-MS/MS"

BASF Study Number 698252

Data Requirements:

EPA Residue Chemistry Test Guidelines,
OPPTS 860.1340 Residue Analytical Method
Residue Analytical Methods, SANCO/825/00 rev.8.1 (16/11/2010)
OECD ENV/JM/MONO(2007)17 – Guidance Document
on Pesticide Residue Analytical Methods

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Study Completion Date:

April 19, 2013

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This report consists of 121 pages.

BASF Registration Document No. 2013/7001488

STATEMENT OF NO DATA CONFIDENTIALITY CLAIMS

No claim of confidentiality, on any basis whatsoever, is made for any information contained in this document. I acknowledge that information not designated as within the scope of FIFRA sec. 10(d)(1)(A), (B), or (C) and which pertains to a registered or previously registered pesticide is not entitled to confidential treatment and may be released to the public, subject to the provisions regarding disclosure to multinational entities under FIFRA 10(g).

Submitter: _____ 19 Apr 2013

Daniel J O'Byrne
BASF Corporation

Date

GOOD LABORATORY COMPLIANCE STATEMENT

This study was conducted in accordance with 40 CFR 160.

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Daniel J O'Byrne
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19 Apr 2013
Date

QUALITY ASSURANCE UNIT STATEMENT

Study Number: 698252

Name/Number of Test Items: Topramezone (BAS 670 H) and M670H05

Type of Study: Method Validation

The Quality Assurance Unit of the testing facility at BASF has inspected/audited the protocol, study, raw data and the final report and reported its findings to the study director and management.

Date of Inspection (Phase)	Date Reported to Study Director and Management
15 Apr 2013 (Protocol)	15 Apr 2013
15 Apr 2013 (Weighing, fortification, extraction)	17 Apr 2013
16 Apr 2013 (LC/MS/MS; matrix effect sample prep.)	17 Apr 2013
18 Apr 2013 (Raw data and final report)	18 Apr 2013

Signature of QAU

CERTIFICATION

We, the undersigned, hereby declare that this study was performed under our supervision according to the procedure described herein, and that this report provides a true and accurate record of the results obtained.

Author/
Study
Director: John E. Jones, III
BASF Crop Protection

4/19/13
Study Completion Date

Approved By: Samy Abdel-Baky
Dr. Samy Abdel-Baky, Group Leader
BASF Crop Protection

4-19-13
Date

ABSTRACT

BASF Analytical Method D1302 determines residues of topramezone (BAS 670 H) in plant commodities by high performance liquid chromatography/negative ion electrospray ionization tandem mass spectrometry (LC/MS/MS-ESI). The method is used as a determinative as well as a confirmatory technique. This study was conducted to validate BASF Analytical Method D1302 using various plant matrices - corn forage and grain, grape fruit, dried bean seed, and sunflower seed. A description of the methodology follows.

Briefly, residues of topramezone are extracted from crop samples by mechanical shaking with water, or for oily matrices (corn grain and sunflower seed), with acetonitrile:water (50:50, v/v). An aliquot is taken from the extract, acidified, partitioned with dichloromethane and then back-partitioned into aqueous ammonium formate (pH 10). The residues in the aqueous phase are then analyzed by LC/MS/MS using the negative ionization mode monitoring ion transitions *m/z* 362→334 for topramezone, and *m/z* 282→238 for M670H05 as the primary transitions for quantitation. Other secondary or "alternate" transitions are available for confirmatory purposes, or can also be used for quantitation. The results are calculated by direct comparison of the sample peak responses to those of external standards.

The method limit of quantitation (LOQ) for residues of topramezone in plant commodities is 0.01 ppm for each analyte. The method limit of detection (LOD) was set at 0.002 ppm, or approximately 20% of the LOQ. This is equivalent to 0.02 ng/mL, which was the low standard used for the analyses. For validation, untreated plant commodity samples were fortified with topramezone and M670H05 and analyzed according to the established method validation guidelines. The analytical sets each consisted of a reagent blank, two controls, five replicates fortified with each analyte at the method limit of quantitation (LOQ, 0.01 ppm), and five replicates fortified at a higher level, corresponding to 10X the LOQ (0.1 ppm) – for each plant matrix tested. In addition, untreated control extract samples (for each matrix) fortified with each analyte to evaluate any potential matrix effects. None (>25%) were noted.

The method validation was run successfully for each plant commodity and the LC/MS/MS ion transitions (primary and secondary, or "alternate") available for the method:

Mean recoveries of topramezone from plant matrices (corn forage, corn grain, grape fruit, dried bean seed, and sunflower seed) fortified at 0.01 and 0.1 ppm ranged from 84 to 99% (RSD, 1-11%) and from 93 to 113% (RSD, 3-13%), respectively, for the primary transition. For the secondary transition, mean recoveries of topramezone at 0.01 and 0.1 ppm ranged from 89 to 101% (RSD, 3-13%) and 93 to 110% (RSD, 3-9%), respectively.

Mean recoveries of M670H05 fortified in plant matrices (corn forage, corn grain, grape fruit, dried bean seed, and sunflower seed) at 0.01 and 0.1 ppm ranged from 83 to 101% (RSD, 3-5%) and 115 to 135% (RSD, 1-6%), respectively, for the primary transition. For the secondary transition, mean recoveries of M670H05 at 0.01 and 0.1 ppm ranged from 75 to 100% (RSD, 2-6%) and 112 to 132% (RSD, 2-7%), respectively.

Apparent residues of topramezone and M670H05 were below the method limit of detection in all of the control plant commodity samples (< 0.002 ppm).

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GENERAL INFORMATION

BASF Registration Document Number:	2013/7004251
BASF Study Number:	698252
Study Title:	Validation of BASF Analytical Method D1302: "Determination of Topramezone (BAS 670 H) and Its Metabolite (M670H05) in Plant Matrices by LC-MS/MS"
Sponsor:	BASF Corporation 26 Davis Drive Research Triangle Park, NC 27709
Study Director :	John E. Jones, III
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Testing Facility/Performing Laboratory:	BASF Crop Protection P.O. Box 13528 26 Davis Drive Research Triangle Park, NC 27709
Supervisor:	Dr. Samy Abdel-Baky

Study Dates:

Study Initiation Date:	April 15, 2013
Experimental Start Date:	April 15, 2013
Experimental Completion Date:	April 18, 2013
Study Completion Date:	Refer to Study Director's Signature, Page 5

I. INTRODUCTION

A. Background and Purpose of Study

Topramezone is an herbicide used to control grassy and broadleaf weeds in corn. The purpose of this study was to validate BASF Analytical Method No. D1302 which determines residues of topramezone (BAS 670 H and its metabolites) in plant commodities to a limit of quantitation (LOQ) of 0.01 ppm.

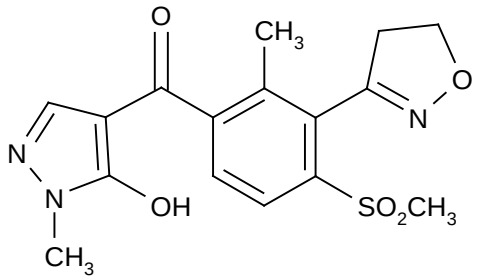
II. MATERIALS / METHODS

A. Test and Reference Items:

The test/reference substances were maintained frozen until use in this study. Characterization and purity were determined prior to the substances being used in this study. Details of these determinations are available to BASF and are located at BASF SE BASF Agricultural Center, Limburgerhof, Germany. The test/reference substances are shown below:

Test and Reference Substances (used for fortification and calibration)

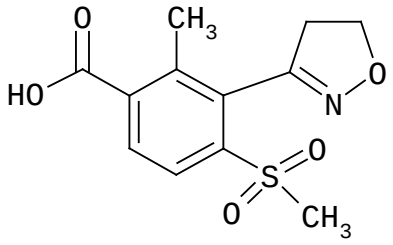
BASF Code Name:	BAS 670 H (Topramezone)
BASF Registry Number:	375080
IUPAC Name	[3-(4,5-Dihydro-isoxazol-3-yl)-4-methanesulfonyl-2-methyl-phenyl]-(5-hydroxy-1-methyl-1H-pyrazol-4-yl)-methanone
CAS Number:	210631-68-8
Molecular Formula:	$C_{16}H_{17}N_3O_5S$
Molecular Weight:	363.39
Lot Number	01882-216
Purity	99.8%
Certificate of Analysis	Refer to Appendix A, page 22
Expiration Date	June 1, 2019
Structural Formula:	



Test and Reference Substances (used for fortification and calibration) - continued

BASF Code Name:	M670H05
BASF Registry Number:	388010
IUPAC Name:	3-(4,5-dihydro-isoxazol-3-yl)-4-methanesulfonyl-2-methyl-benzoic acid
Molecular Formula:	$C_{12}H_{13}NO_5S$
Molecular Weight:	283.3
Lot Number	L76-134
Purity	99.3%
Certificate of Analysis	Refer to Appendix A, page 22
Expiration Date	October 01, 2016

Structural Formula:



The test/reference substances were used in the study to generate data for both instrument and method performance. Quantitation of residues in all samples was achieved using calibration curves calculated by linear regression of instrument responses for the reference substances. The performance of the instrument was evaluated during each injection set.

The standard solutions of topramezone were refrigerated during their use in this study. The stock solutions containing topramezone and M670H05 are made in acetonitrile and methanol, respectively (1 mg/mL each). Aliquots of these stock solutions are combined and serial diluted with acetonitrile to produce intermediate solutions and/or fortification solutions. The calibration solutions are prepared by serial dilution of an intermediate or fortification solution with 4mM ammonium formate in water (pH 10).

The stock solutions are typically made fresh every three months, and fortification and calibration solutions are made fresh every month. The stability of topramezone and its metabolites in standard solutions has been determined (Reference 1 and 2). Preparation and dilution data forms pertaining to the stock and working solutions are located in the raw data. Example standard dilution and use information are provided in Appendix F.

In this study, the standards were used for the analyses of the recovery samples within the stated period of stability. The sample extracts were stored under refrigeration prior to analysis (if stored) and were analyzed within 0-1 day of extraction; acceptable recoveries

obtained during analysis of all samples demonstrated the storage stability of each analyte in the extracts prior to analysis.

B. Test System

The test systems for this study were corn forage and grain, grape fruit, dried bean seed, and sunflower seed.

C. Identification of Samples

The test systems were untreated control crop RAC samples from previously conducted magnitude of the residue studies:

Sample Description	Original Sample Name/ID	Study of Origin (BASF Study no.)	New Sample Name
Corn, forage	R0803890001	347433	698252-0001
Corn, grain	R0803830002	347433	698252-0002
Bean, dried seed	R1000310001	369501	698252-0003
Grape, fruit	R0800440001	249262	698252-0004
Sunflower, seed	R0801520001	347437	698252-0005

The control plant commodity samples were homogenized to a consistency appropriate for analysis and held frozen (generally at temperatures of -20 ± 10 ºC) under the studies of origin and prior to use in this study. Each analysis set was uniquely identified with a Master Sheet Number, which consisted of the study number plus a unique number (e.g., 698252-1). The test system samples were assigned unique numbers according to SOP 10.04.XX and these were recorded in each analytical set or "Master Sheet" (e.g., corn forage sample 698252-0001 "Fort. 1", from Master Sheet, 698252-1).

D. Route of Administration

The test substance was applied to the test system as an analytical standard solution by volumetric pipette to ensure precise delivery of a small amount of test substance.

E. Method of Analysis

The method used in this validation study was BASF Analytical Method D1302: "Determination of Topramezone (BAS 670 H) and Its Metabolite (M670H05) in Plant Matrices by LC-MS/MS" (Reference 1). The method is attached as Appendix B. A brief description of the methodology follows.

The residues of topramezone are extracted from crop samples (5 g each) by mechanical shaking with water or, for oily matrices (corn grain and sunflower seed), with acetonitrile:water (50:50, v/v). An aliquot is taken from the extract, acidified, partitioned with dichloromethane and then back-partitioned into aqueous ammonium formate (pH 10). The residues in the aqueous phase are then determined by LC/MS/MS using the negative ionization mode monitoring ion transitions *m/z* 362→334 for topramezone and *m/z* 282→238 for M670H05 as the primary transitions for quantitation. Other secondary or "alternate" transitions are available for confirmatory purposes, or can also be used for quantitation. The results are calculated by direct comparison of the sample peak responses to those of external standards.

F. Limit of Quantitation / Limit of Detection

The method LOQ for residues of topramezone in plant commodities is 0.01 ppm for each analyte. The method limit of detection set at 0.002 ppm, or approximately 20% of the LOQ. This is equivalent to 0.02 ng/mL, which was the low standard used for the analyses.

G. Validation of Method

To validate the method, a reagent blank, two controls, five fortifications in each plant matrix at the limit of quantitation (0.01 ppm), and five fortifications in each plant matrix corresponding to 10X the method limit of quantitation (0.1 ppm), were analyzed. Acceptable recovery (mean) ranges were to be 70-120 %. Acceptability of mean recoveries outside of this range, should the situation arise, was to be evaluated on an individual basis.

In addition, possible matrix effects were tested by analyzing an aliquot of extract from unfortified control samples worked up through the method and then fortified with a known amount of analyte. These instrument recovery samples were prepared by using 2.5 mL of the 0.1 ng/mL LC/MS/MS standard (instead of 2.5 mL of 4 mM ammonium formate in water) to partition an aliquot of the dichloromethane layer (partitioning step) of a control sample and were compared to the standard curve.

III. RESULTS

The method validation was run successfully for each of the plant commodities and all tested transitions (primary and secondary) available for the method.

A. Method Recoveries

Topramezone. See Table 1 (page 16) for summary of individual recoveries.

Mean recoveries of topramezone from plant matrices (corn forage, corn grain, grape fruit, dried bean seed, and sunflower seed) fortified at 0.01 and 0.1 ppm ranged from 84 to 99% (RSD, 1-11%) and from 93 to 113% (RSD, 3-13%), respectively, for the primary transition. For the secondary transition, mean recoveries of topramezone at 0.01 and 0.1 ppm ranged from 89 to 101% (RSD, 3-13%) and 93 to 110% (RSD, 3-9%), respectively.

M670H05. See Table 2 (page 18) for summary of individual recoveries.

Mean recoveries of M670H05 fortified in plant matrices (corn forage, corn grain, grape fruit, dried bean seed, and sunflower seed) at 0.01 and 0.1 ppm ranged from 83 to 101% (RSD, 3-5%) and 115 to 135% (RSD, 1-6%), respectively, for the primary transition. For the secondary transition, mean recoveries of M670H05 at 0.01 and 0.1 ppm ranged from 75 to 100% (RSD, 2-6%) and 112 to 132% (RSD, 2-7%), respectively.

The method-detector response was linear over the 0.02-0.4 ng/mL range ($r = 0.9918$ to 0.9997).

Apparent residues of topramezone and M670H05 were below the method limit of detection in all of the control plant commodity samples (< 0.002 ppm).

Acceptable recovery data for “matrix effects” samples, also described as “instrument recovery” samples were obtained for each analyte across all matrices using both transitions. As no matrix effects were observed (i.e., were insignificant based on recoveries in the acceptable range of 70-120%), calibration in solvent alone is adequate and matrix-matched calibration standards are not required.

B. Method Modifications

The recovery data for this study was to be generated using Draft Technical Procedure D1302, version dated April 12, 2013. Any changes that occurred during the course of the method validation were to be documented in the raw data, in a protocol change, and then incorporated into the final version of the method. Refer to “Protocol Changes” (p. 21) for a description of any method modifications required to complete the method validation. The final method is attached to this document as Appendix B.

Table 1. Summary of Recoveries of Topramezone from Plant Commodities

Matrix	Fortification Levels (ppm)	N	Recovery (%)	Average Recovery (%)	Standard Deviation	%RSD[a]
Corn, forage	Primary Transition[b] (*m/z* 362→334)					
	0.01	5	91, 91, 91, 92, 94	92	1	1
	0.1	5	109, 114, 121, 108, 114	113	5	5
	Overall	**10**	**Range, 91 - 121**	**103**	**12**	**12**
	Secondary Transition[c] (*m/z* 362→318)					
	0.01	5	98, 93, 98, 103, 98	98	4	4
	0.1	5	112, 109, 114, 108, 105	110	4	3
	Overall	**10**	**Range, 93 - 114**	**104**	**7**	**7**
Corn, grain	Primary Transition[b] (*m/z* 362→334)					
	0.01	5	79, 84, 86, 91, 79	84	5	6
	0.1	5	96, 99, 101, 103, 94	99	4	4
	Overall	**10**	**Range, 79 - 103**	**91**	**9**	**10**
	Secondary Transition[c] (*m/z* 362→318)					
	0.01	5	87, 92, 91, 89, 84	89	3	4
	0.1	5	107, 103, 107, 104, 100	104	3	3
	Overall	**10**	**Range, 84 - 107**	**96**	**9**	**9**
Grape, fruit	Primary Transition[b] (*m/z* 362→334)					
	0.01	5	92, 98, 93, 92, 91	93	3	3
	0.1	5	113, 109, 105, 108, 109	109	3	3
	Overall	**10**	**Range, 91 - 113**	**101**	**9**	**9**
	Secondary Transition[c] (*m/z* 362→318)					
	0.01	5	91, 96, 90, 91, 88	91	3	3
	0.1	5	101, 111, 112, 108, 110	108	4	4
	Overall	**10**	**Range, 88 - 112**	**100**	**10**	**10**

[a] Relative Standard Deviation = (Standard Deviation ÷ Average Recovery) x 100.
[b] The primary transition, which would typically be used for quantification of residues in samples.
[c] The secondary (or alternate) transition, which would typically be used for confirmatory purposes.

Table 1. Summary of Recoveries of Topramezone from Plant Commodities (cont.)

Matrix	Fortification Levels (ppm)	N	Recovery (%)	Average Recovery (%)	Standard Deviation	%RSD[a]
Bean, dried seed	Primary Transition[b] (*m/z* 362→334)					
	0.01	5	99, 97, 95, 104, 99	99	3	3
	0.1	5	124, 107, 110, 109, 86	107	14	13
	Overall	**10**	**Range, 86 - 124**	**103**	**10**	**10**
	Secondary Transition[c] (*m/z* 362→318)					
	0.01	5	112, 94, 84, 104, 111	101	12	12
	0.1	5	117, 107, 113, 108, 91	107	10	9
	Overall	**10**	**Range, 84 - 117**	**104**	**11**	**10**
Sunflower, seed	Primary Transition[b] (*m/z* 362→334)					
	0.01	5	92, 111, 98, 87, [272][d]	97	10	11
	0.1	5	97, 92, 92, 91, 92	93	2	3
	Overall	**10**	**Range, 87 - 111**	**95**	**7**	**7**
	Secondary Transition[c] (*m/z* 362→318)					
	0.01	5	91, 112, 87, 86, [264][d]	94	12	13
	0.1	5	100, 94, 87, 95, 91	93	5	5
	Overall	**10**	**Range, 86 - 112**	**94**	**8**	**9**

[a] Relative Standard Deviation = (Standard Deviation ÷ Average Recovery) x 100.
[b] The primary transition, which would typically be used for quantification of residues in samples.
[c] The secondary (or alternate) transition, which would typically be used for confirmatory purposes.
[d] This anomalous recovery result is not included in the descriptive statistics (range, mean, SD and RSD).

Table 2. Summary of Recoveries of M670H05 from Plant Commodities

Matrix	Fortification Levels (ppm)	N	Recovery (%)	Average Recovery (%)	Standard Deviation	%RSD[a]
Corn, forage	Primary Transition[b] (*m/z* 282→238)					
	0.01	5	84, 87, 83, 78, 83	83	3	4
	0.1	5	135, 133, 135, 131, 133	133	2	1
	Overall	**10**	**Range, 78 - 135**	**108**	**27**	**25**
	Secondary Transition[c] (*m/z* 282→79)					
	0.01	5	87, 85, 78, 93, 85	86	5	6
	0.1	5	133, 129, 137, 132, 131	132	3	2
	Overall	**10**	**Range, 78 - 137**	**109**	**25**	**23**
Corn, grain	Primary Transition[b] (*m/z* 282→238)					
	0.01	5	81, 85, 82, 86, 80	83	3	3
	0.1	5	120, 124, 122, 123, 122	122	1	1
	Overall	**10**	**Range, 80 - 124**	**103**	**21**	**20**
	Secondary Transition[c] (*m/z* 282→79)					
	0.01	5	79, 82, 85, 80, 84	82	3	3
	0.1	5	115, 115, 118, 121, 120	118	3	2
	Overall	**10**	**Range, 79 - 121**	**100**	**19**	**19**
Grape, fruit	Primary Transition[b] (*m/z* 282→238)					
	0.01	5	85, 89, 88, 82, 86	86	3	3
	0.1	5	134, 134, 133, 135, 137	135	2	1
	Overall	**10**	**Range, 82 - 137**	**110**	**26**	**23**
	Secondary Transition[c] (*m/z* 282→79)					
	0.01	5	84, 88, 84, 87, 86	86	2	2
	0.1	5	130, 134, 132, 136, 128	132	3	2
	Overall	**10**	**Range, 84 - 136**	**109**	**24**	**22**

[a] Relative Standard Deviation = (Standard Deviation ÷ Average Recovery) x 100.
[b] The primary transition, which would typically be used for quantification of residues in samples.
[c] The secondary (or alternate) transition, which would typically be used for confirmatory purposes.

Table 2. Summary of Recoveries of M670H05 from Plant Commodities (continued)

Matrix	Fortification Levels (ppm)	N	Recovery (%)	Average Recovery (%)	Standard Deviation	%RSD[a]
Bean, dried seed	Primary Transition[b] (*m/z* 282→238)					
	0.01	5	98, 97, 99, 108, 101	101	4	4
	0.1	5	130, 131, 131, 126, 114	126	7	6
	Overall	**10**	**Range, 97 - 131**	**114**	**15**	**13**
	Secondary Transition[c] (*m/z* 282→79)					
	0.01	5	103, 100, 94, 102, 100	100	3	3
	0.1	5	130, 133, 134, 127, 112	127	9	7
	Overall	**10**	**Range, 94 - 134**	**114**	**16**	**14**
Sunflower, seed	Primary Transition[b] (*m/z* 282→238)					
	0.01	5	86, 82, 80, 88, 78	83	4	5
	0.1	5	112, 118, 112, 116, 115	115	3	2
	Overall	**10**	**Range, 78 - 118**	**99**	**17**	**17**
	Secondary Transition[c] (*m/z* 282→79)					
	0.01	5	75, 72, 71, 82, 75	75	4	6
	0.1	5	110, 115, 113, 112, 111	112	2	2
	Overall	**10**	**Range, 71 - 115**	**94**	**20**	**21**

[a] Relative Standard Deviation = (Standard Deviation ÷ Average Recovery) x 100.
[b] The primary transition, which would typically be used for quantification of residues in samples.
[c] The secondary (or alternate) transition, which would typically be used for confirmatory purposes.

An example calculation is included in Appendix C. Detailed analytical data such as supporting raw data necessary for re-calculations, standards and calibration curve data are provided in Appendix D. Typical standard curves and chromatography are shown in Appendix E.

IV. STATISTICS AND DATA INTEGRITY

Statistical treatment of the data included determinations of averages, standard deviation and relative standard deviation (RSD) for the procedural recoveries. Additionally, a linear regression analysis was performed to define each standard curve.

The statistical calculations throughout this report were performed using an automated computer spreadsheet (Microsoft Excel®) and were rounded for presentation purposes. Slight differences may be noted in hand calculations using the recoveries presented in the tables. These are due to rounding and have no effect on the scientific conclusions presented in this report. The detailed residue data found in the appendices cited above may be consulted for confirmation of the calculated results.

Several measures were taken to ensure the quality of the study results. The quality assurance unit at BASF inspected the analytical procedures for compliance with Good Laboratory Practices that included adherence to the protocol. The dates inspected are detailed in the quality assurance unit statement. Study samples and test and reference items were maintained in secured (i.e. pad-locked) storage with limited access. Freezer temperatures were continuously monitored by electronic means.

V. DISCUSSION/CONCLUSIONS

The method validation of BASF Analytical Method D1302, which measures residues of topramezone in plant commodities, was accomplished successfully for each plant commodity and the LC/MS/MS ion transitions (primary and secondary, or “alternate”) available for the method.

Mean recoveries of topramezone from plant matrices (corn forage, corn grain, grape fruit, dried bean seed, and sunflower seed) fortified at 0.01 and 0.1 ppm ranged from 84 to 99% (RSD, 1-11%) and from 93 to 113% (RSD, 3-13%), respectively, for the primary transition. For the secondary transition, mean recoveries of topramezone at 0.01 and 0.1 ppm ranged from 89 to 101% (RSD, 3-13%) and 93 to 110% (RSD, 3-9%), respectively.

Mean recoveries of M670H05 fortified in plant matrices (corn forage, corn grain, grape fruit, dried bean seed, and sunflower seed) at 0.01 and 0.1 ppm ranged from 83 to 101% (RSD, 3-5%) and 115 to 135% (RSD, 1-6%), respectively, for the primary transition. For the secondary transition, mean recoveries of M670H05 at 0.01 and 0.1 ppm ranged from 75 to 100% (RSD, 2-6%) and 112 to 132% (RSD, 2-7%), respectively.

Apparent residues of topramezone and its metabolite were below the method limit of detection (< 0.002 ppm) in all control plant commodity samples.

VI. PROTOCOL CHANGES

There were several protocol changes issued for this study:

1. This change attached a corrected version of the method to the protocol. In Section 3.5 (Preparation for Measurement) there were several typographical errors and these were corrected. In addition, acceptable stopping points for the method were added).

2. This change documented that an additional analyte topramezone metabolite M670H02) was included in the standard mixtures for LC/MS/MS analysis, but was not used for the analysis. M670H02 was needed for a different analysis involving topramezone that utilized the same standard concentrations.

3. This change documented the following: (a) the stock solution for M670H05 was dissolved in methanol. The method implies that the stock solution should be made in acetonitrile. Methanol is an appropriate solvent for the stock solution of M670H05. The final version of the method will be updated to reflect this. (b) Standards used for fortifications were not assayed before use in the study. Due to time constraints, the analysis was started with existing standards. The fortification solutions were assayed after analysis and the results included in the raw data. (c) The “Study of Origin” for dry bean was different than stated in the original Protocol, due to a typographical error (the Study of Origin for dry bean 369501). (d) Fortifications solutions were made in acetonitrile instead of water. Acetonitrile is an appropriate solvent for the analytes, however, the method will not be updated to include it for the fortification solutions to avoid confusion in standard preparation. (e) The ion source temperature was different than stated in the method. The method had a typographical error in Sections 3.9.1 and 3.9.2 for temperature. The method has been updated

   None of the changes noted above affected the validity of the study.

VII. ARCHIVING / LOCATION OF THE RAW DATA

The final report, protocol, raw data pertaining to this study, and a retention sample of test/reference substances will be stored in the archives of the sponsor (BASF Corporation, BASF Crop Protection, 26 Davis Drive, Research Triangle Park, NC, 27709, USA) at least for the period of time specified in the GLP regulations. Specimens will be stored after completion of the final report for the period of time specified in the GLP regulations.

VIII. REFERENCES

1. Jones, III, John E. (2012). BASF Analytical Method D1302: "Determination of Topramezone (BAS 670 H) and Its Metabolite (M670H05) in Plant Matrices by LC-MS/MS”, Version April 15, 2013.

2. Jones, J. Determination of the Stability of BAS 670 H and M670H05 in Various Solvents. BASF Study Code 56755. BASF DocID: 2002/5003843. U.S. EPA MRID 45902408

Appendix A. Reference Substance Certificate of Analysis

BASF
The Chemical Company

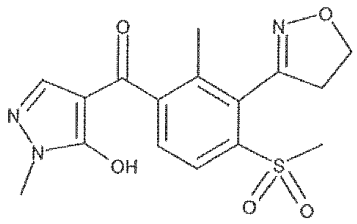
BASF SE
APR/DA - Analytics

BASF SE - Crop Protection - Speyerer Strasse 2, D-67117 Limburgerhof, Germany

Certificate of Analysis

BAS Code :	**BAS 670 H**	**Common Name :**	**Topramezone**
Reg.No. :	375080	Batch No. :	01882-216
Substance Type :	PAI	Date of Production :	June 25, 2003
Date of Initial Analysis :	July 18, 2003	Study Code :	56730_1
Date of Reanalysis :	June 07, 2011	Study Code :	ASAP11_106

Purity : 99,8 % (tolerance ± 1.0%)



CL-No.	
CAS No.	210631-68-8
Core Project	670H
Internal (Metabolite) Code	M670H00
Molecular Formula	$C_{16}H_{17}N_3O_5S$
Molecular Weight	363,4

IUPAC-Name :	[3-(4,5-dihydro-isoxazol-3-yl)-4-methylsulfonyl-2-methylphenyl](5-hydroxy-1-methyl-1H-pyrazol-4-yl)methanone
Determination by :	HPLC, determination of water (coulometric)
Homogeneity :	given

Additional Information

Storage Advice :	keep at room temperature (typically +25°C) or cooler
Expiration Date :	June 01, 2019

Recipients should ensure that the label information on the corresponding substance container(s) correspond(s) with that on this Certificate of Analysis

Study Director : Deissroth, Steffen

Study Completion Date : July 05, 2011

Issued on : July 07, 2011

Issued by : S. Deissroth

This document has been computer generated and is based on the GLP-studies cited above
Released July 05, 2011 by Deissroth, Steffen

BASF
The Chemical Company

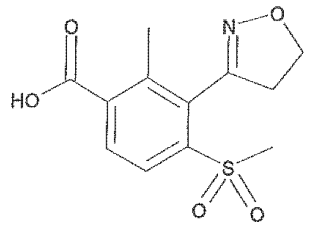
BASF SE
APR/DA - Global Analytics

BASF Agricultural Center Limburgerhof - P.O. Box 120, D-67114 Limburgerhof, Germany

Certificate of Analysis

Reg.No. :	388010	**Batch No. :**	L76-134
Substance Type :	ME	**Date of Production :**	September 16, 2008
Date of Initial Analysis :	September 22, 2008	**Study Code :**	357073_1

Purity : 99,3 % (tolerance ± 1.0%)



CL-No.
CAS No. 223646-24-0
Core Project 670H
Internal (Metabolite) Code M670H05
Molecular Formula $C_{12}H_{13}NO_5S$
Molecular Weight 283,3

IUPAC-Name : 3-(4,5-dihydro-isoxazol-3-yl)-4-methylsulfonyl-2-methyl-benzoic acid
Determination by : HPLC, determination of water (coulometric)
Homogeneity : given

Additional Information
Storage Advice : keep at room temperature (typically +25°C) or cooler
Expiration Date : October 01, 2016

Recipients should ensure that the label information on the corresponding substance container(s) correspond(s) with that on this Certificate of Analysis

Study Director : Baumann, Ernst, Dr. **Study Completion Date :** October 31, 2008

Issued on : March 23, 2010 **Issued by :** S Schweiz

This document has been computer generated and is based on the GLP-studies cited above
Released October 31, 2008 by Baumann, Ernst, Dr.

Appendix B. BASF Analytical Method Number D1302

BASF
The Chemical Company

Technical Procedure:

Determination of Topramezone(BAS 670 H) and Its Metabolite M670H05(Reg. No. 388010) in Plant Matrices by LC-MS/MS

BASF Method Number D1302

Author(s)
John E. Jones III

Date
April 15, 2013

Test Facility
BASF Crop Protection
26 Davis Drive
Research Triangle Park, NC 27709
USA

Number of Pages
19

ABSTRACT

BASF Method D1302 is developed to determine the residues of topramezone (BAS 670 H) and its metabolite, M670H05 in various plant matrices using LC-MS/MS at BASF Corporation, Research Triangle Park, N.C.

A brief description of the method is described below:

The analytes are extracted from plant matrices using water (50:50 acetonitrile:water for oily matrices). An aliquot is taken from the extract, acidified, partitioned with dichloromethane and then back-partitioned into pH 10 aqueous ammonium formate. The aqueous phase is then injected directly into LC/MS/MS for determination of BAS 670 H and M670H05. The limit of quantitation of the method for the analytes in all matrices is 0.01 mg/kg. The limit of detection for in plant matrices for the analytes is 0.002 mg/kg.

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DEFINITIONS AND ACRONYMS

Sample Set:	A group of samples that are extracted and cleaned up at the same time using the same method represented.
Untreated Sample:	A sample that has not been treated with the test substance.
Control Sample:	Usually an untreated sample used for fortification experiments (can be acquired from same study or from a different source).
Unknown Sample:	The samples with unknown residues.
Treated Sample:	A sample that has been treated with the test substance.
Blank:	Solvent, solution or mobile phase injected together with a sample set.
Reagent Blank:	A complete analysis conducted using solvents and reagents only in absence of any sample. Also known as blank of reagents or procedural blank. This sample is analyzed within the sample set in order to evaluate possible contamination on chemicals/reagents.
Procedural Recovery:	A control sample to which a known amount of analyte has been added before sample work up. This sample is then carried through the method and analyzed with the unknown samples in order to determine the reliability of the method.
Instrument Recovery:	A control sample which is carried through the method and to which a known amount of analyte has been added before injection. This sample is analyzed within the sample set in order to evaluate the matrix effect in the instrument.
Analytical Run:	A group of samples that undergo a determinative measurement on an analytical instrument (eg. LC/MS/MS) in a defined and continuous sequence under identical instrumental conditions.
Limit of Quantitation (LOQ):	Lowest tested concentration of the analyte in a sample that can be determined with acceptable accuracy and precision according to the method.
Limit of Detection (LOD):	Concentration of analyte equivalent to a defined percentage of the limit of quantitation of the method (e.g 20% of LOQ). At this concentration, the analyte must be qualitatively detectable in sample matrix (analyte peak height at least 3-5 x baseline noise).

1 INTRODUCTION

Topramezone (BAS 670 H) is an herbicide used against grassy and broadleaf weeds in corn. The technical procedure D1302 offers the ability to determine BAS 670 H and M670H05 residues in plant matrices including corn forage, dry bean, grape, and soybean seed. The method D1302 has a limit of quantitation of 0.01 mg/kg for the active ingredient and its metabolite in the plant matrices.

Method D1302 was successfully tested during method development in corn forage, corn grain, dry bean, grape, and sunflower seed for all analytes.

This method was developed at BASF Corporation, Research Triangle Park, N.C.

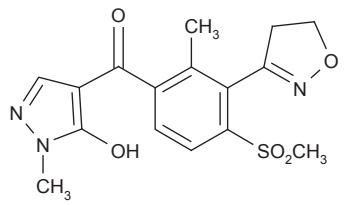
2 MATERIALS

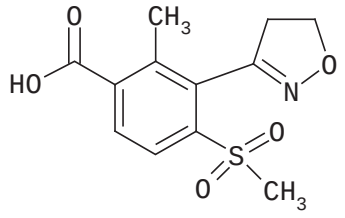
2.1 Safety

The test and reference items, as well as the chemicals required for this analysis, should be handled in accordance with good industrial hygiene and safety practice. Avoid contact with the skin, eyes and clothing. Wearing of closed work clothing is recommended. Remove contaminated clothing. Store work clothing separately. Keep away from food, drink and plant feed stuffs. No eating, drinking, smoking or tobacco use at the place of work. Hands and/or face should be washed before breaks and at the end of the shift. Details are given in the Materials Safety Data Sheets (MSDS) of the individual substances. All procedures involving organic solvents should be performed in a well-ventilated hood.
Disposal of samples and chemicals must be done in compliance with on-site safety policies and procedures.

2.2 Test and Reference Items

Test and reference items should be stored according to the information provided in the certificate of analysis.

BAS-Code	BAS 670 H	
Common Name	Topramezone	
IUPAC Name	[3-(4,5-Dihydro-isoxazol-3-yl)-4-methanesulfonyl-2-methyl-phenyl]-(5-hydroxy-1-methyl-1H-pyrazol-4-yl)-methanone	
BASF Reg. No.	375080	
CAS-No.	210631-68-8	
Molecular Formula	$C_{16}H_{17}N_3O_5S$	
Molecular Weight	363.39	

BAS-Code	M670H05	
IUPAC Name	3-(4,5-dihydro-isoxazol-3-yl)-4-methanesulfonyl-2-methyl-benzoic acid	
BASF Reg. No.	388010	
CAS-No.	223646-24-0	
Molecular Formula	$C_{12}H_{13}NO_5S$	
Molecular Weight	283.3	

2.3 Equipment

Equipment	Size, Description	Manufacturer	Catalog No.
Beakers	50, 100, 250 mL	VWR	
Spatula	Various	VWR	
Volumetric pipettes	0.5, 1, 2, 2.5, 5, 10, 25 mL	Schott Glaswerke, Mainz	
Volumetric Flasks	10, 25, 50, 100 mL	VWR	
MicroMan pipettes	10 μL – 1000 μL	Gilson	M-25, M-50, M-250, M-1000
Wide Mouth Bottle	1, 2L		
Analytical Balance	PM 4800 Delta Range	Mettler Toledo	
Culture Tubes	16 X 100 mm	VWR	
Teflon® Bottle	125 mL Nalgene	VWR	16071-041
Culture Tubes,caps	16 mm	VWR	60828-768
Multitube Vortexer	VX-2500	VWR	58816-116
Centrifuge	Refrigerated Centrifuge Model CS-6KR	Beckmann	
Mechanical shaker	KS501 digital	IKA Labortechnik	
Retsch Ultra Centrifugal Mill	Mdl Zm100 0r Zm200	Retsch	
Stephan Floor Chopper	Homoloid Machine, Model J.	Fitzpatrick, Co.	
LC-MS/MS Instrument	Acquity-API 5500	Waters-ABSciex	
LC Vials	2 mL Amber	National Scientific	C4000-76W
LC Column	Atlantis T3, 100 X 2.1 mm, 3u	Waters	186003718

Note: The equipment and instrumentation listed above may be substituted by that of similar specifications. The applicability is confirmed if the recoveries of the fortification experiments are in the expected concentration range.

2.4 Reagents

2.4.1 Chemicals

Chemical	Grade	Manufacturer/Supplier	Catalog No.
Water	High Purity	B & J	365-4
Methanol	High Purity	B & J	230-4
Dichloromethane	High Purity	B & J	300-4
Acetonitrile	High Purity	EMD	AX0145P-1
Ammonium Hydroxide	28-30%	EM Science	AX1303-13
Ammonium Formate	MicroSelect >99%	Fluka	09735
Hydrochloric Acid	37%	Sigma Aldrich	258148
Sodium Chloride	≥98%	Sigma Aldrich	310166-1KG

Note: Equivalent reagents and chemicals from other suppliers may be substituted.

2.4.2 Solutions and Solvent Mixtures

Description	Code	Composition
Extraction solvent	S1	Water
Extraction Solvent	S2	50:50 Acetonitrile:Water Add 500 mL of acetonitrile and 500 mL of water into a vessel, e.g. 1 L wide mouth bottle and mix well to ensure complete homogeneous
Partitioning Additive	S3	1N HCl in Saturated NaCl solution Add 300 g of NaCl to 917 mL water, then add 83 mL of concentrated HCl
LC/MS/MS Standard and Sample Dilution Solvent	S4	4mM Ammonium Formate in Water, pH 10 Add 0.25 g of ammonium formate and 1 mL of 28-30% ammonium hydroxide into 1L of water, e.g., 1L wide mouth bottle and mix well to ensure complete homogeneous solution.
HPLC mobile phase A	LC1	4mM Ammonium Formate in Water Add 0.25 g of ammonium formate into 1L of water , e.g., 1L wide mouth bottle and mix well to ensure complete homogeneous solution.
HPLC mobile phase B	LC2	4mM Ammonium Formate in Methanol Add 0.25 g of ammonium formate into 1L of methanol , e.g., 1L wide mouth bottle and mix well to ensure complete homogeneous solution.

Note: If necessary, the solutions may also be prepared in different volumes as long as the proportions are not modified.

2.4.3 Standard Solutions

Stock Solutions

Prepare a 1.0 mg/mL stock solution individually by weighing an appropriate amount of each analyte into a flask and add the required volume.
For example, to prepare 10 mL of 1.0 mg/mL stock solution of BAS 670 H in acetonitrile or methanol, weigh 10 mg BAS 670 H into a 10 mL volumetric flask. Dissolve and dilute to mark with acetonitrile or methanol. Ensure a complete homogeneous solution (e.g. by sonication or vortexing). The stock solutions for all other analytes are made in a similar fashion.

Independence of standard calibration and fortification solutions should initially be confirmed to show correct preparation of the solutions. This can be achieved for example using one of the following approaches:

- Two stock solutions are independently prepared. One is used for preparation of fortification solutions, the other for calibration standard solutions.
- Fortification and calibration standard solutions should be prepared from one stock solution in separate dilution series.

For subsequent preparations of solutions, freshly prepared solutions can be compared directly to previous standard solutions.

A correction for purity is done if the purity is ≤ 95%. If the purity is > 95 % correction is optional.

Fortification Solutions

Prepare mixed standard solutions for fortification by combining stock solutions of each analyte (see above) in a flask. Dilute volumetrically with appropriate solvents as exemplified in the table below and ensure a complete homogeneous solution (e.g. by sonication or vortexing).

Preparation of mixed Fortification solutions

Take solution (µg/mL)	Volume (mL)	Dilute with Water to a final volume of (mL)	Concentration (µg/mL)
1000	0.5	50	10.0
10	5.0	50	1.0
10	0.5	50	0.10

Note: A different concentration scheme may be used, if other fortification levels are needed for the analysis.

If necessary, the volume of solution prepared may be changed as long as the proportions are not modified.

Calibration Standard Solutions

Prepare mixed standard calibration solutions for LC-MS/MS analysis by using the solutions that were prepared in Section "stock solutions" or "standard fortification solutions" in flasks. Dilute volumetrically with appropriate solvents as exemplified in the table below and ensure a complete homogeneous solution (e.g. by sonication or vortexing).

Preparation of standard solutions for calibration

Take solution (ng/mL)	Volume (mL)	Dilute with S4* to a final volume of (mL)	Concentration (ng/mL)
100	1	50	2.0
2.0	40	100	0.4
0.4	25	50	0.20
0.4	12.5	50	0.10
0.4	5	50	0.04
0.4	2.5	50	0.020

* If matrix-matched standards (= instrument recovery samples) are needed for successful analysis, calibration standard solution are prepared in matrix solution, i.e., final volume of a control sample carried through the analytical procedure. Matrix-matched standards should be prepared in a way that the matrix load is at least 90% of the matrix load in the unknown samples. In addition the matrix load should be the same in all calibration standard solutions.

Note: A different concentration scheme may be used and additional standards may be prepared as needed.
If necessary, the volume of solution prepared may be changed as long as the proportions are not modified.

- Use amber bottles with Teflon-lined screw caps as storage containers for standard solutions.

2.4.4 Stability of Standard Solutions

The reference item solutions are stable under the specifications described below, which means no decline below 90 % of the nominal concentration after the described storage period. If solutions are stored at different conditions or/and for a longer time, the stability of the reference items has to be confirmed.

Reference Item	Concentration [µg/mL]	Solvents	Storage conditions	Time Interval [days]	Reference No.
Reg.No.375080 Reg.No.388010	1	Water	Appr. 4°C	90	[1]
Reg.No.375080	1	Methanol	Appr. 4°C	90	[1]
Reg.No.375080	1	Acetonitrile	Appr. 4°C	60	[1]
Reg.No.375080	1	0.05% NH4OH Solution	Appr. 4°C	90	[1]
Reg.No.388010	1	Methanol	Appr. 4°C	30	[1]
Reg.No.388010	1	Acetonitrile	Appr. 4°C	30	[1]
Reg.No.388010	1	0.05% NH4OH Solution	Appr. 4°C	62	[1]

BASF recommends that stock solutions (1 mg/mL) in acetonitrile be made fresh every three months. Dilution of stock solutions should be stored refrigerated no longer than one month or according to their established storage stability in a particular solvent.

3 ANALYTICAL PROCEDURE

3.1 Sample Preparation

Samples have to be sufficiently homogenized beforehand, in order to assure that the aliquot taken for residue analysis is representative for the whole sample. Plant samples are homogenized using a Stephan floor chopper with dry ice. If additional grinding is necessary to achieve a smaller particle size the sample can be homogenized in a Retsch Ultra Centrifugal Mill equipped with a 1.0 mm screen. After homogenization and evaporation of dry ice or liquid nitrogen, matrices are stored frozen until analysis.

Samples are generally stored at about -20°C

Storage stability of BAS 670 H and M670H05 have been determined in a GLP study[3].

3.2 Weighing and Fortification

For treated samples and control samples, weigh 5.0 g (+/- 0.1 g) of sample into a 125 mL Teflon® coated plastic bottle.

For fortified samples, weigh at this stage 5.0 g (+/- 0.1 g) of control sample into a 125 mL Teflon® coated plastic bottle and add fortification solutions on the matrix.

The following scheme may be used:

Sample Type	Sample Weight	Concentration of Spiking Solution	Volume of Spiking Solution	Level of Fortification
Control	5 g	-	-	0.00 mg/kg
Fortification (LOQ)	5 g	100 ng/mL	0.5 mL	0.010 mg/kg *
Fortification (10xLOQ)	5 g	1.0 µg/mL	0.5 mL	0.1 mg/kg
Treated	5 g	-	-	-

* Limit of quantification

Note: Volume of spiking solution added to generate the fortified sample should not exceed 10% of sample weight or volume.

3.3 Extraction of Sample Material

Add 50 mL of S1 (or S2 for oily matrices, 49.0 or 49.5 mL in the case of a fortification samples) to the plant matrix. For watery matrices (such as fruits and juices), add 45 mL of S1(44 or 44.5 mL in case of fortification). Cap tightly and shake on a mechanical shaker for 30 minutes. Remove approximately 5-10 mL (precise volume is not important) from the extract and transfer to a 16 X 100 mm culture tube. When removing the aliquot, try to avoid transferring pieces of the plant material into the culture tube. Centrifuge at 1500 rpm for 5 minutes.

The capability of solvent S1 and S2 to extract residues of BAS 670 H and M670H05 was proven within a pre-registration metabolism study using radioactive test substance (Metabolism of [^{14}C]-BAS 670 H in Corn, BASF Study No.: 98129[2]).

Note: In case of watery matrices, a volumetric flask may be used for the extraction vessel. Weigh out 5 grams of the matrix into a 50 mL volumetric flask and adjust to mark with S1

3.4 Sample Clean-up

Transfer exactly 1.0 mL of the centrifuged extract into another 16 X 100 mm culture tube and add 1 mL 1N HCl in saturated NaCl solution(S3). Add exactly 4 mL of dichloromethane to the culture tube and cap firmly using a culture tube cap. Vortex @ 2400 rpm for 1-2 minutes on a multitube vortexer. Centrifuge at 1500 rpm for 5 minutes*. Take exactly a 1 mL aliquot of the dichloromethane phase using a glass pipette or a 1000 μL MicroMan positive pressure pipette and transfer into another 16 X 100 mm culture tube.

Note: A solid layer may form between the aqueous phase and the dichloromethane phase. When removing the aliquot from the lower dichloromethane phase, avoid including any of the solids.

Add exactly 2.5 mL of S4 and firmly cap with a culture tube cap. Vortex @ 2400 rpm for 1-2 minutes. Centrifuge at 1500 rpm for 5 minutes*.

Note: A mechanical shaker may be used in lieu of a multitube vortexer. If a shaker is used, caps should be firmly secured. Samples should be laid horizontally perpendicular to the direction of the shaking. Sample should be shaken @~300 rpm for 15 minutes. Additionally, if an individual sample vortexer such as the Vortex Genie II is used, each sample should be vortexed at maximum speed for at least 1 minute.

*** Appropriate stopping point for the method**

3.5 Preparation for Measurement

Transfer an aliquot of the top layer (S4) into an LC vial and cap. No dilution is necessary for control and LOQ samples. For 10XLOQ fortifications, dilutions can be made by removing an aliquot of the top layer and diluting further with S4. (For example, remove 100 μL from the top layer then add 900 μL of S4)

3.6 Influence of matrix effects on analysis

During method development, it was demonstrated that the matrix load in the samples from the matrices had no significant influence on the analysis (i.e., matrix effects < 20%). Therefore, samples can be analyzed using calibration standard solutions prepared in solvent S4 or in matrix solution (see 2.4.3).
If less sensitive instruments are used and matrix effects are observed (>20%), matrix-matched calibration standard solutions must be used.

3.7 Stability of Extracts and Final Volumes

Storage stability has not been established for the extracts or the final volumes. Procedural recoveries can be used to prove the stability, if necessary.

QUANTIFICATION AND CALCULATION

3.8 Set-up of the analytical run

A sequence for measurement generally consists of:
- Calibration standards
- Control samples
- Procedural recovery samples
- Unknown samples
- Instrument recovery sample

Reagent Blanks or blanks can also be injected if necessary. Each injection set should begin and end with an injection of a calibration standard. Standards should be interspersed with samples. Each calibration standard should be at least injected twice. At least 5 calibration levels need to be injected.

3.9 Instrumental analysis

3.9.1 Instrumentation and Conditions for Grape, Forage, Seed, and Grain

	Parameter		
Chromatographic System	Waters Acquity		
Analytical-column	Atlantis T3, 100 X 2.1, 3µ		
Column Temperature	50°C		
Injection Volume	25 µL		
Mobile Phase A Mobile Phase B	4 mM Ammonium Formate in Water 4 mM Ammonium Formate in Methanol		
Flow Rate	600 µL/min		
Gradient (including wash and equilibration)	Time (min)	Phase A	Phase B
	0.0	95	5
	0.2	95	5
	1.5	40	60
	2.5	5	95
	3.45	5	95
	3.5	95	5
	4.0	95	5
Detection System	AB Sciex API 5500 Mass Spectrometer		
Ionisation	Turbo Ion Spray (ESI)		
Ionization Temperature	450 °C		
Analyte	**Transitions**	**Polarity**	**Expected Retention Time**
BAS 670 H	362 --> 334* 362 --> 318	Negative	approx. 1.6 min
M670H05	282 --> 237.9* 282 --> 79.1	Negative	approx. 1.3 min.

* proposed as quantification transition. Any of these transitions could be used for quantitation in case interference is observed at the same retention time

Note: Instruments with similar specifications may substitute the equipment listed above. The instruments used are applicable for analysis if the recoveries of the fortification experiments are in the acceptable range.

In general a divert valve is used to reduce the matrix load on the detection system.

Instrument conditions, e.g. injection volumes, columns, gradient steps or mass transitions may be modified, but any changes must be recorded in the raw data. Changes are acceptable, when the recoveries of the fortification experiments are in the acceptable range.

Other parameters like gas flows and voltages are depended of the equipment used and therefore not listed. Those parameters may need to be adapted for the used instrument.

3.9.2 Instrumentation and Conditions for Dry Bean

	Parameter		
Chromatographic System	Waters Acquity		
Analytical-column	Atlantis T3, 100 X 2.1, 3µ		
Column Temperature	50°C		
Injection Volume	25 µL		
Mobile Phase A Mobile Phase B	4 mM Ammonium Formate in Water 4 mM Ammonium Formate in Methanol		
Flow Rate	600 µL/min		
Gradient (including wash and equilibration)	Time (min)	Phase A	Phase B
	0.0	95	5
	0.2	95	5
	4.5	65	35
	6.0	5	95
	7.45	5	95
	7.5	95	5
	8.0	95	5
Detection System	AB Sciex API 5500 Mass Spectrometer		
Ionisation	Turbo Ion Spray (ESI)		
Ionization Temperature	450 °C		
Analyte	**Transitions**	**Polarity**	**Expected Retention Time**
BAS 670 H	362 --> 334* 362 --> 318	Negative	approx. 3.3 min
M670H05	282 --> 237.9* 282 --> 79.1	Negative	approx. 1.8 min.

* proposed as quantification transition. Any of these transitions could be used for quantitation in case interference is observed at the same retention time

Note: Instruments with similar specifications may substitute the equipment listed above. The instruments used are applicable for analysis if the recoveries of the fortification experiments are in the acceptable range.

In general a divert valve is used to reduce the matrix load on the detection system.

Instrument conditions, e.g. injection volumes, columns, gradient steps or mass transitions may be modified, but any changes must be recorded in the raw data. Changes are acceptable, when the recoveries of the fortification experiments are in the acceptable range.

Other parameters like gas flows and voltages are depended of the equipment used and therefore not listed. Those parameters may need to be adapted for the used instrument.

3.9.3 Calibration procedures

Calculation of results is based on peak area measurements using a calibration curve. At least 5 calibration levels need to be injected (e.g., required for enforcement). The calibration curve is obtained by direct injection of BAS 670 H and M670H05 standards for LC-MS/MS in the range of 0.4 ng/mL to 0.02 ng/mL. The calibration curve is obtained by direct injection of In a given injection run, the same injection volume is used for all samples and standards.

Linear calibration functions are preferred for evaluation. If other functions are used (e.g. quadratic), this should be fully justified.

3.9.4 Calculation of Residues and Recoveries

Calculation of results is based on area measurements.
For the procedural recoveries, the sample weight will be considered 5 g in the final calculation of residues [mg/kg]. The method requires that the sample weight to be 5 ± 0.1 g for fortification samples. The recovery is the percentage of the fortified amount (µg or ng), which is recovered through the method and the weights cancels out, as shown in the equation below, during the final calculation step.

The residues of BAS XXX F in mg/kg are calculated as shown in equations **I** and **II**:

I. **Concentration [ng/mL]** $= \dfrac{\text{Response} - \textit{Intercept}}{\textit{Slope}} = C_A$

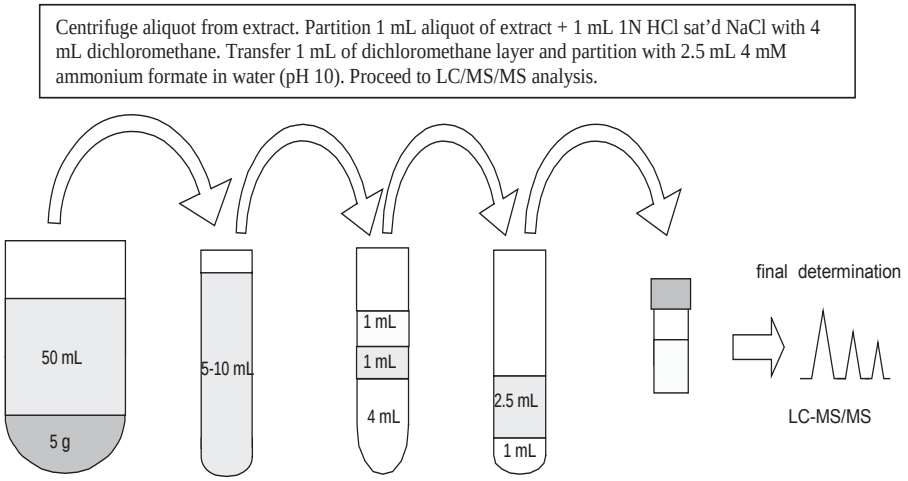
II. **Residue [mg/kg]** $= \dfrac{V_{end} \times C_A}{G \times A_F \times 1000}$

V_{end}	=	Final volume of the extract after all dilution steps [mL]
C_A	=	Concentration of analyte as read from the calibration curve [ng/mL]
G	=	Weight of the sample extracted [g]
A_F	=	Aliquotation factor
1000	=	Factor remaining after all unit conversions

The recoveries of spiked compounds are calculated according to equation **III**:

III. **Recovery %** $= \dfrac{(\text{Residue in fortified sample} - \text{Residue in control}) \times 100}{\text{Amount of analyte fortified}}$

4 FLOWCHART


Centrifuge aliquot from extract. Partition 1 mL aliquot of extract + 1 mL 1N HCl sat'd NaCl with 4 mL dichloromethane. Transfer 1 mL of dichloromethane layer and partition with 2.5 mL 4 mM ammonium formate in water (pH 10). Proceed to LC/MS/MS analysis.
50 mL
5 g
5-10 mL
1 mL
1 mL
4 mL
2.5 mL
1 mL
final determination
LC-MS/MS

5 METHOD MANAGEMENT AND TIME REQUIREMENTS

The analysis of one series of samples (= 20 unknown samples, 2 fortified samples for recovery experiments, 1 blank sample) requires 1.0 working days (8 hours) per laboratory assistant. This time includes the calculation of the results, the preparation of the equipment as well as the reporting of all raw data under GLP.

6 CONCLUSION AND METHOD CAPABILITIES

Recoveries, Chromatograms, and Calibration Curves
Recovery data will be provided in the validation part of the analytical method D1302.

Limit of Quantification (LOQ) and Limit of Detection (LOD)
The limit of quantification is defined as the lowest fortification level successfully tested. The limit of quantification is 0.01 mg/kg for all analytes. The limit of detection was estimated at 20% of the limit of quantification, equivalent to 0.002 mg/kg for all analytes. The lowest standard for each analyte in the calibration curve has good detectability (signal to noise ratio greater than 3:1).

Selectivity
The tested untreated plant samples showed no significant interferences (< 20 or 30 %) at the retention time of the analytes.
Justification of selection of ions is shown in the appendix.

Confirmatory Techniques
The HPLC-MS/MS final determination for BAS 670 H and M670H05 is a highly selective detection technique. For every compound the quantitation is possible at two different transitions. Therefore, no additional confirmatory technique is required.

Potential Problems
There is the potential for losses during the partition steps if the agitation of the samples is inadequate. Vortexing is recommended as opposed to shaking.

7 REFERENCES

[1] Jones, J.: Determination of the Stability of BAS 670 H and M670H05 in Various Solvents. Study Code: 56755, BASF DocID: 2002/5003843.

[2] Ellenson, J: Metabolism of [^{14}C]-BAS 670 H in Corn. Study Code: 98129. BASF DocID: 2002/5002068.

[3] Jordan, J.: Storage Stability of BAS 670 H and Its Cleaved Acid Metabolite M670H05 in Plant Matrices. Study Code: 56750. BASF DocID: 2003/5000339

APPENDIX

7.1 Example of Calculation

Example: BAS 670 H, 362 → 334; corn forage sample fortified at 0.01 mg/kg:

Concentration in the final volume [ng/mL]

$$\textbf{Concentration [ng/mL]} = \frac{\text{Response} - \textit{Intercept}}{\textit{Slope}} = C_A$$

Residue in the sample [mg/kg]

$$\textbf{Residue [mg/kg]} = \frac{V_{end} \times C_A}{G \times A_F \times 1000}$$

$$\textbf{Recovery \%} = \frac{\text{Residue in fortified sample} - \text{Residue in control} \times 100}{\text{Amount of analyte fortified}}$$

The following values were used in this calculation:

Response of fortified sample	11108
Response of control sample	0
Slope:	110000
Intercept:	335
Sample Weight (G):	5 g
Final Volume (V_{end}):	2.5 mL
Aliquotation factor A_F:	0.005 (= 0.5%)
Conversion factor ng → µg:	1000

$$\textbf{Concentration (ng/mL)} = \frac{11108 - 335}{110000} = 0.098\ ng/ml$$

Residue (mg/kg)

$$= \frac{2.5ml \times 0.098\ ng/ml}{5\ g \times 0.005 \times 1000} = 0.00980\ \mu g/g = 0.00980\ mg/kg$$

$$\textbf{Recovery \%} = \frac{\left(0.00980\ mg/kg - 0.00000\ mg/kg\right) \times 100}{0.0100\ mg/kg} = 98.0\%$$

Appendix C. Typical Recovery Calculation for LC/MS/MS Quantitation

Sample No. 698252-0001. Control forage ("Fort. 1") fortified with topramezone and M670H05 at 0.01 ppm each, from Master Sheet Number 698252-1.

$$\text{Concentration of Analyte } (C_A) \text{ (ng/mL)} = \frac{\text{peak area - intercept}}{\text{slope}}$$

	Topramezone	M670H05
Peak Area =	12,994.0	33,980.0
Intercept =	-196.0577	-160.0119
Slope =	145308.6624	406021.8216
C_A (ng/mL) =	0.0908	0.0841

$$\text{Residue (ppm)} = \frac{FV \times C_A}{G \times A_F}$$

	Topramezone	M670H05
Final Vol. (FV) mL =	2.5	2.5
C_A (ng/mL) =	0.0908	0.0841
Sample Wt. (g) =	5.00	5.00
Aliquot Factor (A_F) =	0.5%	0.5%
Residue (ppm) =	0.0091	0.0084

A_F = [Aliquot taken from initial extract (1 mL) / Volume of initial extract (50 mL)] x [Aliquot taken from liquid-liquid partition (1 mL) / dichloromethane layer (4 mL)] = 0.005 (0.5%)

Net residue (ppm of analyte) = Residue (ppm of analyte) - Residue in Control (ppm)

$$\text{Recovery of analyte (\%)} = \frac{\text{Residue (ppm of analyte) - Residue in Control (ppm)}}{\text{Amount Fortified (ppm)}} \times 100$$

	Topramezone	M670H05
Amount fortified	0.01	0.01
Residue (ppm) =	0.0091	0.0084
Residue in control =	0.000	0.000
%Recovery	91%	84%

Use full computer/calculator precision in any intermediate calculations.
Round only the final value.

Appendix D. Detailed Analytical Data for Recoveries of Topramezone

Notes:

Dates of extraction and analysis are given in "day-month-year" format.

Use full computer/calculator precision in any intermediate calculations. Round only the final value. Slight differences may be noted in hand calculations versus calculations in the individual data tables presented in this report due to rounding.

Master Sheet No.: 698252-01
Filename:1304\20130415BKDa.wiff
Analyte: Parent Topramezone (BAS 670H)
Comment: Primary transition, m/z 362 to 334

Sample Description	Sample ID	Date Analyzed	Date Extracted	Sample Type	QC [mg/kg] conc. [ng/ml]	Peak Count	Calc. conc. [ng/ml]	Sample wt [g]	Final vol.[mL]	Aliquot factor	Calc. res. [mg/kg]	Result [mg/kg]	Rec. %
ERS13-383	0.02 ng/mL	15.4.2013 8:04:26 PM		Standard	0.020	2,584.0	0.0191						
ERS13-382	0.04 ng/mL	15.4.2013 8:09:14 PM		Standard	0.040	5,170.0	0.0369						
ERS13-381	0.1 ng/mL	15.4.2013 8:14:03 PM		Standard	0.100	13,474.0	0.0941						
ERS13-380	0.2 ng/mL	15.4.2013 8:18:51 PM		Standard	0.200	26,158.0	0.1814						
ERS13-379	0.4 ng/mL	15.4.2013 8:23:38 PM		Standard	0.400	51,254.0	0.3541						
Blank	Blank	15.4.2013 8:28:27 PM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
698252-0001	Corn Forage Control	15.4.2013 8:33:14 PM	15-Apr-13	Control	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
698252-0001	Corn Forage Control	15.4.2013 8:38:01 PM	15-Apr-13	Control	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
698252-0001	Corn Forage 0.01 ppm Fort 1	15.4.2013 8:42:48 PM	15-Apr-13	Fortification	0.010	12,994.0	0.0908	5.0	2.5	0.5%	0.0091	0.0091	91%
Blank	Blank	15.4.2013 8:47:35 PM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
ERS13-383	0.02 ng/mL	15.4.2013 8:52:24 PM		Standard	0.020	3,139.0	0.0230						
698252-0001	Corn Forage 0.01 ppm Fort 2	15.4.2013 8:57:12 PM	15-Apr-13	Fortification	0.010	12,955.0	0.0905	5.0	2.5	0.5%	0.0091	0.0091	91%
698252-0001	Corn Forage 0.01 ppm Fort 3	15.4.2013 9:02:00 PM	15-Apr-13	Fortification	0.010	13,080.0	0.0914	5.0	2.5	0.5%	0.0091	0.0091	91%
698252-0001	Corn Forage 0.01 ppm Fort 4	15.4.2013 9:06:48 PM	15-Apr-13	Fortification	0.010	13,140.0	0.0918	5.0	2.5	0.5%	0.0092	0.0092	92%
698252-0001	Corn Forage 0.01 ppm Fort 5	15.4.2013 9:11:36 PM	15-Apr-13	Fortification	0.010	13,425.0	0.0937	5.0	2.5	0.5%	0.0094	0.0094	94%
Blank	Blank	15.4.2013 9:16:23 PM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
ERS13-382	0.04 ng/mL	15.4.2013 9:21:09 PM		Standard	0.040	5,823.0	0.0414						
698252-0001	Corn Forage 0.1 ppm Fort 1	15.4.2013 9:25:57 PM	15-Apr-13	Fortification	0.100	31,539.0	0.2184	5.0	12.5	0.5%	0.1092	0.1092	109%
698252-0001	Corn Forage 0.1 ppm Fort 2	15.4.2013 9:30:45 PM	15-Apr-13	Fortification	0.100	32,844.0	0.2274	5.0	12.5	0.5%	0.1137	0.1137	114%
698252-0001	Corn Forage 0.1 ppm Fort 3	15.4.2013 9:35:32 PM	15-Apr-13	Fortification	0.100	34,905.0	0.2416	5.0	12.5	0.5%	0.1208	0.1208	121%
698252-0001	Corn Forage 0.1 ppm Fort 4	15.4.2013 9:40:22 PM	15-Apr-13	Fortification	0.100	31,325.0	0.2169	5.0	12.5	0.5%	0.1085	0.1085	108%
Blank	Blank	15.4.2013 9:45:10 PM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
ERS13-381	0.1 ng/mL	15.4.2013 9:49:56 PM		Standard	0.100	15,371.0	0.1071						
698252-0001	Corn Forage 0.1 ppm Fort 5	15.4.2013 9:54:46 PM	15-Apr-13	Fortification	0.100	33,073.0	0.2290	5.0	12.5	0.5%	0.1145	0.1145	114%
698252-0004	Grape Control	15.4.2013 9:59:32 PM	15-Apr-13	Control	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
698252-0004	Grape Control	15.4.2013 10:04:22 PM	15-Apr-13	Control	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
698252-0004	Grape 0.01 ppm Fort 1	15.4.2013 10:09:14 PM	15-Apr-13	Fortification	0.010	13,212.0	0.0923	5.0	2.5	0.5%	0.0092	0.0092	92%
Blank	Blank	15.4.2013 10:14:02 PM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
ERS13-380	0.2 ng/mL	15.4.2013 10:18:53 PM		Standard	0.200	27,375.0	0.1897						
698252-0004	Grape 0.01 ppm Fort 2	15.4.2013 10:23:44 PM	15-Apr-13	Fortification	0.010	14,036.0	0.0979	5.0	2.5	0.5%	0.0098	0.0098	98%
698252-0004	Grape 0.01 ppm Fort 3	15.4.2013 10:28:34 PM	15-Apr-13	Fortification	0.010	13,292.0	0.0928	5.0	2.5	0.5%	0.0093	0.0093	93%
698252-0004	Grape 0.01 ppm Fort 4	15.4.2013 10:33:21 PM	15-Apr-13	Fortification	0.010	13,181.0	0.0921	5.0	2.5	0.5%	0.0092	0.0092	92%
698252-0004	Grape 0.01 ppm Fort 5	15.4.2013 10:38:09 PM	15-Apr-13	Fortification	0.010	13,009.0	0.0909	5.0	2.5	0.5%	0.0091	0.0091	91%
Blank	Blank	15.4.2013 10:42:57 PM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
ERS13-379	0.4 ng/mL	15.4.2013 10:47:44 PM		Standard	0.400	63,076.0	0.4354						
698252-0004	Grape 0.1 ppm Fort 1	15.4.2013 10:52:32 PM	15-Apr-13	Fortification	0.100	32,614.0	0.2258	5.0	12.5	0.5%	0.1129	0.1129	113%
698252-0004	Grape 0.1 ppm Fort 2	15.4.2013 10:57:19 PM	15-Apr-13	Fortification	0.100	31,451.0	0.2178	5.0	12.5	0.5%	0.1089	0.1089	109%
698252-0004	Grape 0.1 ppm Fort 3	15.4.2013 11:02:07 PM	15-Apr-13	Fortification	0.100	30,240.0	0.2095	5.0	12.5	0.5%	0.1047	0.1047	105%
698252-0004	Grape 0.1 ppm Fort 4	15.4.2013 11:06:54 PM	15-Apr-13	Fortification	0.100	31,058.0	0.2151	5.0	12.5	0.5%	0.1075	0.1075	108%
Blank	Blank	15.4.2013 11:11:42 PM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
ERS13-383	0.02 ng/mL	15.4.2013 11:16:32 PM		Standard	0.020	3,054.0	0.0224						
698252-0004	Grape 0.1 ppm Fort 5	15.4.2013 11:21:24 PM	15-Apr-13	Fortification	0.100	31,383.0	0.2173	5.0	12.5	0.5%	0.1087	0.1087	109%
Reagent Blank	Reagent Blank	15.4.2013 11:26:14 PM	15-Apr-13	Control	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
698252-0001	Corn Forage Control Spike	15.4.2013 11:31:02 PM	15-Apr-13	Instrument Rec.	0.010	13,920.0	0.0971	5.0	2.5	0.5%	0.0097	0.0097	97%
698252-0004	Grape Control Spike	15.4.2013 11:35:49 PM	15-Apr-13	Instrument Rec.	0.010	14,854.0	0.1036	5.0	2.5	0.5%	0.0104	0.0104	104%
Blank	Blank	15.4.2013 11:40:37 PM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
ERS13-383	0.02 ng/mL	15.4.2013 11:45:24 PM		Standard	0.020	2,998.0	0.0220						
ERS13-382	0.04 ng/mL	15.4.2013 11:50:12 PM		Standard	0.040	6,002.0	0.0427						
ERS13-381	0.1 ng/mL	15.4.2013 11:54:59 PM		Standard	0.100	14,651.0	0.1022						
ERS13-380	0.2 ng/mL	15.4.2013 11:59:49 PM		Standard	0.200	30,288.0	0.2098						
ERS13-379	0.4 ng/mL	16.4.2013 12:04:36 AM		Standard	0.400	60,656.0	0.4188						

	correlation function:
concentration range from:	0.020
concentration range to:	0.400
intercept A =	-196.0577
slope B =	145308.6624
correlation coefficient	0.9929

limit of quantitation (LOQ) [mg/kg] = 0.01
limit of detection (LOD) [mg/kg] = 0.002

Instrument Rec. = Instrument recovery sample (used to test for matrix effects)

Master Sheet No.: 698252-01
Filename:1304\20130415BKDb.wiff
Analyte: Parent Topramezone (BAS 670H)
Comment: Primary transition, m/z 362 to 334

Sample Description	Sample ID	Date Analyzed	Date Extracted	Sample Type	QC [mg/kg] conc. [ng/ml]	Peak Count	Calc. conc. [ng/ml]	Sample wt [g]	Final vol.[mL]	Aliquot factor	Calc. res. [mg/kg]	Result [mg/kg]	Rec. %
ERS13-383	0.02 ng/mL	16.4.2013 5:31:23 AM		Standard	0.020	2,140.0	0.0145						
ERS13-382	0.04 ng/mL	16.4.2013 5:40:11 AM		Standard	0.040	4,843.0	0.0392						
ERS13-381	0.1 ng/mL	16.4.2013 5:48:59 AM		Standard	0.100	11,820.0	0.1029						
ERS13-380	0.2 ng/mL	16.4.2013 5:57:47 AM		Standard	0.200	21,526.0	0.1915						
ERS13-379	0.4 ng/mL	16.4.2013 6:06:34 AM		Standard	0.400	41,740.0	0.3761						
Blank	Blank	16.4.2013 6:15:22 AM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
698252-0003	Dry Bean Control	16.4.2013 6:24:09 AM	15-Apr-13	Control	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
698252-0003	Dry Bean Control	16.4.2013 6:32:57 AM	15-Apr-13	Control	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
698252-0003	Dry Bean 0.01 ppm Fort 1	16.4.2013 6:41:45 AM	15-Apr-13	Fortification	0.010	11,354.0	0.0986	5.0	2.5	0.5%	0.0099	0.0099	99%
Blank	Blank	16.4.2013 6:50:33 AM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
ERS13-383	0.02 ng/mL	16.4.2013 6:59:20 AM		Standard	0.020	2,526.0	0.0180						
698252-0003	Dry Bean 0.01 ppm Fort 2	16.4.2013 7:08:08 AM	15-Apr-13	Fortification	0.010	11,148.0	0.0967	5.0	2.5	0.5%	0.0097	0.0097	97%
698252-0003	Dry Bean 0.01 ppm Fort 3	16.4.2013 7:16:55 AM	15-Apr-13	Fortification	0.010	10,930.0	0.0947	5.0	2.5	0.5%	0.0095	0.0095	95%
698252-0003	Dry Bean 0.01 ppm Fort 4	16.4.2013 7:25:43 AM	15-Apr-13	Fortification	0.010	11,977.0	0.1043	5.0	2.5	0.5%	0.0104	0.0104	104%
698252-0003	Dry Bean 0.01 ppm Fort 5	16.4.2013 7:34:30 AM	15-Apr-13	Fortification	0.010	11,426.0	0.0993	5.0	2.5	0.5%	0.0099	0.0099	99%
Blank	Blank	16.4.2013 7:43:17 AM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
ERS13-382	0.04 ng/mL	16.4.2013 7:52:04 AM		Standard	0.040	4,505.0	0.0361						
698252-0003	Dry Bean 0.1 ppm Fort 1	16.4.2013 8:00:51 AM	15-Apr-13	Fortification	0.100	27,634.0	0.2473	5.0	12.5	0.5%	0.1236	0.1236	124%
698252-0003	Dry Bean 0.1 ppm Fort 2	16.4.2013 8:09:39 AM	15-Apr-13	Fortification	0.100	24,069.0	0.2147	5.0	12.5	0.5%	0.1074	0.1074	107%
698252-0003	Dry Bean 0.1 ppm Fort 3	16.4.2013 8:18:27 AM	15-Apr-13	Fortification	0.100	24,753.0	0.2209	5.0	12.5	0.5%	0.1105	0.1105	110%
698252-0003	Dry Bean 0.1 ppm Fort 4	16.4.2013 8:27:15 AM	15-Apr-13	Fortification	0.100	24,386.0	0.2176	5.0	12.5	0.5%	0.1088	0.1088	109%
Blank	Blank	16.4.2013 8:36:02 AM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
ERS13-381	0.1 ng/mL	16.4.2013 8:44:50 AM		Standard	0.100	11,310.0	0.0982						
698252-0003	Dry Bean 0.1 ppm Fort 5	16.4.2013 8:53:37 AM	15-Apr-13	Fortification	0.100	19,444.0	0.1725	5.0	12.5	0.5%	0.0862	0.0862	86%
698252-0003	Dry Bean Control Spike	16.4.2013 9:02:26 AM	15-Apr-13	Instrument Rec.	0.010	11,056.0	0.0959	5.0	2.5	0.5%	0.0096	0.0096	96%
Blank	Blank	16.4.2013 9:11:13 AM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
ERS13-383	0.02 ng/mL	16.4.2013 9:20:02 AM		Standard	0.020	2,559.0	0.0183						
ERS13-382	0.04 ng/mL	16.4.2013 9:28:49 AM		Standard	0.040	4,848.0	0.0392						
ERS13-381	0.1 ng/mL	16.4.2013 9:37:36 AM		Standard	0.100	12,545.0	0.1095						
ERS13-380	0.2 ng/mL	16.4.2013 9:46:27 AM		Standard	0.200	24,681.0	0.2203						
ERS13-379	0.4 ng/mL	16.4.2013 9:55:16 AM		Standard	0.400	46,163.0	0.4164						

	correlation function:
concentration range from:	0.020
concentration range to:	0.400
intercept A =	555.0031
slope B =	109518.4285
correlation coefficient	0.9966

limit of quantitation (LOQ) [mg/kg] = 0.01
limit of detection (LOD) [mg/kg] = 0.002

Instrument Rec. = Instrument recovery sample (used to test for matrix effects)

Master Sheet No.: 698252-01
Filename:1304\20130415BKDa.wiff
Analyte: Parent Topramezone (BAS 670H)
Comment: Secondary transition, m/z 362 to 318

Sample Description	Sample ID	Date Analyzed	Date Extracted	Sample Type	QC [mg/kg] conc. [ng/ml]	Peak Count	Calc. conc. [ng/ml]	Sample wt [g]	Final vol.[mL]	Aliquot factor	Calc. res. [mg/kg]	Result [mg/kg]	Rec. %
ERS13-383	0.02 ng/mL	15.4.2013 8:04:26 PM		Standard	0.020	1,633.0	0.0240						
ERS13-382	0.04 ng/mL	15.4.2013 8:09:14 PM		Standard	0.040	2,697.0	0.0383						
ERS13-381	0.1 ng/mL	15.4.2013 8:14:03 PM		Standard	0.100	7,044.0	0.0969						
ERS13-380	0.2 ng/mL	15.4.2013 8:18:51 PM		Standard	0.200	12,570.0	0.1715						
ERS13-379	0.4 ng/mL	15.4.2013 8:23:38 PM		Standard	0.400	25,994.0	0.3525						
Blank	Blank	15.4.2013 8:28:27 PM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
698252-0001	Corn Forage Control	15.4.2013 8:33:14 PM	15-Apr-13	Control	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
698252-0001	Corn Forage Control	15.4.2013 8:38:01 PM	15-Apr-13	Control	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
698252-0001	Corn Forage 0.01 ppm Fort 1	15.4.2013 8:42:48 PM	15-Apr-13	Fortification	0.010	7,121.0	0.0980	5.0	2.5	0.5%	0.0098	0.0098	98%
Blank	Blank	15.4.2013 8:47:35 PM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
ERS13-383	0.02 ng/mL	15.4.2013 8:52:24 PM		Standard	0.020	1,429.0	0.0212						
698252-0001	Corn Forage 0.01 ppm Fort 2	15.4.2013 8:57:12 PM	15-Apr-13	Fortification	0.010	6,776.0	0.0933	5.0	2.5	0.5%	0.0093	0.0093	93%
698252-0001	Corn Forage 0.01 ppm Fort 3	15.4.2013 9:02:00 PM	15-Apr-13	Fortification	0.010	7,122.0	0.0980	5.0	2.5	0.5%	0.0098	0.0098	98%
698252-0001	Corn Forage 0.01 ppm Fort 4	15.4.2013 9:06:48 PM	15-Apr-13	Fortification	0.010	7,465.0	0.1026	5.0	2.5	0.5%	0.0103	0.0103	103%
698252-0001	Corn Forage 0.01 ppm Fort 5	15.4.2013 9:11:36 PM	15-Apr-13	Fortification	0.010	7,119.0	0.0980	5.0	2.5	0.5%	0.0098	0.0098	98%
Blank	Blank	15.4.2013 9:16:23 PM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
ERS13-382	0.04 ng/mL	15.4.2013 9:21:09 PM		Standard	0.040	2,898.0	0.0410						
698252-0001	Corn Forage 0.1 ppm Fort 1	15.4.2013 9:25:57 PM	15-Apr-13	Fortification	0.100	16,475.0	0.2241	5.0	12.5	0.5%	0.1121	0.1121	112%
698252-0001	Corn Forage 0.1 ppm Fort 2	15.4.2013 9:30:45 PM	15-Apr-13	Fortification	0.100	16,051.0	0.2184	5.0	12.5	0.5%	0.1092	0.1092	109%
698252-0001	Corn Forage 0.1 ppm Fort 3	15.4.2013 9:35:32 PM	15-Apr-13	Fortification	0.100	16,801.0	0.2285	5.0	12.5	0.5%	0.1143	0.1143	114%
698252-0001	Corn Forage 0.1 ppm Fort 4	15.4.2013 9:40:22 PM	15-Apr-13	Fortification	0.100	15,894.0	0.2163	5.0	12.5	0.5%	0.1081	0.1081	108%
Blank	Blank	15.4.2013 9:45:10 PM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
ERS13-381	0.1 ng/mL	15.4.2013 9:49:56 PM		Standard	0.100	7,120.0	0.0980						
698252-0001	Corn Forage 0.1 ppm Fort 5	15.4.2013 9:54:46 PM	15-Apr-13	Fortification	0.100	15,467.0	0.2105	5.0	12.5	0.5%	0.1053	0.1053	105%
698252-0004	Grape Control	15.4.2013 9:59:32 PM	15-Apr-13	Control	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
698252-0004	Grape Control	15.4.2013 10:04:22 PM	15-Apr-13	Control	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
698252-0004	Grape 0.01 ppm Fort 1	15.4.2013 10:09:14 PM	15-Apr-13	Fortification	0.010	6,595.0	0.0909	5.0	2.5	0.5%	0.0091	0.0091	91%
Blank	Blank	15.4.2013 10:14:02 PM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
ERS13-380	0.2 ng/mL	15.4.2013 10:18:53 PM		Standard	0.200	14,171.0	0.1930						
698252-0004	Grape 0.01 ppm Fort 2	15.4.2013 10:23:44 PM	15-Apr-13	Fortification	0.010	6,961.0	0.0958	5.0	2.5	0.5%	0.0096	0.0096	96%
698252-0004	Grape 0.01 ppm Fort 3	15.4.2013 10:28:34 PM	15-Apr-13	Fortification	0.010	6,495.0	0.0895	5.0	2.5	0.5%	0.0090	0.0090	90%
698252-0004	Grape 0.01 ppm Fort 4	15.4.2013 10:33:21 PM	15-Apr-13	Fortification	0.010	6,629.0	0.0913	5.0	2.5	0.5%	0.0091	0.0091	91%
698252-0004	Grape 0.01 ppm Fort 5	15.4.2013 10:38:09 PM	15-Apr-13	Fortification	0.010	6,403.0	0.0883	5.0	2.5	0.5%	0.0088	0.0088	88%
Blank	Blank	15.4.2013 10:42:57 PM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
ERS13-379	0.4 ng/mL	15.4.2013 10:47:44 PM		Standard	0.400	31,728.0	0.4298						
698252-0004	Grape 0.1 ppm Fort 1	15.4.2013 10:52:32 PM	15-Apr-13	Fortification	0.100	14,825.0	0.2019	5.0	12.5	0.5%	0.1009	0.1009	101%
698252-0004	Grape 0.1 ppm Fort 2	15.4.2013 10:57:19 PM	15-Apr-13	Fortification	0.100	16,257.0	0.2212	5.0	12.5	0.5%	0.1106	0.1106	111%
698252-0004	Grape 0.1 ppm Fort 3	15.4.2013 11:02:07 PM	15-Apr-13	Fortification	0.100	16,534.0	0.2249	5.0	12.5	0.5%	0.1125	0.1125	112%
698252-0004	Grape 0.1 ppm Fort 4	15.4.2013 11:06:54 PM	15-Apr-13	Fortification	0.100	15,873.0	0.2160	5.0	12.5	0.5%	0.1080	0.1080	108%
Blank	Blank	15.4.2013 11:11:42 PM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
ERS13-383	0.02 ng/mL	15.4.2013 11:16:32 PM		Standard	0.020	1,564.0	0.0231						
698252-0004	Grape 0.1 ppm Fort 5	15.4.2013 11:21:24 PM	15-Apr-13	Fortification	0.100	16,158.0	0.2198	5.0	12.5	0.5%	0.1099	0.1099	110%
Reagent Blank	Reagent Blank	15.4.2013 11:26:14 PM	15-Apr-13	Control	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
698252-0001	Corn Forage Control Spike	15.4.2013 11:31:02 PM	15-Apr-13	Instrument Rec.	0.010	6,873.0	0.0946	5.0	2.5	0.5%	0.0095	0.0095	95%
698252-0004	Grape Control Spike	15.4.2013 11:35:49 PM	15-Apr-13	Instrument Rec.	0.010	7,523.0	0.1034	5.0	2.5	0.5%	0.0103	0.0103	103%
Blank	Blank	15.4.2013 11:40:37 PM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
ERS13-383	0.02 ng/mL	15.4.2013 11:45:24 PM		Standard	0.020	1,386.0	0.0207						
ERS13-382	0.04 ng/mL	15.4.2013 11:50:12 PM		Standard	0.040	3,344.0	0.0471						
ERS13-381	0.1 ng/mL	15.4.2013 11:54:59 PM		Standard	0.100	7,456.0	0.1025						
ERS13-380	0.2 ng/mL	15.4.2013 11:59:49 PM		Standard	0.200	15,495.0	0.2109						
ERS13-379	0.4 ng/mL	16.4.2013 12:04:36 AM		Standard	0.400	31,719.0	0.4296						

	correlation function:
concentration range from:	0.020
concentration range to:	0.400
intercept A =	-145.6939
slope B =	74164.8271
correlation coefficient	0.9918

limit of quantitation (LOQ) [mg/kg] = 0.01
limit of detection (LOD) [mg/kg] = 0.002

Instrument Rec. = Instrument recovery sample (used to test for matrix effects)

Master Sheet No.: 698252-01
Filename:1304\20130415BKDb.wiff
Analyte: Parent Topramezone (BAS 670H)
Comment: Secondary transition, m/z 362 to 318

Sample Description	Sample ID	Date Analyzed	Date Extracted	Sample Type	QC [mg/kg] conc. [ng/ml]	Peak Count	Calc. conc. [ng/ml]	Sample wt [g]	Final vol.[mL]	Aliquot factor	Calc. res. [mg/kg]	Result [mg/kg]	Rec. %
ERS13-383	0.02 ng/mL	16.4.2013 5:31:23 AM		Standard	0.020	955.0	0.0098						
ERS13-382	0.04 ng/mL	16.4.2013 5:40:11 AM		Standard	0.040	2,431.0	0.0380						
ERS13-381	0.1 ng/mL	16.4.2013 5:48:59 AM		Standard	0.100	6,089.0	0.1078						
ERS13-380	0.2 ng/mL	16.4.2013 5:57:47 AM		Standard	0.200	10,376.0	0.1897						
ERS13-379	0.4 ng/mL	16.4.2013 6:06:34 AM		Standard	0.400	19,704.0	0.3677						
Blank	Blank	16.4.2013 6:15:22 AM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
698252-0003	Dry Bean Control	16.4.2013 6:24:09 AM	15-Apr-13	Control	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
698252-0003	Dry Bean Control	16.4.2013 6:32:57 AM	15-Apr-13	Control	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
698252-0003	Dry Bean 0.01 ppm Fort 1	16.4.2013 6:41:45 AM	15-Apr-13	Fortification	0.010	6,329.0	0.1124	5.0	2.5	0.5%	0.0112	0.0112	112%
Blank	Blank	16.4.2013 6:50:33 AM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
ERS13-383	0.02 ng/mL	16.4.2013 6:59:20 AM		Standard	0.020	1,355.0	0.0174						
698252-0003	Dry Bean 0.01 ppm Fort 2	16.4.2013 7:08:08 AM	15-Apr-13	Fortification	0.010	5,389.0	0.0944	5.0	2.5	0.5%	0.0094	0.0094	94%
698252-0003	Dry Bean 0.01 ppm Fort 3	16.4.2013 7:16:55 AM	15-Apr-13	Fortification	0.010	4,841.0	0.0840	5.0	2.5	0.5%	0.0084	0.0084	84%
698252-0003	Dry Bean 0.01 ppm Fort 4	16.4.2013 7:25:43 AM	15-Apr-13	Fortification	0.010	5,904.0	0.1043	5.0	2.5	0.5%	0.0104	0.0104	104%
698252-0003	Dry Bean 0.01 ppm Fort 5	16.4.2013 7:34:30 AM	15-Apr-13	Fortification	0.010	6,253.0	0.1109	5.0	2.5	0.5%	0.0111	0.0111	111%
Blank	Blank	16.4.2013 7:43:17 AM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
ERS13-382	0.04 ng/mL	16.4.2013 7:52:04 AM		Standard	0.040	2,459.0	0.0385						
698252-0003	Dry Bean 0.1 ppm Fort 1	16.4.2013 8:00:51 AM	15-Apr-13	Fortification	0.100	12,710.0	0.2342	5.0	12.5	0.5%	0.1171	0.1171	117%
698252-0003	Dry Bean 0.1 ppm Fort 2	16.4.2013 8:09:39 AM	15-Apr-13	Fortification	0.100	11,680.0	0.2145	5.0	12.5	0.5%	0.1073	0.1073	107%
698252-0003	Dry Bean 0.1 ppm Fort 3	16.4.2013 8:18:27 AM	15-Apr-13	Fortification	0.100	12,290.0	0.2262	5.0	12.5	0.5%	0.1131	0.1131	113%
698252-0003	Dry Bean 0.1 ppm Fort 4	16.4.2013 8:27:15 AM	15-Apr-13	Fortification	0.100	11,730.0	0.2155	5.0	12.5	0.5%	0.1078	0.1078	108%
Blank	Blank	16.4.2013 8:36:02 AM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
ERS13-381	0.1 ng/mL	16.4.2013 8:44:50 AM		Standard	0.100	5,723.0	0.1008						
698252-0003	Dry Bean 0.1 ppm Fort 5	16.4.2013 8:53:37 AM	15-Apr-13	Fortification	0.100	9,968.0	0.1819	5.0	12.5	0.5%	0.0909	0.0909	91%
698252-0003	Dry Bean Control Spike	16.4.2013 9:02:26 AM	15-Apr-13	Instrument Rec.	0.010	5,990.0	0.1059	5.0	2.5	0.5%	0.0106	0.0106	106%
Blank	Blank	16.4.2013 9:11:13 AM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
ERS13-383	0.02 ng/mL	16.4.2013 9:20:02 AM		Standard	0.020	1,335.0	0.0171						
ERS13-382	0.04 ng/mL	16.4.2013 9:28:49 AM		Standard	0.040	2,451.0	0.0384						
ERS13-381	0.1 ng/mL	16.4.2013 9:37:36 AM		Standard	0.100	6,590.0	0.1174						
ERS13-380	0.2 ng/mL	16.4.2013 9:46:27 AM		Standard	0.200	11,465.0	0.2104						
ERS13-379	0.4 ng/mL	16.4.2013 9:55:16 AM		Standard	0.400	22,809.0	0.4270						

	correlation function:
concentration range from:	0.020
concentration range to:	0.400
intercept A =	441.6913
slope B =	52380.9601
correlation coefficient	0.9944

limit of quantitation (LOQ) [mg/kg] = 0.01
limit of detection (LOD) [mg/kg] = 0.002

Instrument Rec. = Instrument recovery sample (used to test for matrix effects)

Master Sheet No.: 698252-01
Filename:1304\20130415BKDa.wiff
Analyte: M670H05
Comment: Primary transition, m/z 282 to 238

Sample Description	Sample ID	Date Analyzed	Date Extracted	Sample Type	QC [mg/kg] conc. [ng/ml]	Peak Count	Calc. conc. [ng/ml]	Sample wt [g]	Final vol.[mL]	Aliquot factor	Calc. res. [mg/kg]	Result [mg/kg]	Rec. %
ERS13-383	0.02 ng/mL	15.4.2013 8:04:26 PM		Standard	0.020	7,867.0	0.0198						
ERS13-382	0.04 ng/mL	15.4.2013 8:09:14 PM		Standard	0.040	15,801.0	0.0393						
ERS13-381	0.1 ng/mL	15.4.2013 8:14:03 PM		Standard	0.100	39,645.0	0.0980						
ERS13-380	0.2 ng/mL	15.4.2013 8:18:51 PM		Standard	0.200	78,720.0	0.1943						
ERS13-379	0.4 ng/mL	15.4.2013 8:23:38 PM		Standard	0.400	156,464.0	0.3858						
Blank	Blank	15.4.2013 8:28:27 PM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
698252-0001	Corn Forage Control	15.4.2013 8:33:14 PM	15-Apr-13	Control	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
698252-0001	Corn Forage Control	15.4.2013 8:38:01 PM	15-Apr-13	Control	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
698252-0001	Corn Forage 0.01 ppm Fort 1	15.4.2013 8:42:48 PM	15-Apr-13	Fortification	0.010	33,980.0	0.0841	5.0	2.5	0.5%	0.0084	0.0084	84%
Blank	Blank	15.4.2013 8:47:35 PM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
ERS13-383	0.02 ng/mL	15.4.2013 8:52:24 PM		Standard	0.020	7,629.0	0.0192						
698252-0001	Corn Forage 0.01 ppm Fort 2	15.4.2013 8:57:12 PM	15-Apr-13	Fortification	0.010	35,354.0	0.0875	5.0	2.5	0.5%	0.0087	0.0087	87%
698252-0001	Corn Forage 0.01 ppm Fort 3	15.4.2013 9:02:00 PM	15-Apr-13	Fortification	0.010	33,517.0	0.0829	5.0	2.5	0.5%	0.0083	0.0083	83%
698252-0001	Corn Forage 0.01 ppm Fort 4	15.4.2013 9:06:48 PM	15-Apr-13	Fortification	0.010	31,708.0	0.0785	5.0	2.5	0.5%	0.0078	0.0078	78%
698252-0001	Corn Forage 0.01 ppm Fort 5	15.4.2013 9:11:36 PM	15-Apr-13	Fortification	0.010	33,501.0	0.0829	5.0	2.5	0.5%	0.0083	0.0083	83%
Blank	Blank	15.4.2013 9:16:23 PM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
ERS13-382	0.04 ng/mL	15.4.2013 9:21:09 PM		Standard	0.040	16,915.0	0.0421						
698252-0001	Corn Forage 0.1 ppm Fort 1	15.4.2013 9:25:57 PM	15-Apr-13	Fortification	0.100	109,113.0	0.2691	5.0	12.5	0.5%	0.1346	0.1346	135%
698252-0001	Corn Forage 0.1 ppm Fort 2	15.4.2013 9:30:45 PM	15-Apr-13	Fortification	0.100	107,736.0	0.2657	5.0	12.5	0.5%	0.1329	0.1329	133%
698252-0001	Corn Forage 0.1 ppm Fort 3	15.4.2013 9:35:32 PM	15-Apr-13	Fortification	0.100	109,599.0	0.2703	5.0	12.5	0.5%	0.1352	0.1352	135%
698252-0001	Corn Forage 0.1 ppm Fort 4	15.4.2013 9:40:22 PM	15-Apr-13	Fortification	0.100	106,012.0	0.2615	5.0	12.5	0.5%	0.1307	0.1307	131%
Blank	Blank	15.4.2013 9:45:10 PM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
ERS13-381	0.1 ng/mL	15.4.2013 9:49:56 PM		Standard	0.100	41,088.0	0.1016						
698252-0001	Corn Forage 0.1 ppm Fort 5	15.4.2013 9:54:46 PM	15-Apr-13	Fortification	0.100	108,008.0	0.2664	5.0	12.5	0.5%	0.1332	0.1332	133%
698252-0004	Grape Control	15.4.2013 9:59:32 PM	15-Apr-13	Control	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
698252-0004	Grape Control	15.4.2013 10:04:22 PM	15-Apr-13	Control	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
698252-0004	Grape 0.01 ppm Fort 1	15.4.2013 10:09:14 PM	15-Apr-13	Fortification	0.010	34,291.0	0.0849	5.0	2.5	0.5%	0.0085	0.0085	85%
Blank	Blank	15.4.2013 10:14:02 PM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
ERS13-380	0.2 ng/mL	15.4.2013 10:18:53 PM		Standard	0.200	82,724.0	0.2041						
698252-0004	Grape 0.01 ppm Fort 2	15.4.2013 10:23:44 PM	15-Apr-13	Fortification	0.010	36,103.0	0.0893	5.0	2.5	0.5%	0.0089	0.0089	89%
698252-0004	Grape 0.01 ppm Fort 3	15.4.2013 10:28:34 PM	15-Apr-13	Fortification	0.010	35,382.0	0.0875	5.0	2.5	0.5%	0.0088	0.0088	88%
698252-0004	Grape 0.01 ppm Fort 4	15.4.2013 10:33:21 PM	15-Apr-13	Fortification	0.010	33,329.0	0.0825	5.0	2.5	0.5%	0.0082	0.0082	82%
698252-0004	Grape 0.01 ppm Fort 5	15.4.2013 10:38:09 PM	15-Apr-13	Fortification	0.010	34,869.0	0.0863	5.0	2.5	0.5%	0.0086	0.0086	86%
Blank	Blank	15.4.2013 10:42:57 PM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
ERS13-379	0.4 ng/mL	15.4.2013 10:47:44 PM		Standard	0.400	164,196.0	0.4048						
698252-0004	Grape 0.1 ppm Fort 1	15.4.2013 10:52:32 PM	15-Apr-13	Fortification	0.100	108,762.0	0.2683	5.0	12.5	0.5%	0.1341	0.1341	134%
698252-0004	Grape 0.1 ppm Fort 2	15.4.2013 10:57:19 PM	15-Apr-13	Fortification	0.100	109,025.0	0.2689	5.0	12.5	0.5%	0.1345	0.1345	134%
698252-0004	Grape 0.1 ppm Fort 3	15.4.2013 11:02:07 PM	15-Apr-13	Fortification	0.100	107,622.0	0.2655	5.0	12.5	0.5%	0.1327	0.1327	133%
698252-0004	Grape 0.1 ppm Fort 4	15.4.2013 11:06:54 PM	15-Apr-13	Fortification	0.100	109,375.0	0.2698	5.0	12.5	0.5%	0.1349	0.1349	135%
Blank	Blank	15.4.2013 11:11:42 PM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
ERS13-383	0.02 ng/mL	15.4.2013 11:16:32 PM		Standard	0.020	7,845.0	0.0197						
698252-0004	Grape 0.1 ppm Fort 5	15.4.2013 11:21:24 PM	15-Apr-13	Fortification	0.100	111,462.0	0.2749	5.0	12.5	0.5%	0.1375	0.1375	137%
Reagent Blank	Reagent Blank	15.4.2013 11:26:14 PM	15-Apr-13	Control	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
698252-0001	Corn Forage Control Spike	15.4.2013 11:31:02 PM	15-Apr-13	Instrument Rec.	0.010	31,537.0	0.0781	5.0	2.5	0.5%	0.0078	0.0078	78%
698252-0004	Grape Control Spike	15.4.2013 11:35:49 PM	15-Apr-13	Instrument Rec.	0.010	33,528.0	0.0830	5.0	2.5	0.5%	0.0083	0.0083	83%
Blank	Blank	15.4.2013 11:40:37 PM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
ERS13-383	0.02 ng/mL	15.4.2013 11:45:24 PM		Standard	0.020	7,633.0	0.0192						
ERS13-382	0.04 ng/mL	15.4.2013 11:50:12 PM		Standard	0.040	15,916.0	0.0396						
ERS13-381	0.1 ng/mL	15.4.2013 11:54:59 PM		Standard	0.100	41,578.0	0.1028						
ERS13-380	0.2 ng/mL	15.4.2013 11:59:49 PM		Standard	0.200	81,160.0	0.2003						
ERS13-379	0.4 ng/mL	16.4.2013 12:04:36 AM		Standard	0.400	166,109.0	0.4095						

	correlation function:
concentration range from:	0.020
concentration range to:	0.400
intercept A =	-160.0119
slope B =	406021.8216
correlation coefficient	0.9994

limit of quantitation (LOQ) [mg/kg] = 0.01
limit of detection (LOD) [mg/kg] = 0.002

Instrument Rec. = Instrument recovery sample (used to test for matrix effects)

Master Sheet No.: 698252-01
Filename:1304\20130415BKDb.wiff
Analyte: M670H05
Comment: Primary transition, m/z 282 to 238

Sample Description	Sample ID	Date Analyzed	Date Extracted	Sample Type	QC [mg/kg] conc. [ng/ml]	Peak Count	Calc. conc. [ng/ml]	Sample wt [g]	Final vol.[mL]	Aliquot factor	Calc. res. [mg/kg]	Result [mg/kg]	Rec. %
ERS13-383	0.02 ng/mL	16.4.2013 5:31:23 AM		Standard	0.020	7,967.0	0.0185						
ERS13-382	0.04 ng/mL	16.4.2013 5:40:11 AM		Standard	0.040	15,961.0	0.0388						
ERS13-381	0.1 ng/mL	16.4.2013 5:48:59 AM		Standard	0.100	41,748.0	0.1043						
ERS13-380	0.2 ng/mL	16.4.2013 5:57:47 AM		Standard	0.200	82,159.0	0.2068						
ERS13-379	0.4 ng/mL	16.4.2013 6:06:34 AM		Standard	0.400	159,076.0	0.4020						
Blank	Blank	16.4.2013 6:15:22 AM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
698252-0003	Dry Bean Control	16.4.2013 6:24:09 AM	15-Apr-13	Control	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
698252-0003	Dry Bean Control	16.4.2013 6:32:57 AM	15-Apr-13	Control	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
698252-0003	Dry Bean 0.01 ppm Fort 1	16.4.2013 6:41:45 AM	15-Apr-13	Fortification	0.010	39,253.0	0.0979	5.0	2.5	0.5%	0.0098	0.0098	98%
Blank	Blank	16.4.2013 6:50:33 AM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
ERS13-383	0.02 ng/mL	16.4.2013 6:59:20 AM		Standard	0.020	7,936.0	0.0185						
698252-0003	Dry Bean 0.01 ppm Fort 2	16.4.2013 7:08:08 AM	15-Apr-13	Fortification	0.010	38,788.0	0.0968	5.0	2.5	0.5%	0.0097	0.0097	97%
698252-0003	Dry Bean 0.01 ppm Fort 3	16.4.2013 7:16:55 AM	15-Apr-13	Fortification	0.010	39,746.0	0.0992	5.0	2.5	0.5%	0.0099	0.0099	99%
698252-0003	Dry Bean 0.01 ppm Fort 4	16.4.2013 7:25:43 AM	15-Apr-13	Fortification	0.010	43,339.0	0.1083	5.0	2.5	0.5%	0.0108	0.0108	108%
698252-0003	Dry Bean 0.01 ppm Fort 5	16.4.2013 7:34:30 AM	15-Apr-13	Fortification	0.010	40,325.0	0.1007	5.0	2.5	0.5%	0.0101	0.0101	101%
Blank	Blank	16.4.2013 7:43:17 AM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
ERS13-382	0.04 ng/mL	16.4.2013 7:52:04 AM		Standard	0.040	15,799.0	0.0384						
698252-0003	Dry Bean 0.1 ppm Fort 1	16.4.2013 8:00:51 AM	15-Apr-13	Fortification	0.100	103,143.0	0.2601	5.0	12.5	0.5%	0.1300	0.1300	130%
698252-0003	Dry Bean 0.1 ppm Fort 2	16.4.2013 8:09:39 AM	15-Apr-13	Fortification	0.100	103,796.0	0.2618	5.0	12.5	0.5%	0.1309	0.1309	131%
698252-0003	Dry Bean 0.1 ppm Fort 3	16.4.2013 8:18:27 AM	15-Apr-13	Fortification	0.100	103,719.0	0.2616	5.0	12.5	0.5%	0.1308	0.1308	131%
698252-0003	Dry Bean 0.1 ppm Fort 4	16.4.2013 8:27:15 AM	15-Apr-13	Fortification	0.100	99,564.0	0.2510	5.0	12.5	0.5%	0.1255	0.1255	126%
Blank	Blank	16.4.2013 8:36:02 AM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
ERS13-381	0.1 ng/mL	16.4.2013 8:44:50 AM		Standard	0.100	41,284.0	0.1031						
698252-0003	Dry Bean 0.1 ppm Fort 5	16.4.2013 8:53:37 AM	15-Apr-13	Fortification	0.100	90,815.0	0.2288	5.0	12.5	0.5%	0.1144	0.1144	114%
698252-0003	Dry Bean Control Spike	16.4.2013 9:02:26 AM	15-Apr-13	Instrument Rec.	0.010	39,076.0	0.0975	5.0	2.5	0.5%	0.0097	0.0097	97%
Blank	Blank	16.4.2013 9:11:13 AM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
ERS13-383	0.02 ng/mL	16.4.2013 9:20:02 AM		Standard	0.020	8,155.0	0.0190						
ERS13-382	0.04 ng/mL	16.4.2013 9:28:49 AM		Standard	0.040	16,117.0	0.0392						
ERS13-381	0.1 ng/mL	16.4.2013 9:37:36 AM		Standard	0.100	40,647.0	0.1015						
ERS13-380	0.2 ng/mL	16.4.2013 9:46:27 AM		Standard	0.200	77,017.0	0.1938						
ERS13-379	0.4 ng/mL	16.4.2013 9:55:16 AM		Standard	0.400	156,683.0	0.3960						

	correlation function:
concentration range from:	0.020
concentration range to:	0.400
intercept A =	661.5212
slope B =	394017.3953
correlation coefficient	0.9997

limit of quantitation (LOQ) [mg/kg] = 0.01
limit of detection (LOD) [mg/kg] = 0.002

Instrument Rec. = Instrument recovery sample (used to test for matrix effects)

Master Sheet No.: 698252-01
Filename:1304\20130415BKDa.wiff
Analyte: M670H05
Comment: Secondary transition, m/z 282 to 79

Sample Description	Sample ID	Date Analyzed	Date Extracted	Sample Type	QC [mg/kg] conc. [ng/ml]	Peak Count	Calc. conc. [ng/ml]	Sample wt [g]	Final vol.[mL]	Aliquot factor	Calc. res. [mg/kg]	Result [mg/kg]	Rec. %
ERS13-383	0.02 ng/mL	15.4.2013 8:04:26 PM		Standard	0.020	1,275.0	0.0190						
ERS13-382	0.04 ng/mL	15.4.2013 8:09:14 PM		Standard	0.040	2,428.0	0.0362						
ERS13-381	0.1 ng/mL	15.4.2013 8:14:03 PM		Standard	0.100	6,269.0	0.0936						
ERS13-380	0.2 ng/mL	15.4.2013 8:18:51 PM		Standard	0.200	12,655.0	0.1889						
ERS13-379	0.4 ng/mL	15.4.2013 8:23:38 PM		Standard	0.400	25,491.0	0.3806						
Blank	Blank	15.4.2013 8:28:27 PM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
698252-0001	Corn Forage Control	15.4.2013 8:33:14 PM	15-Apr-13	Control	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
698252-0001	Corn Forage Control	15.4.2013 8:38:01 PM	15-Apr-13	Control	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
698252-0001	Corn Forage 0.01 ppm Fort 1	15.4.2013 8:42:48 PM	15-Apr-13	Fortification	0.010	5,813.0	0.0868	5.0	2.5	0.5%	0.0087	0.0087	87%
Blank	Blank	15.4.2013 8:47:35 PM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
ERS13-383	0.02 ng/mL	15.4.2013 8:52:24 PM		Standard	0.020	1,407.0	0.0210						
698252-0001	Corn Forage 0.01 ppm Fort 2	15.4.2013 8:57:12 PM	15-Apr-13	Fortification	0.010	5,673.0	0.0847	5.0	2.5	0.5%	0.0085	0.0085	85%
698252-0001	Corn Forage 0.01 ppm Fort 3	15.4.2013 9:02:00 PM	15-Apr-13	Fortification	0.010	5,220.0	0.0779	5.0	2.5	0.5%	0.0078	0.0078	78%
698252-0001	Corn Forage 0.01 ppm Fort 4	15.4.2013 9:06:48 PM	15-Apr-13	Fortification	0.010	6,230.0	0.0930	5.0	2.5	0.5%	0.0093	0.0093	93%
698252-0001	Corn Forage 0.01 ppm Fort 5	15.4.2013 9:11:36 PM	15-Apr-13	Fortification	0.010	5,705.0	0.0852	5.0	2.5	0.5%	0.0085	0.0085	85%
Blank	Blank	15.4.2013 9:16:23 PM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
ERS13-382	0.04 ng/mL	15.4.2013 9:21:09 PM		Standard	0.040	2,796.0	0.0417						
698252-0001	Corn Forage 0.1 ppm Fort 1	15.4.2013 9:25:57 PM	15-Apr-13	Fortification	0.100	17,798.0	0.2657	5.0	12.5	0.5%	0.1329	0.1329	133%
698252-0001	Corn Forage 0.1 ppm Fort 2	15.4.2013 9:30:45 PM	15-Apr-13	Fortification	0.100	17,237.0	0.2574	5.0	12.5	0.5%	0.1287	0.1287	129%
698252-0001	Corn Forage 0.1 ppm Fort 3	15.4.2013 9:35:32 PM	15-Apr-13	Fortification	0.100	18,336.0	0.2738	5.0	12.5	0.5%	0.1369	0.1369	137%
698252-0001	Corn Forage 0.1 ppm Fort 4	15.4.2013 9:40:22 PM	15-Apr-13	Fortification	0.100	17,622.0	0.2631	5.0	12.5	0.5%	0.1316	0.1316	132%
Blank	Blank	15.4.2013 9:45:10 PM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
ERS13-381	0.1 ng/mL	15.4.2013 9:49:56 PM		Standard	0.100	6,856.0	0.1023						
698252-0001	Corn Forage 0.1 ppm Fort 5	15.4.2013 9:54:46 PM	15-Apr-13	Fortification	0.100	17,609.0	0.2629	5.0	12.5	0.5%	0.1315	0.1315	131%
698252-0004	Grape Control	15.4.2013 9:59:32 PM	15-Apr-13	Control	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
698252-0004	Grape Control	15.4.2013 10:04:22 PM	15-Apr-13	Control	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
698252-0004	Grape 0.01 ppm Fort 1	15.4.2013 10:09:14 PM	15-Apr-13	Fortification	0.010	5,653.0	0.0844	5.0	2.5	0.5%	0.0084	0.0084	84%
Blank	Blank	15.4.2013 10:14:02 PM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
ERS13-380	0.2 ng/mL	15.4.2013 10:18:53 PM		Standard	0.200	13,494.0	0.2015						
698252-0004	Grape 0.01 ppm Fort 2	15.4.2013 10:23:44 PM	15-Apr-13	Fortification	0.010	5,910.0	0.0882	5.0	2.5	0.5%	0.0088	0.0088	88%
698252-0004	Grape 0.01 ppm Fort 3	15.4.2013 10:28:34 PM	15-Apr-13	Fortification	0.010	5,648.0	0.0843	5.0	2.5	0.5%	0.0084	0.0084	84%
698252-0004	Grape 0.01 ppm Fort 4	15.4.2013 10:33:21 PM	15-Apr-13	Fortification	0.010	5,827.0	0.0870	5.0	2.5	0.5%	0.0087	0.0087	87%
698252-0004	Grape 0.01 ppm Fort 5	15.4.2013 10:38:09 PM	15-Apr-13	Fortification	0.010	5,761.0	0.0860	5.0	2.5	0.5%	0.0086	0.0086	86%
Blank	Blank	15.4.2013 10:42:57 PM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
ERS13-379	0.4 ng/mL	15.4.2013 10:47:44 PM		Standard	0.400	28,150.0	0.4203						
698252-0004	Grape 0.1 ppm Fort 1	15.4.2013 10:52:32 PM	15-Apr-13	Fortification	0.100	17,438.0	0.2604	5.0	12.5	0.5%	0.1302	0.1302	130%
698252-0004	Grape 0.1 ppm Fort 2	15.4.2013 10:57:19 PM	15-Apr-13	Fortification	0.100	17,952.0	0.2680	5.0	12.5	0.5%	0.1340	0.1340	134%
698252-0004	Grape 0.1 ppm Fort 3	15.4.2013 11:02:07 PM	15-Apr-13	Fortification	0.100	17,723.0	0.2646	5.0	12.5	0.5%	0.1323	0.1323	132%
698252-0004	Grape 0.1 ppm Fort 4	15.4.2013 11:06:54 PM	15-Apr-13	Fortification	0.100	18,180.0	0.2714	5.0	12.5	0.5%	0.1357	0.1357	136%
Blank	Blank	15.4.2013 11:11:42 PM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
ERS13-383	0.02 ng/mL	15.4.2013 11:16:32 PM		Standard	0.020	1,291.0	0.0192						
698252-0004	Grape 0.1 ppm Fort 5	15.4.2013 11:21:24 PM	15-Apr-13	Fortification	0.100	17,191.0	0.2567	5.0	12.5	0.5%	0.1283	0.1283	128%
Reagent Blank	Reagent Blank	15.4.2013 11:26:14 PM	15-Apr-13	Control	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
698252-0001	Corn Forage Control Spike	15.4.2013 11:31:02 PM	15-Apr-13	Instrument Rec.	0.010	5,405.0	0.0807	5.0	2.5	0.5%	0.0081	0.0081	81%
698252-0004	Grape Control Spike	15.4.2013 11:35:49 PM	15-Apr-13	Instrument Rec.	0.010	5,748.0	0.0858	5.0	2.5	0.5%	0.0086	0.0086	86%
Blank	Blank	15.4.2013 11:40:37 PM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
ERS13-383	0.02 ng/mL	15.4.2013 11:45:24 PM		Standard	0.020	1,328.0	0.0198						
ERS13-382	0.04 ng/mL	15.4.2013 11:50:12 PM		Standard	0.040	2,836.0	0.0423						
ERS13-381	0.1 ng/mL	15.4.2013 11:54:59 PM		Standard	0.100	7,263.0	0.1084						
ERS13-380	0.2 ng/mL	15.4.2013 11:59:49 PM		Standard	0.200	13,703.0	0.2046						
ERS13-379	0.4 ng/mL	16.4.2013 12:04:36 AM		Standard	0.400	26,825.0	0.4005						

	correlation function:
concentration range from:	0.020
concentration range to:	0.400
intercept A =	2.6557
slope B =	66967.1777
correlation coefficient	0.9982

limit of quantitation (LOQ) [mg/kg] = 0.01
limit of detection (LOD) [mg/kg] = 0.002

Instrument Rec. = Instrument recovery sample (used to test for matrix effects)

Master Sheet No.: 698252-01
Filename:1304\20130415BKDb.wiff
Analyte: M670H05
Comment: Secondary transition, m/z 282 to 79

Sample Description	Sample ID	Date Analyzed	Date Extracted	Sample Type	QC [mg/kg] conc. [ng/ml]	Peak Count	Calc. conc. [ng/ml]	Sample wt [g]	Final vol.[mL]	Aliquot factor	Calc. res. [mg/kg]	Result [mg/kg]	Rec. %
ERS13-383	0.02 ng/mL	16.4.2013 5:31:23 AM		Standard	0.020	1,404.0	0.0197						
ERS13-382	0.04 ng/mL	16.4.2013 5:40:11 AM		Standard	0.040	2,606.0	0.0383						
ERS13-381	0.1 ng/mL	16.4.2013 5:48:59 AM		Standard	0.100	7,217.0	0.1100						
ERS13-380	0.2 ng/mL	16.4.2013 5:57:47 AM		Standard	0.200	13,532.0	0.2082						
ERS13-379	0.4 ng/mL	16.4.2013 6:06:34 AM		Standard	0.400	25,949.0	0.4012						
Blank	Blank	16.4.2013 6:15:22 AM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
698252-0003	Dry Bean Control	16.4.2013 6:24:09 AM	15-Apr-13	Control	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
698252-0003	Dry Bean Control	16.4.2013 6:32:57 AM	15-Apr-13	Control	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
698252-0003	Dry Bean 0.01 ppm Fort 1	16.4.2013 6:41:45 AM	15-Apr-13	Fortification	0.010	6,779.0	0.1032	5.0	2.5	0.5%	0.0103	0.0103	103%
Blank	Blank	16.4.2013 6:50:33 AM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
ERS13-383	0.02 ng/mL	16.4.2013 6:59:20 AM		Standard	0.020	1,176.0	0.0161						
698252-0003	Dry Bean 0.01 ppm Fort 2	16.4.2013 7:08:08 AM	15-Apr-13	Fortification	0.010	6,580.0	0.1001	5.0	2.5	0.5%	0.0100	0.0100	100%
698252-0003	Dry Bean 0.01 ppm Fort 3	16.4.2013 7:16:55 AM	15-Apr-13	Fortification	0.010	6,184.0	0.0940	5.0	2.5	0.5%	0.0094	0.0094	94%
698252-0003	Dry Bean 0.01 ppm Fort 4	16.4.2013 7:25:43 AM	15-Apr-13	Fortification	0.010	6,679.0	0.1017	5.0	2.5	0.5%	0.0102	0.0102	102%
698252-0003	Dry Bean 0.01 ppm Fort 5	16.4.2013 7:34:30 AM	15-Apr-13	Fortification	0.010	6,600.0	0.1004	5.0	2.5	0.5%	0.0100	0.0100	100%
Blank	Blank	16.4.2013 7:43:17 AM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
ERS13-382	0.04 ng/mL	16.4.2013 7:52:04 AM		Standard	0.040	2,522.0	0.0370						
698252-0003	Dry Bean 0.1 ppm Fort 1	16.4.2013 8:00:51 AM	15-Apr-13	Fortification	0.100	16,873.0	0.2601	5.0	12.5	0.5%	0.1301	0.1301	130%
698252-0003	Dry Bean 0.1 ppm Fort 2	16.4.2013 8:09:39 AM	15-Apr-13	Fortification	0.100	17,239.0	0.2658	5.0	12.5	0.5%	0.1329	0.1329	133%
698252-0003	Dry Bean 0.1 ppm Fort 3	16.4.2013 8:18:27 AM	15-Apr-13	Fortification	0.100	17,442.0	0.2690	5.0	12.5	0.5%	0.1345	0.1345	134%
698252-0003	Dry Bean 0.1 ppm Fort 4	16.4.2013 8:27:15 AM	15-Apr-13	Fortification	0.100	16,541.0	0.2550	5.0	12.5	0.5%	0.1275	0.1275	127%
Blank	Blank	16.4.2013 8:36:02 AM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
ERS13-381	0.1 ng/mL	16.4.2013 8:44:50 AM		Standard	0.100	6,667.0	0.1015						
698252-0003	Dry Bean 0.1 ppm Fort 5	16.4.2013 8:53:37 AM	15-Apr-13	Fortification	0.100	14,555.0	0.2241	5.0	12.5	0.5%	0.1120	0.1120	112%
698252-0003	Dry Bean Control Spike	16.4.2013 9:02:26 AM	15-Apr-13	Instrument Rec.	0.010	6,120.0	0.0930	5.0	2.5	0.5%	0.0093	0.0093	93%
Blank	Blank	16.4.2013 9:11:13 AM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
ERS13-383	0.02 ng/mL	16.4.2013 9:20:02 AM		Standard	0.020	1,102.0	0.0150						
ERS13-382	0.04 ng/mL	16.4.2013 9:28:49 AM		Standard	0.040	2,595.0	0.0382						
ERS13-381	0.1 ng/mL	16.4.2013 9:37:36 AM		Standard	0.100	7,104.0	0.1083						
ERS13-380	0.2 ng/mL	16.4.2013 9:46:27 AM		Standard	0.200	12,445.0	0.1913						
ERS13-379	0.4 ng/mL	16.4.2013 9:55:16 AM		Standard	0.400	25,563.0	0.3952						

	correlation function:
concentration range from:	0.020
concentration range to:	0.400
intercept A =	138.9975
slope B =	64330.3767
correlation coefficient	0.9991

limit of quantitation (LOQ) [mg/kg] = 0.01
limit of detection (LOD) [mg/kg] = 0.002

Instrument Rec. = Instrument recovery sample (used to test for matrix effects)

Master Sheet No.: 698252-02
Filename:1304\20130415BKDc.wiff
Analyte: Parent Topramezone (BAS 670H)
Comment: Primary transition, m/z 362 to 334

Sample Description	Sample ID	Date Analyzed	Date Extracted	Sample Type	QC [mg/kg] conc. [ng/ml]	Peak Count	Calc. conc. [ng/ml]	Sample wt [g]	Final vol.[mL]	Aliquot factor	Calc. res. [mg/kg]	Result [mg/kg]	Rec. %
ERS13-383	0.02 ng/mL	16.4.2013 12:38:16 AM		Standard	0.020	3,315.0	0.0215						
ERS13-382	0.04 ng/mL	16.4.2013 12:43:03 AM		Standard	0.040	6,238.0	0.0414						
ERS13-381	0.1 ng/mL	16.4.2013 12:47:51 AM		Standard	0.100	15,530.0	0.1044						
ERS13-380	0.2 ng/mL	16.4.2013 12:52:38 AM		Standard	0.200	29,242.0	0.1975						
ERS13-379	0.4 ng/mL	16.4.2013 12:57:28 AM		Standard	0.400	58,705.0	0.3975						
Blank	Blank	16.4.2013 1:02:15 AM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
698252-0002	Corn Grain Control	16.4.2013 1:07:05 AM	15-Apr-13	Control	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
698252-0002	Corn Grain Control	16.4.2013 1:11:56 AM	15-Apr-13	Control	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
698252-0002	Corn Grain 0.01 ppm Fort 1	16.4.2013 1:16:43 AM	15-Apr-13	Fortification	0.010	11,823.0	0.0793	5.0	2.5	0.5%	0.0079	0.0079	79%
Blank	Blank	16.4.2013 1:21:31 AM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
ERS13-383	0.02 ng/mL	16.4.2013 1:26:19 AM		Standard	0.020	3,610.0	0.0235						
698252-0002	Corn Grain 0.01 ppm Fort 2	16.4.2013 1:31:07 AM	15-Apr-13	Fortification	0.010	12,512.0	0.0840	5.0	2.5	0.5%	0.0084	0.0084	84%
698252-0002	Corn Grain 0.01 ppm Fort 3	16.4.2013 1:35:55 AM	15-Apr-13	Fortification	0.010	12,837.0	0.0862	5.0	2.5	0.5%	0.0086	0.0086	86%
698252-0002	Corn Grain 0.01 ppm Fort 4	16.4.2013 1:40:42 AM	15-Apr-13	Fortification	0.010	13,505.0	0.0907	5.0	2.5	0.5%	0.0091	0.0091	91%
698252-0002	Corn Grain 0.01 ppm Fort 5	16.4.2013 1:45:29 AM	15-Apr-13	Fortification	0.010	11,788.0	0.0790	5.0	2.5	0.5%	0.0079	0.0079	79%
Blank	Blank	16.4.2013 1:50:20 AM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
ERS13-382	0.04 ng/mL	16.4.2013 1:55:08 AM		Standard	0.040	5,829.0	0.0386						
698252-0002	Corn Grain 0.1 ppm Fort 1	16.4.2013 1:59:56 AM	15-Apr-13	Fortification	0.100	28,429.0	0.1920	5.0	12.5	0.5%	0.0960	0.0960	96%
698252-0002	Corn Grain 0.1 ppm Fort 2	16.4.2013 2:04:43 AM	15-Apr-13	Fortification	0.100	29,391.0	0.1985	5.0	12.5	0.5%	0.0993	0.0993	99%
698252-0002	Corn Grain 0.1 ppm Fort 3	16.4.2013 2:09:30 AM	15-Apr-13	Fortification	0.100	29,803.0	0.2013	5.0	12.5	0.5%	0.1007	0.1007	101%
698252-0002	Corn Grain 0.1 ppm Fort 4	16.4.2013 2:14:18 AM	15-Apr-13	Fortification	0.100	30,573.0	0.2065	5.0	12.5	0.5%	0.1033	0.1033	103%
Blank	Blank	16.4.2013 2:19:06 AM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
ERS13-381	0.1 ng/mL	16.4.2013 2:23:53 AM		Standard	0.100	13,591.0	0.0913						
698252-0002	Corn Grain 0.1 ppm Fort 5	16.4.2013 2:28:41 AM	15-Apr-13	Fortification	0.100	27,786.0	0.1876	5.0	12.5	0.5%	0.0938	0.0938	94%
698252-0005	Sunflower Seed Control	16.4.2013 2:33:32 AM	15-Apr-13	Control	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
698252-0005	Sunflower Seed Control	16.4.2013 2:38:19 AM	15-Apr-13	Control	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
698252-0005	Sunflower Seed 0.01 ppm Fort 1	16.4.2013 2:43:07 AM	15-Apr-13	Fortification	0.010	13,623.0	0.0915	5.0	2.5	0.5%	0.0092	0.0092	92%
Blank	Blank	16.4.2013 2:47:55 AM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
ERS13-380	0.2 ng/mL	16.4.2013 2:52:43 AM		Standard	0.200	31,754.0	0.2146						
698252-0005	Sunflower Seed 0.01 ppm Fort 2	16.4.2013 2:57:31 AM	15-Apr-13	Fortification	0.010	16,560.0	0.1114	5.0	2.5	0.5%	0.0111	0.0111	111%
698252-0005	Sunflower Seed 0.01 ppm Fort 3	16.4.2013 3:02:19 AM	15-Apr-13	Fortification	0.010	14,625.0	0.0983	5.0	2.5	0.5%	0.0098	0.0098	98%
698252-0005	Sunflower Seed 0.01 ppm Fort 4	16.4.2013 3:07:08 AM	15-Apr-13	Fortification	0.010	13,031.0	0.0875	5.0	2.5	0.5%	0.0087	0.0087	87%
698252-0005	Sunflower Seed 0.01 ppm Fort 5	16.4.2013 3:11:56 AM	15-Apr-13	Fortification	0.010	40,277.0	0.2724	5.0	2.5	0.5%	0.0272	0.0272	272%
Blank	Blank	16.4.2013 3:16:45 AM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
ERS13-379	0.4 ng/mL	16.4.2013 3:21:35 AM		Standard	0.400	59,740.0	0.4045						
698252-0005	Sunflower Seed 0.1 ppm Fort 1	16.4.2013 3:26:23 AM	15-Apr-13	Fortification	0.100	28,849.0	0.1948	5.0	12.5	0.5%	0.0974	0.0974	97%
698252-0005	Sunflower Seed 0.1 ppm Fort 2	16.4.2013 3:31:10 AM	15-Apr-13	Fortification	0.100	27,200.0	0.1836	5.0	12.5	0.5%	0.0918	0.0918	92%
698252-0005	Sunflower Seed 0.1 ppm Fort 3	16.4.2013 3:35:57 AM	15-Apr-13	Fortification	0.100	27,178.0	0.1835	5.0	12.5	0.5%	0.0917	0.0917	92%
698252-0005	Sunflower Seed 0.1 ppm Fort 4	16.4.2013 3:40:45 AM	15-Apr-13	Fortification	0.100	26,999.0	0.1823	5.0	12.5	0.5%	0.0911	0.0911	91%
Blank	Blank	16.4.2013 3:45:35 AM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
ERS13-383	0.02 ng/mL	16.4.2013 3:50:23 AM		Standard	0.020	3,263.0	0.0212						
698252-0005	Sunflower Seed 0.1 ppm Fort 5	16.4.2013 3:55:11 AM	15-Apr-13	Fortification	0.100	27,165.0	0.1834	5.0	12.5	0.5%	0.0917	0.0917	92%
Reagent Blank	Reagent Blank	16.4.2013 3:59:59 AM	15-Apr-13	Control	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
698252-0002	Corn Grain Control Spike	16.4.2013 4:04:50 AM	15-Apr-13	Instrument Rec.	0.010	14,004.0	0.0941	5.0	2.5	0.5%	0.0094	0.0094	94%
Blank	Blank	16.4.2013 4:09:38 AM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
ERS13-383	0.02 ng/mL	16.4.2013 4:14:29 AM		Standard	0.020	3,388.0	0.0220						
ERS13-382	0.04 ng/mL	16.4.2013 4:19:18 AM		Standard	0.040	5,454.0	0.0361						
ERS13-381	0.1 ng/mL	16.4.2013 4:24:05 AM		Standard	0.100	13,529.0	0.0909						
ERS13-380	0.2 ng/mL	16.4.2013 4:28:56 AM		Standard	0.200	29,515.0	0.1994						
ERS13-379	0.4 ng/mL	16.4.2013 4:33:47 AM		Standard	0.400	58,441.0	0.3957						

	correlation function:
concentration range from:	0.020
concentration range to:	0.400
intercept A =	140.6127
slope B =	147345.3029
correlation coefficient	0.9992

limit of quantitation (LOQ) [mg/kg] = 0.01
limit of detection (LOD) [mg/kg] = 0.002

Instrument Rec. = Instrument recovery sample (used to test for matrix effects)

Master Sheet No.: 698252-02
Filename:1304\20130415BKDc.wiff
Analyte: Parent Topramezone (BAS 670H)
Comment: Secondary transition, m/z 362 to 318

Sample Description	Sample ID	Date Analyzed	Date Extracted	Sample Type	QC [mg/kg] conc. [ng/ml]	Peak Count	Calc. conc. [ng/ml]	Sample wt [g]	Final vol.[mL]	Aliquot factor	Calc. res. [mg/kg]	Result [mg/kg]	Rec. %
ERS13-383	0.02 ng/mL	16.4.2013 12:38:16 AM		Standard	0.020	1,677.0	0.0220						
ERS13-382	0.04 ng/mL	16.4.2013 12:43:03 AM		Standard	0.040	2,832.0	0.0378						
ERS13-381	0.1 ng/mL	16.4.2013 12:47:51 AM		Standard	0.100	7,727.0	0.1047						
ERS13-380	0.2 ng/mL	16.4.2013 12:52:38 AM		Standard	0.200	15,327.0	0.2085						
ERS13-379	0.4 ng/mL	16.4.2013 12:57:28 AM		Standard	0.400	29,038.0	0.3958						
Blank	Blank	16.4.2013 1:02:15 AM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
698252-0002	Corn Grain Control	16.4.2013 1:07:05 AM	15-Apr-13	Control	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
698252-0002	Corn Grain Control	16.4.2013 1:11:56 AM	15-Apr-13	Control	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
698252-0002	Corn Grain 0.01 ppm Fort 1	16.4.2013 1:16:43 AM	15-Apr-13	Fortification	0.010	6,439.0	0.0871	5.0	2.5	0.5%	0.0087	0.0087	87%
Blank	Blank	16.4.2013 1:21:31 AM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
ERS13-383	0.02 ng/mL	16.4.2013 1:26:19 AM		Standard	0.020	1,631.0	0.0214						
698252-0002	Corn Grain 0.01 ppm Fort 2	16.4.2013 1:31:07 AM	15-Apr-13	Fortification	0.010	6,781.0	0.0917	5.0	2.5	0.5%	0.0092	0.0092	92%
698252-0002	Corn Grain 0.01 ppm Fort 3	16.4.2013 1:35:55 AM	15-Apr-13	Fortification	0.010	6,744.0	0.0912	5.0	2.5	0.5%	0.0091	0.0091	91%
698252-0002	Corn Grain 0.01 ppm Fort 4	16.4.2013 1:40:42 AM	15-Apr-13	Fortification	0.010	6,557.0	0.0887	5.0	2.5	0.5%	0.0089	0.0089	89%
698252-0002	Corn Grain 0.01 ppm Fort 5	16.4.2013 1:45:29 AM	15-Apr-13	Fortification	0.010	6,191.0	0.0837	5.0	2.5	0.5%	0.0084	0.0084	84%
Blank	Blank	16.4.2013 1:50:20 AM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
ERS13-382	0.04 ng/mL	16.4.2013 1:55:08 AM		Standard	0.040	3,129.0	0.0419						
698252-0002	Corn Grain 0.1 ppm Fort 1	16.4.2013 1:59:56 AM	15-Apr-13	Fortification	0.100	15,670.0	0.2132	5.0	12.5	0.5%	0.1066	0.1066	107%
698252-0002	Corn Grain 0.1 ppm Fort 2	16.4.2013 2:04:43 AM	15-Apr-13	Fortification	0.100	15,126.0	0.2058	5.0	12.5	0.5%	0.1029	0.1029	103%
698252-0002	Corn Grain 0.1 ppm Fort 3	16.4.2013 2:09:30 AM	15-Apr-13	Fortification	0.100	15,699.0	0.2136	5.0	12.5	0.5%	0.1068	0.1068	107%
698252-0002	Corn Grain 0.1 ppm Fort 4	16.4.2013 2:14:18 AM	15-Apr-13	Fortification	0.100	15,303.0	0.2082	5.0	12.5	0.5%	0.1041	0.1041	104%
Blank	Blank	16.4.2013 2:19:06 AM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
ERS13-381	0.1 ng/mL	16.4.2013 2:23:53 AM		Standard	0.100	7,616.0	0.1032						
698252-0002	Corn Grain 0.1 ppm Fort 5	16.4.2013 2:28:41 AM	15-Apr-13	Fortification	0.100	14,663.0	0.1994	5.0	12.5	0.5%	0.0997	0.0997	100%
698252-0005	Sunflower Seed Control	16.4.2013 2:33:32 AM	15-Apr-13	Control	0.000	139.0	0.0010	5.0	2.5	0.5%	0.0001	< LOD	
698252-0005	Sunflower Seed Control	16.4.2013 2:38:19 AM	15-Apr-13	Control	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
698252-0005	Sunflower Seed 0.01 ppm Fort 1	16.4.2013 2:43:07 AM	15-Apr-13	Fortification	0.010	6,722.0	0.0909	5.0	2.5	0.5%	0.0091	0.0091	91%
Blank	Blank	16.4.2013 2:47:55 AM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
ERS13-380	0.2 ng/mL	16.4.2013 2:52:43 AM		Standard	0.200	14,586.0	0.1984						
698252-0005	Sunflower Seed 0.01 ppm Fort 2	16.4.2013 2:57:31 AM	15-Apr-13	Fortification	0.010	8,276.0	0.1122	5.0	2.5	0.5%	0.0112	0.0112	112%
698252-0005	Sunflower Seed 0.01 ppm Fort 3	16.4.2013 3:02:19 AM	15-Apr-13	Fortification	0.010	6,441.0	0.0871	5.0	2.5	0.5%	0.0087	0.0087	87%
698252-0005	Sunflower Seed 0.01 ppm Fort 4	16.4.2013 3:07:08 AM	15-Apr-13	Fortification	0.010	6,362.0	0.0860	5.0	2.5	0.5%	0.0086	0.0086	86%
698252-0005	Sunflower Seed 0.01 ppm Fort 5	16.4.2013 3:11:56 AM	15-Apr-13	Fortification	0.010	19,358.0	0.2636	5.0	2.5	0.5%	0.0264	0.0264	264%
Blank	Blank	16.4.2013 3:16:45 AM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
ERS13-379	0.4 ng/mL	16.4.2013 3:21:35 AM		Standard	0.400	29,898.0	0.4076						
698252-0005	Sunflower Seed 0.1 ppm Fort 1	16.4.2013 3:26:23 AM	15-Apr-13	Fortification	0.100	14,648.0	0.1992	5.0	12.5	0.5%	0.0996	0.0996	100%
698252-0005	Sunflower Seed 0.1 ppm Fort 2	16.4.2013 3:31:10 AM	15-Apr-13	Fortification	0.100	13,850.0	0.1883	5.0	12.5	0.5%	0.0942	0.0942	94%
698252-0005	Sunflower Seed 0.1 ppm Fort 3	16.4.2013 3:35:57 AM	15-Apr-13	Fortification	0.100	12,816.0	0.1742	5.0	12.5	0.5%	0.0871	0.0871	87%
698252-0005	Sunflower Seed 0.1 ppm Fort 4	16.4.2013 3:40:45 AM	15-Apr-13	Fortification	0.100	13,911.0	0.1892	5.0	12.5	0.5%	0.0946	0.0946	95%
Blank	Blank	16.4.2013 3:45:35 AM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
ERS13-383	0.02 ng/mL	16.4.2013 3:50:23 AM		Standard	0.020	1,512.0	0.0198						
698252-0005	Sunflower Seed 0.1 ppm Fort 5	16.4.2013 3:55:11 AM	15-Apr-13	Fortification	0.100	13,379.0	0.1819	5.0	12.5	0.5%	0.0909	0.0909	91%
Reagent Blank	Reagent Blank	16.4.2013 3:59:59 AM	15-Apr-13	Control	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
698252-0002	Corn Grain Control Spike	16.4.2013 4:04:50 AM	15-Apr-13	Instrument Rec.	0.010	6,216.0	0.0840	5.0	2.5	0.5%	0.0084	0.0084	84%
Blank	Blank	16.4.2013 4:09:38 AM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
ERS13-383	0.02 ng/mL	16.4.2013 4:14:29 AM		Standard	0.020	1,528.0	0.0200						
ERS13-382	0.04 ng/mL	16.4.2013 4:19:18 AM		Standard	0.040	2,845.0	0.0380						
ERS13-381	0.1 ng/mL	16.4.2013 4:24:05 AM		Standard	0.100	7,047.0	0.0954						
ERS13-380	0.2 ng/mL	16.4.2013 4:28:56 AM		Standard	0.200	13,725.0	0.1866						
ERS13-379	0.4 ng/mL	16.4.2013 4:33:47 AM		Standard	0.400	29,281.0	0.3991						

correlation function:	
concentration range from:	0.020
concentration range to:	0.400
intercept A =	65.5187
slope B =	73195.9570
correlation coefficient	0.9993

limit of quantitation (LOQ) [mg/kg] = 0.01
limit of detection (LOD) [mg/kg] = 0.002

Instrument Rec. = Instrument recovery sample (used to test for matrix effects)

Master Sheet No.: 698252-02
Filename:1304\20130415BKDc.wiff
Analyte: M670H05
Comment: Primary transition, m/z 282 to 238

Sample Description	Sample ID	Date Analyzed	Date Extracted	Sample Type	QC [mg/kg] conc. [ng/ml]	Peak Count	Calc. conc. [ng/ml]	Sample wt [g]	Final vol.[mL]	Aliquot factor	Calc. res. [mg/kg]	Result [mg/kg]	Rec. %
ERS13-383	0.02 ng/mL	16.4.2013 12:38:16 AM		Standard	0.020	8,175.0	0.0200						
ERS13-382	0.04 ng/mL	16.4.2013 12:43:03 AM		Standard	0.040	16,030.0	0.0396						
ERS13-381	0.1 ng/mL	16.4.2013 12:47:51 AM		Standard	0.100	41,837.0	0.1040						
ERS13-380	0.2 ng/mL	16.4.2013 12:52:38 AM		Standard	0.200	81,347.0	0.2027						
ERS13-379	0.4 ng/mL	16.4.2013 12:57:28 AM		Standard	0.400	163,687.0	0.4082						
Blank	Blank	16.4.2013 1:02:15 AM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
698252-0002	Corn Grain Control	16.4.2013 1:07:05 AM	15-Apr-13	Control	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
698252-0002	Corn Grain Control	16.4.2013 1:11:56 AM	15-Apr-13	Control	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
698252-0002	Corn Grain 0.01 ppm Fort 1	16.4.2013 1:16:43 AM	15-Apr-13	Fortification	0.010	32,619.0	0.0810	5.0	2.5	0.5%	0.0081	0.0081	81%
Blank	Blank	16.4.2013 1:21:31 AM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
ERS13-383	0.02 ng/mL	16.4.2013 1:26:19 AM		Standard	0.020	8,241.0	0.0202						
698252-0002	Corn Grain 0.01 ppm Fort 2	16.4.2013 1:31:07 AM	15-Apr-13	Fortification	0.010	34,164.0	0.0849	5.0	2.5	0.5%	0.0085	0.0085	85%
698252-0002	Corn Grain 0.01 ppm Fort 3	16.4.2013 1:35:55 AM	15-Apr-13	Fortification	0.010	32,868.0	0.0816	5.0	2.5	0.5%	0.0082	0.0082	82%
698252-0002	Corn Grain 0.01 ppm Fort 4	16.4.2013 1:40:42 AM	15-Apr-13	Fortification	0.010	34,683.0	0.0862	5.0	2.5	0.5%	0.0086	0.0086	86%
698252-0002	Corn Grain 0.01 ppm Fort 5	16.4.2013 1:45:29 AM	15-Apr-13	Fortification	0.010	32,316.0	0.0803	5.0	2.5	0.5%	0.0080	0.0080	80%
Blank	Blank	16.4.2013 1:50:20 AM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
ERS13-382	0.04 ng/mL	16.4.2013 1:55:08 AM		Standard	0.040	16,791.0	0.0415						
698252-0002	Corn Grain 0.1 ppm Fort 1	16.4.2013 1:59:56 AM	15-Apr-13	Fortification	0.100	96,541.0	0.2406	5.0	12.5	0.5%	0.1203	0.1203	120%
698252-0002	Corn Grain 0.1 ppm Fort 2	16.4.2013 2:04:43 AM	15-Apr-13	Fortification	0.100	99,778.0	0.2487	5.0	12.5	0.5%	0.1243	0.1243	124%
698252-0002	Corn Grain 0.1 ppm Fort 3	16.4.2013 2:09:30 AM	15-Apr-13	Fortification	0.100	98,098.0	0.2445	5.0	12.5	0.5%	0.1222	0.1222	122%
698252-0002	Corn Grain 0.1 ppm Fort 4	16.4.2013 2:14:18 AM	15-Apr-13	Fortification	0.100	98,968.0	0.2467	5.0	12.5	0.5%	0.1233	0.1233	123%
Blank	Blank	16.4.2013 2:19:06 AM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
ERS13-381	0.1 ng/mL	16.4.2013 2:23:53 AM		Standard	0.100	42,291.0	0.1052						
698252-0002	Corn Grain 0.1 ppm Fort 5	16.4.2013 2:28:41 AM	15-Apr-13	Fortification	0.100	98,203.0	0.2447	5.0	12.5	0.5%	0.1224	0.1224	122%
698252-0005	Sunflower Seed Control	16.4.2013 2:33:32 AM	15-Apr-13	Control	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
698252-0005	Sunflower Seed Control	16.4.2013 2:38:19 AM	15-Apr-13	Control	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
698252-0005	Sunflower Seed 0.01 ppm Fort 1	16.4.2013 2:43:07 AM	15-Apr-13	Fortification	0.010	34,563.0	0.0859	5.0	2.5	0.5%	0.0086	0.0086	86%
Blank	Blank	16.4.2013 2:47:55 AM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
ERS13-380	0.2 ng/mL	16.4.2013 2:52:43 AM		Standard	0.200	78,537.0	0.1957						
698252-0005	Sunflower Seed 0.01 ppm Fort 2	16.4.2013 2:57:31 AM	15-Apr-13	Fortification	0.010	33,197.0	0.0825	5.0	2.5	0.5%	0.0082	0.0082	82%
698252-0005	Sunflower Seed 0.01 ppm Fort 3	16.4.2013 3:02:19 AM	15-Apr-13	Fortification	0.010	32,126.0	0.0798	5.0	2.5	0.5%	0.0080	0.0080	80%
698252-0005	Sunflower Seed 0.01 ppm Fort 4	16.4.2013 3:07:08 AM	15-Apr-13	Fortification	0.010	35,414.0	0.0880	5.0	2.5	0.5%	0.0088	0.0088	88%
698252-0005	Sunflower Seed 0.01 ppm Fort 5	16.4.2013 3:11:56 AM	15-Apr-13	Fortification	0.010	31,458.0	0.0781	5.0	2.5	0.5%	0.0078	0.0078	78%
Blank	Blank	16.4.2013 3:16:45 AM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
ERS13-379	0.4 ng/mL	16.4.2013 3:21:35 AM		Standard	0.400	159,383.0	0.3975						
698252-0005	Sunflower Seed 0.1 ppm Fort 1	16.4.2013 3:26:23 AM	15-Apr-13	Fortification	0.100	90,059.0	0.2244	5.0	12.5	0.5%	0.1122	0.1122	112%
698252-0005	Sunflower Seed 0.1 ppm Fort 2	16.4.2013 3:31:10 AM	15-Apr-13	Fortification	0.100	94,703.0	0.2360	5.0	12.5	0.5%	0.1180	0.1180	118%
698252-0005	Sunflower Seed 0.1 ppm Fort 3	16.4.2013 3:35:57 AM	15-Apr-13	Fortification	0.100	90,026.0	0.2243	5.0	12.5	0.5%	0.1122	0.1122	112%
698252-0005	Sunflower Seed 0.1 ppm Fort 4	16.4.2013 3:40:45 AM	15-Apr-13	Fortification	0.100	93,197.0	0.2322	5.0	12.5	0.5%	0.1161	0.1161	116%
Blank	Blank	16.4.2013 3:45:35 AM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
ERS13-383	0.02 ng/mL	16.4.2013 3:50:23 AM		Standard	0.020	7,887.0	0.0193						
698252-0005	Sunflower Seed 0.1 ppm Fort 5	16.4.2013 3:55:11 AM	15-Apr-13	Fortification	0.100	92,463.0	0.2304	5.0	12.5	0.5%	0.1152	0.1152	115%
Reagent Blank	Reagent Blank	16.4.2013 3:59:59 AM	15-Apr-13	Control	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
698252-0002	Corn Grain Control Spike	16.4.2013 4:04:50 AM	15-Apr-13	Instrument Rec.	0.010	34,164.0	0.0849	5.0	2.5	0.5%	0.0085	0.0085	85%
Blank	Blank	16.4.2013 4:09:38 AM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
ERS13-383	0.02 ng/mL	16.4.2013 4:14:29 AM		Standard	0.020	7,694.0	0.0188						
ERS13-382	0.04 ng/mL	16.4.2013 4:19:18 AM		Standard	0.040	16,349.0	0.0404						
ERS13-381	0.1 ng/mL	16.4.2013 4:24:05 AM		Standard	0.100	39,525.0	0.0983						
ERS13-380	0.2 ng/mL	16.4.2013 4:28:56 AM		Standard	0.200	76,750.0	0.1912						
ERS13-379	0.4 ng/mL	16.4.2013 4:33:47 AM		Standard	0.400	159,438.0	0.3976						

correlation function:	
concentration range from:	0.020
concentration range to:	0.400
intercept A =	166.5287
slope B =	400564.1485
correlation coefficient	0.9996

limit of quantitation (LOQ) [mg/kg] = 0.01
limit of detection (LOD) [mg/kg] = 0.002

Instrument Rec. = Instrument recovery sample (used to test for matrix effects)

Master Sheet No.: 698252-02
Filename:1304\20130415BKDc.wiff
Analyte: M670H05
Comment: Secondary transition, m/z 282 to 79

Sample Description	Sample ID	Date Analyzed	Date Extracted	Sample Type	QC [mg/kg] conc. [ng/ml]	Peak Count	Calc. conc. [ng/ml]	Sample wt [g]	Final vol.[mL]	Aliquot factor	Calc. res. [mg/kg]	Result [mg/kg]	Rec. %
ERS13-383	0.02 ng/mL	16.4.2013 12:38:16 AM		Standard	0.020	1,466.0	0.0241						
ERS13-382	0.04 ng/mL	16.4.2013 12:43:03 AM		Standard	0.040	2,672.0	0.0418						
ERS13-381	0.1 ng/mL	16.4.2013 12:47:51 AM		Standard	0.100	6,654.0	0.1004						
ERS13-380	0.2 ng/mL	16.4.2013 12:52:38 AM		Standard	0.200	12,790.0	0.1906						
ERS13-379	0.4 ng/mL	16.4.2013 12:57:28 AM		Standard	0.400	26,924.0	0.3984						
Blank	Blank	16.4.2013 1:02:15 AM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
698252-0002	Corn Grain Control	16.4.2013 1:07:05 AM	15-Apr-13	Control	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
698252-0002	Corn Grain Control	16.4.2013 1:11:56 AM	15-Apr-13	Control	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
698252-0002	Corn Grain 0.01 ppm Fort 1	16.4.2013 1:16:43 AM	15-Apr-13	Fortification	0.010	5,179.0	0.0787	5.0	2.5	0.5%	0.0079	0.0079	79%
Blank	Blank	16.4.2013 1:21:31 AM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
ERS13-383	0.02 ng/mL	16.4.2013 1:26:19 AM		Standard	0.020	1,337.0	0.0222						
698252-0002	Corn Grain 0.01 ppm Fort 2	16.4.2013 1:31:07 AM	15-Apr-13	Fortification	0.010	5,396.0	0.0819	5.0	2.5	0.5%	0.0082	0.0082	82%
698252-0002	Corn Grain 0.01 ppm Fort 3	16.4.2013 1:35:55 AM	15-Apr-13	Fortification	0.010	5,630.0	0.0853	5.0	2.5	0.5%	0.0085	0.0085	85%
698252-0002	Corn Grain 0.01 ppm Fort 4	16.4.2013 1:40:42 AM	15-Apr-13	Fortification	0.010	5,288.0	0.0803	5.0	2.5	0.5%	0.0080	0.0080	80%
698252-0002	Corn Grain 0.01 ppm Fort 5	16.4.2013 1:45:29 AM	15-Apr-13	Fortification	0.010	5,524.0	0.0838	5.0	2.5	0.5%	0.0084	0.0084	84%
Blank	Blank	16.4.2013 1:50:20 AM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
ERS13-382	0.04 ng/mL	16.4.2013 1:55:08 AM		Standard	0.040	2,755.0	0.0431						
698252-0002	Corn Grain 0.1 ppm Fort 1	16.4.2013 1:59:56 AM	15-Apr-13	Fortification	0.100	15,432.0	0.2295	5.0	12.5	0.5%	0.1147	0.1147	115%
698252-0002	Corn Grain 0.1 ppm Fort 2	16.4.2013 2:04:43 AM	15-Apr-13	Fortification	0.100	15,500.0	0.2305	5.0	12.5	0.5%	0.1152	0.1152	115%
698252-0002	Corn Grain 0.1 ppm Fort 3	16.4.2013 2:09:30 AM	15-Apr-13	Fortification	0.100	15,875.0	0.2360	5.0	12.5	0.5%	0.1180	0.1180	118%
698252-0002	Corn Grain 0.1 ppm Fort 4	16.4.2013 2:14:18 AM	15-Apr-13	Fortification	0.100	16,299.0	0.2422	5.0	12.5	0.5%	0.1211	0.1211	121%
Blank	Blank	16.4.2013 2:19:06 AM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
ERS13-381	0.1 ng/mL	16.4.2013 2:23:53 AM		Standard	0.100	7,048.0	0.1062						
698252-0002	Corn Grain 0.1 ppm Fort 5	16.4.2013 2:28:41 AM	15-Apr-13	Fortification	0.100	16,122.0	0.2396	5.0	12.5	0.5%	0.1198	0.1198	120%
698252-0005	Sunflower Seed Control	16.4.2013 2:33:32 AM	15-Apr-13	Control	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
698252-0005	Sunflower Seed Control	16.4.2013 2:38:19 AM	15-Apr-13	Control	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
698252-0005	Sunflower Seed 0.01 ppm Fort 1	16.4.2013 2:43:07 AM	15-Apr-13	Fortification	0.010	4,958.0	0.0755	5.0	2.5	0.5%	0.0075	0.0075	75%
Blank	Blank	16.4.2013 2:47:55 AM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
ERS13-380	0.2 ng/mL	16.4.2013 2:52:43 AM		Standard	0.200	12,586.0	0.1876						
698252-0005	Sunflower Seed 0.01 ppm Fort 2	16.4.2013 2:57:31 AM	15-Apr-13	Fortification	0.010	4,736.0	0.0722	5.0	2.5	0.5%	0.0072	0.0072	72%
698252-0005	Sunflower Seed 0.01 ppm Fort 3	16.4.2013 3:02:19 AM	15-Apr-13	Fortification	0.010	4,682.0	0.0714	5.0	2.5	0.5%	0.0071	0.0071	71%
698252-0005	Sunflower Seed 0.01 ppm Fort 4	16.4.2013 3:07:08 AM	15-Apr-13	Fortification	0.010	5,372.0	0.0815	5.0	2.5	0.5%	0.0082	0.0082	82%
698252-0005	Sunflower Seed 0.01 ppm Fort 5	16.4.2013 3:11:56 AM	15-Apr-13	Fortification	0.010	4,916.0	0.0748	5.0	2.5	0.5%	0.0075	0.0075	75%
Blank	Blank	16.4.2013 3:16:45 AM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
ERS13-379	0.4 ng/mL	16.4.2013 3:21:35 AM		Standard	0.400	27,674.0	0.4095						
698252-0005	Sunflower Seed 0.1 ppm Fort 1	16.4.2013 3:26:23 AM	15-Apr-13	Fortification	0.100	14,787.0	0.2200	5.0	12.5	0.5%	0.1100	0.1100	110%
698252-0005	Sunflower Seed 0.1 ppm Fort 2	16.4.2013 3:31:10 AM	15-Apr-13	Fortification	0.100	15,471.0	0.2300	5.0	12.5	0.5%	0.1150	0.1150	115%
698252-0005	Sunflower Seed 0.1 ppm Fort 3	16.4.2013 3:35:57 AM	15-Apr-13	Fortification	0.100	15,175.0	0.2257	5.0	12.5	0.5%	0.1128	0.1128	113%
698252-0005	Sunflower Seed 0.1 ppm Fort 4	16.4.2013 3:40:45 AM	15-Apr-13	Fortification	0.100	15,034.0	0.2236	5.0	12.5	0.5%	0.1118	0.1118	112%
Blank	Blank	16.4.2013 3:45:35 AM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
ERS13-383	0.02 ng/mL	16.4.2013 3:50:23 AM		Standard	0.020	1,235.0	0.0207						
698252-0005	Sunflower Seed 0.1 ppm Fort 5	16.4.2013 3:55:11 AM	15-Apr-13	Fortification	0.100	14,871.0	0.2212	5.0	12.5	0.5%	0.1106	0.1106	111%
Reagent Blank	Reagent Blank	16.4.2013 3:59:59 AM	15-Apr-13	Control	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
698252-0002	Corn Grain Control Spike	16.4.2013 4:04:50 AM	15-Apr-13	Instrument Rec.	0.010	5,373.0	0.0816	5.0	2.5	0.5%	0.0082	0.0082	82%
Blank	Blank	16.4.2013 4:09:38 AM		Blank	0.000	0.0	0.0000	5.0	2.5	0.5%	0.0000	< LOD	
ERS13-383	0.02 ng/mL	16.4.2013 4:14:29 AM		Standard	0.020	1,306.0	0.0218						
ERS13-382	0.04 ng/mL	16.4.2013 4:19:18 AM		Standard	0.040	2,513.0	0.0395						
ERS13-381	0.1 ng/mL	16.4.2013 4:24:05 AM		Standard	0.100	6,443.0	0.0973						
ERS13-380	0.2 ng/mL	16.4.2013 4:28:56 AM		Standard	0.200	12,834.0	0.1913						
ERS13-379	0.4 ng/mL	16.4.2013 4:33:47 AM		Standard	0.400	27,405.0	0.4055						

	correlation function:
concentration range from:	0.020
concentration range to:	0.400
intercept A =	-173.9934
slope B =	68011.2583
correlation coefficient	0.9991

limit of quantitation (LOQ) [mg/kg] = 0.01
limit of detection (LOD) [mg/kg] = 0.002

Instrument Rec. = Instrument recovery sample (used to test for matrix effects)

Master Sheet No.: 698252-02
Analytical batch: 1304\20130416JEJb.wiff
Analyte: Parent Topramezone (BAS 670H)
Comment: Primary transition, m/z 362 to 334

Sample Description	Sample ID	Date Analyzed	Date Extracted	Sample Type	QC [mg/kg] conc. [ng/ml]	Peak Count	Calc. conc. [ng/ml]	Sample wt [g]	Final vol.[mL]	Aliquot factor	Calc. res. [mg/kg]	Result [mg/kg]	Rec. %
ERS13-383	0.02 ng/mL	16.4.2013 4:11:44 PM		Standard	0.020	2,437.0	0.0191						
ERS13-382	0.04 ng/mL	16.4.2013 4:16:32 PM		Standard	0.040	4,813.0	0.0386						
ERS13-381	0.1 ng/mL	16.4.2013 4:21:19 PM		Standard	0.100	12,144.0	0.0988						
ERS13-380	0.2 ng/mL	16.4.2013 4:26:06 PM		Standard	0.200	26,383.0	0.2157						
ERS13-379	0.4 ng/mL	16.4.2013 4:30:58 PM		Standard	0.400	50,513.0	0.4139						
Blank	Blank	16.4.2013 4:35:45 PM		Blank	0.000	0.0	0.0000	5.0	2.50	0.50%	0.0000	< LOD	
698252-0005	Sunflower Seed Control Spike	16.4.2013 4:40:36 PM	15-Apr-13	Instrument Rec.	0.010	11,405.0	0.0928	5.0	2.50	0.50%	0.0093	0.0093	93%
Blank	Blank	16.4.2013 4:45:27 PM		Blank	0.000	0.0	0.0000	5.0	2.50	0.50%	0.0000	< LOD	
ERS13-383	0.02 ng/mL	16.4.2013 4:50:18 PM		Standard	0.020	2,309.0	0.0181						
ERS13-382	0.04 ng/mL	16.4.2013 4:55:05 PM		Standard	0.040	5,253.0	0.0422						
ERS13-381	0.1 ng/mL	16.4.2013 4:59:52 PM		Standard	0.100	11,617.0	0.0945						
ERS13-380	0.2 ng/mL	16.4.2013 5:04:40 PM		Standard	0.200	24,239.0	0.1981						
ERS13-379	0.4 ng/mL	16.4.2013 5:09:27 PM		Standard	0.400	46,496.0	0.3809						

	correlation function
concentration range from:	0.020
concentration range to:	0.400
intercept A:	109.8532
slope B:	121779.9129
correlation coefficient:	0.9978

limit of quantitation (LOQ) [mg/kg] = 0.01
limit of detection (LOD) [mg/kg] = 0.002

Instrument Rec. = Instrument recovery sample (used to test for matrix effects)

Master Sheet No.: 698252-02
Analytical batch: 1304\20130416JEJb.wiff
Analyte: Parent Topramezone (BAS 670H)
Comment: Secondary transition, m/z 362 to 318

Sample Description	Sample ID	Date Analyzed	Date Extracted	Sample Type	QC [mg/kg] conc. [ng/ml]	Peak Count	Calc. conc. [ng/ml]	Sample wt [g]	Final vol.[mL]	Aliquot factor	Calc. res. [mg/kg]	Result [mg/kg]	Rec. %
ERS13-383	0.02 ng/mL	16.4.2013 4:11:44 PM		Standard	0.020	1,071.0	0.0172						
ERS13-382	0.04 ng/mL	16.4.2013 4:16:32 PM		Standard	0.040	2,248.0	0.0363						
ERS13-381	0.1 ng/mL	16.4.2013 4:21:19 PM		Standard	0.100	6,038.0	0.0976						
ERS13-380	0.2 ng/mL	16.4.2013 4:26:06 PM		Standard	0.200	12,745.0	0.2062						
ERS13-379	0.4 ng/mL	16.4.2013 4:30:58 PM		Standard	0.400	24,520.0	0.3969						
Blank	Blank	16.4.2013 4:35:45 PM		Blank	0.000	0.0	0.0000	5.0	2.50	0.50%	0.0000	< LOD	
698252-0005	Sunflower Seed Control Spike	16.4.2013 4:40:36 PM	15-Apr-13	Instrument Rec.	0.010	5,705.0	0.0923	5.0	2.50	0.50%	0.0092	0.0092	92%
Blank	Blank	16.4.2013 4:45:27 PM		Blank	0.000	0.0	0.0000	5.0	2.50	0.50%	0.0000	< LOD	
ERS13-383	0.02 ng/mL	16.4.2013 4:50:18 PM		Standard	0.020	1,328.0	0.0214						
ERS13-382	0.04 ng/mL	16.4.2013 4:55:05 PM		Standard	0.040	2,617.0	0.0423						
ERS13-381	0.1 ng/mL	16.4.2013 4:59:52 PM		Standard	0.100	5,871.0	0.0949						
ERS13-380	0.2 ng/mL	16.4.2013 5:04:40 PM		Standard	0.200	12,986.0	0.2101						
ERS13-379	0.4 ng/mL	16.4.2013 5:09:27 PM		Standard	0.400	24,529.0	0.3970						

	correlation function
concentration range from:	0.020
concentration range to:	0.400
intercept A:	6.8234
slope B:	61766.2935
correlation coefficient:	0.9994

limit of quantitation (LOQ) [mg/kg] = 0.01
limit of detection (LOD) [mg/kg] = 0.002

Instrument Rec. = Instrument recovery sample (used to test for matrix effects)

Master Sheet No.: 698252-02
Analytical batch: 1304\20130416JEJa.wiff
Analyte: M670H05
Comment: Primary transition, m/z 282 to 238

Sample Description	Sample ID	Date Analyzed	Date Extracted	Sample Type	QC [mg/kg] conc. [ng/ml]	Peak Count	Calc. conc. [ng/ml]	Sample wt [g]	Final vol.[mL]	Aliquot factor	Calc. res. [mg/kg]	Result [mg/kg]	Rec. %
ERS13-383	0.02 ng/mL	16.4.2013 2:25:34 PM		Standard	0.020	6,860.0	0.0177						
ERS13-382	0.04 ng/mL	16.4.2013 2:30:22 PM		Standard	0.040	15,089.0	0.0393						
ERS13-381	0.1 ng/mL	16.4.2013 2:35:09 PM		Standard	0.100	40,471.0	0.1056						
ERS13-380	0.2 ng/mL	16.4.2013 2:40:00 PM		Standard	0.200	74,120.0	0.1936						
ERS13-379	0.4 ng/mL	16.4.2013 2:44:47 PM		Standard	0.400	153,839.0	0.4021						
Blank	Blank	16.4.2013 2:49:35 PM		Blank	0.000	0.0	0.0000	5.0	2.50	0.50%	0.0000	< LOD	
698252-0005	Sunflower Seed Control Spike	16.4.2013 2:54:22 PM	15-Apr-13	Instrument Rec.	0.010	28,737.0	0.0749	5.0	2.50	0.50%	0.0075	0.0075	75%
Blank	Blank	16.4.2013 2:59:12 PM		Blank	0.000	0.0	0.0000	5.0	2.50	0.50%	0.0000	< LOD	
ERS13-383	0.02 ng/mL	16.4.2013 3:04:04 PM		Standard	0.020	7,189.0	0.0186						
ERS13-382	0.04 ng/mL	16.4.2013 3:08:53 PM		Standard	0.040	15,217.0	0.0396						
ERS13-381	0.1 ng/mL	16.4.2013 3:13:40 PM		Standard	0.100	40,215.0	0.1050						
ERS13-380	0.2 ng/mL	16.4.2013 3:18:28 PM		Standard	0.200	76,376.0	0.1995						
ERS13-379	0.4 ng/mL	16.4.2013 3:23:17 PM		Standard	0.400	152,608.0	0.3989						

	correlation function
concentration range from:	0.020
concentration range to:	0.400
intercept A:	81.2032
slope B:	382349.9793
correlation coefficient:	0.9997

limit of quantitation (LOQ) [mg/kg] = 0.01
limit of detection (LOD) [mg/kg] = 0.002

Instrument Rec. = Instrument recovery sample (used to test for matrix effects)

Master Sheet No.: 698252-02
Analytical batch: 1304\20130416JEJa.wiff
Analyte: M670H05
Comment: Secondary transition, m/z 282 to 79

Sample Description	Sample ID	Date Analyzed	Date Extracted	Sample Type	QC [mg/kg] conc. [ng/ml]	Peak Count	Calc. conc. [ng/ml]	Sample wt [g]	Final vol.[mL]	Aliquot factor	Calc. res. [mg/kg]	Result [mg/kg]	Rec. %
ERS13-383	0.02 ng/mL	16.4.2013 2:25:34 PM		Standard	0.020	1,324.0	0.0210						
ERS13-382	0.04 ng/mL	16.4.2013 2:30:22 PM		Standard	0.040	2,518.0	0.0402						
ERS13-381	0.1 ng/mL	16.4.2013 2:35:09 PM		Standard	0.100	6,520.0	0.1043						
ERS13-380	0.2 ng/mL	16.4.2013 2:40:00 PM		Standard	0.200	12,448.0	0.1993						
ERS13-379	0.4 ng/mL	16.4.2013 2:44:47 PM		Standard	0.400	24,827.0	0.3978						
Blank	Blank	16.4.2013 2:49:35 PM		Blank	0.000	0.0	0.0000	5.0	2.50	0.50%	0.0000	< LOD	
698252-0005	Sunflower Seed Control Spike	16.4.2013 2:54:22 PM	15-Apr-13	Instrument Rec.	0.010	4,438.0	0.0709	5.0	2.50	0.50%	0.0071	0.0071	71%
Blank	Blank	16.4.2013 2:59:12 PM		Blank	0.000	0.0	0.0000	5.0	2.50	0.50%	0.0000	< LOD	
ERS13-383	0.02 ng/mL	16.4.2013 3:04:04 PM		Standard	0.020	1,107.0	0.0175						
ERS13-382	0.04 ng/mL	16.4.2013 3:08:53 PM		Standard	0.040	2,470.0	0.0394						
ERS13-381	0.1 ng/mL	16.4.2013 3:13:40 PM		Standard	0.100	6,642.0	0.1063						
ERS13-380	0.2 ng/mL	16.4.2013 3:18:28 PM		Standard	0.200	11,760.0	0.1883						
ERS13-379	0.4 ng/mL	16.4.2013 3:23:17 PM		Standard	0.400	25,329.0	0.4058						

	correlation function
concentration range from:	0.020
concentration range to:	0.400
intercept A:	12.8541
slope B:	62379.2496
correlation coefficient:	0.9994

limit of quantitation (LOQ) [mg/kg] = 0.01
limit of detection (LOD) [mg/kg] = 0.002

Instrument Rec. = Instrument recovery sample (used to test for matrix effects)

Appendix E. Example Standard Curves and Chromatography

Note : The examples presented include the standard curves and chromatography for the primary transition and secondary transition. The detailed information on the chromatograms indicates the transition that is depicted.

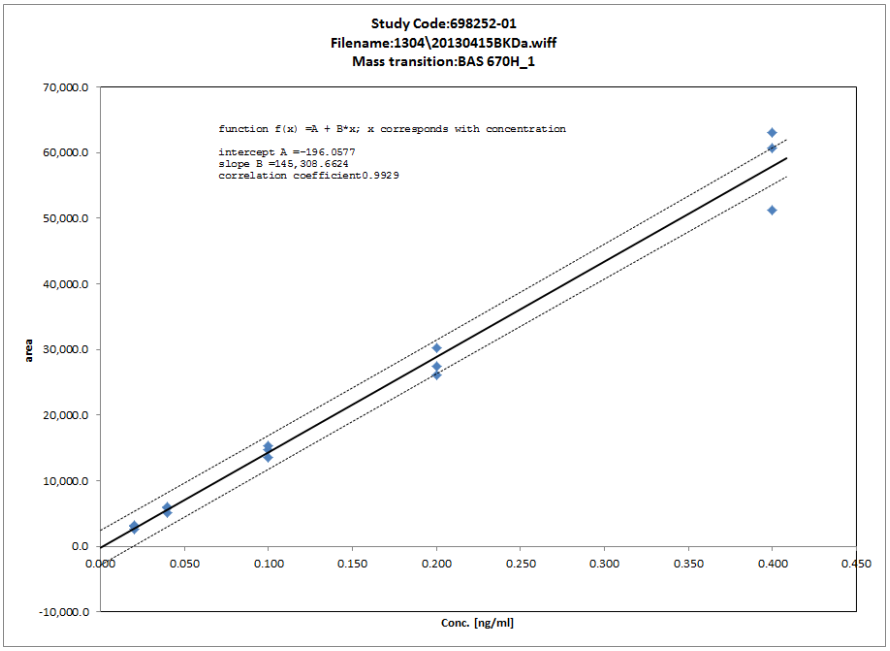
For the example standards shown in Figure 3, the incorrect “sample name” is shown for each standard. The following “cross-walk” is provided showing the correct standard sample names:

“Sample Name” on Example Standard Chromatogram	The correct standard “Sample Name”	Standard Concentration
ERS13-335	ERS13-379	0.4 ng/mL
ERS13-336	ERS13-380	0.2 ng/mL
ERS13-337	ERS13-381	0.1 ng/mL
ERS13-338	ERS13-382	0.04 ng/mL
ERS13-339	ERS13-383	0.02 ng/mL

The standard concentrations shown in the example chromatograms are correct.

Figure 1. Calibration curves for topramezone

Master Sheet 698252-1 (primary transition, 362→334)



Master Sheet 698252-1 (secondary transition, 362→318)

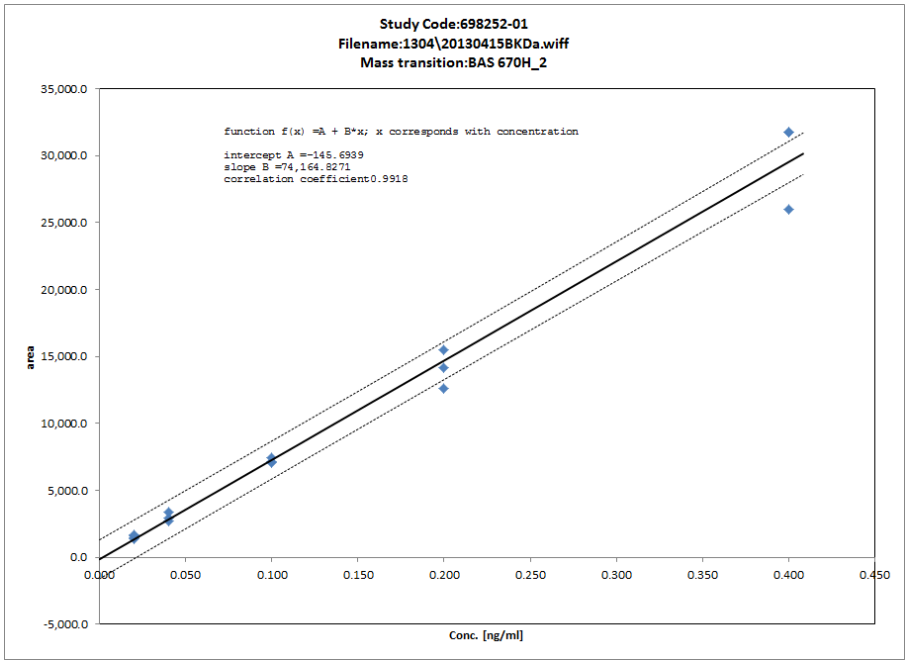
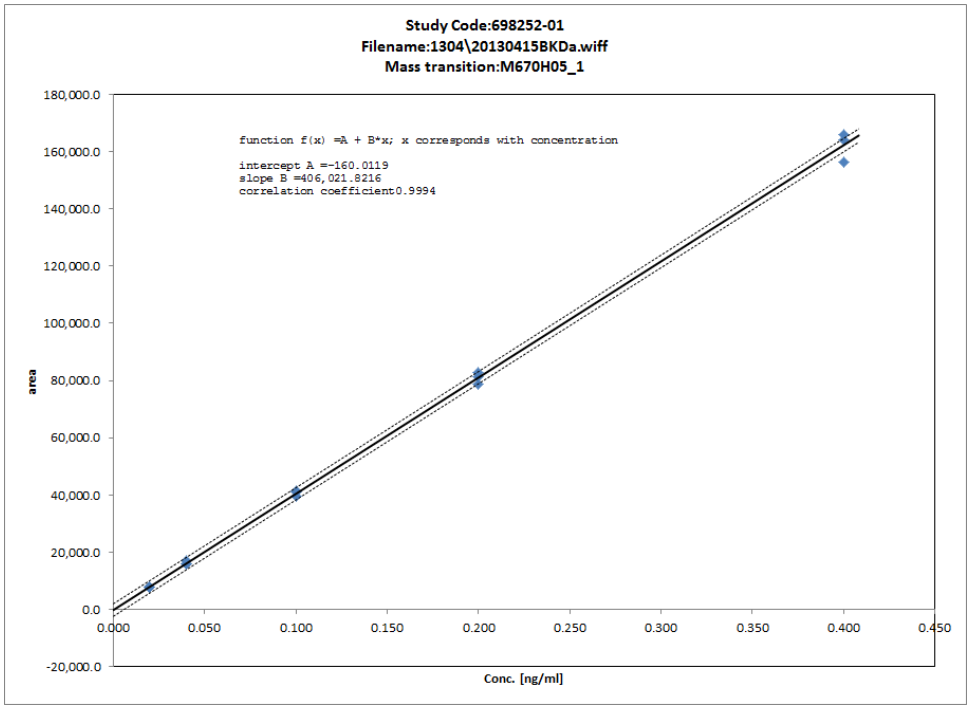


Figure 2. Calibration curves for M670H05

Master Sheet 698252-1 (primary transition, 282→318)



Master Sheet 698252-1 (secondary transition, 282→79)

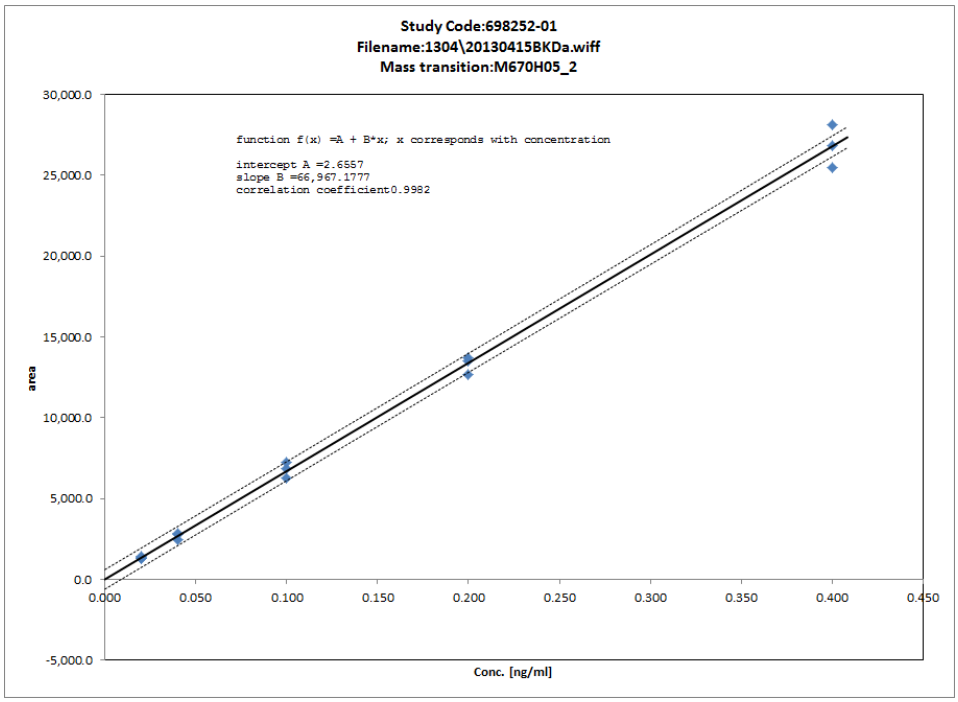
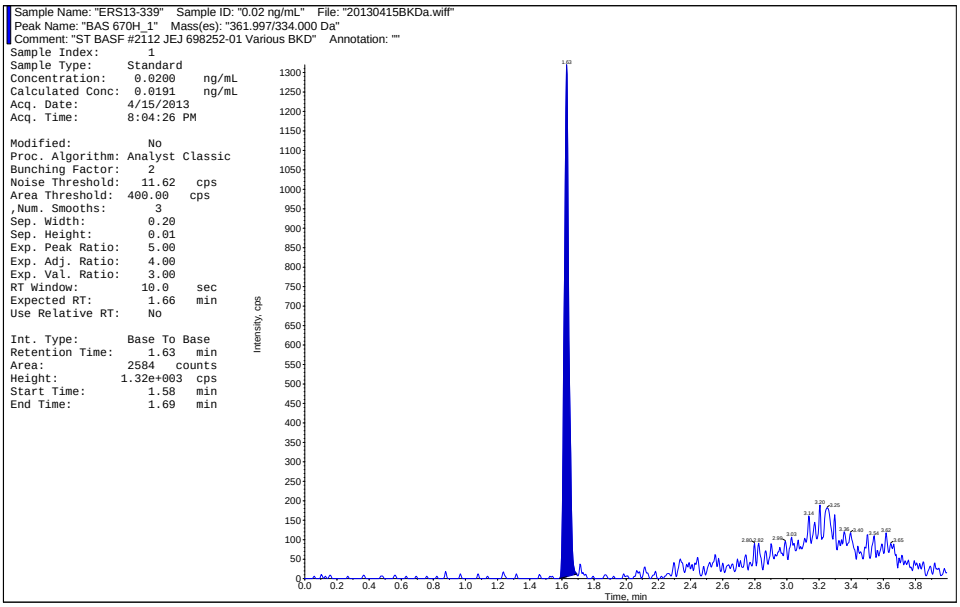
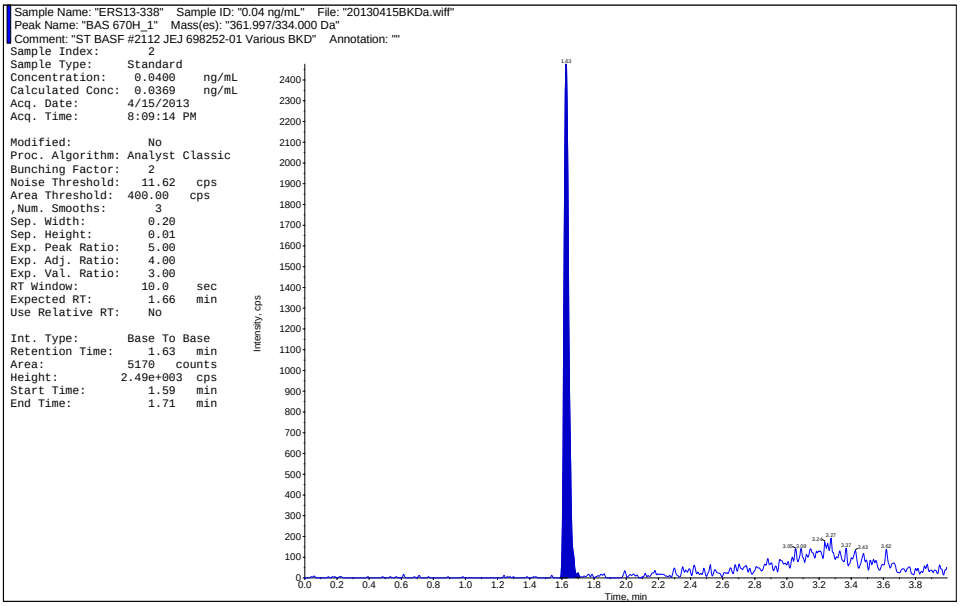


Figure 3. Example standard chromatograms for topramezone

Primary transition, Topramezone, from Master Sheet 698252-1

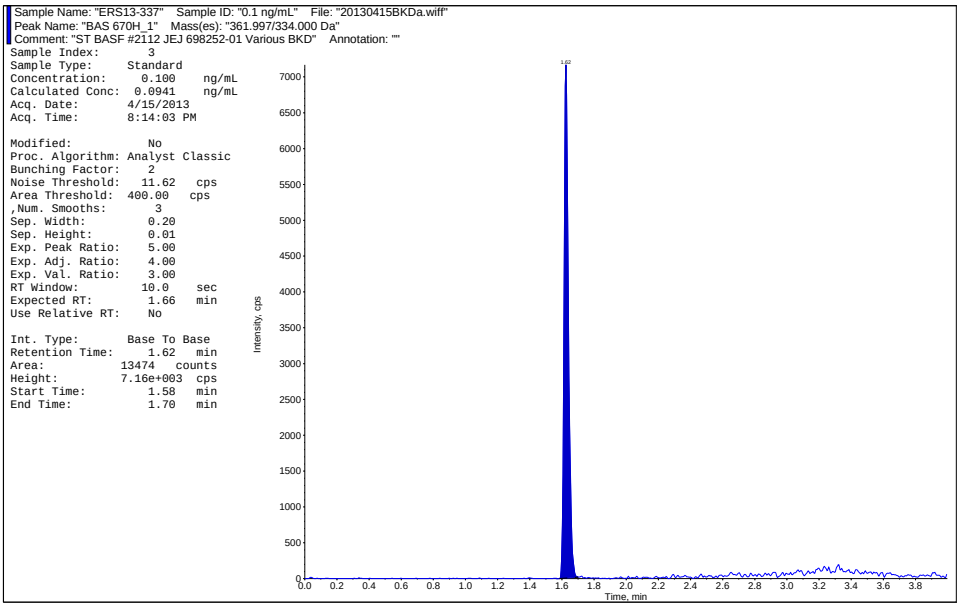


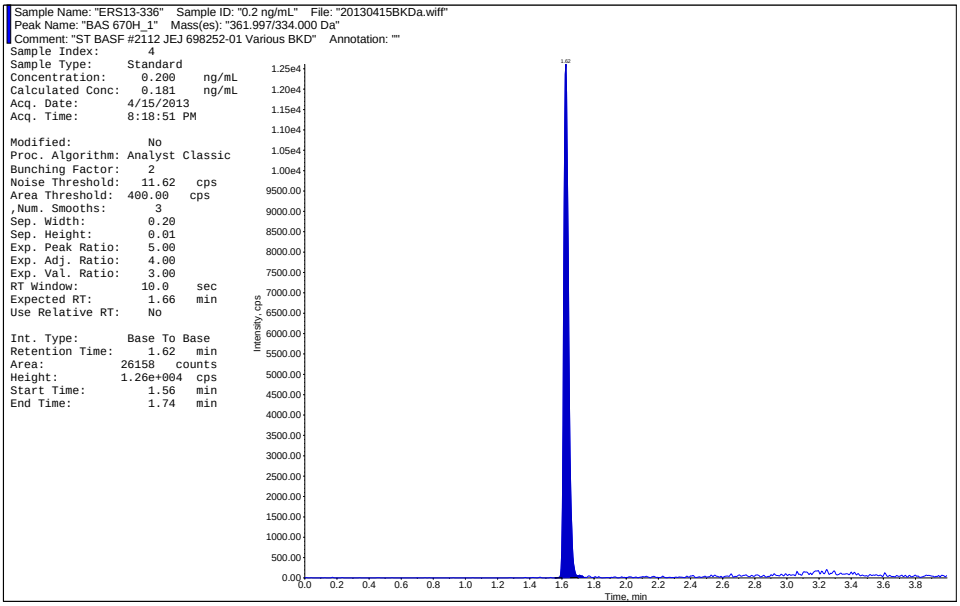


0.02 ng/mL (ERS13-383) and 0.04 ng/mL standards (ERS13-382), respectively

Figure Continued.

Standards chromatograms, Primary transition, Topramezone, from Master Sheet 698252-1

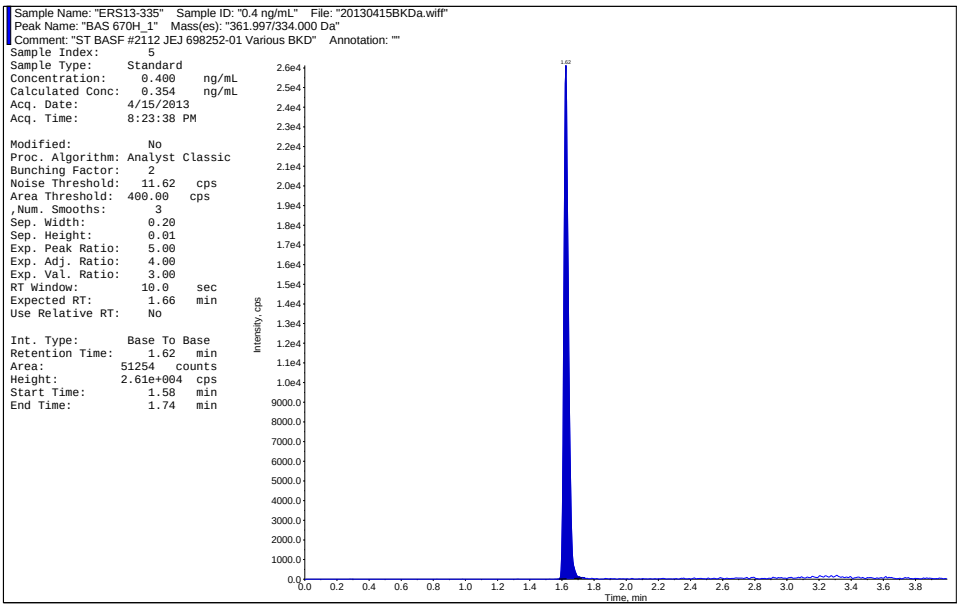




0.1 ng/mL (ERS13-381) and 0.2 ng/mL standards (ERS13-380), respectively

Figure Continued.

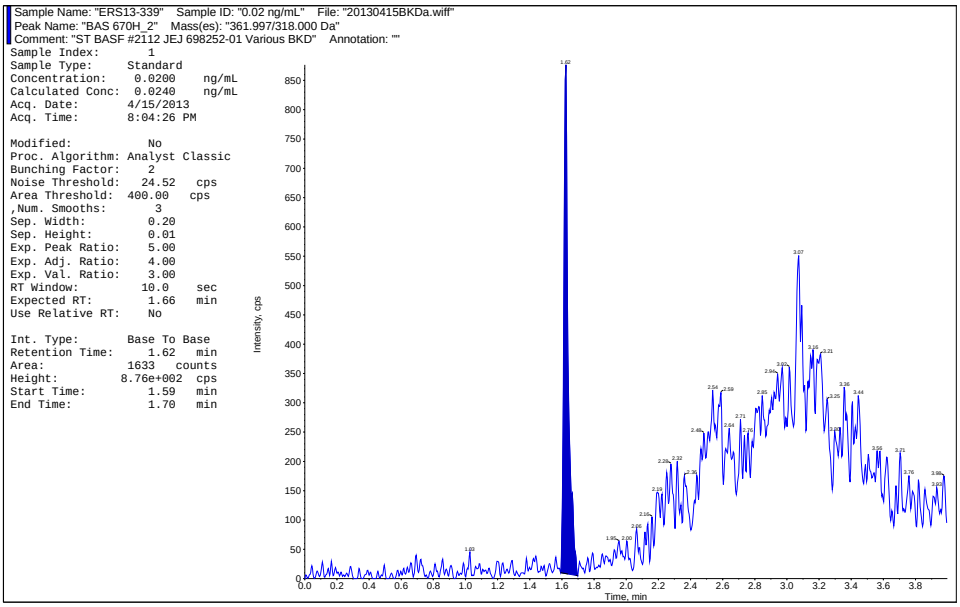
Standards chromatograms, Primary transition, Topramezone, from Master Sheet 698252-1

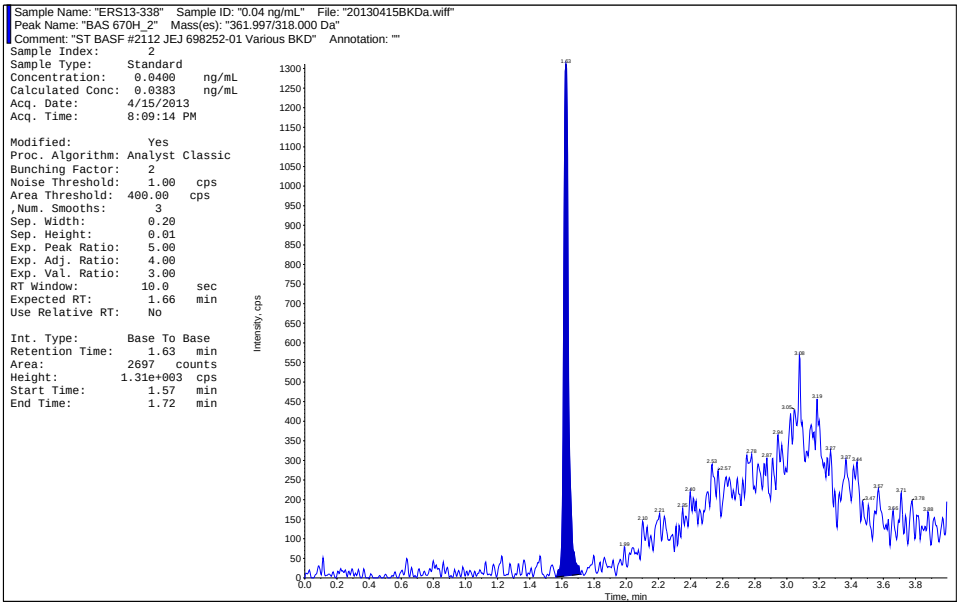


0.4 ng/mL standard (ERS13-379)

Figure Continued.

Standards chromatograms, Secondary transition, Topramezone, from Master Sheet 698252-1

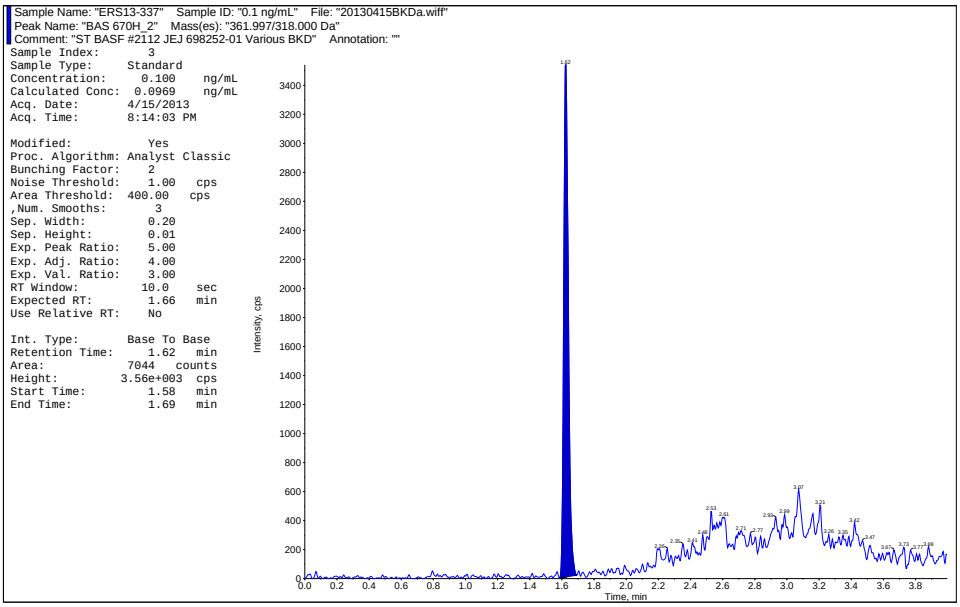


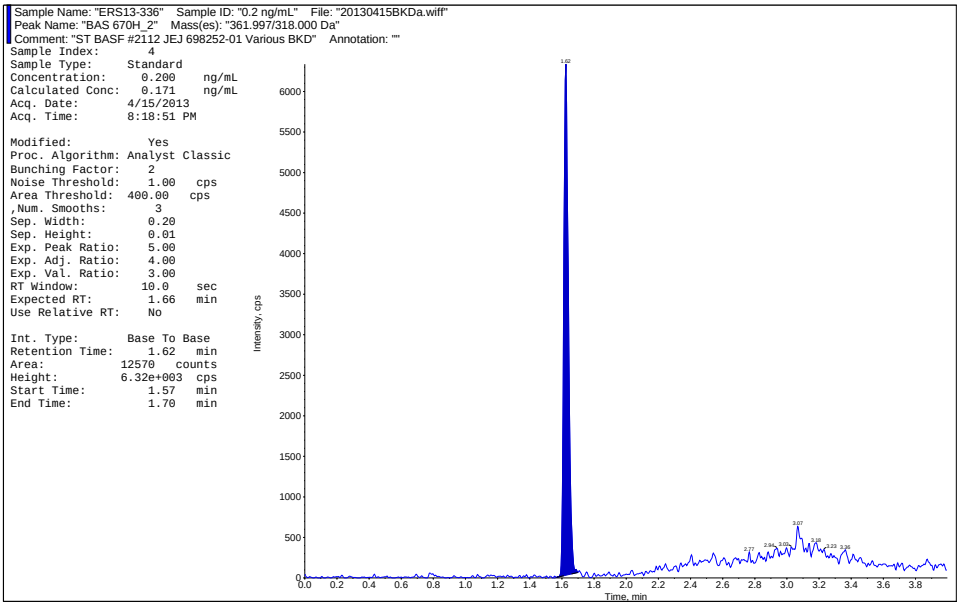


0.02 ng/mL (ERS13-383) and 0.04 ng/mL standards (ERS13-382), respectively

Figure Continued.

Standards chromatograms, Secondary transition, Topramezone, from Master Sheet 698252-1

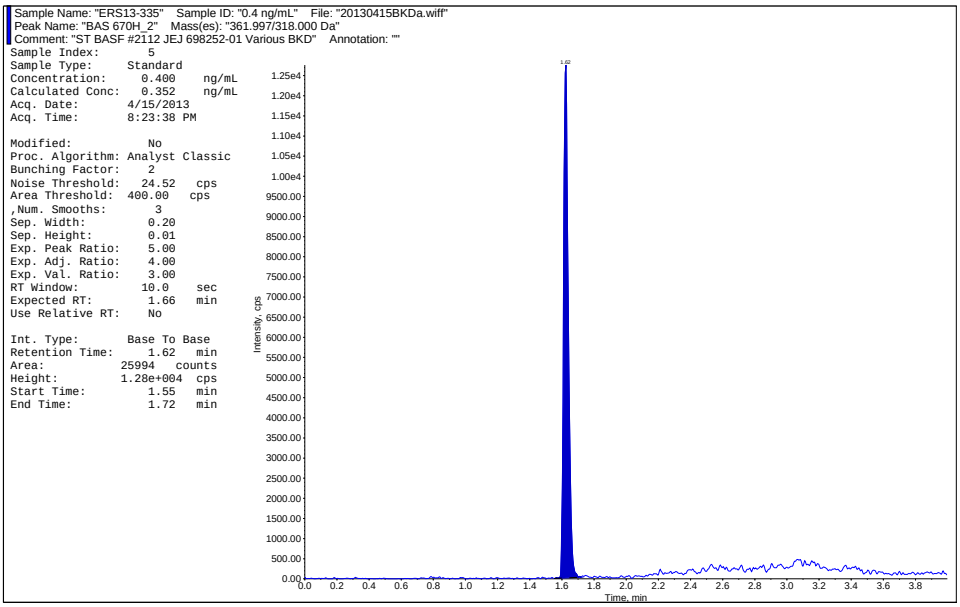




0.1 ng/mL (ERS13-381) and 0.2 ng/mL standards (ERS13-380), respectively

Figure Continued.

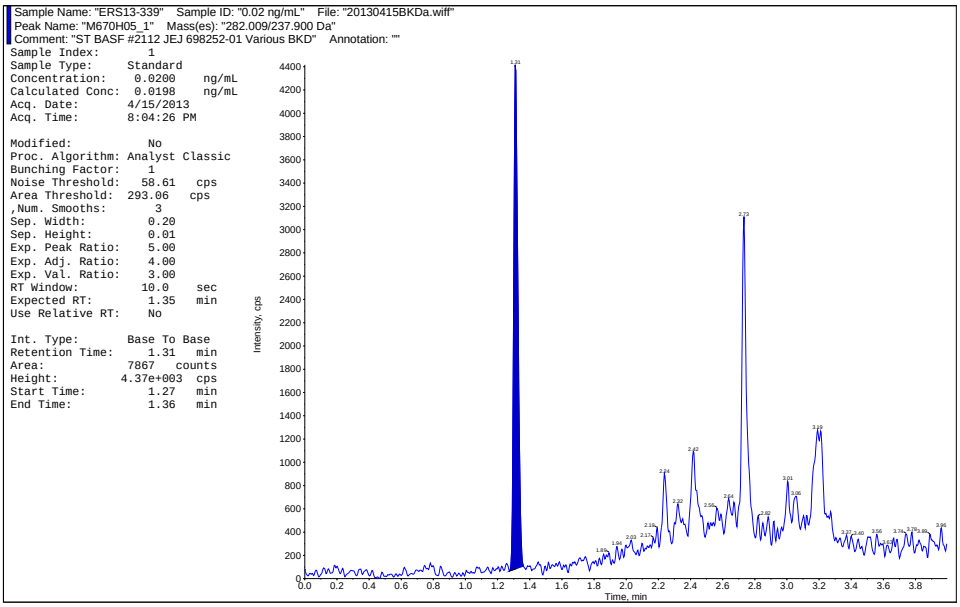
Standards chromatograms, Secondary transition, Topramezone, from Master Sheet 698252-1

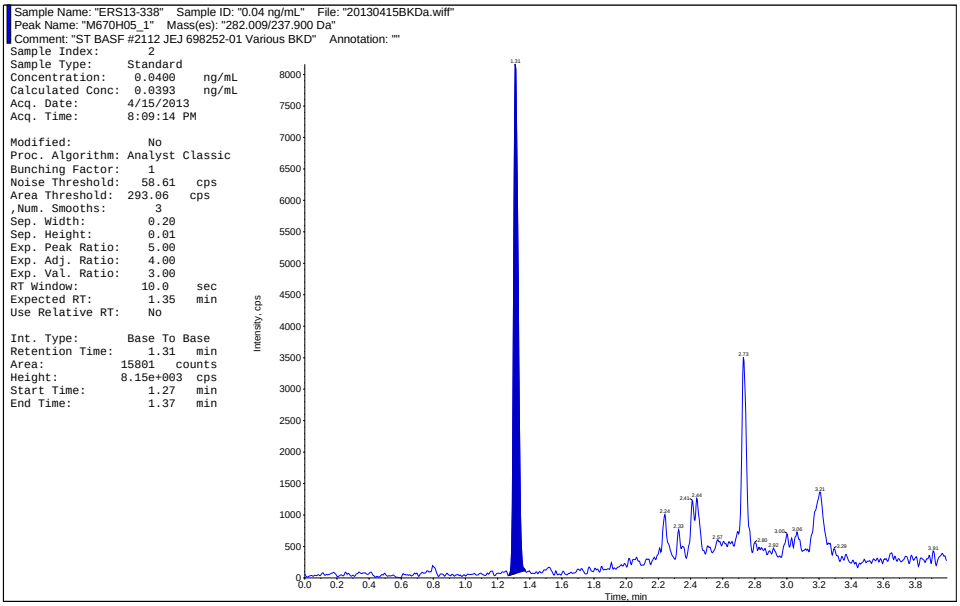


0.4 ng/mL standard (ERS13-379)

Figure 4. Example standard chromatograms for M670H05

Primary transition, M670H05, from Master Sheet 698252-1

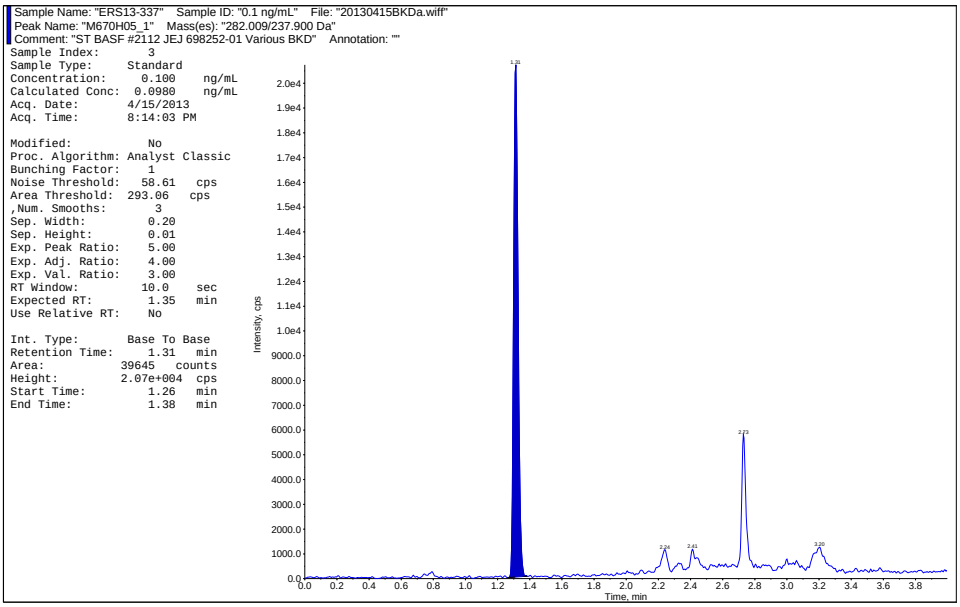


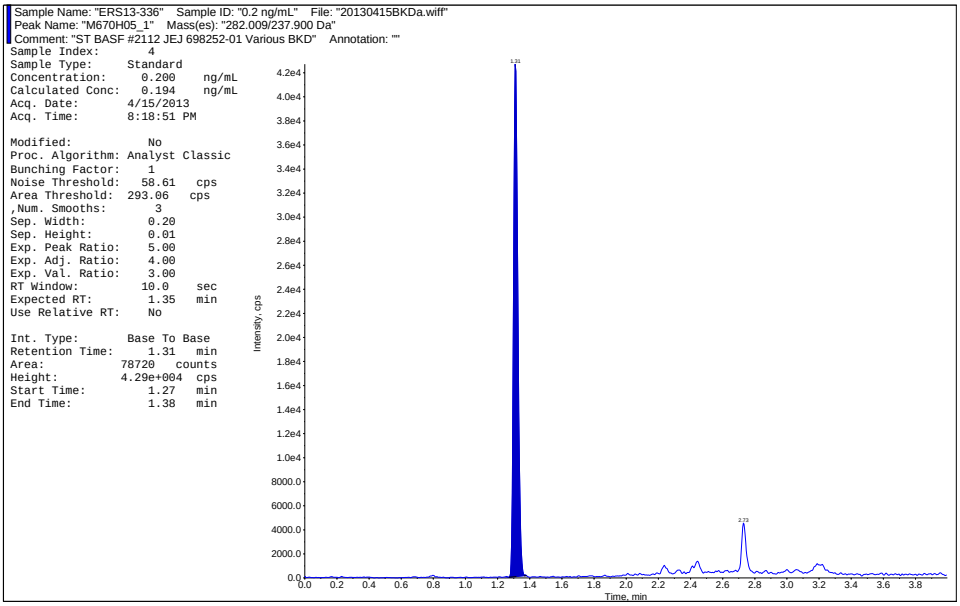


0.02 ng/mL (ERS13-383) and 0.04 ng/mL standards (ERS13-382), respectively

Figure Continued.

Standards chromatograms, Primary transition, M670H05, from Master Sheet 698252-1

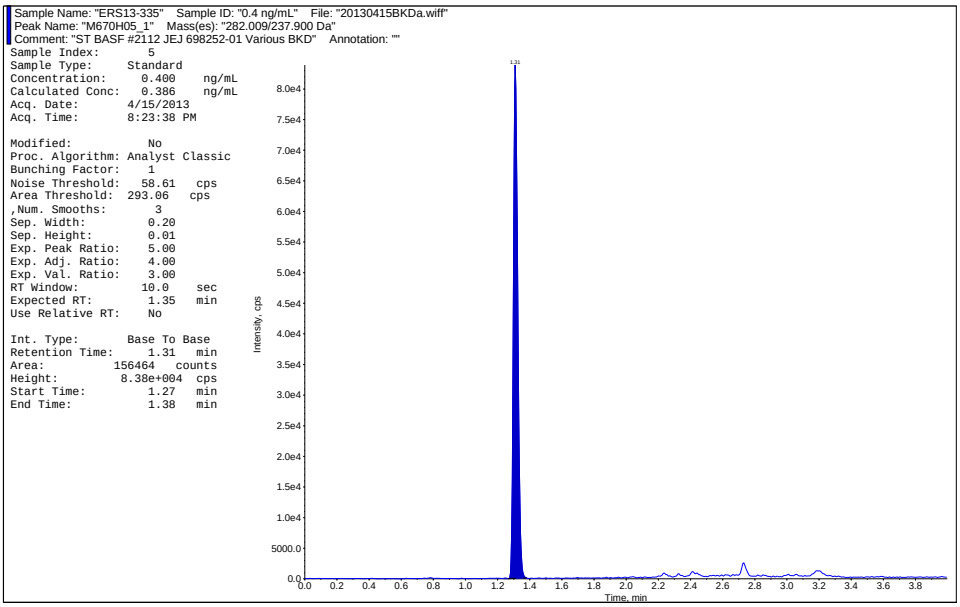




0.1 ng/mL (ERS13-381) and 0.2 ng/mL standards (ERS13-380), respectively

Figure Continued.

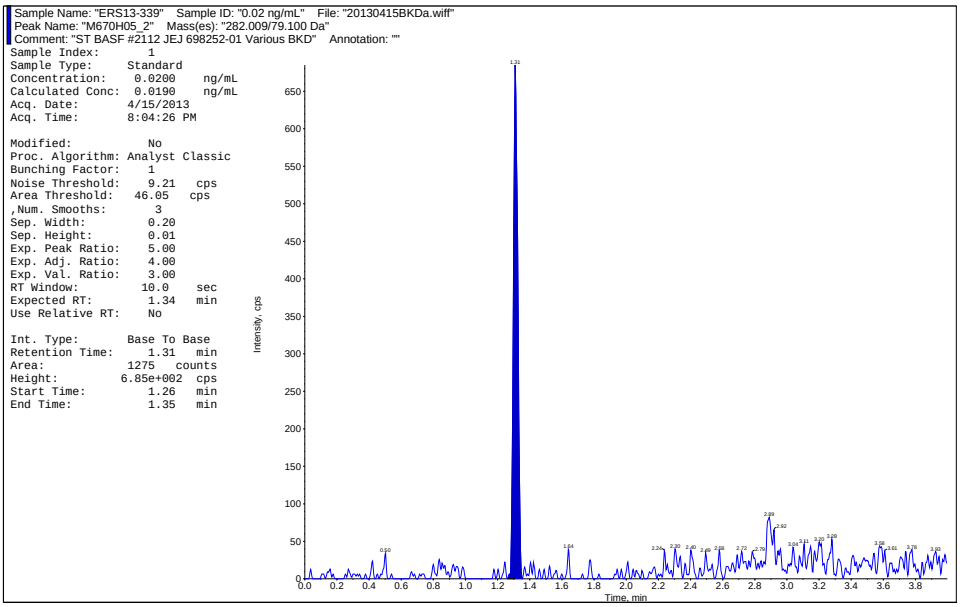
Standards chromatograms, Primary transition, M670H05, from Master Sheet 698252-1

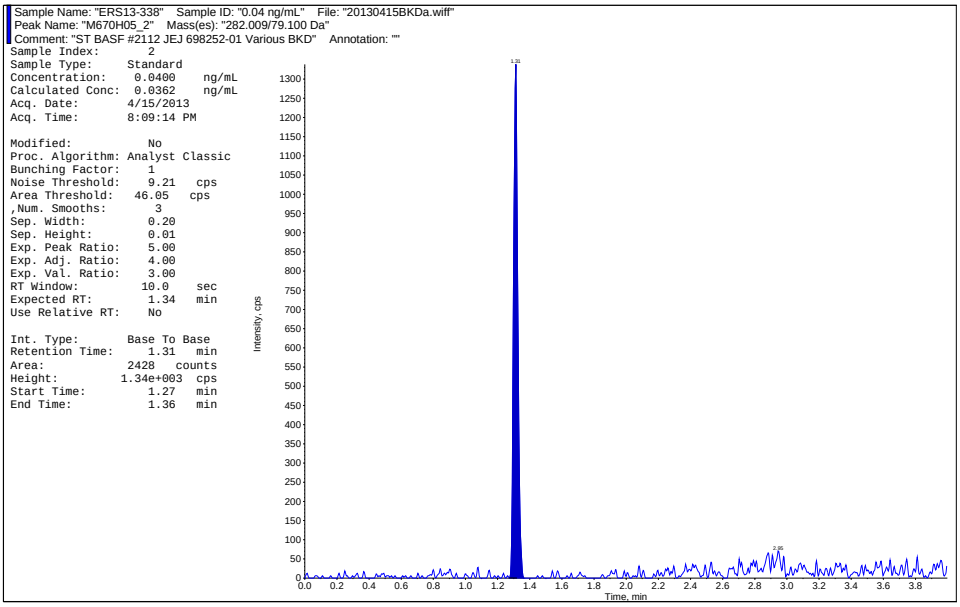


0.4 ng/mL standard (ERS13-379)

Figure Continued.

Standards chromatograms, Secondary transition, M670H05, from Master Sheet 698252-1

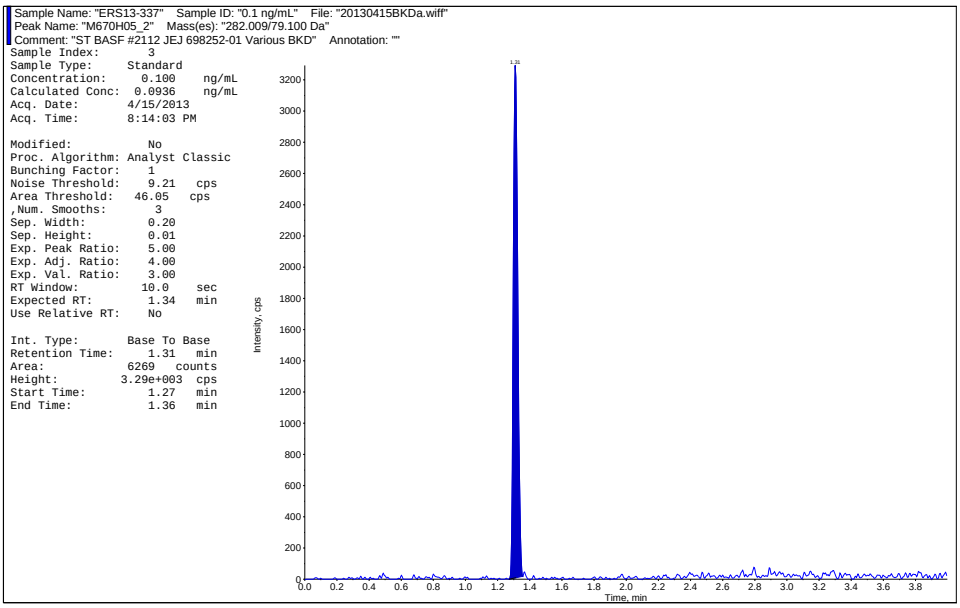


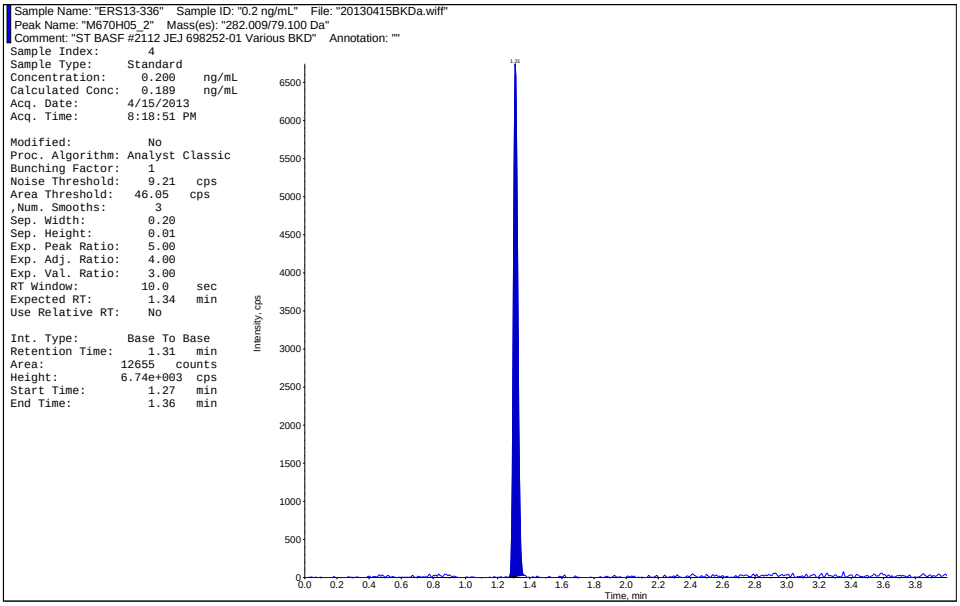


0.02 ng/mL (ERS13-383) and 0.04 ng/mL standards (ERS13-382), respectively

Figure Continued.

Standards chromatograms, Secondary transition, M670H05, from Master Sheet 698252-1

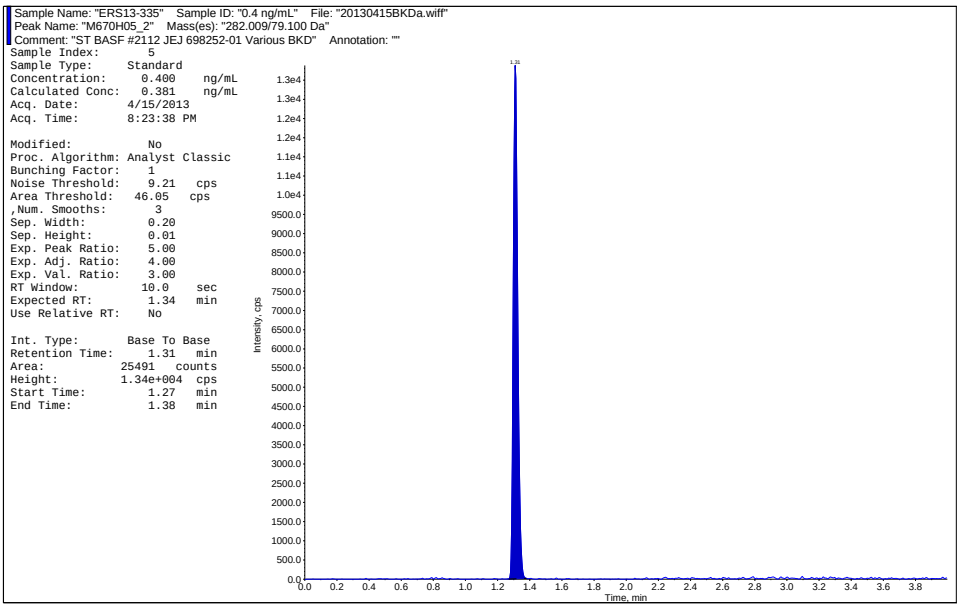




0.1 ng/mL (ERS13-381) and 0.2 ng/mL standards (ERS13-380), respectively

Figure Continued.

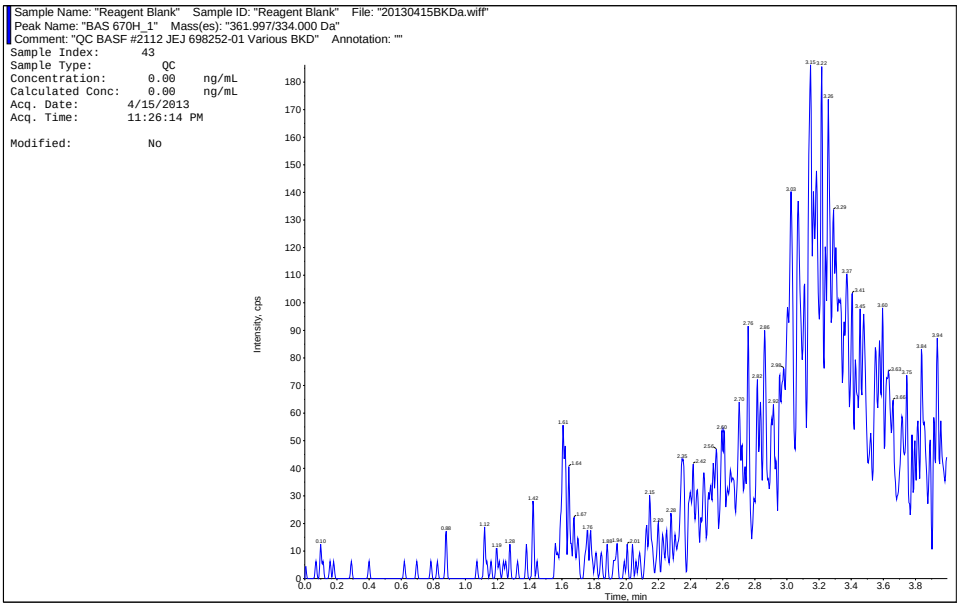
Standards chromatograms, Secondary transition, M670H05, from Master Sheet 698252-1



0.4 ng/mL standard (ERS13-379)

Figure 5. Chromatograms of a reagent blank

Analyzed for Topramezone, from Master Sheet 698252-1



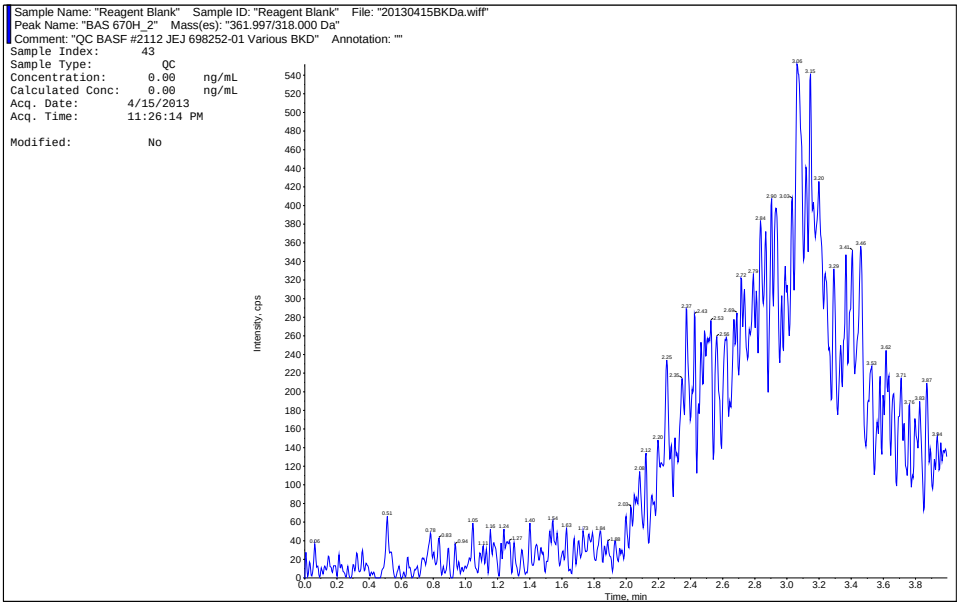
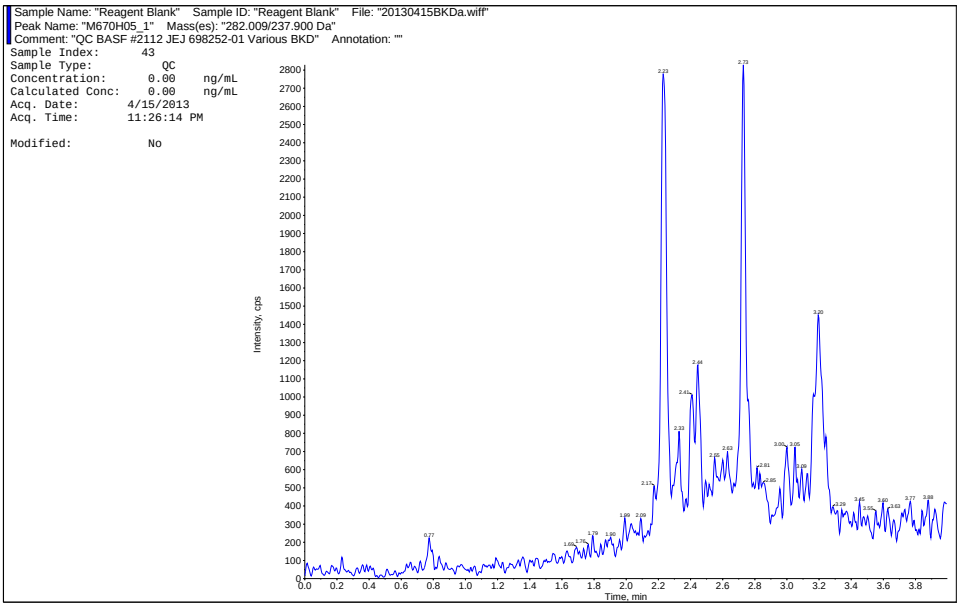


Figure Continued.

Reagent blank, Analyzed for M670H05, from Master Sheet 698252-1



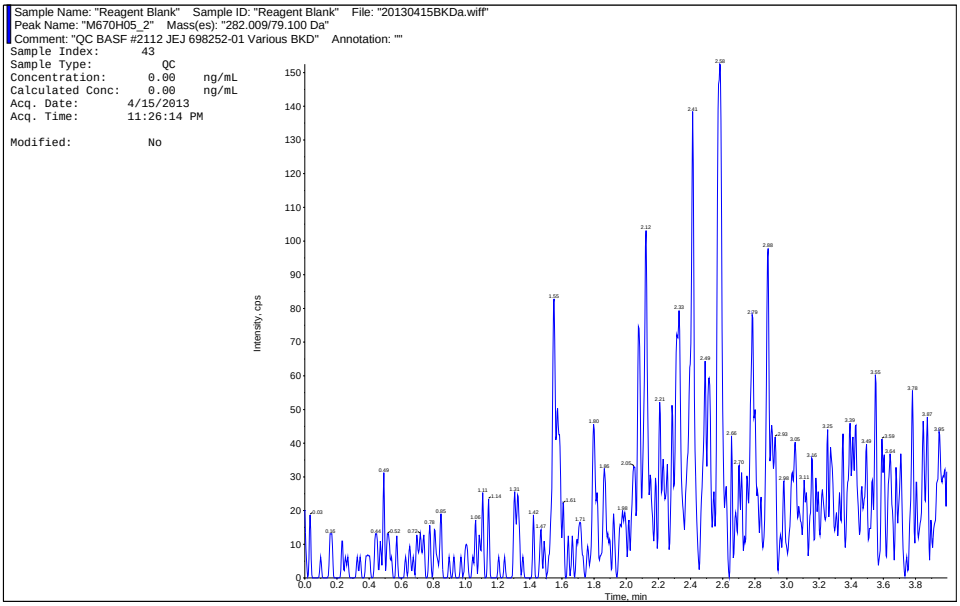
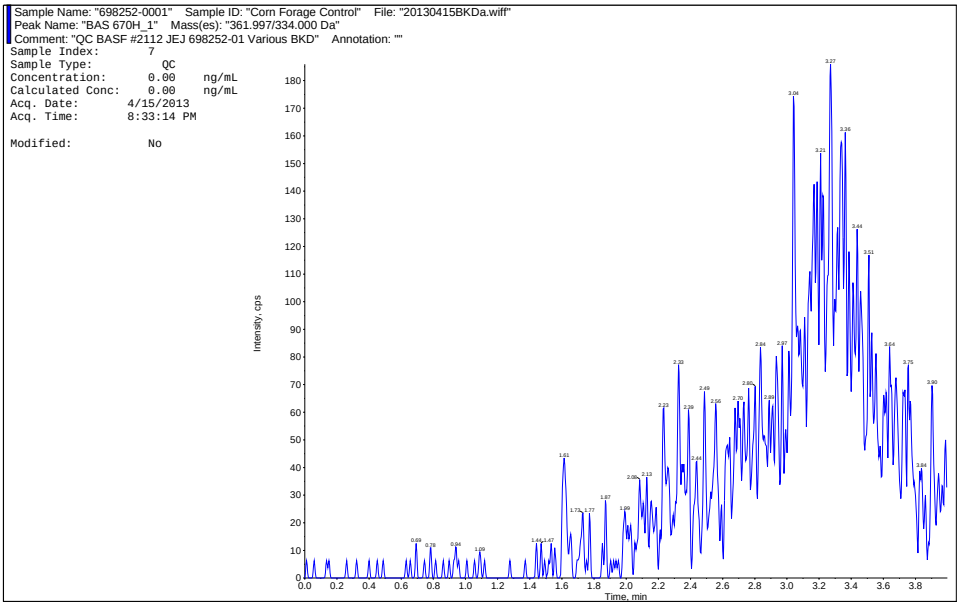


Figure 6. Chromatograms of a control corn forage sample, Sample No. 698252-0001, from Master Sheet 698252-1

Analyzed for Topramezone



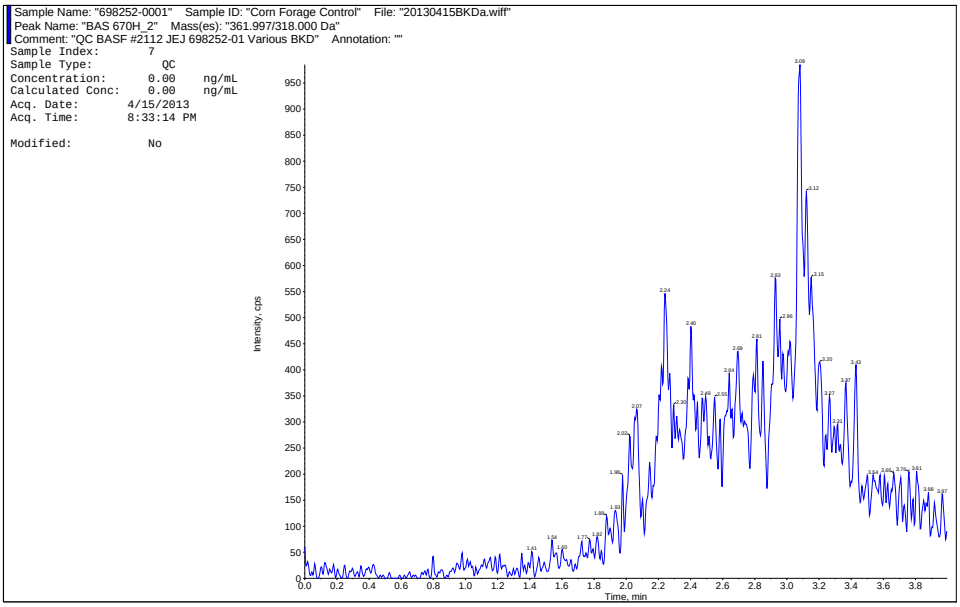
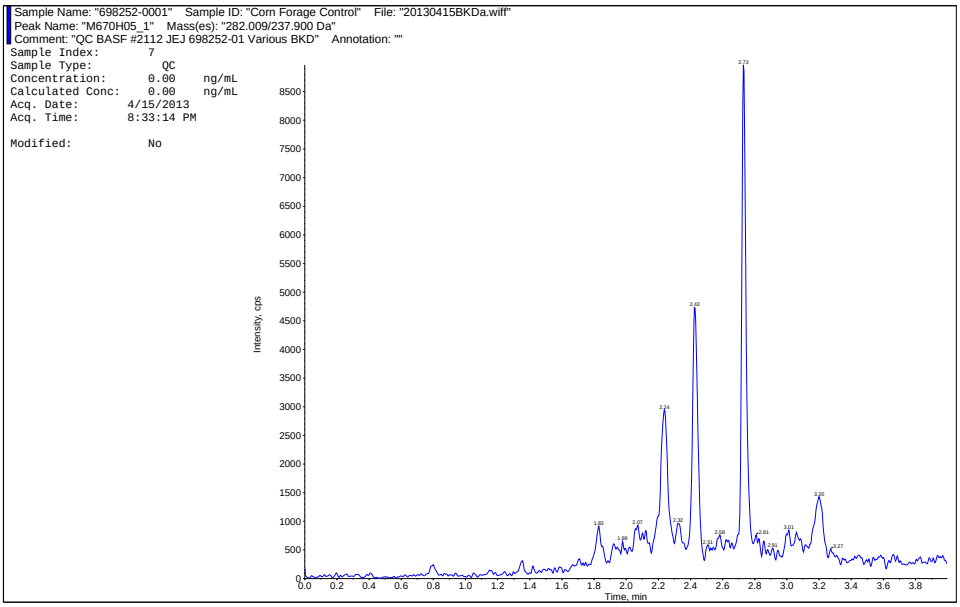


Figure Continued.

Control corn forage sample, analyzed for M670H05



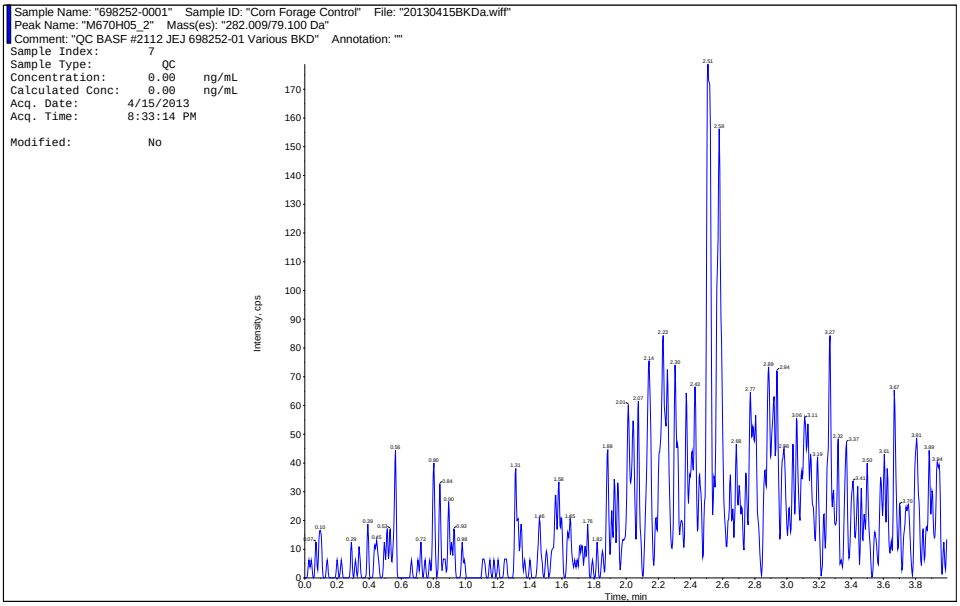
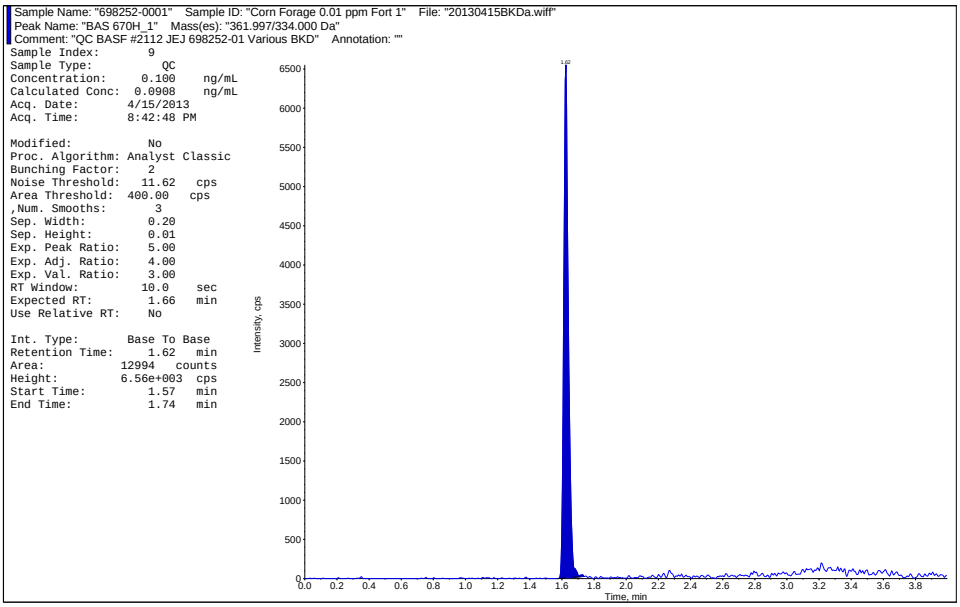


Figure 7. Chromatograms of a control corn forage sample fortified with each analyte at 0.01 ppm, Sample No. 698252-0001, from Master Sheet 698252-1



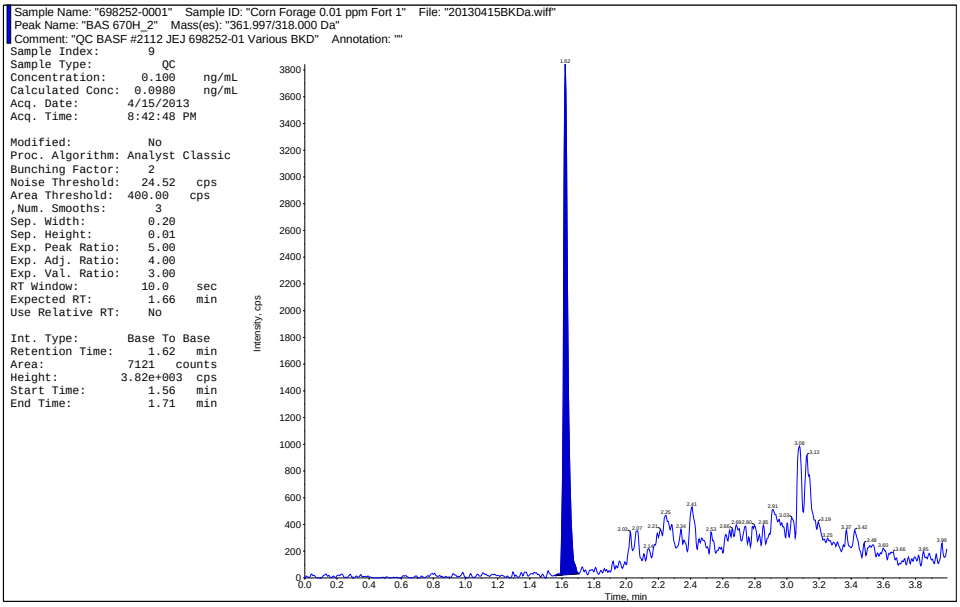
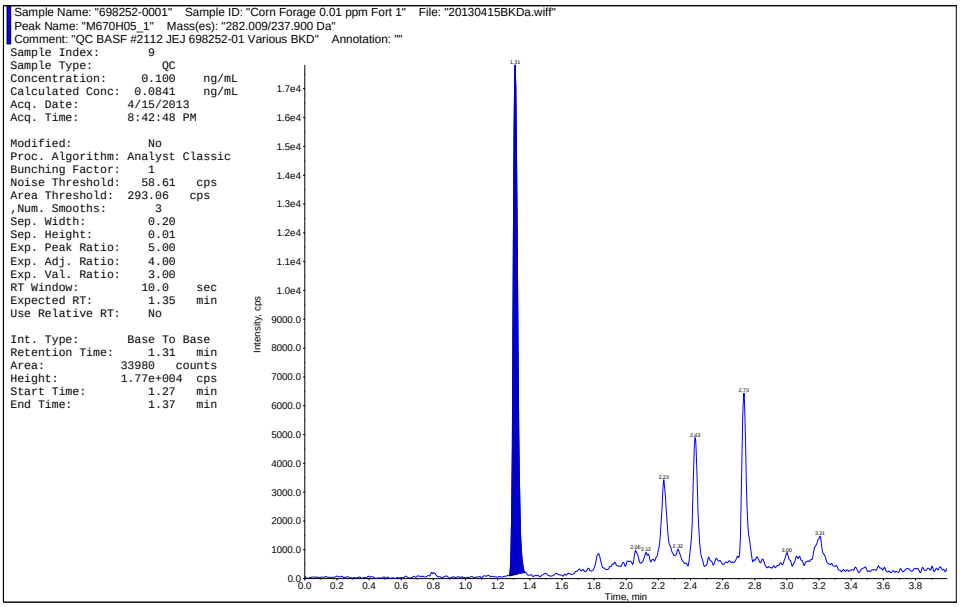


Figure Continued.

Low fortification (0.01 ppm), M670H05



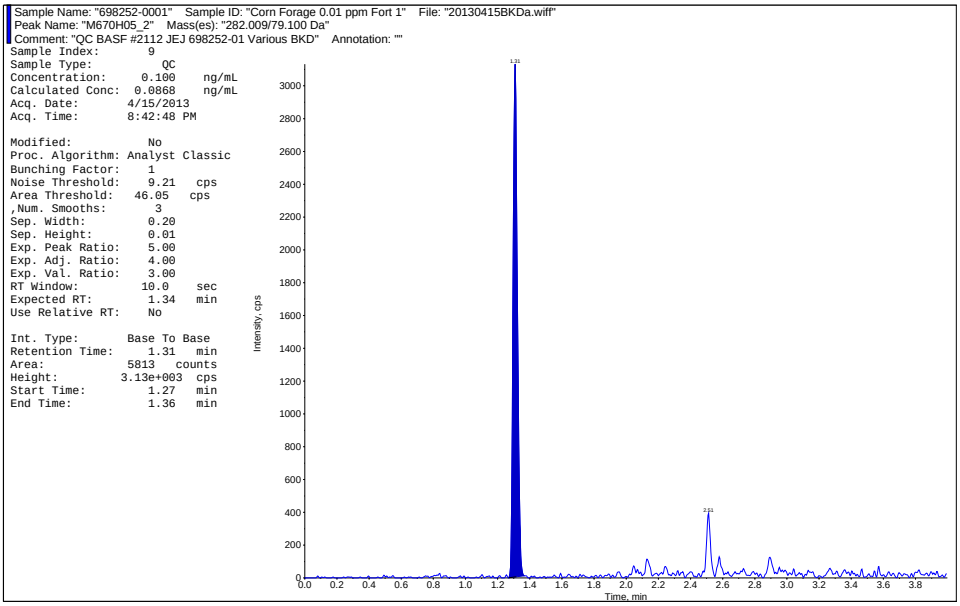
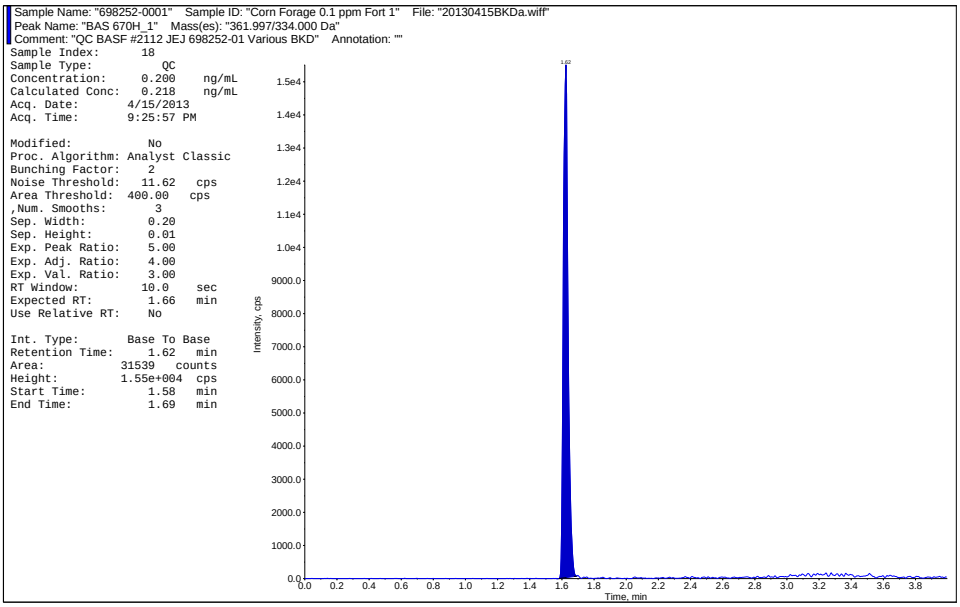


Figure 8. Chromatograms of a control corn forage sample fortified with each analyte at 0.1 ppm, Sample No. 698252-0001, from Master Sheet 698252-1



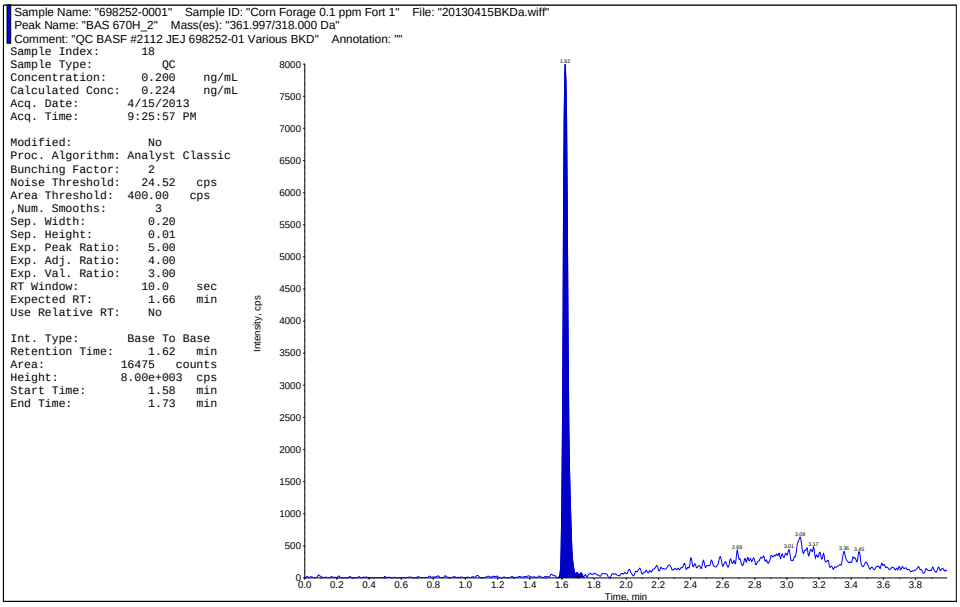
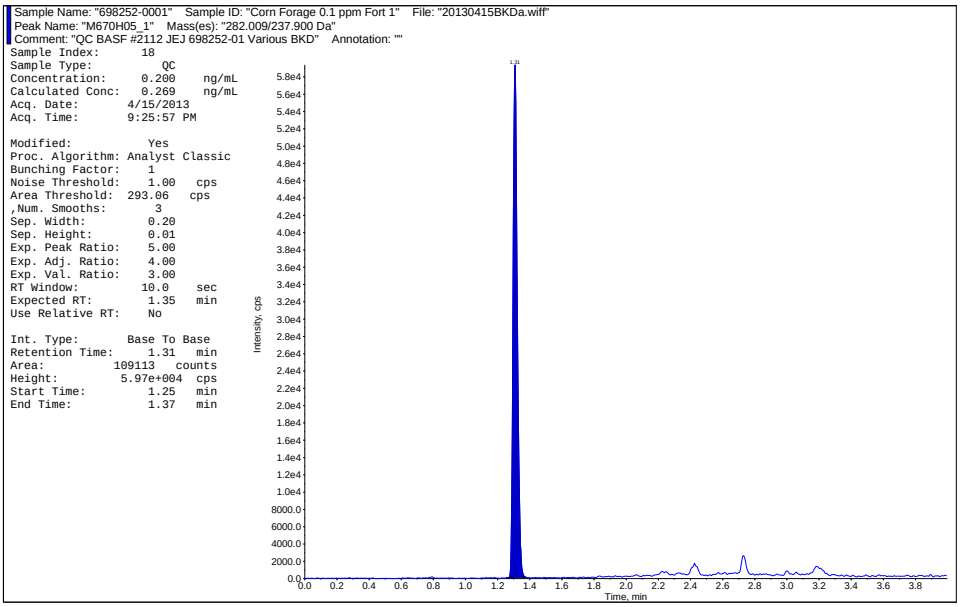


Figure Continued.

High fortification (0.1 ppm), M670H05



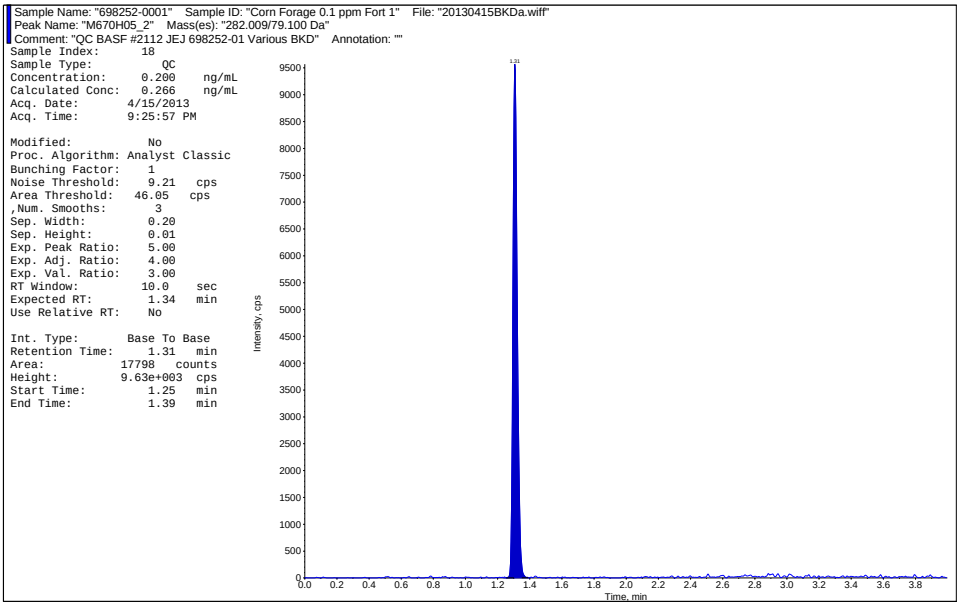
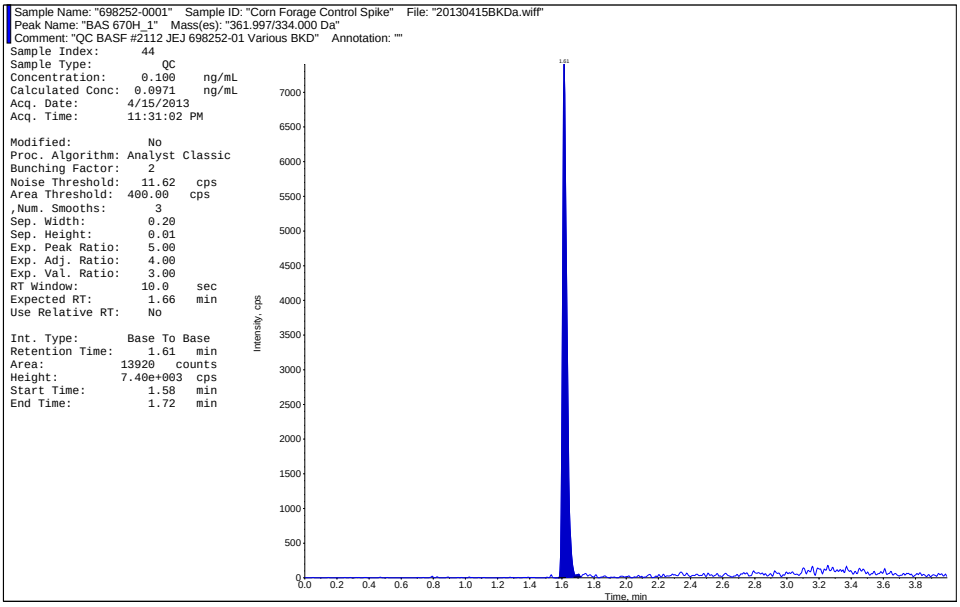


Figure 9. Chromatograms of control extract of corn forage fortified with each analyte at 0.1 ng/mL, Sample No. 698252-0001, from Master Sheet 698252-1



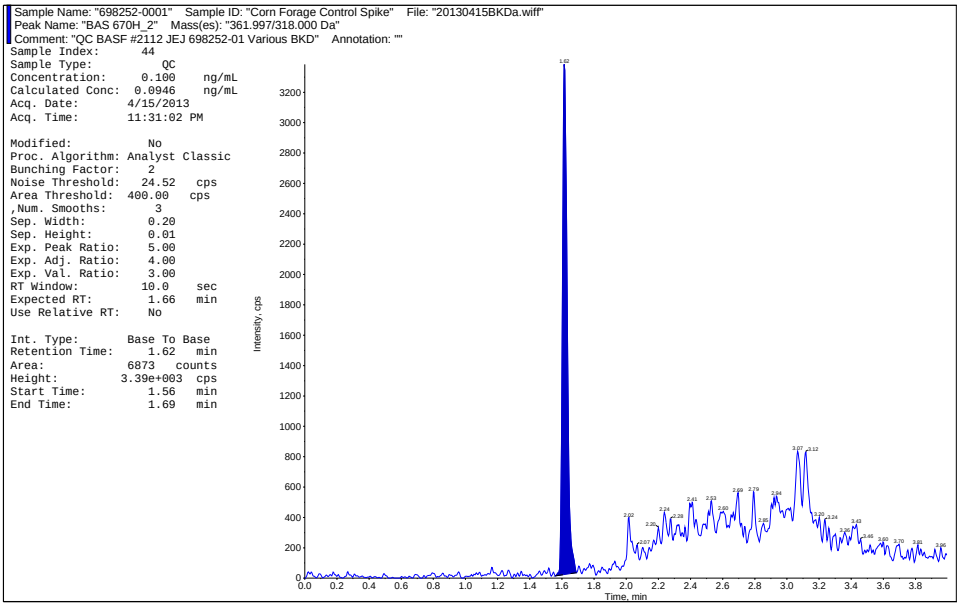
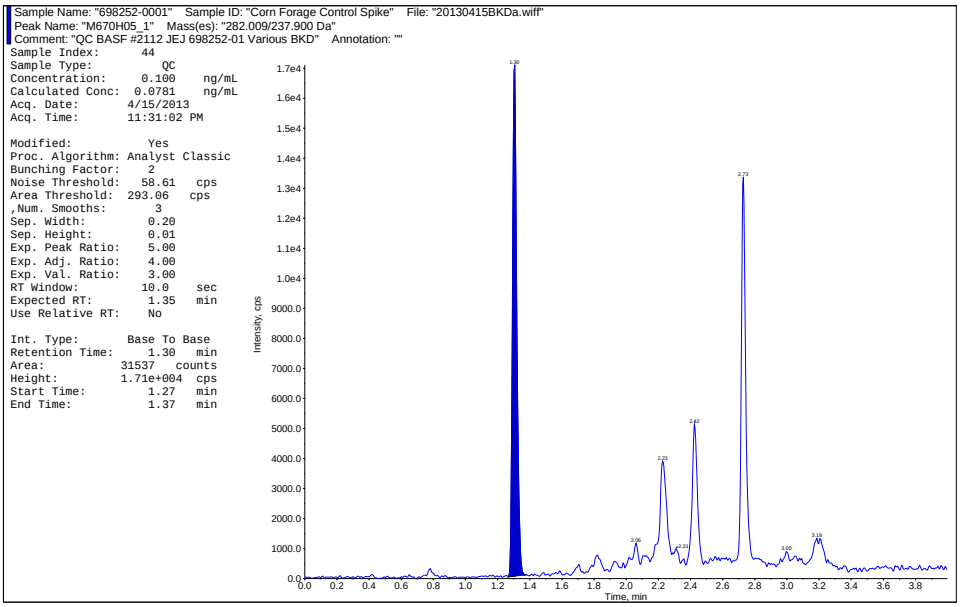


Figure Continued.

Control extract of corn forage fortified with M670H05 (Matrix Effects Test)



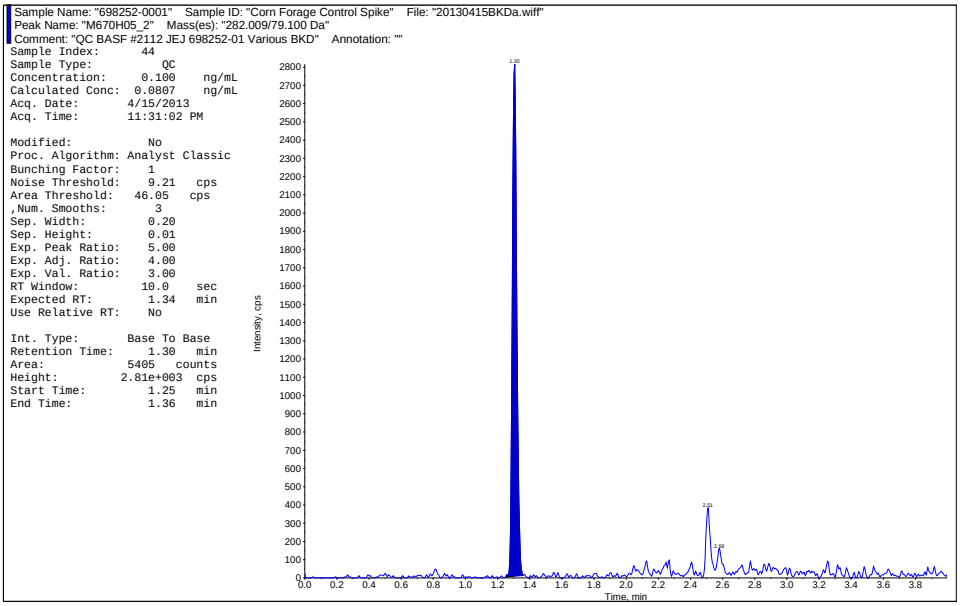
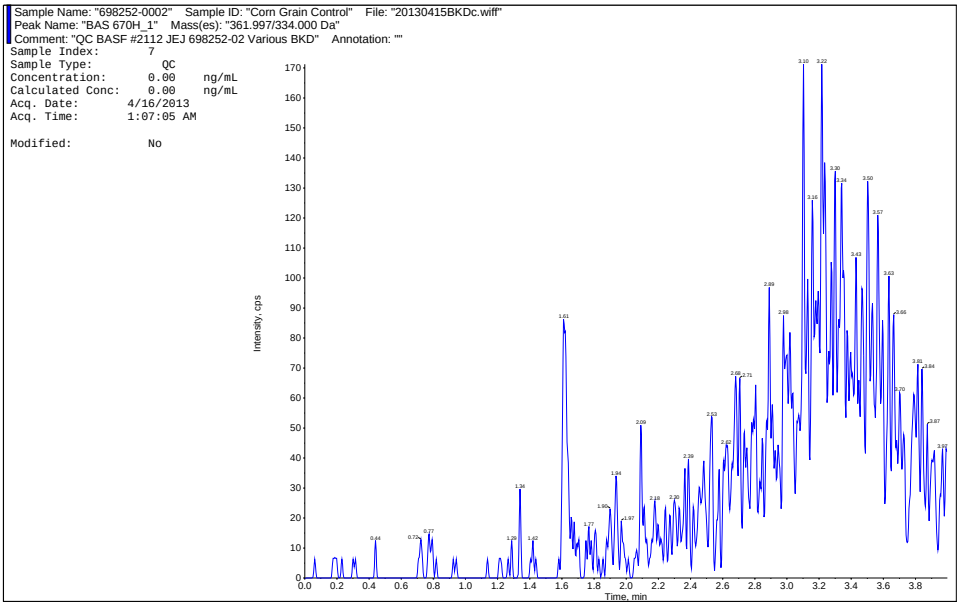


Figure 10. Chromatograms of a control corn grain sample, Sample No. 698252-0002, from Master Sheet 698252-2

Analyzed for Topramezone



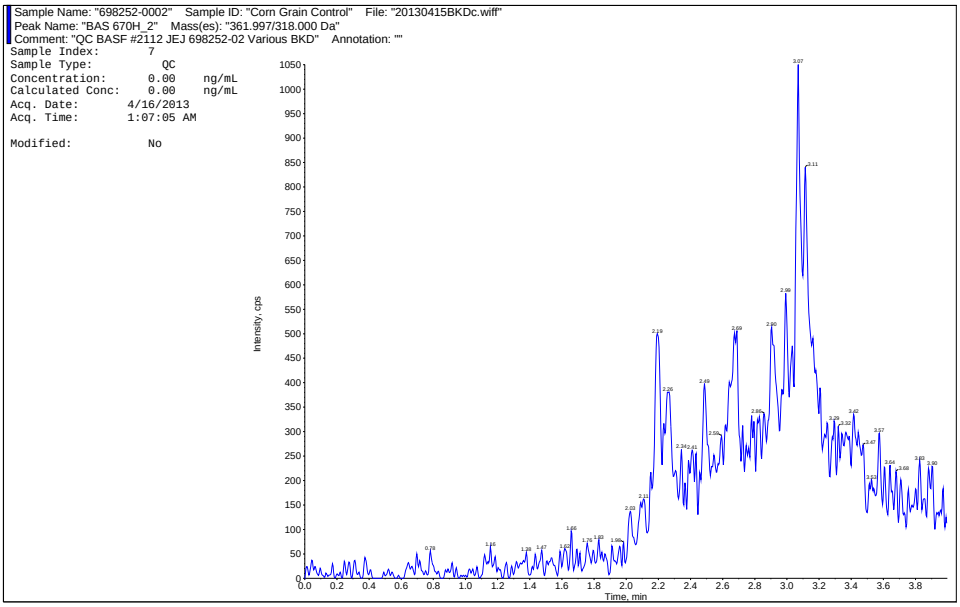
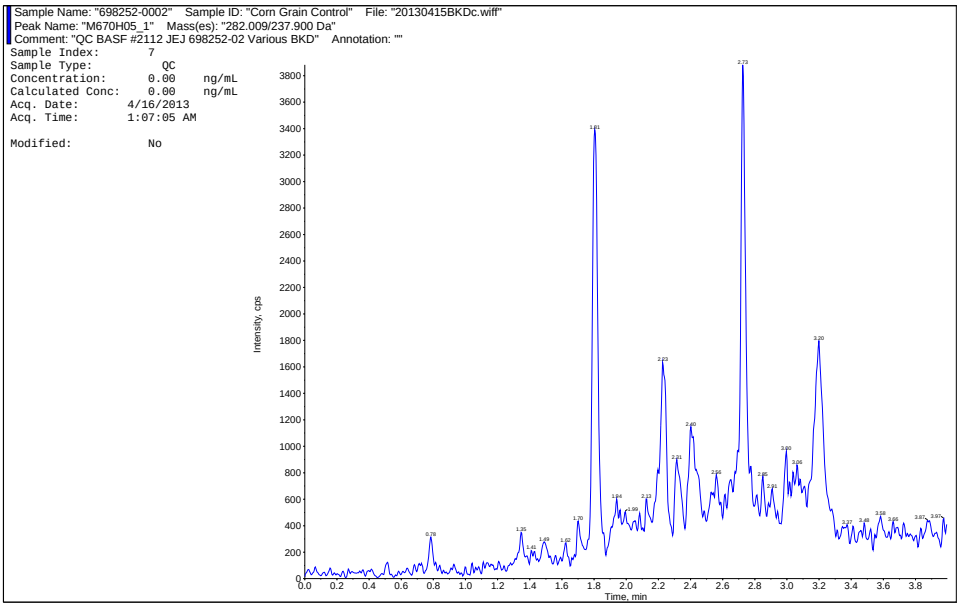


Figure Continued.

Control corn grain sample, analyzed for M670H05



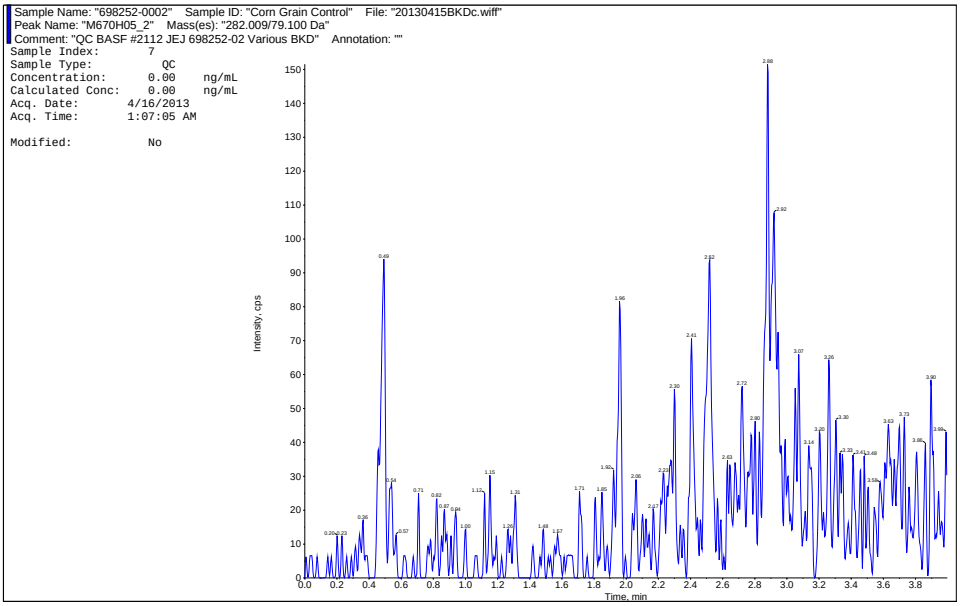
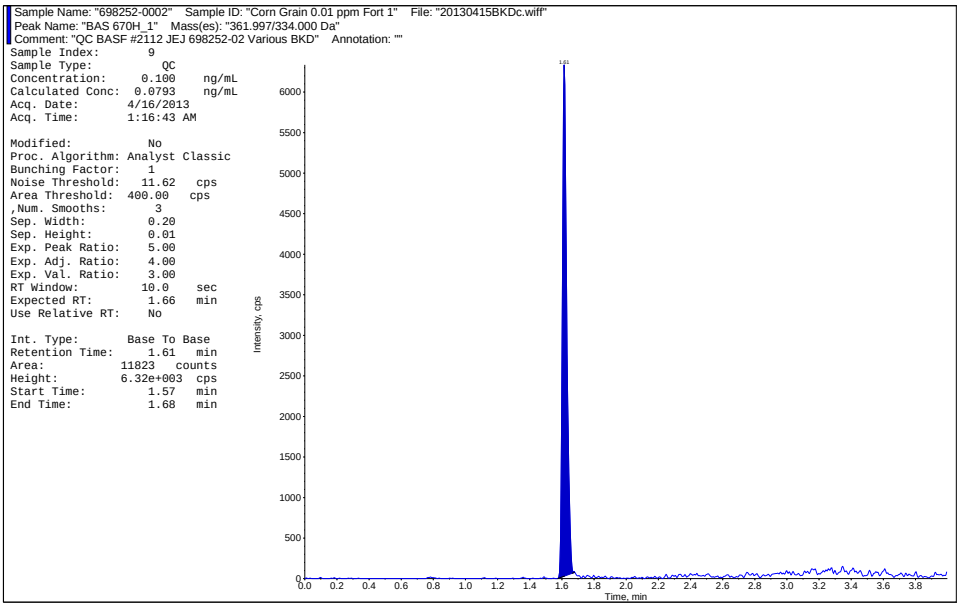


Figure 11. Chromatograms of a control corn grain sample fortified with each analyte at 0.01 ppm, Sample No. 698252-0002, from Master Sheet 698252-2



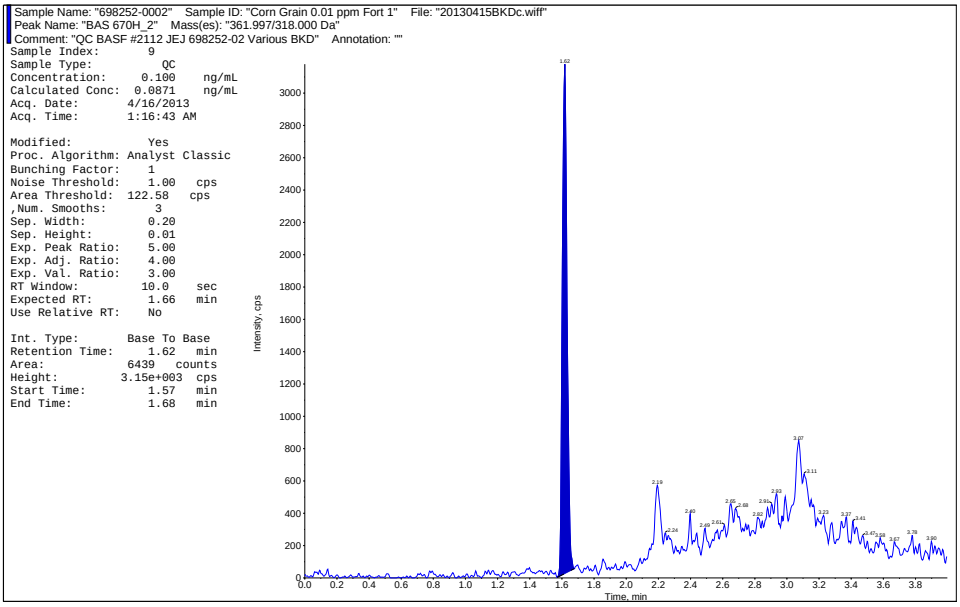
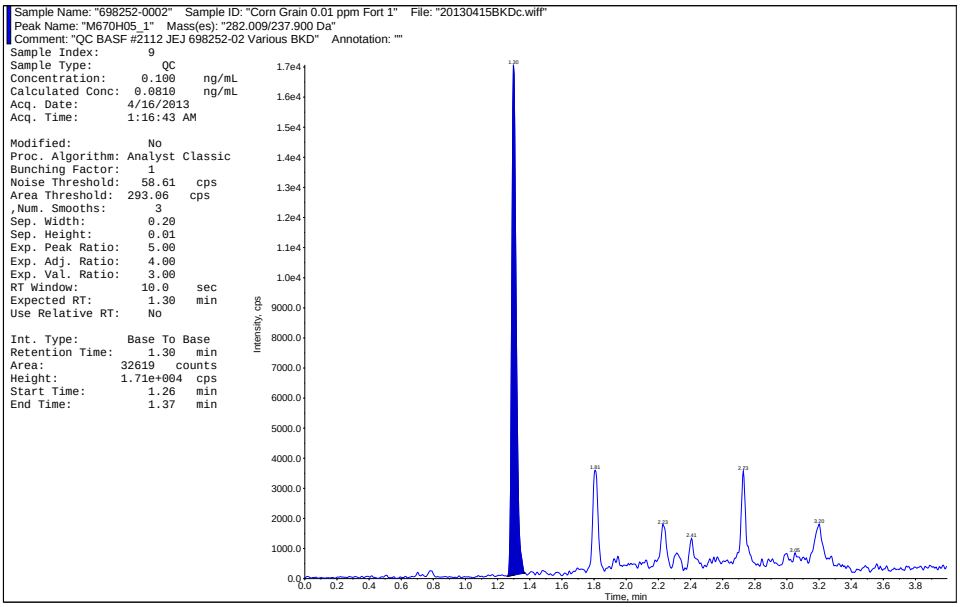


Figure Continued.

Low fortification (0.01 ppm), M670H05



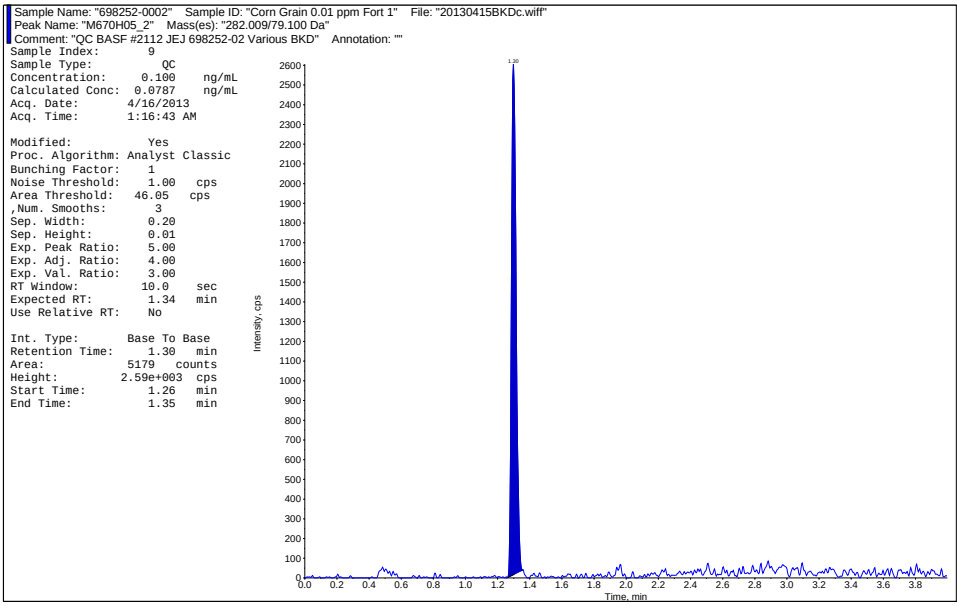
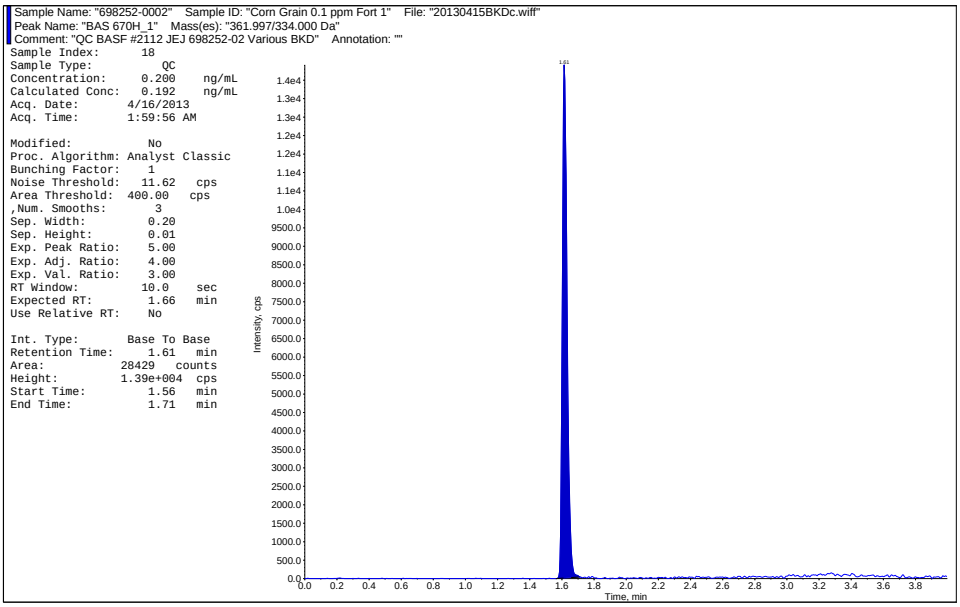


Figure 12. Chromatograms of a control corn grain sample fortified with each analyte at 0.1 ppm, Sample No. 698252-0002, from Master Sheet 698252-2



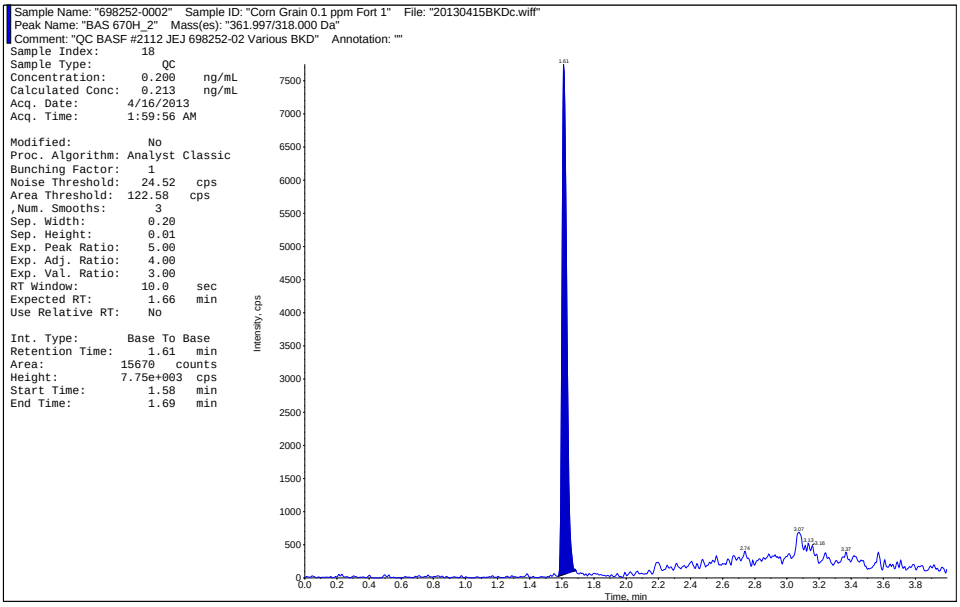
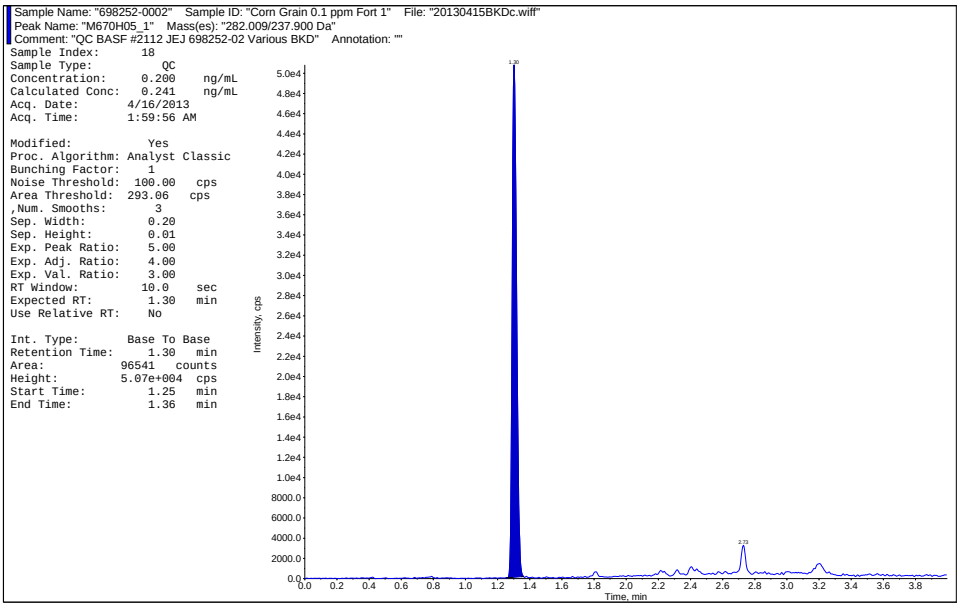


Figure Continued.

High fortification (0.1 ppm), M670H05



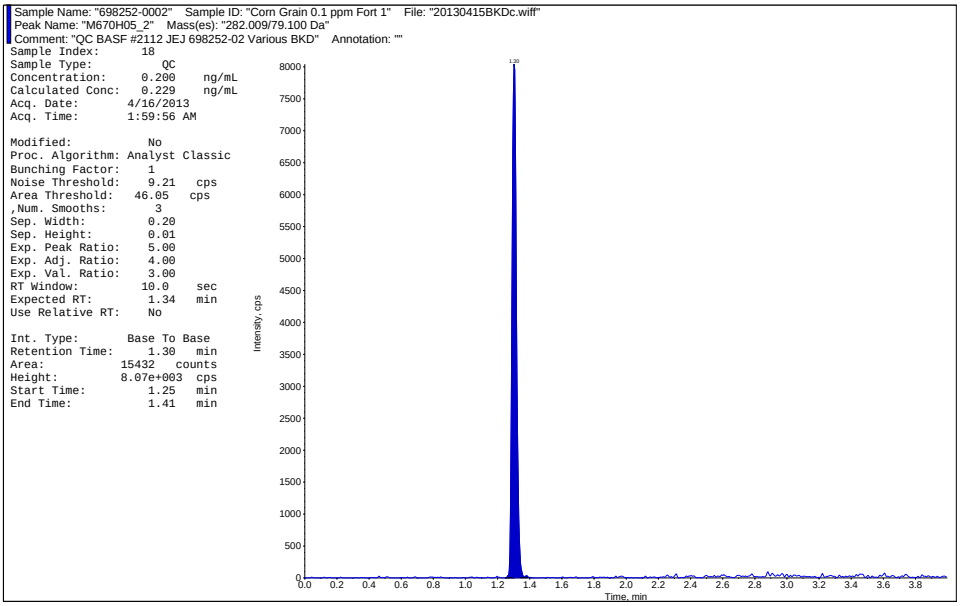
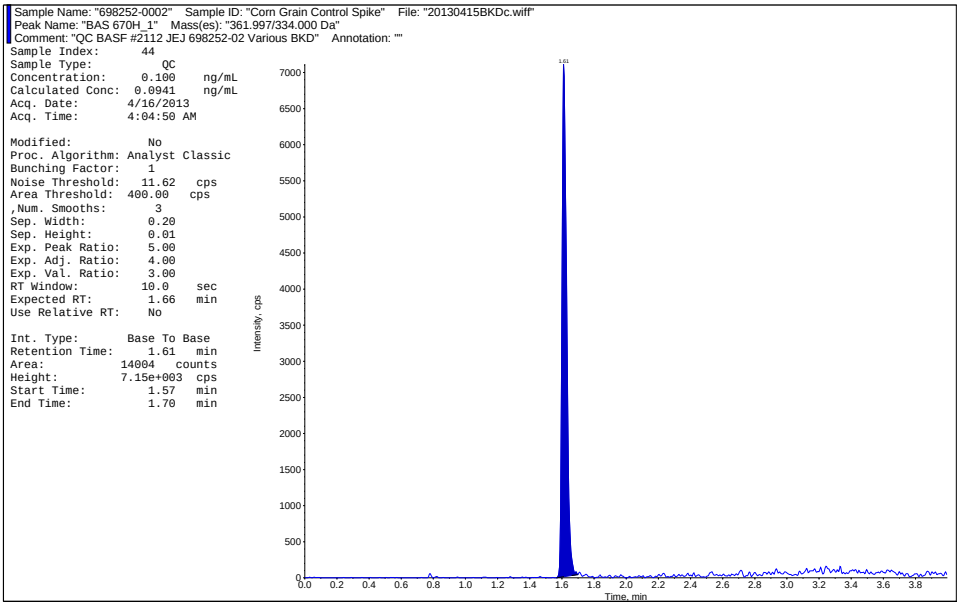


Figure 13. Chromatograms of control extract of corn grain fortified with each analyte at 0.1 ng/mL, Sample No. 698252-0002, from Master Sheet 698252-2



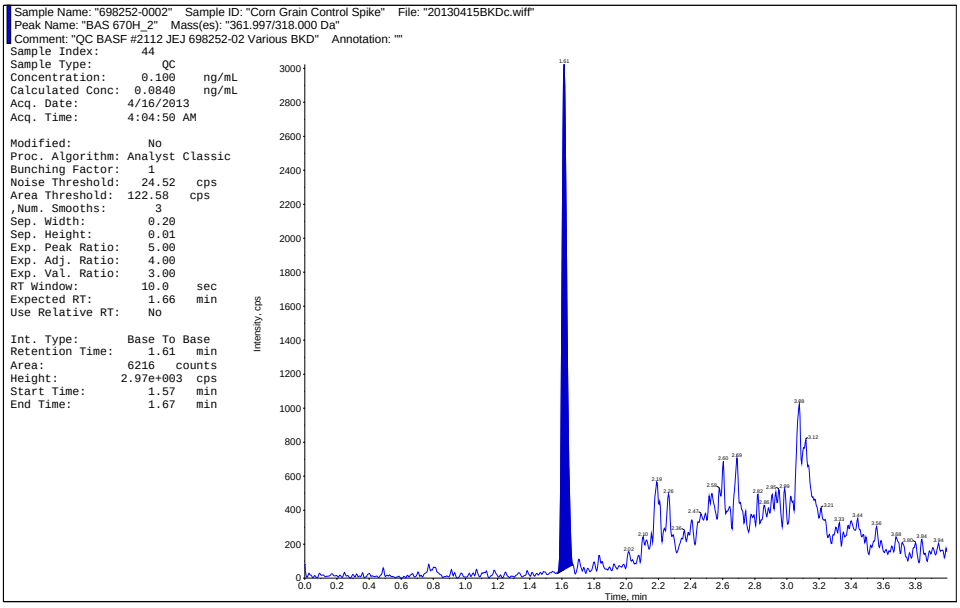
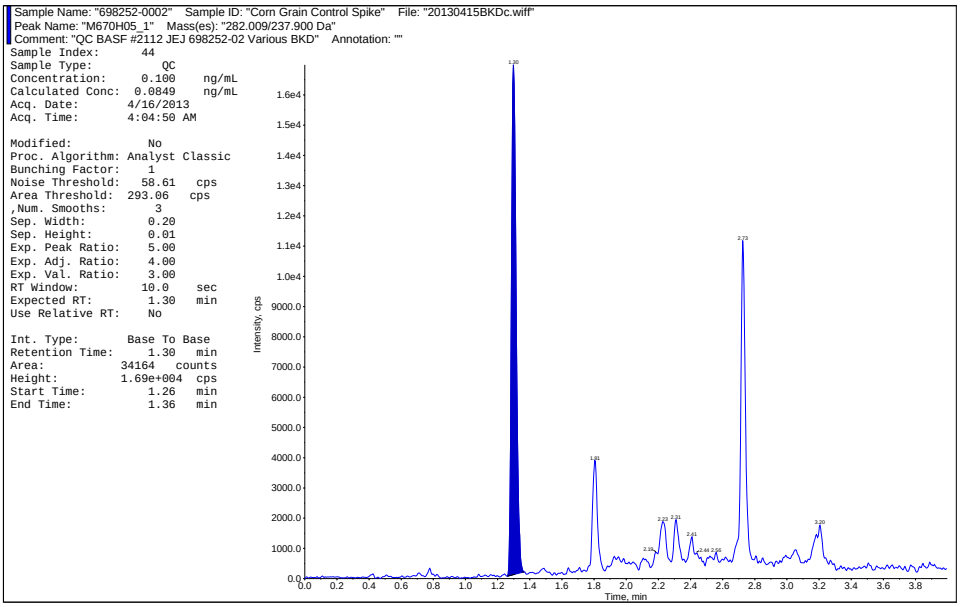


Figure Continued.

Control extract of corn grain fortified with M670H05 (Matrix Effects Test)



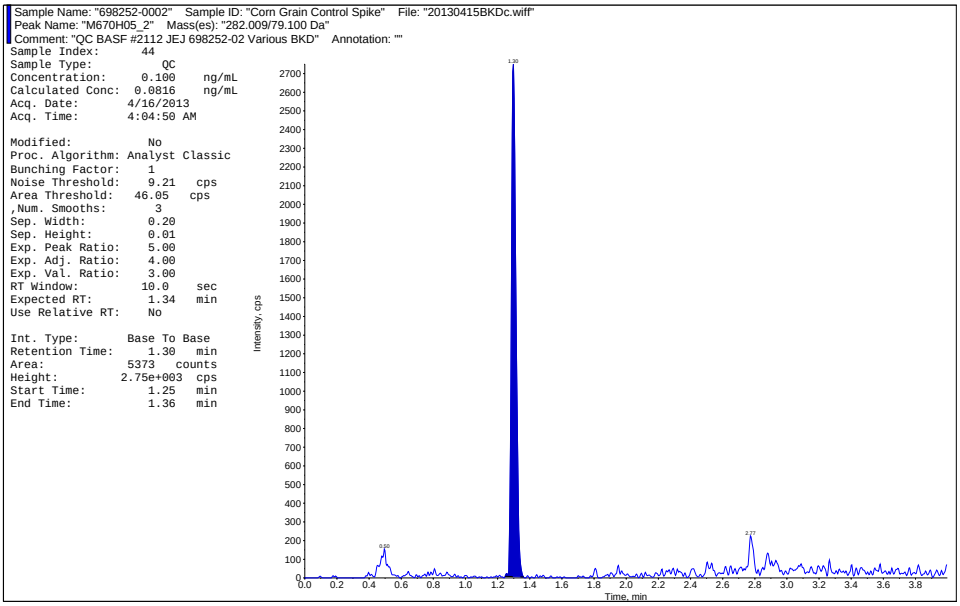
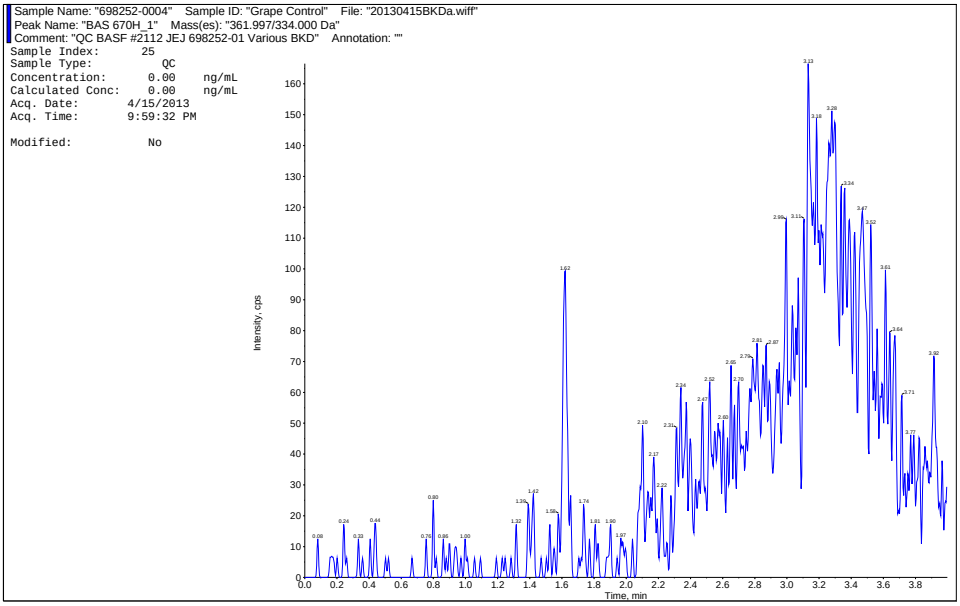


Figure 14. Chromatograms of a control grape (fruit) sample, Sample No. 698252-0004, from Master Sheet 698252-1

Analyzed for Topramezone



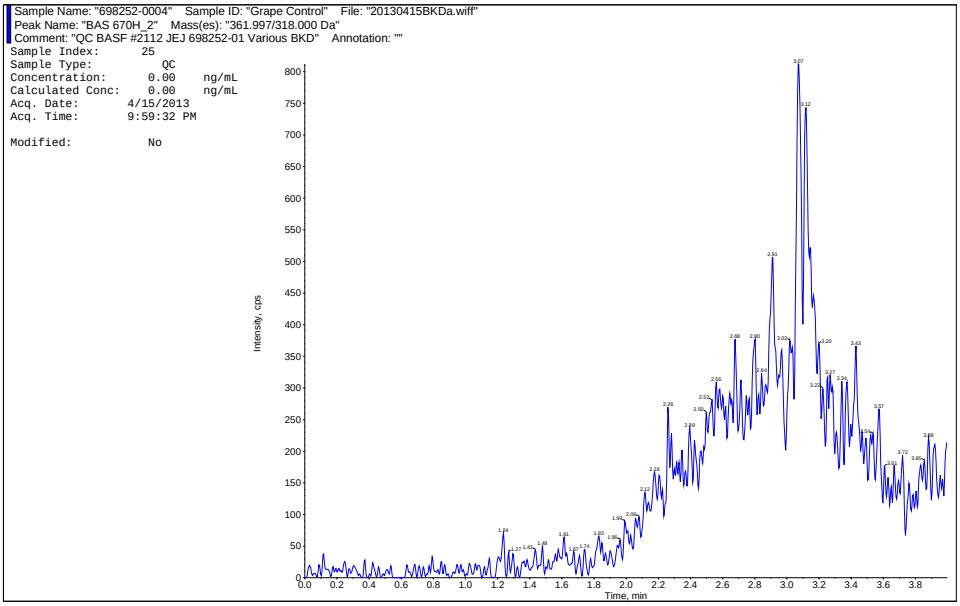
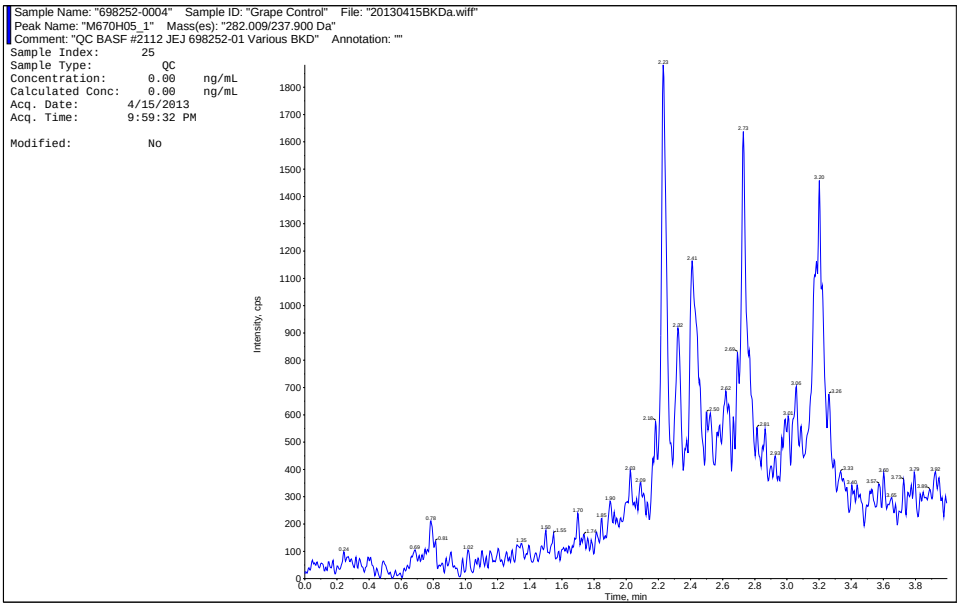


Figure Continued.

Control Grape (fruit) sample, analyzed for M670H05



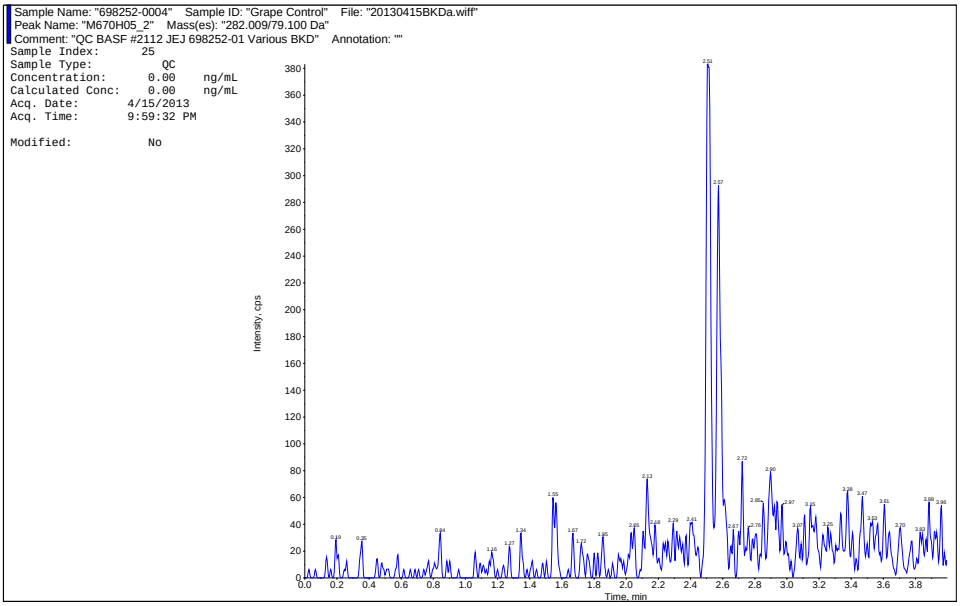
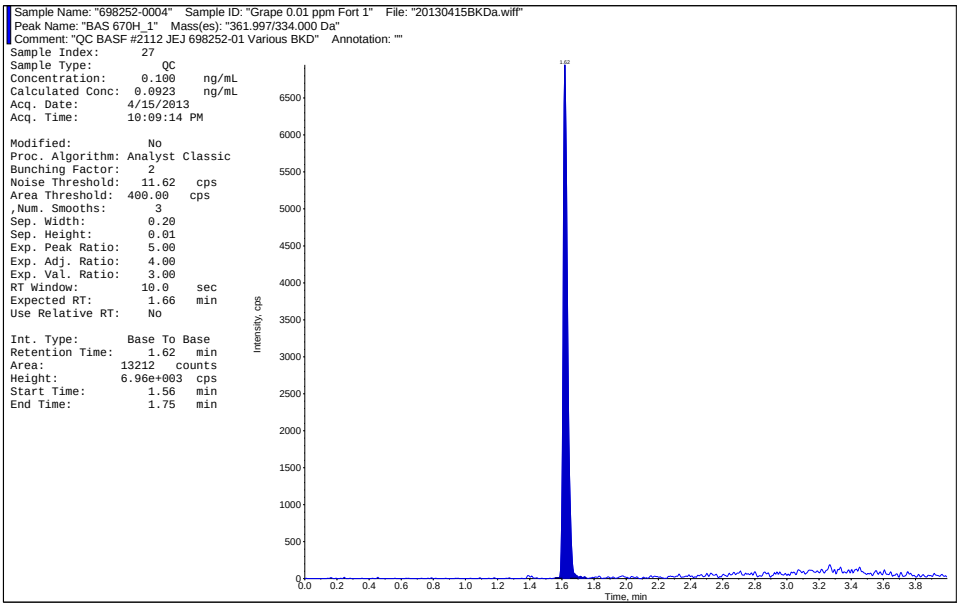


Figure 15. Chromatograms of a control grape (fruit) sample fortified with each analyte at 0.01 ppm, Sample No. 698252-0004, from Master Sheet 698252-1



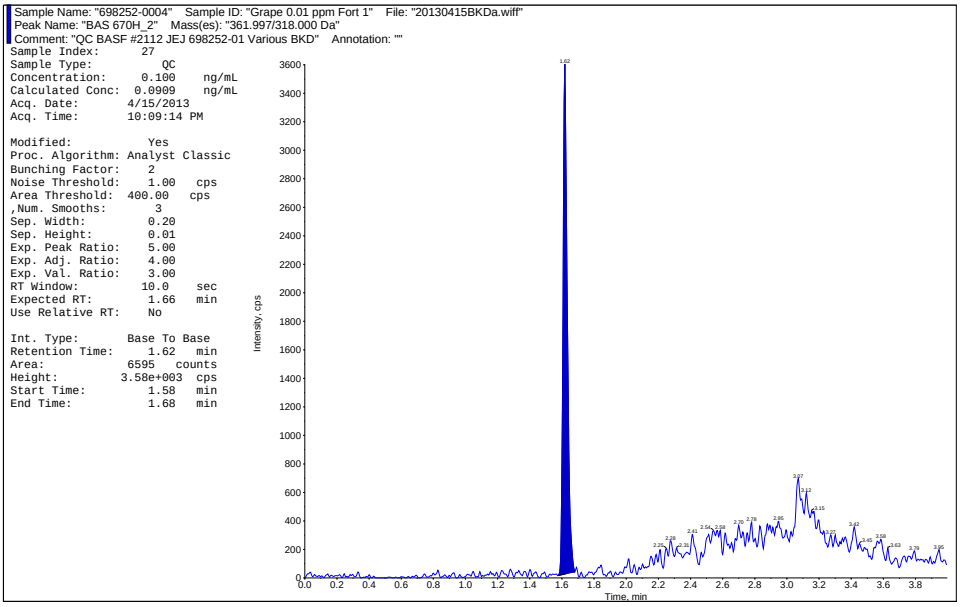
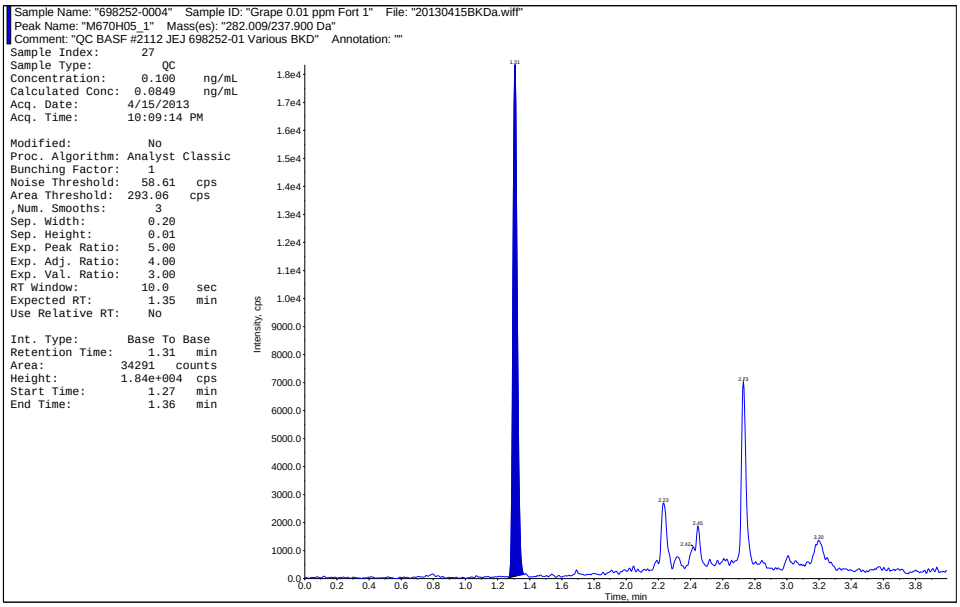


Figure Continued.

Low fortification (0.01 ppm), M670H05



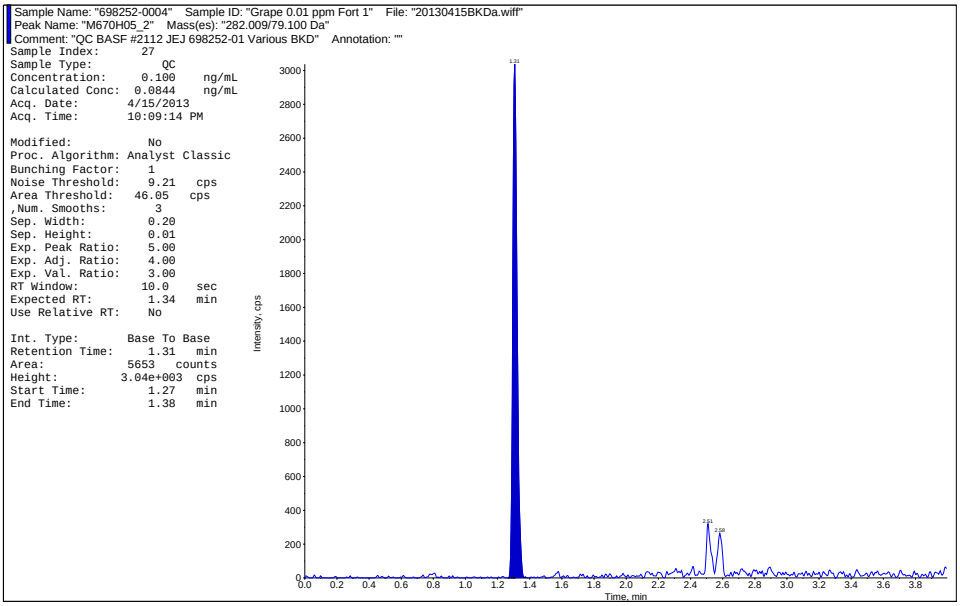
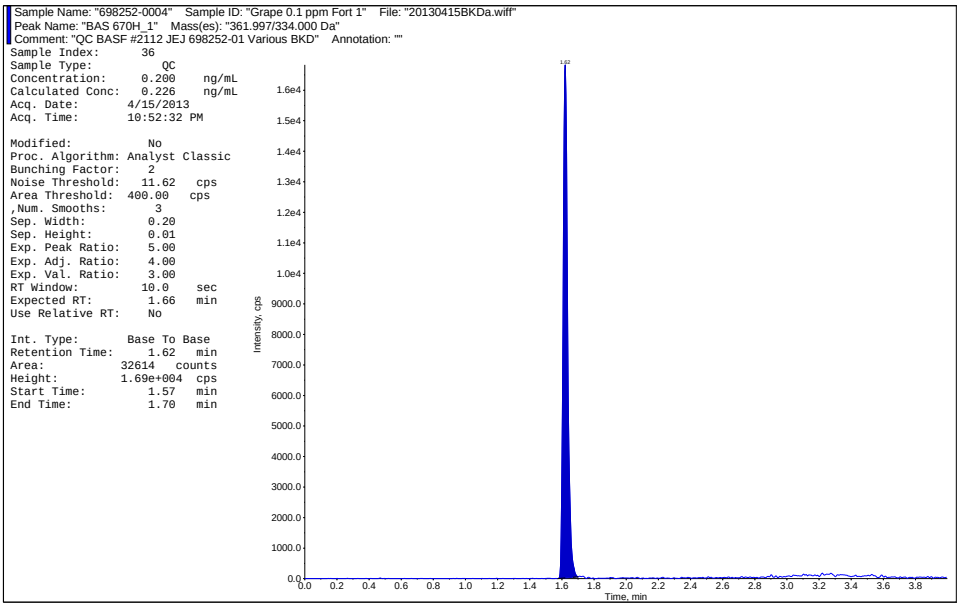


Figure 16. Chromatograms of a control grape (fruit) sample fortified with each analyte at 0.1 ppm, Sample No. 698252-0004, from Master Sheet 698252-1



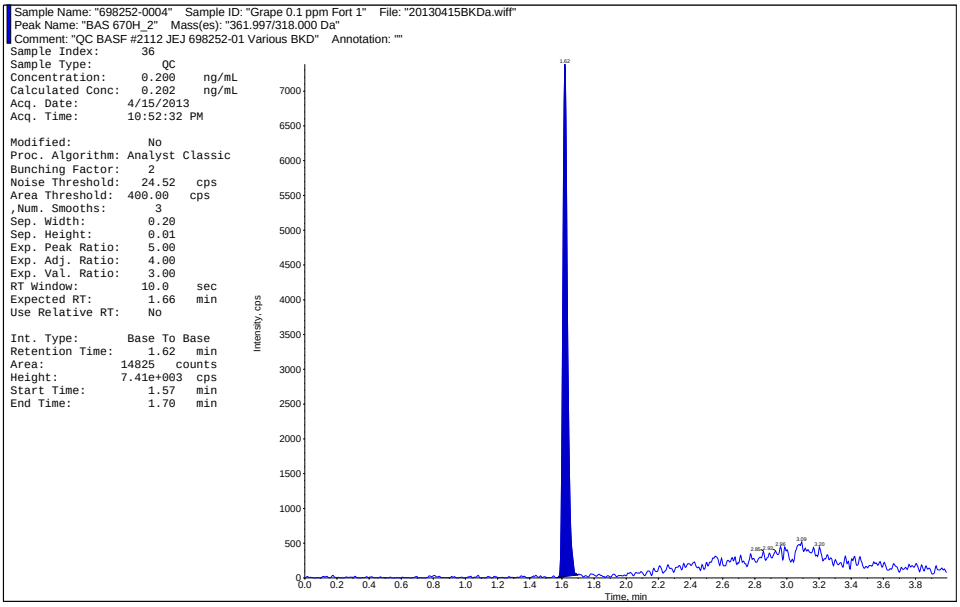
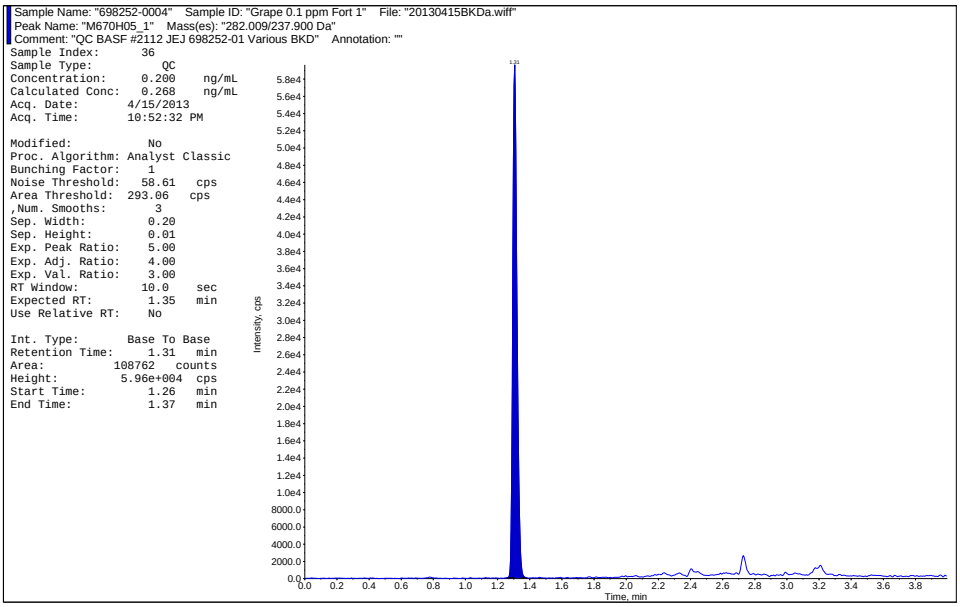


Figure Continued.

High fortification (0.1 ppm), M670H05



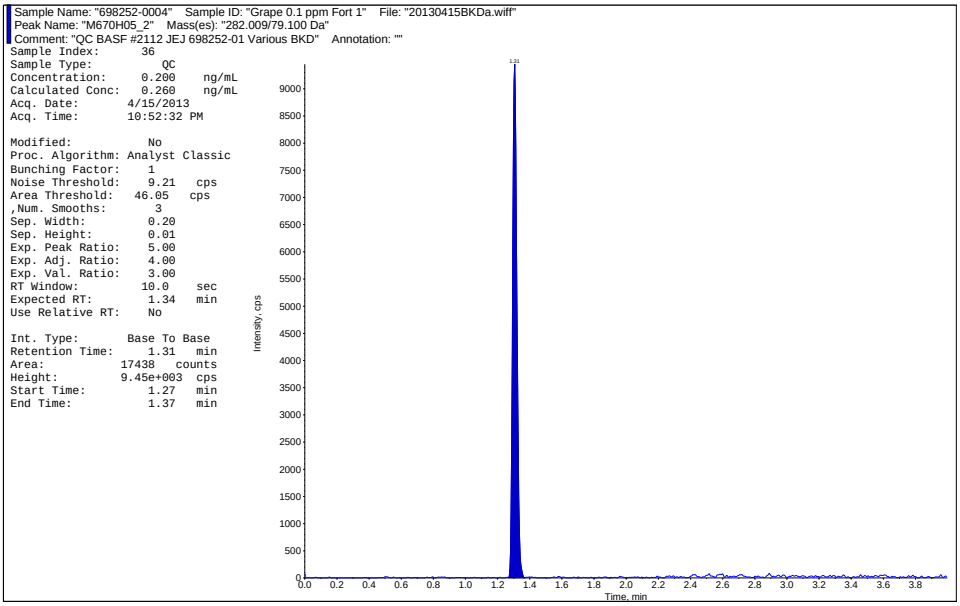
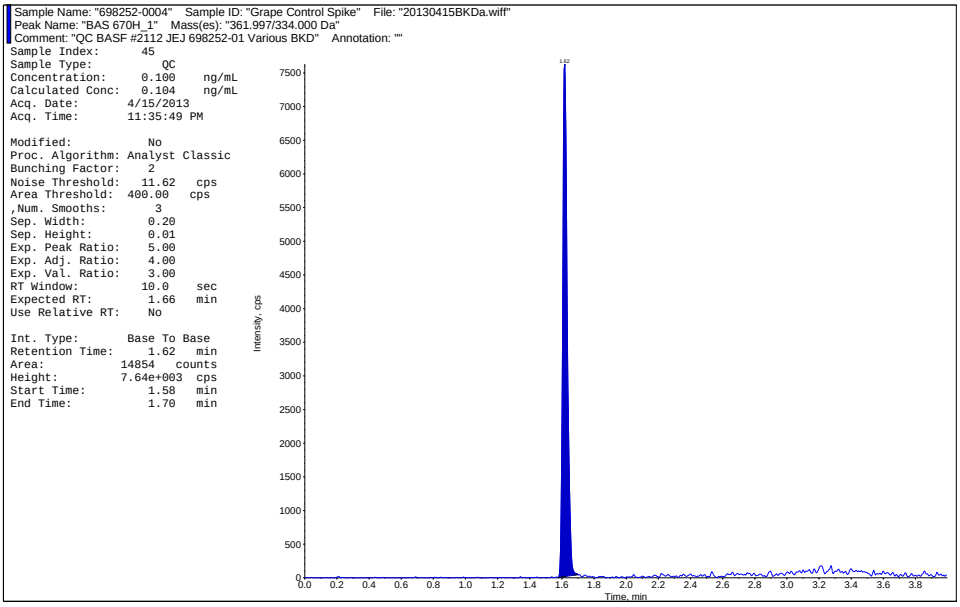


Figure 17. Chromatograms of control extract of grape fortified with each analyte at 0.1 ng/mL, Sample No. 698252-0004, from Master Sheet 698252-1



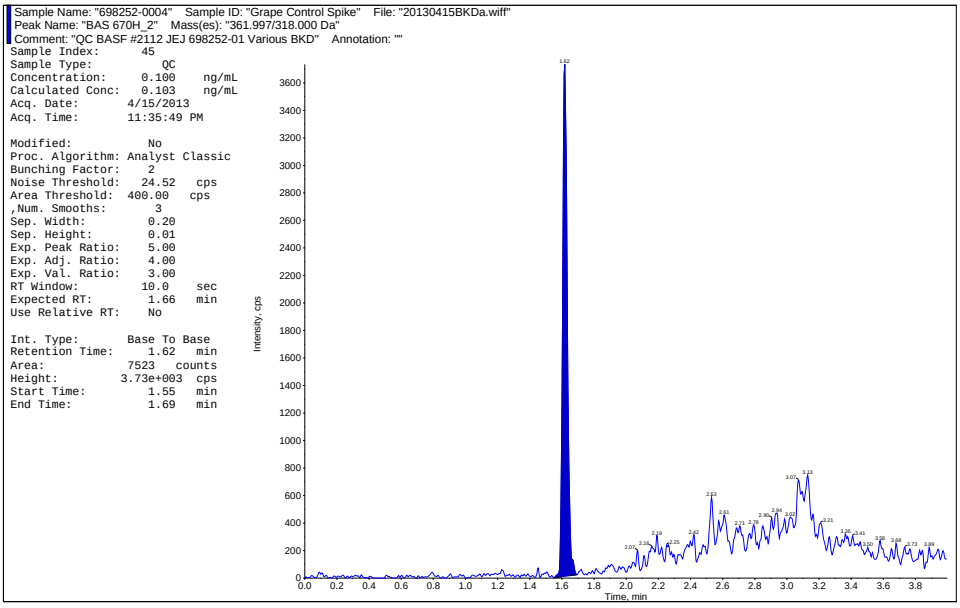
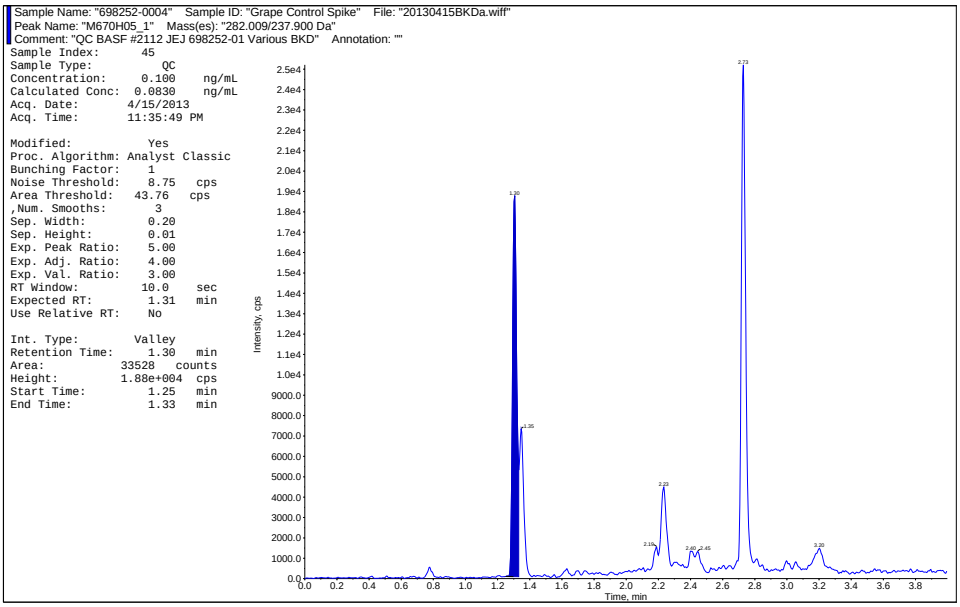


Figure Continued.

Control extract of Grape (fruit) fortified with M670H05 (Matrix Effects Test)



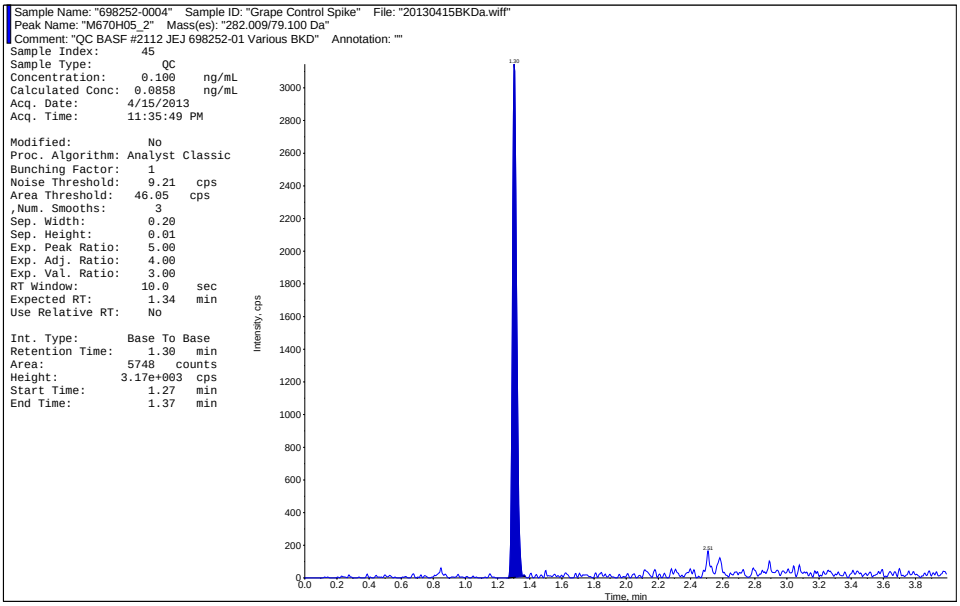
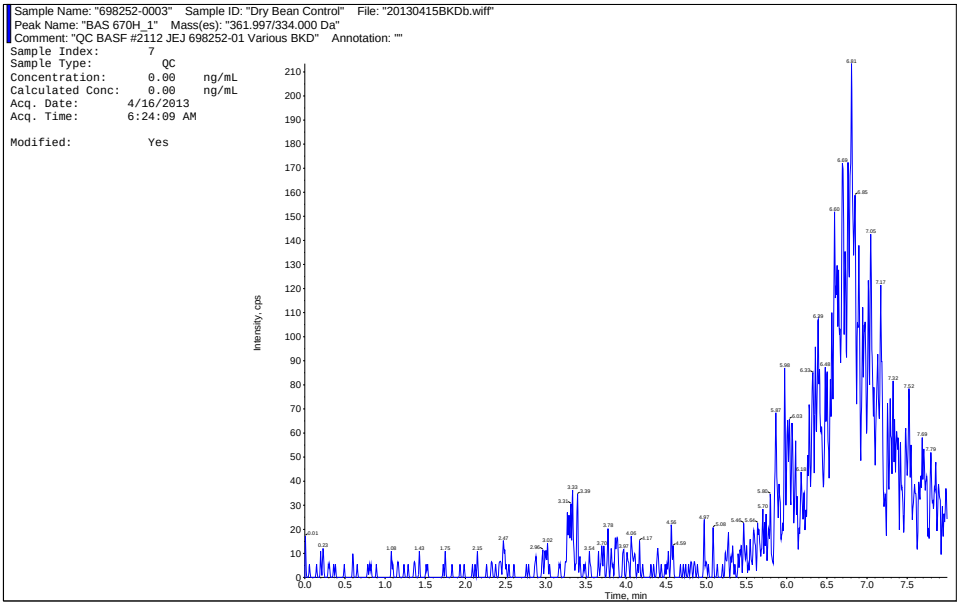


Figure 18. Chromatograms of a control bean seed sample, Sample No. 698252-0003, from Master Sheet 698252-1

Analyzed for Topramezone



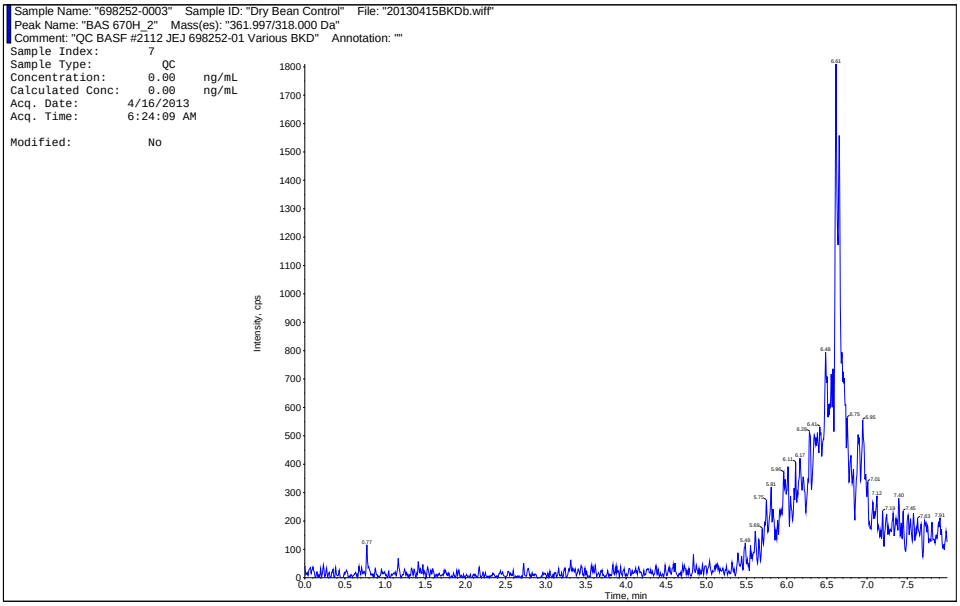
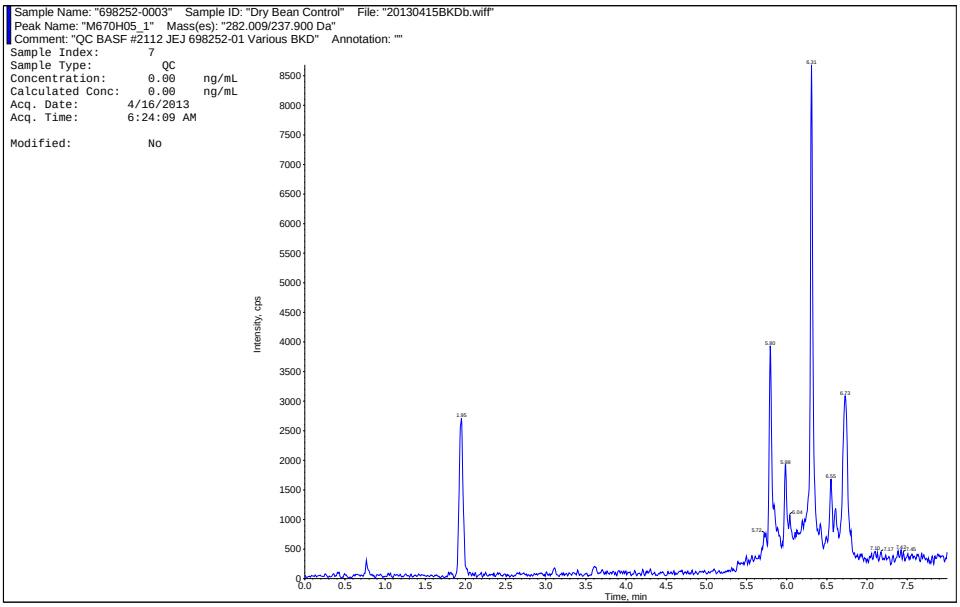


Figure Continued.

Control Bean seed sample, analyzed for M670H05



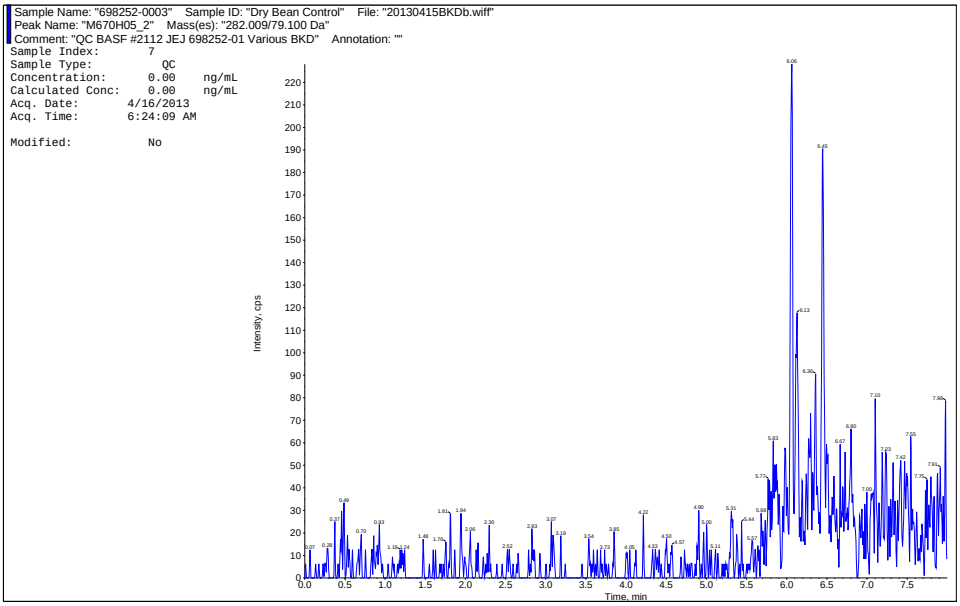
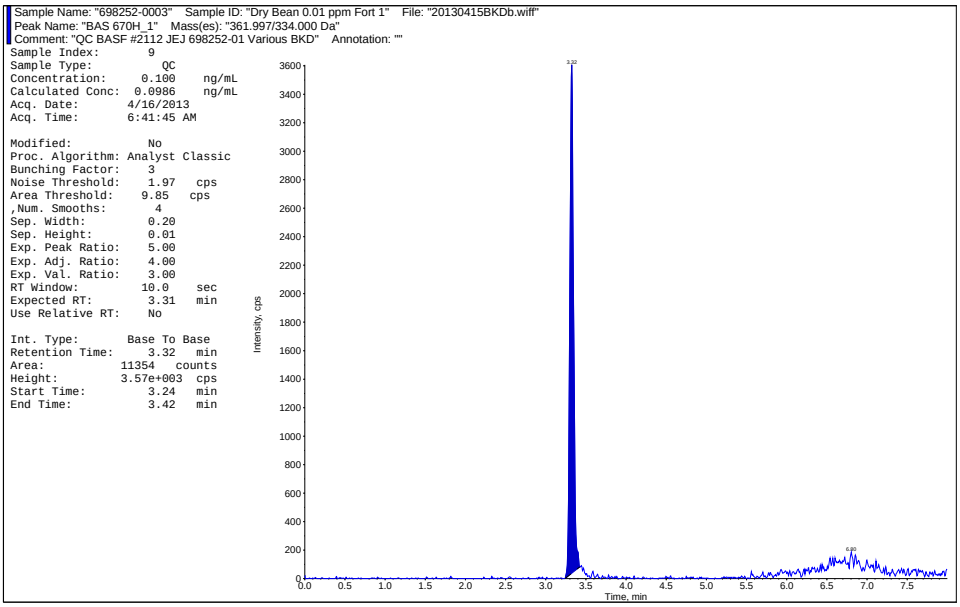


Figure 19. Chromatograms of a control bean seed sample fortified with each analyte at 0.01 ppm, Sample No. 698252-0003, from Master Sheet 698252-1



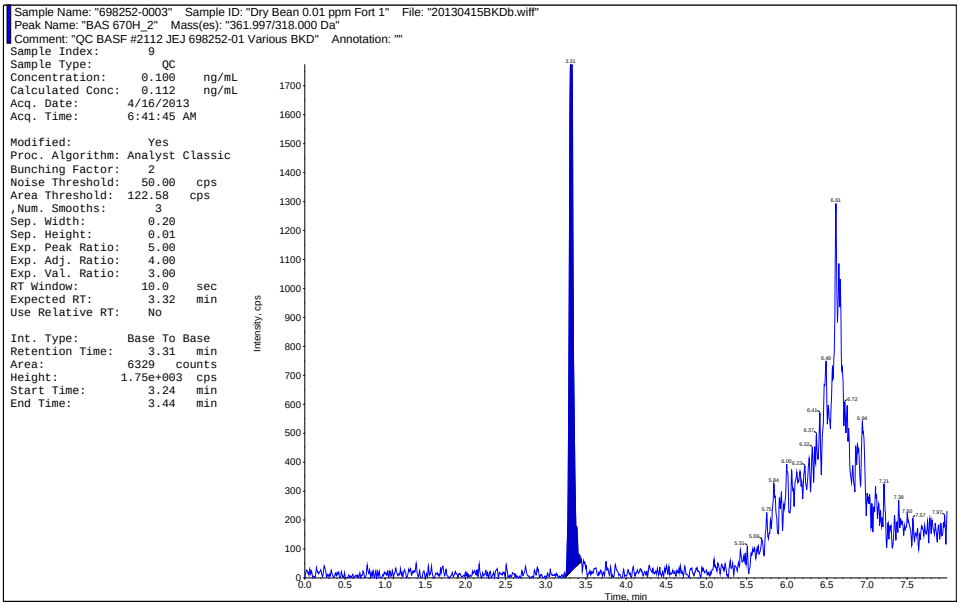
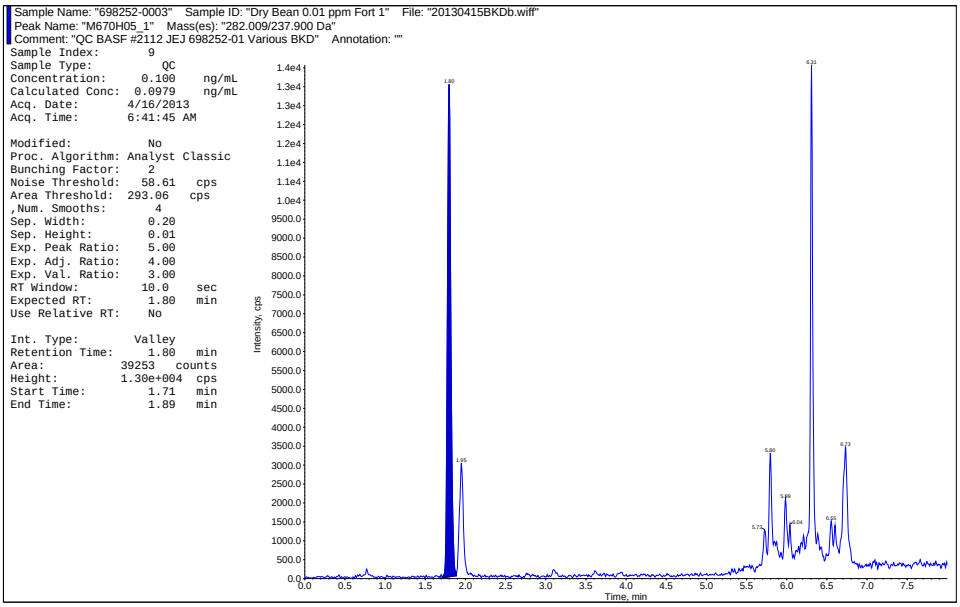


Figure Continued.

Low fortification (0.01 ppm), M670H05



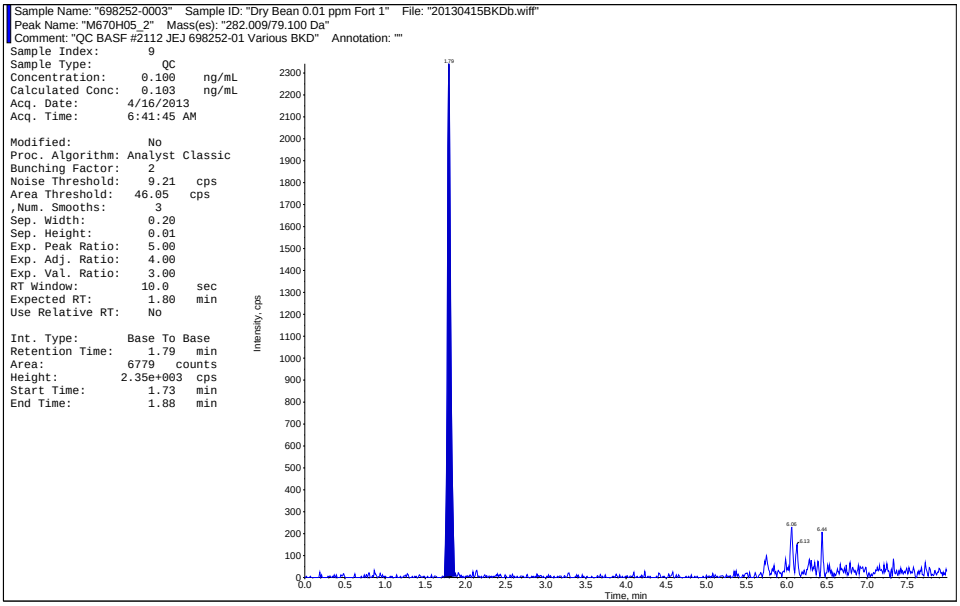
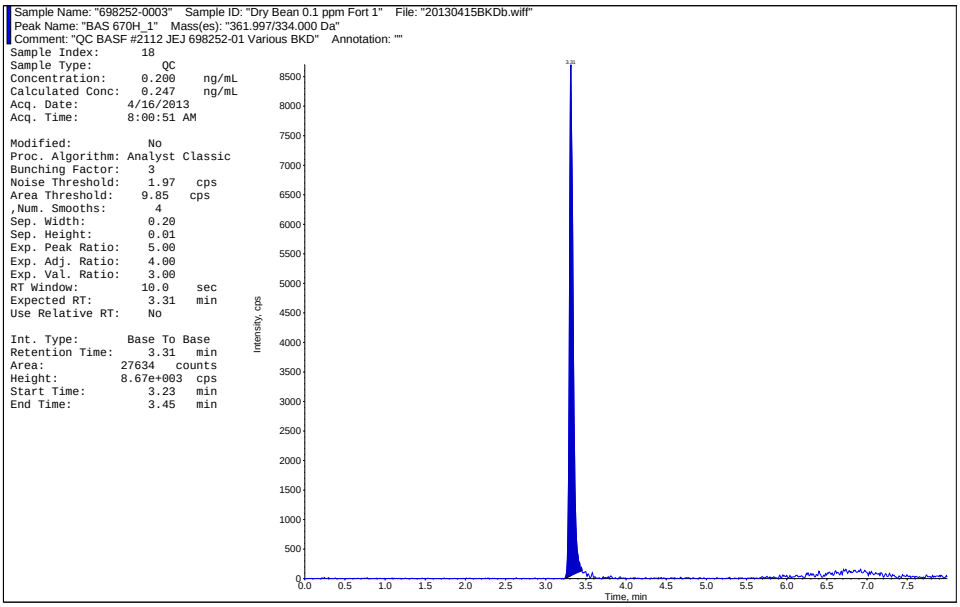


Figure 20. Chromatograms of a control bean seed sample fortified with each analyte at 0.1 ppm, Sample No. 698252-0003, from Master Sheet 698252-1



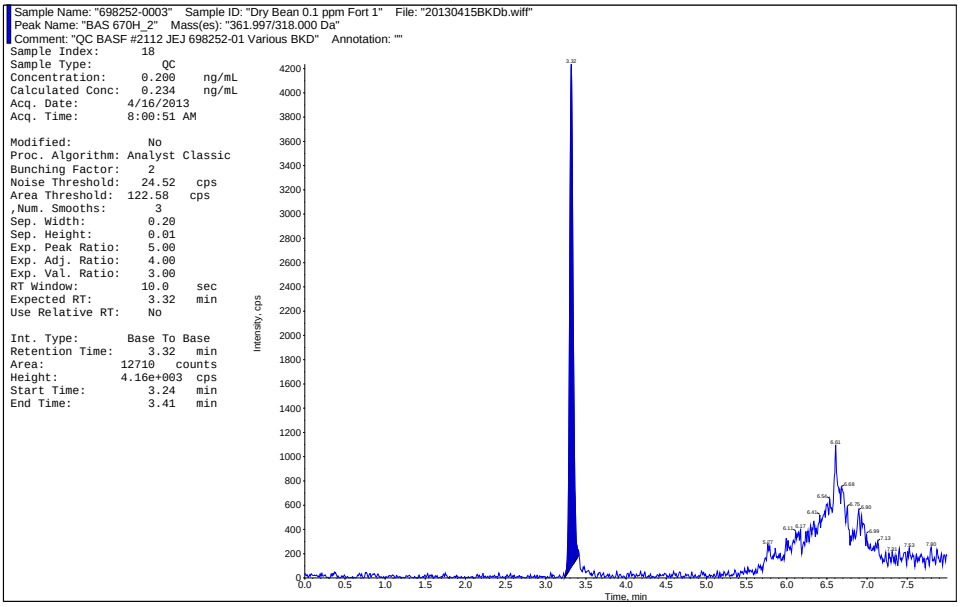
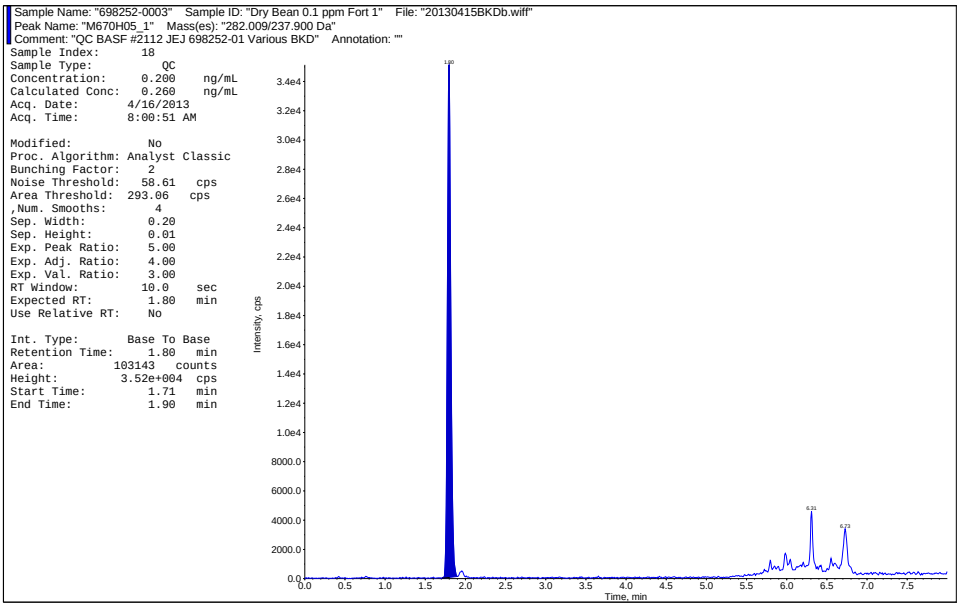


Figure Continued.

High fortification (0.1 ppm), M670H05



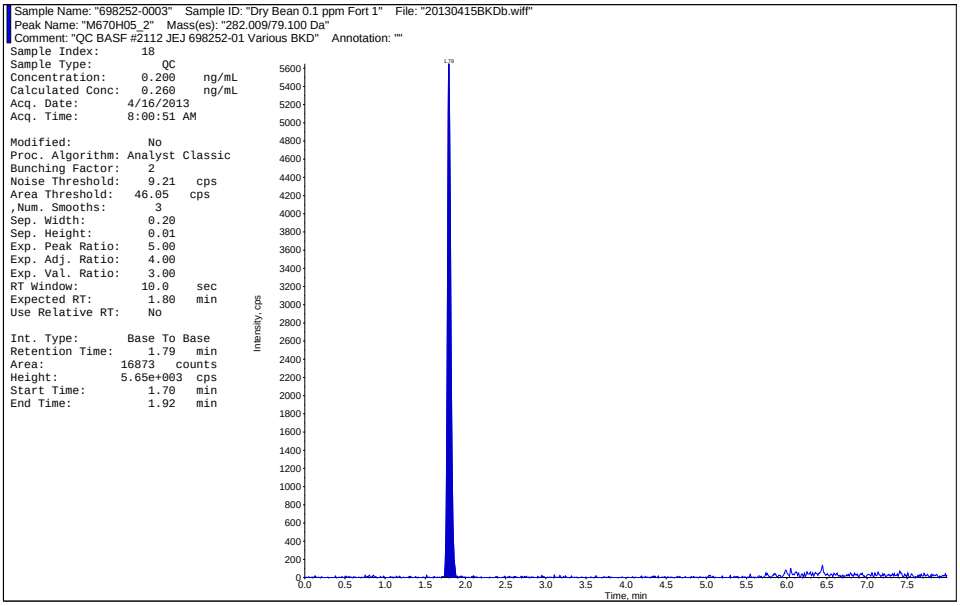
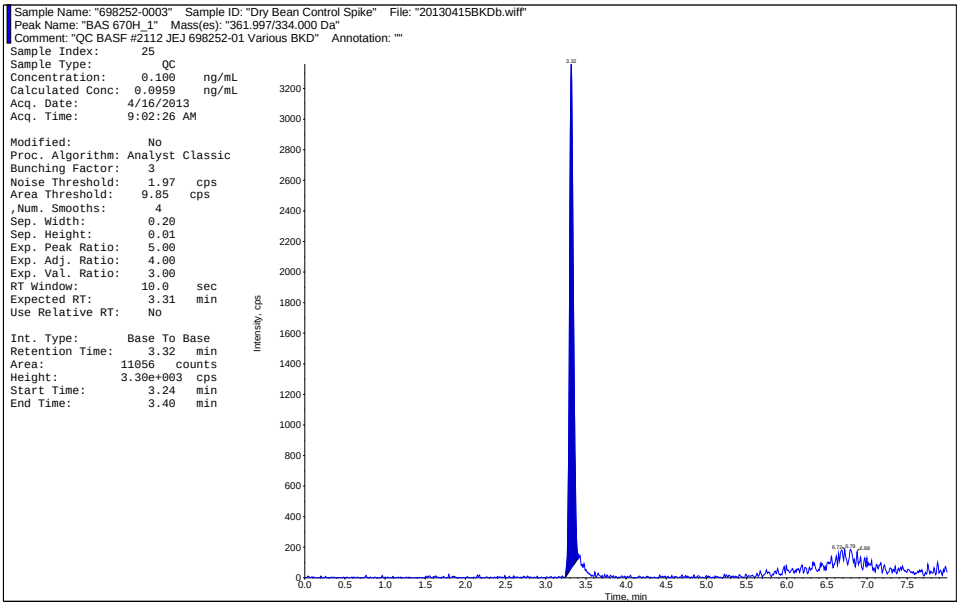


Figure 21. Chromatograms of control extract of bean seed fortified with each analyte at 0.1 ng/mL, Sample No. 698252-0003, from Master Sheet 698252-1



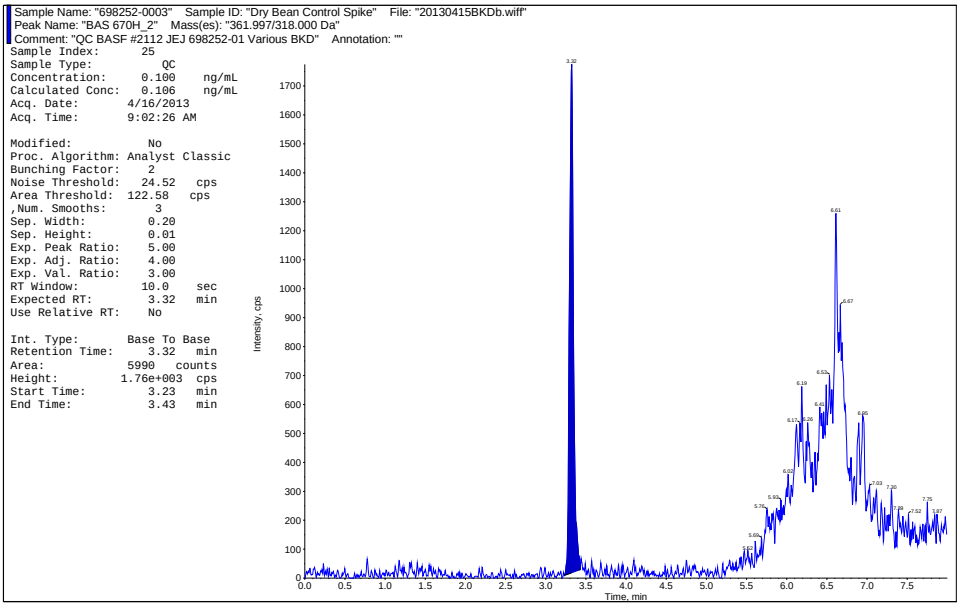
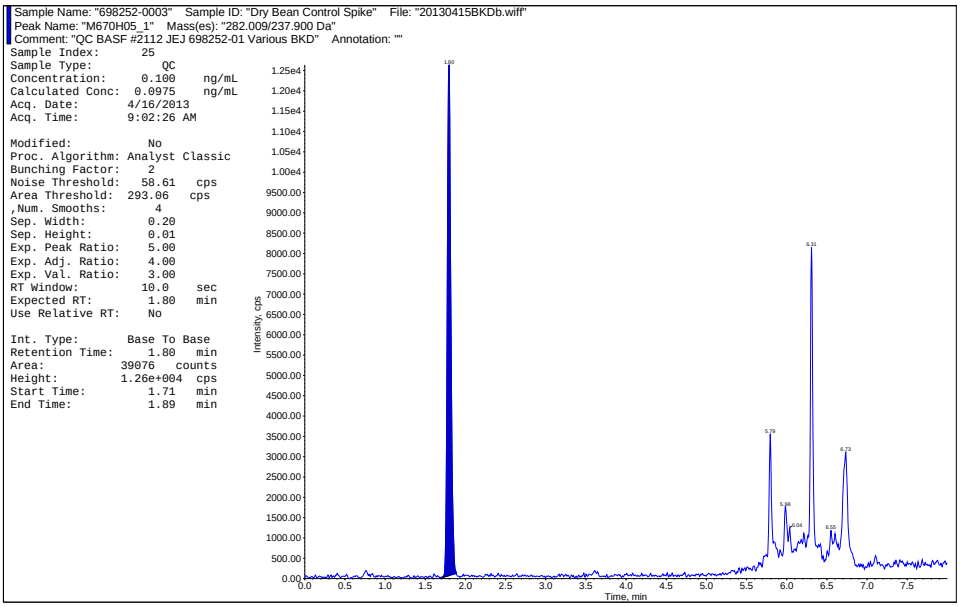


Figure Continued.

Control extract of Bean seed fortified with M670H05 (Matrix Effects Test)



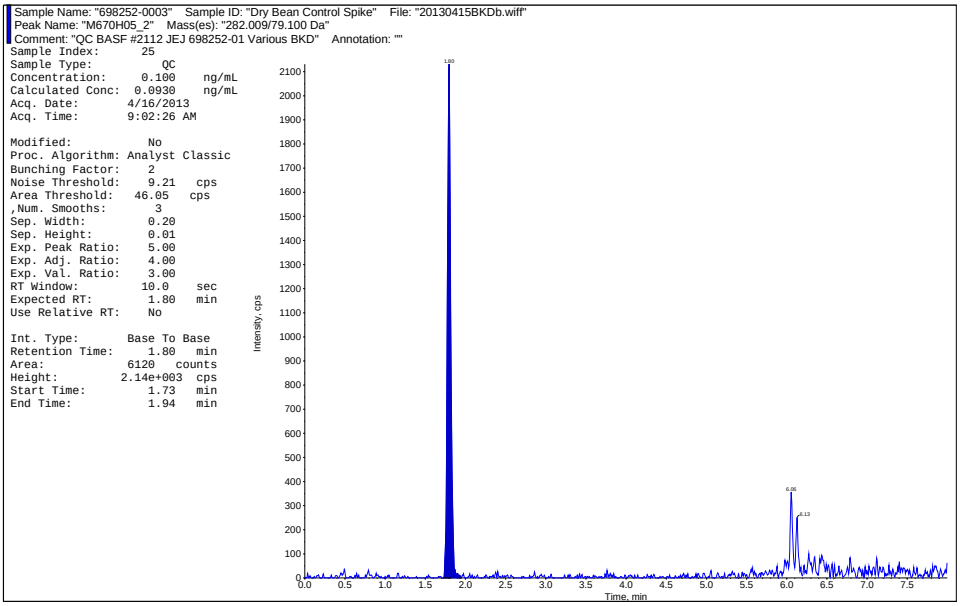
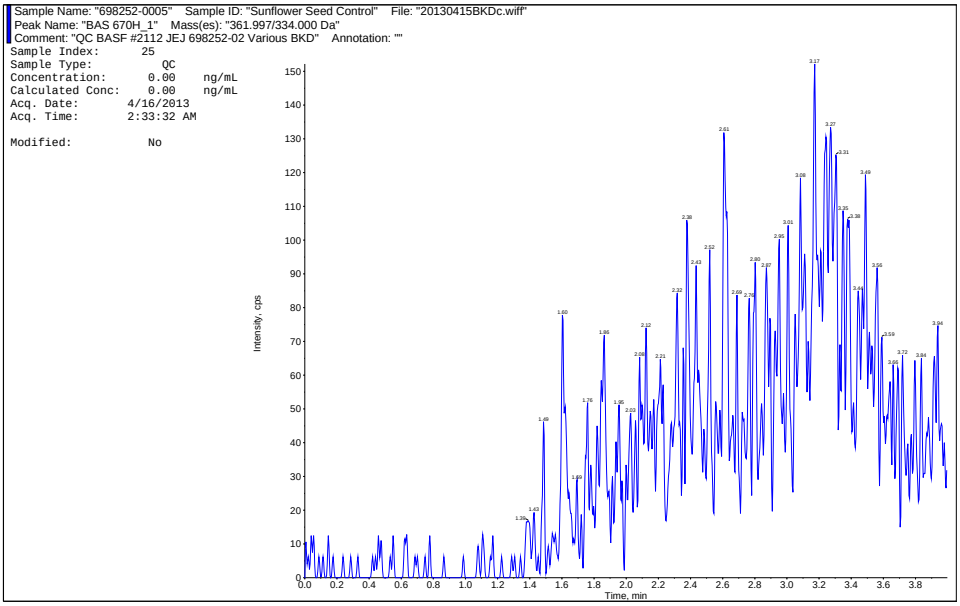


Figure 22. Chromatograms of a control sunflower seed sample, Sample No. 698252-0005, from Master Sheet 698252-2

Analyzed for Topramezone



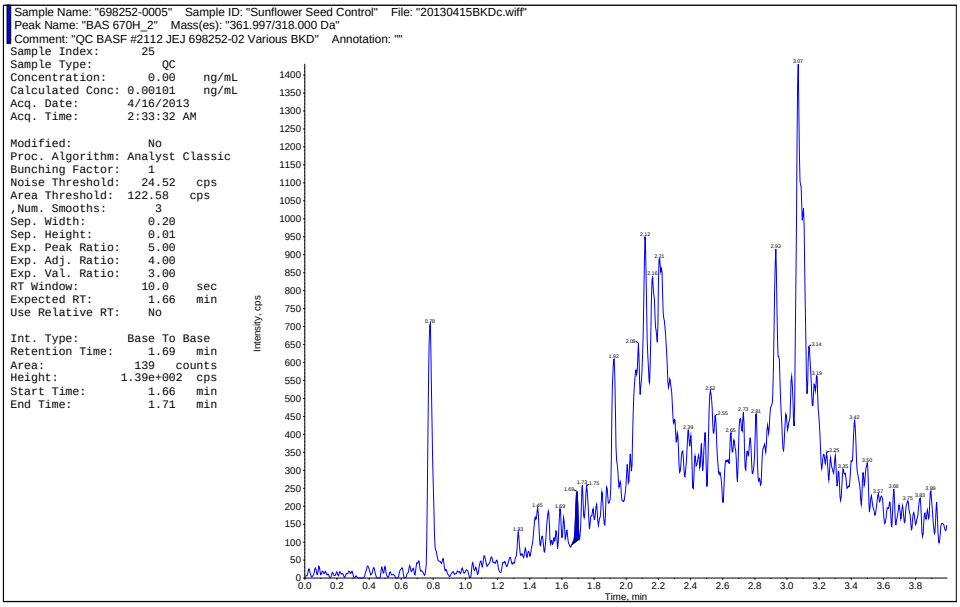
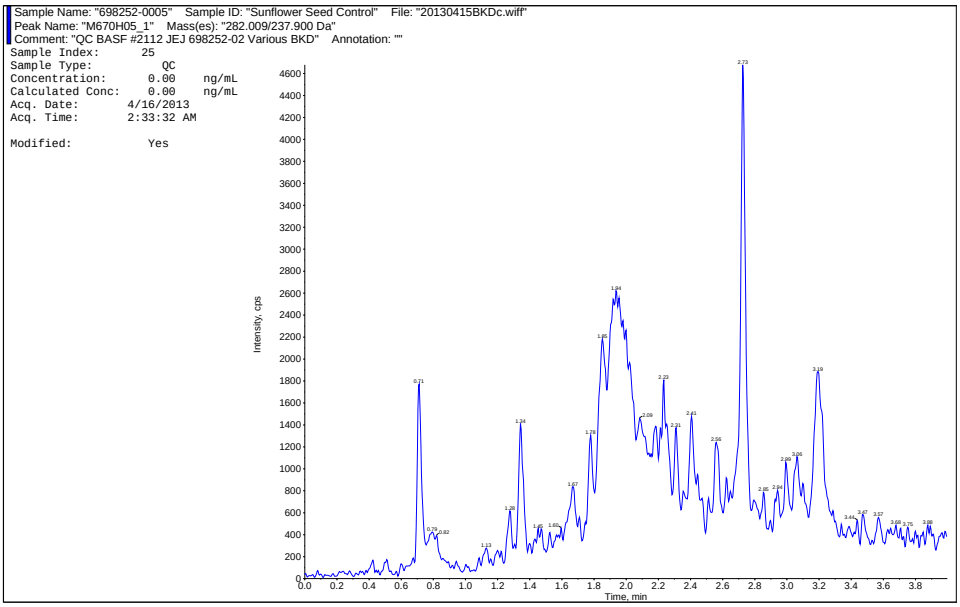


Figure Continued.

Control Sunflower seed sample, analyzed for M670H05



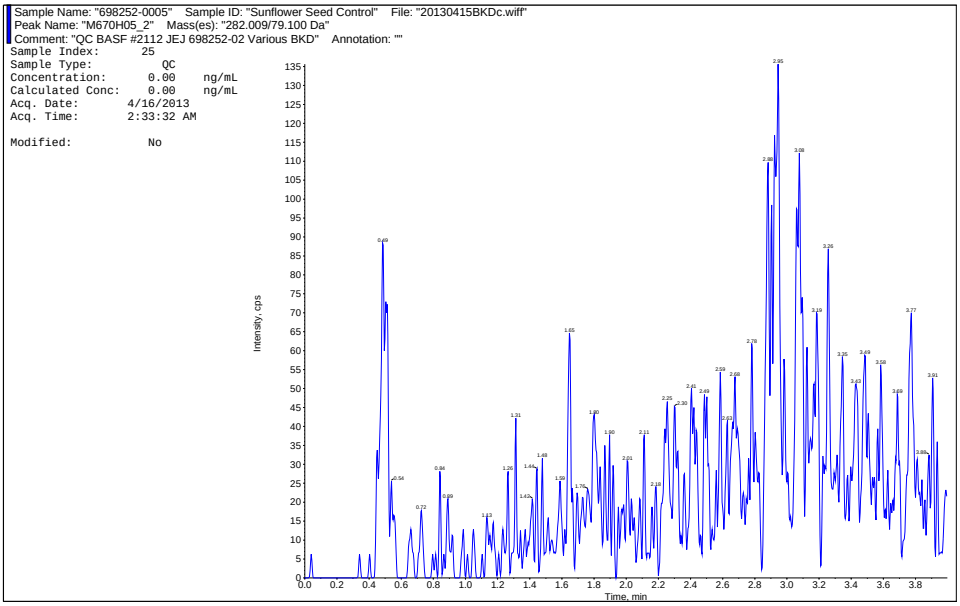
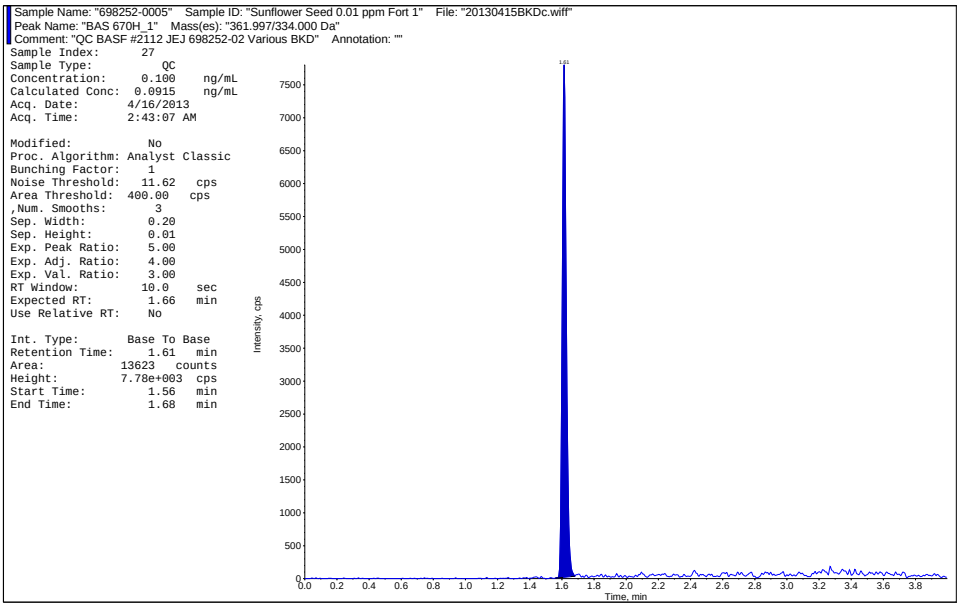


Figure 23. Chromatograms of a control sunflower seed sample fortified with each analyte at 0.01 ppm, Sample No. 698252-0005, from Master Sheet 698252-2



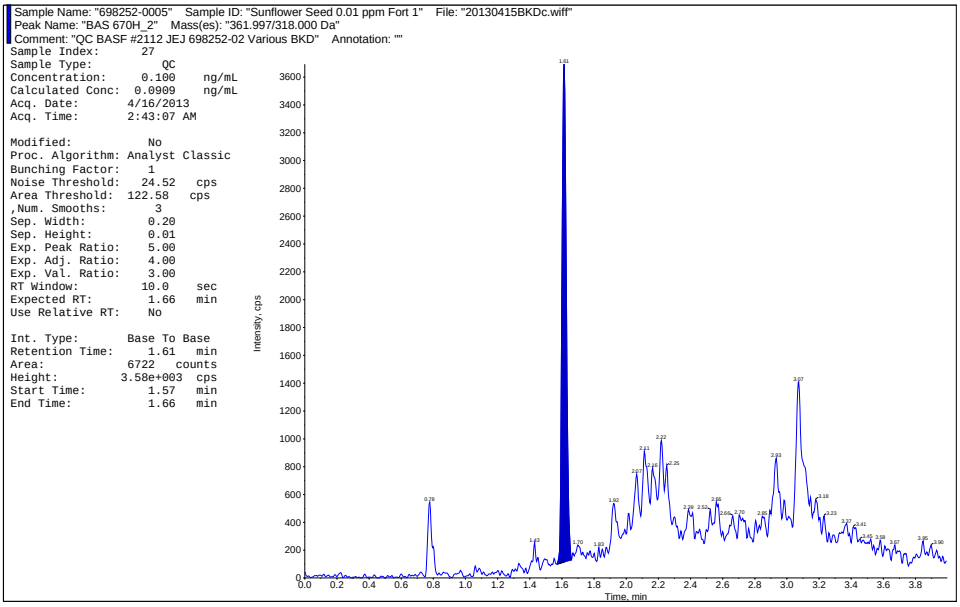
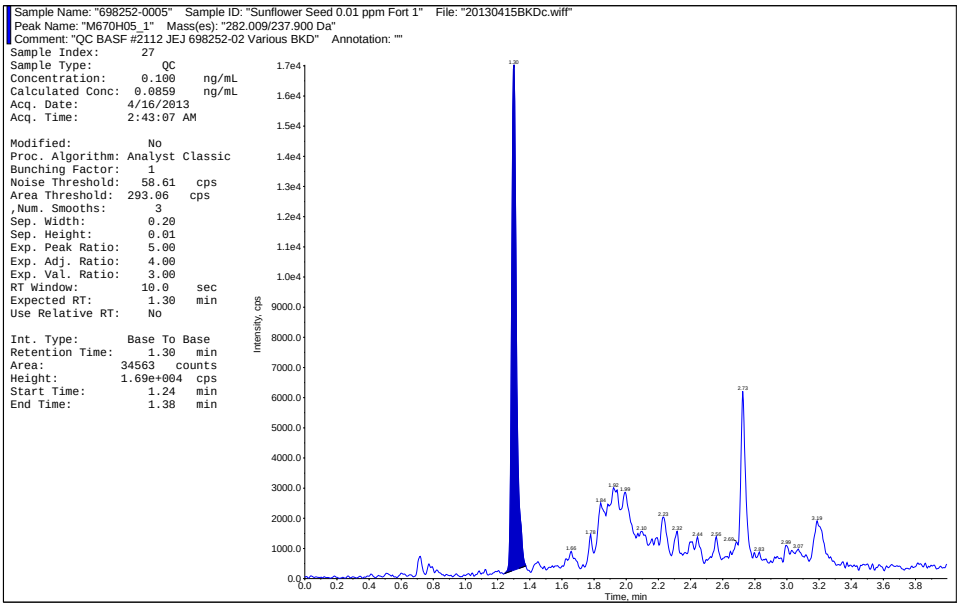


Figure Continued.

Low fortification (0.01 ppm), M670H05



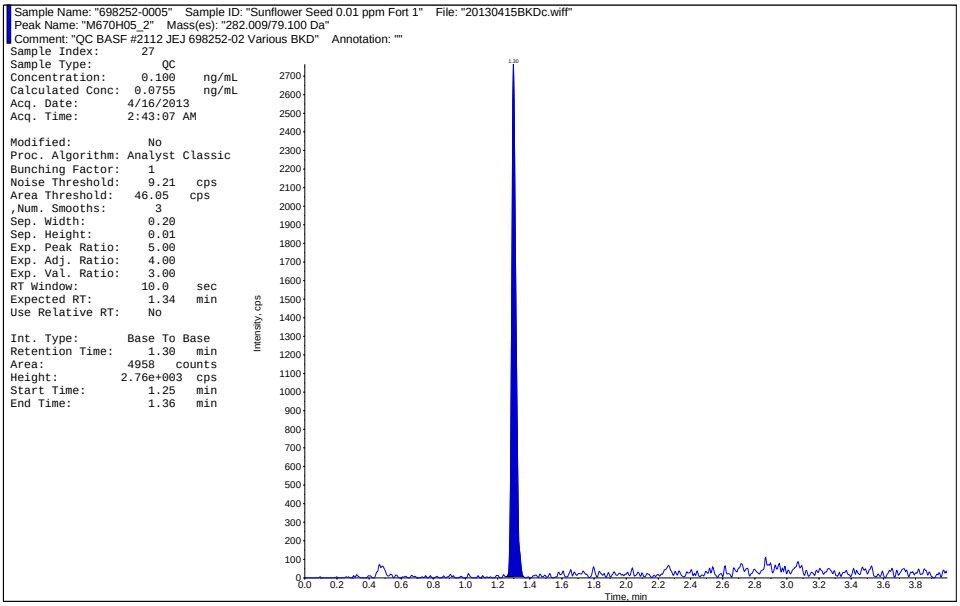
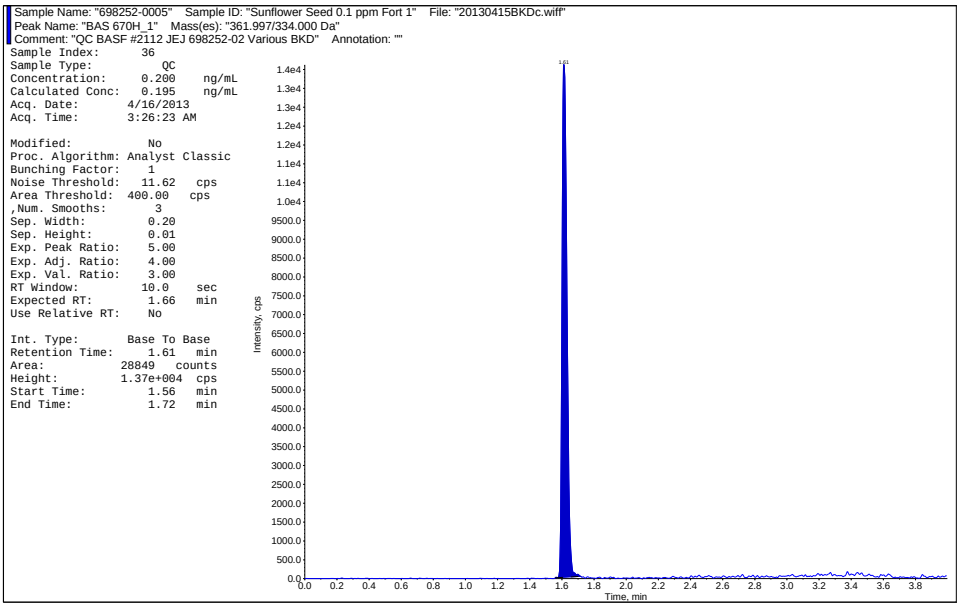


Figure 24. Chromatograms of a control sunflower seed sample fortified with each analyte at 0.1 ppm, Sample No. 698252-0005, from Master Sheet 698252-2



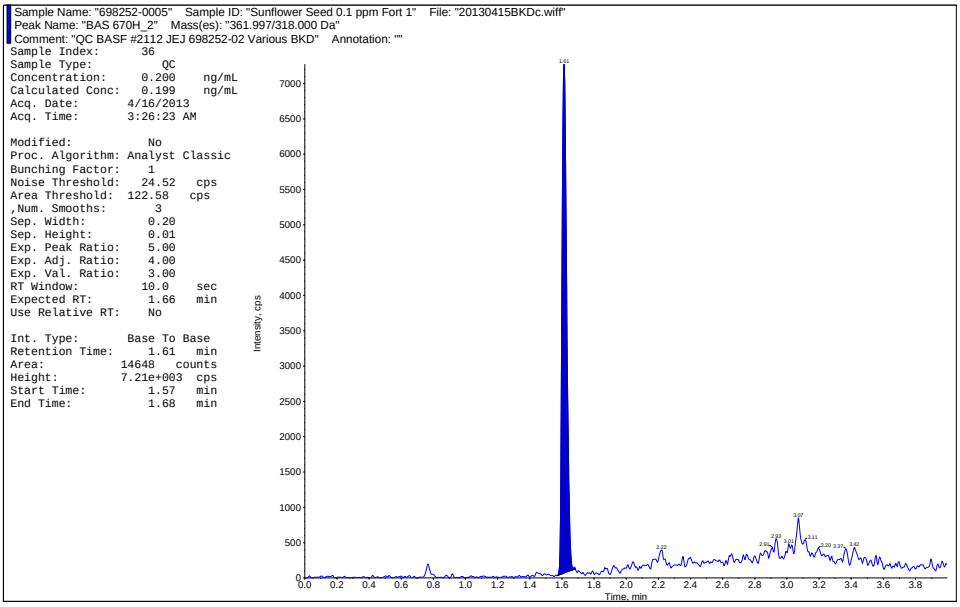
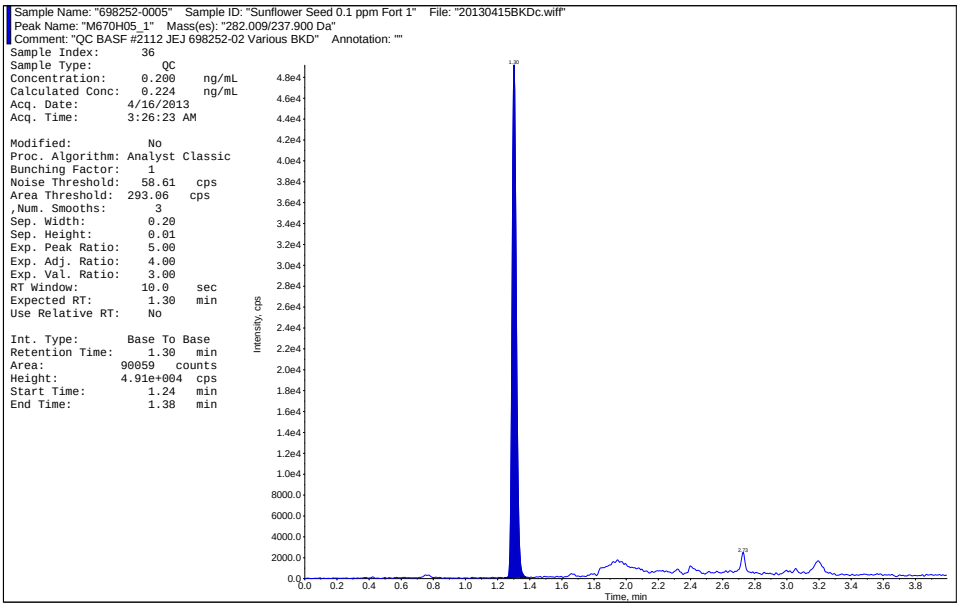


Figure Continued.

High fortification (0.1 ppm), M670H05



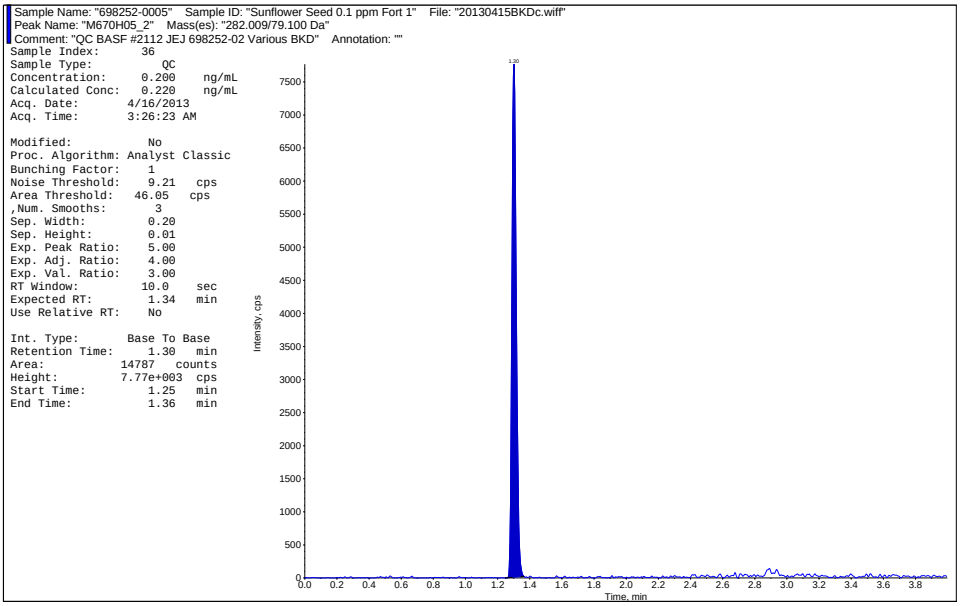
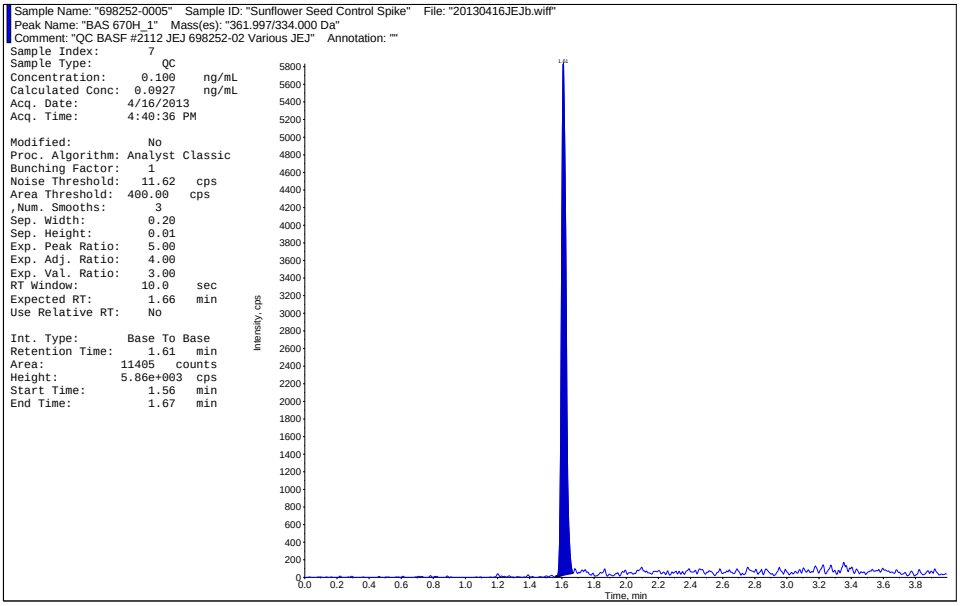


Figure 25. Chromatograms of control extract of sunflower seed fortified with each analyte at 0.1 ng/mL, Sample No. 698252-0005, from Master Sheet 698252-2



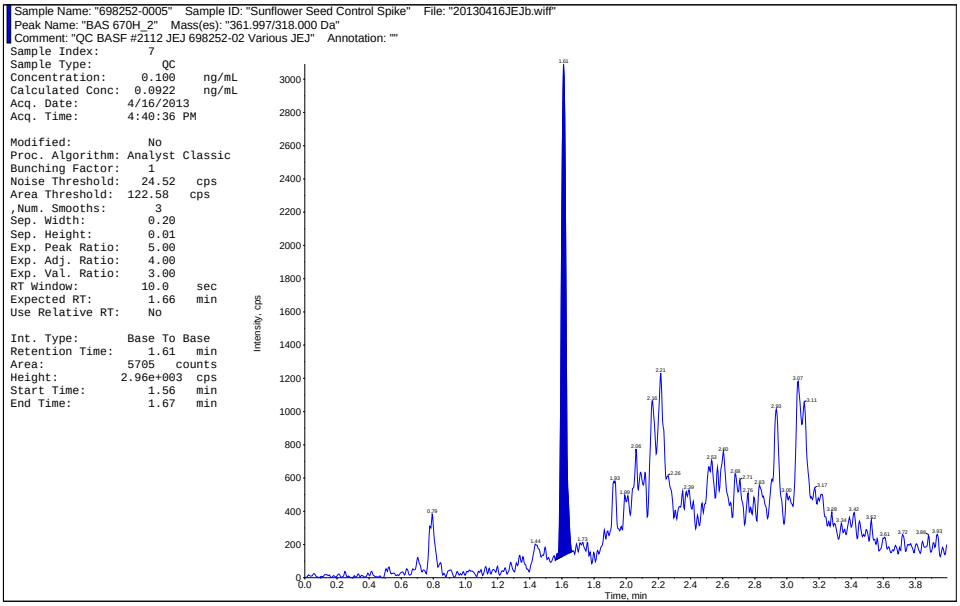
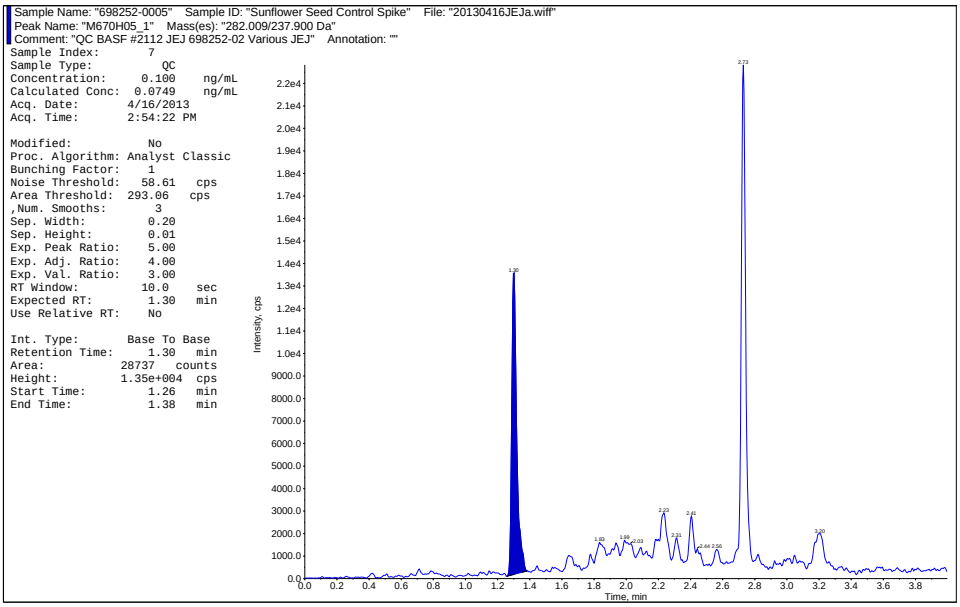
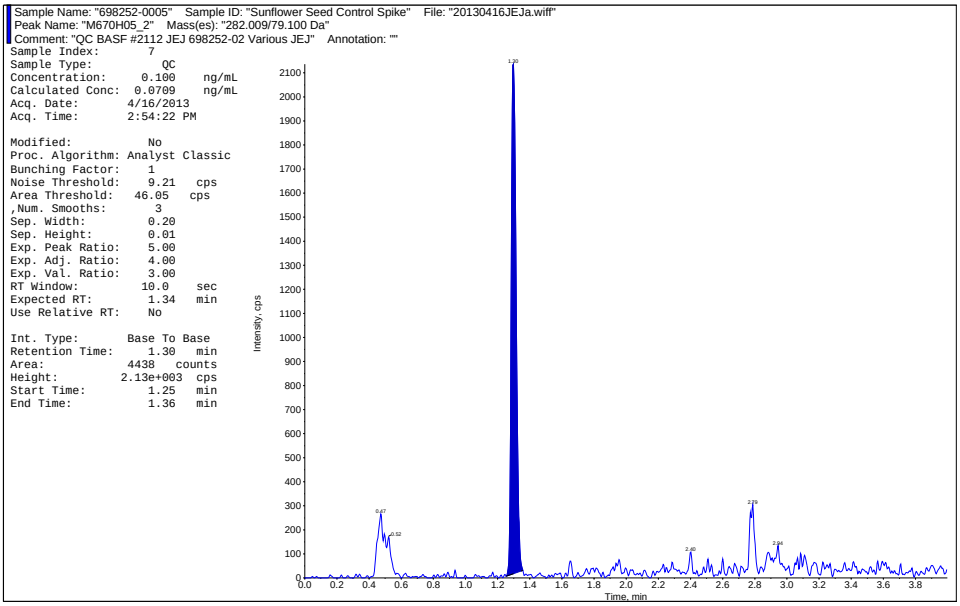


Figure Continued.

Control extract of sunflower seed fortified with M670H05 (Matrix Effects Test)





Appendix F. Example Standards Preparation and Dilution Data

Example Standards Preparation and Dilution Data

Typical analytical standards dilution and use records (used for all sets)

Standard Number	Analyte [1]	Standard (Lot # used) [2]	Amount Weighed / Volume	Final Dilution Vol. (mL)	Final Conc.	Solvent[3]	Prep. Date	Expiration Date
Stock solutions								
ERS13-330	1	01882-216	5.0 mg	5	1 mg/mL	ACN	3/6/13	6/6/13
ERS13-374	2	L76-134	10 mg	10	1 mg/mL	MeoH	4/3/13	5/3/13
Serial dilutions								
ERS13-375	1, 2	ERS13-330 ERS13-374	0.25 mL 0.25 mL	25	10 µg/mL*	ACN	4/3/13	5/3/13
ERS13-376	1, 2	ERS13-375	5 mL	50	1 µg/mL	ACN	4/3/13	5/3/13
ERS13-377	1, 2	ERS13-376	5 mL	50	0.1 µg/mL	ACN	4/3/13	5/3/13
Calibration								
ERS13-378	1, 2	ERS13-377	1 mL	50	2 ng/mL	Mixture	4/3/13	5/3/13
ERS13-379	1, 2	ERS13-378	20 mL	100	0.4 ng/mL	Mixture	4/3/13	5/3/13
ERS13-380	1, 2	ERS13-379	25 mL	50	0.2 ng/mL	Mixture	4/3/13	5/3/13
ERS13-381	1, 2	ERS13-379	12.5 mL	50	0.1 ng/mL	Mixture	4/3/13	5/3/13
ERS13-382	1, 2	ERS13-379	5 mL	50	0.04 ng/mL	Mixture	4/3/13	5/3/13
ERS13-383	1, 2	ERS13-379	2.5 mL	50	0.02 ng/mL	Mixture	4/3/13	5/3/13

1. 1 = topramezone, 2 = metabolite M670H05,
2. Refer to "Appendix A" for COA of lot number used.
3. ACN = acetonitrile. MeOH = methanol "Mixture" = 4mM ammonium formate in water, pH 10.
   *From this point forward in the serial dilutions and calibration standards, the concentration stated is for "each analyte."

The table shown below depicts the fortification scheme used for analysis.

Sample Data	Fortification data						Final Volume (mL)
Description	Spike Level (ppm)	Sample weight (g)	Analyte	Vol. of Standard Used	Standard Conc.	Standard Number	
Control	Control	5.0	None	NA	NA	NA	2.5
Reagent Blank	0.00	NA	None	NA	NA	NA	2.5
0.01 ppm Fort	0.01	5.0	Each analyte	0.5 mL	0.1 µg/mL	ERS13-377	2.5
0.10 ppm Fort	0.1	5.0	Each analyte	0.5 mL	1 µg/mL	ERS13-376	12.5