

TITLE: Famoxadone + Cymoxanil: Magnitude of Residue on Hops

PROJECT No.: 07796

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Laboratory Identification Number

07796.00-DPR01

Field Identification Numbers

07796.00-WA21
07796.00-OR29
07796.00-ID17

GLP COMPLIANCE STATEMENT

Protocol No. 07796

Study Title

Famoxadone + Cymoxanil: Magnitude of Residue on Hops

The Analytical Phase including residue analysis of cymoxanil and famoxadone in hops was conducted and reported in compliance with U.S. EPA FIFRA (40 CFR Part 160) Good Laboratory Practice Standards, which are consistent with the OECD Principles of Good Laboratory Practice (ENV/MC/CHEM(98)17).

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RESIDUE STUDY QUALITY ASSURANCE REPORT SHEET**Protocol No. 07796****Study Title**

Famoxadone + Cymoxanil: Magnitude of Residue on Hops

<i>Audit Dates</i>	<i>Dates Findings Reported to Study Director</i>	<i>Dates Findings Reported to Management</i>
13, 20 Mar 2001	30 Mar 2001	30 Mar 2001
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E. Maria Lopez
Quality Assurance Auditor

Date

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LOCATION OF RAW DATA

When the final analytical summary report is completed and sent to the Study Director, all original raw data including a “true copy” of the final analytical summary report will be archived at:

E. I. du Pont de Nemours and Company
DuPont Crop Protection
Global Technology Division
Stine-Haskell Research Center
Newark, Delaware 19714-0030

SUMMARY

1. Objective/Introduction

At the request of IR-4, Dupont Stine Haskell Research Center has assayed **Hops** for residues of **famoxadone and cymoxanil** to provide residue chemistry data to support pesticide tolerances. Samples were analyzed by the following procedures described in DuPont Report No. AMR 3705-95, Revision No. 2, "Analytical Method for the Determination of DPX-JE874 and Cymoxanil Residues in Various Matrices". A detailed method summary and modifications to the method, including a description of analysis by liquid chromatography using a single quadrupole mass spectrometer (LC/MS) are included in this report.

The study was conducted according to IR-4 National Pesticide Clearance Protocol PR No.: 07796.

2. Sample Inventory/History

This study consisted of residue analysis of samples from the following field trials:

Field ID No. 07796.00-WA21, 07796.00-OR29, and 07796.00-ID17

Upon receipt of the samples in the laboratory, an inspection indicated that the samples were frozen, the sample bags were intact and that the numbers on the samples matched the Residue Chain of Custody Form. The unique sample numbers assigned in the field were used throughout the analysis.

Samples were homogenized using a Hobart Processor as they arrived from the field on the following dates:

<u>Field ID No.</u>	<u>Processing Date</u>
07796.00-WA21	10 Oct 2000
07796.00-OR29	18 Sep 2000
07796.00-ID17	09 Oct 2000

Once samples were homogenized they were stored frozen until analysis at approximately -20°C until analysis.

3. Materials and Methods

Reference Analytical Standard

The reference analytical standards of cymoxanil (Lot No. IN-T3217-151, purity 99.6%) and famoxadone (Lot No. IN-JE874-313, purity 99.4%) were provided by DuPont. Both standards were received on 20 July 2000. The famoxadone standard had an expiration date of 09 April 2001, but was re-characterized and the purity was remained constant at 99.4%. The expiration date was extended to 16 April 2004. Cymoxanil has an expiration date of 13 October 2001, the experimental portion of the study was completed before this date. GLP-Characterization data are archived by DuPont Crop Protection, E. I. du Pont de Nemours and Company, Newark, DE.

Sample Analysis

Method Principle

Hop samples were extracted and analyzed for famoxadone and cymoxanil following procedures described in DuPont Report No. AMR 3705-95, Revision No. 2, "Analytical Method for the Determination of DPX-JE874 and Cymoxanil Residues in Various Matrices." One major change was that both cymoxanil and famoxadone were analyzed by LC/MS. In the original method, cymoxanil was analyzed by LC/UV and famoxadone by GC/NPD. See the method modification section for a complete list of changes. Bridging has been performed to demonstrate equivalence between the detection methods. Hops are a difficult matrix to analyze, only 2 ml of the extract was loaded onto the florisil column to prevent signal suppression. The LOQ for famoxadone was 0.5 ppm.

Ground samples were weighed into pint extraction jars, deionized water was added, and samples were allowed to re-hydrate prior to extraction. Acetonitrile was added and samples were ground with a Tissumizer[®]. Macerated plant matrix was allowed to settle, liquid extracts were filtered and collected in mixing cylinders containing sodium chloride. The mixing cylinders were capped, shaken, and inverted to allow the sodium chloride to dissolve. The cylinders were then allowed to stand while the acetonitrile (upper layer) and water phases separated. Separate acetonitrile aliquots were taken for cymoxanil and famoxadone analysis.

For analysis of cymoxanil, the acetonitrile aliquot was back extracted with hexane, concentrated, diluted with water and passed through a conditioned strong anion exchange (SAX) solid phase extraction (SPE) cartridge stacked on top of a conditioned carbon black SPE cartridge. Cymoxanil passed through the SAX cartridge and was retained on the

carbon black cartridge. Cymoxanil was selectively eluted, the cartridge eluate was concentrated, and cymoxanil residues were exchanged into hexane/ethyl acetate. The resulting solutions were passed through silica SPE cartridges. Cymoxanil was retained, selectively eluted, and cartridge eluates were concentrated and exchanged into methanol/water adjusted to pH 3. Samples were filtered and analyzed by LC/MS.

For analysis of famoxadone, the acetonitrile aliquot was back extracted with hexane, the acetonitrile fraction was concentrated to approximately 2 mL and carefully taken to dryness. The residue was dissolved in 10% ethyl ether/90% hexane (v/v) and passed through a glass chromatography column packed with a layer of sodium sulfate, Florisil, and sodium sulfate. Each column was washed with additional 10% ethyl acetate/90% hexane (v/v). The eluate was concentrated, carefully taken to dryness, reconstituted in acetonitrile and water and analyzed by LC/MS.

Extraction Procedures

Five-gram samples of homogenized hop samples were weighed into 1-pt extraction jars. Samples were fortified with the appropriate level of cymoxanil or famoxadone, if necessary. Sixty milliliters of deionized water was added. The samples were allowed to soak for at least 10 minutes before adding 120 mL of acetonitrile. Samples were homogenized for 3 minutes at medium speed. After solids settled, the liquid extract was poured through fluted filter paper and collected in 250-mL mixing cylinders containing 30 g of sodium chloride. The mixing cylinders were capped and shaken for approximately 1 minute to dissolve the salt. After shaking, the mixing cylinders were allowed to stand for an additional 15-30 minutes to allow the acetonitrile and water phases to separate. A 7.2-mL acetonitrile aliquot (upper layer, equivalent to 1.2 g of sample) was removed for cymoxanil analysis, and a 2-mL aliquot was removed for famoxadone analysis. After hexane extraction, the cymoxanil aliquot was concentrated to 2 mL and diluted with 11 mL of Milli-Q[®] water in preparation for cartridge cleanup. The famoxadone aliquot was evaporated to dryness. Five milliliters of 10% ethyl ether/90% hexane (v/v) was added to redissolve residue in preparation for column chromatography cleanup.

Clean-up Procedures

Cymoxanil

The cymoxanil sample prepared above was passed through a conditioned 1-g SAX SPE cartridge stacked on top of a conditioned 500-mg carbon black SPE cartridge. Cymoxanil passed through the SAX cartridge and was retained on the carbon black cartridge. The SAX cartridge was discarded. The carbon black cartridge was washed in turn with 5 mL of Milli-Q[®] water and 1 mL of methanol. The cartridge was dried using vacuum and eluted with 90% methylene

chloride/10% methanol (v/v). The resulting eluate was concentrated, exchanged into ethyl acetate, diluted with hexane and passed through a conditioned 500-mg silica SPE cartridge. Cymoxanil was retained.

The silica cartridge was washed with 90% hexane/10% ethyl acetate (v/v), and then washed with 80% hexane/20% ethyl acetate. Cymoxanil was eluted with 60% hexane/40% ethyl acetate containing a small volume of methanol (v/v). The resulting eluate was concentrate, transferred into methanol, and diluted with water adjusted to pH 3. The resulting solution was filtered through a 0.45- μ m filter prior to LC/MS analysis.

Famoxadone

The famoxadone sample prepared above was passed through a 12 mL bond elute reservoir containing 1 cm of sodium sulfate, 5 cm of activated Florisil, and 1 cm of sodium sulfate. Residue remaining in the flasks used to concentrate samples was transferred to the column with 2-mL portions of 90% hexane/10% ethyl ether (v/v). The column was rinsed with an additional 15 mL of 90% hexane/10% ethyl ether (v/v). Famoxadone was eluted with 50 mL of 80% hexane/20% ethyl acetate (v/v). The eluates were evaporated to dryness, reconstituted in acetonitrile and water and analyzed by LC/MS.

Method modifications

The major change is that cymoxanil and famoxadone were analyzed by LC/MS. In the original method, cymoxanil was analyzed by LC/UV and famoxadone by GC/NPD.

Because of the bulk of hops, 5 grams of sample rather than 20 grams were extracted. For cymoxanil, the remainder of the method was the same except that the samples were diluted with water adjusted to pH 3 with acetic acid in the final step before analysis because phosphate buffer could not be used for LC/MS.

For famoxadone there were additional changes mostly related to switching the detection from GC to MS. Standards were prepared in acetonitrile rather than ethyl acetate. Only 2 ml of extract rather than 60ml was loaded onto the florisil column because of interferences. This raised the LOQ to 0.5 ppm instead of 0.02 ppm. Further explanation for this is given in the Results and Discussion Section. Finally, samples were diluted with water adjusted to pH 3 with acetic acid in the final step before analysis because ethyl acetate is not a suitable solvent for reverse phase LC/MS.

*Chromatography*Analysis

Hop samples were analyzed for both cymoxanil (DPX-T3217) and famoxadone (DPX-JE874) by HPLC with a single quadrupole mass spectrometer, (LC/MS).

Typical equipment operating conditions were as follows:

Instrument:	Agilent 1100 Series LC/MSD
Injection Volume:	40 μ L
Column I:	Agilent Zorbax [®] XDB-C8 (15.0 cm x 4.6 mm i.d., 5 μ)
Column Temperature:	30°C
Mobile Phases:	A = 0.01 M Acetic Acid C = Acetonitrile
Detector Setting:	
Ionization	electrospray
Polarity	negative
Mode	Selected ion monitoring
Mass/charge	197 (7-14 min) M-1 cymoxanil 373 (14-20 minutes) M-1 famoxadone

Column Gradient	Time (min)	% A	% C	Flow (mL/min)
	0	80	20	1.0
	5	80	20	1.0
	15	20	80	1.0
	19.99	20	80	1.0
	20	80	20	1.0
	26	80	20	1.0

A 100- μ g/mL standard solution was prepared by dissolving the cymoxanil and famoxadone reference standards in acetonitrile. A 1.0- μ g/mL intermediate standard solution was prepared by diluting the standard solution with additional acetonitrile. Typically, fortification solutions were prepared by diluting the 1.0- μ g/mL intermediate standard solution with additional acetonitrile. Chromatographic standards ranging from 0.0050 to 0.25 μ g/mL were prepared from appropriate volumes of the 1.0- μ g/mL standard solution, water adjusted to pH 3.0 with acetic acid, and additional acetonitrile.

A typical analytical set consisted of at least two treated samples, at least one unfortified control sample, at least two fortified control samples, and at least five standard injections. Standard injections bracketed every one to three samples.

3.2.8 Method of Analytical Calculations

The $\mu\text{g/mL}$ detected in the extract was calculated using the response factor of bracketing standards, peak response (area) in integrator units (IU), and the following equations:

$$\text{Response Factor} = \frac{\text{Standard Concentration } (\mu\text{g/mL})}{\text{Standard Peak Response (IU)}}$$

$$\text{Avg. Resp. Factor} = \frac{\text{Sum of Response Factors of Bracketing Standards} \left(\frac{\mu\text{g/mL}}{\text{IU}} \right)}{2 \text{ (Number of Standards)}}$$

$$\mu\text{g/mL extract} = \text{Peak Area (IU)} \times \text{Avg. Resp. Factor} \left(\frac{\mu\text{g/mL}}{\text{IU}} \right)$$

$$\begin{aligned} \text{Analyte Found (ppm)} = \\ \mu\text{g/mL extract} \times \frac{\text{Final Volume (mL)} \times \text{Aliquot Factor (AF)}}{\text{Sample Mass (g)}} \end{aligned}$$

The famoxadone and cymoxanil (ppm) found were reported to two significant figures using Excel rounding. Residue values detected at or above the LOD and below the LOQ are rounded to one significant figure because these values are less reliable and less reproducible. Levels less than the limit of detection (LOD) are reported as 'ND' (not detected).

Percent recoveries from fortified samples were calculated as follows:

$$\% \text{ Recovery} = \frac{\text{ppm famoxadone found}}{\text{Fortification level (ppm)}} \times 100$$

Percent recoveries calculated and reported by the analytical laboratory were based on ppm famoxadone and cymoxanil found, rounded to excessive decimal places. Percent recoveries reported on the analytical data summary sheets and in this report were rounded to the nearest whole number using Excel default rounding procedures. Mean percent recoveries were calculated from the reported percent recoveries.

Sample Calculation

The concentration of famoxadone found in treated hop sample 7796.00-WA21C-C (Washington Test Site, PHI 3 days, Residue Data Sheets Famoxadone Set 1) was calculated as follows.

$$\text{Standard Response Factor} = \frac{0.010 (\mu\text{g/mL})}{18207 (\text{IU})}$$

$$\begin{aligned} \text{Average Response Factor} &= \frac{\frac{0.01 \text{ standard}}{18207 (\text{peak area})} + \frac{0.05 \text{ standard}}{108234 (\text{peak area})}}{2} \\ &= 0.000000509 \end{aligned}$$

Famoxadone ppm =

$$654786 \times 0.000000509 \times \frac{4 \text{ ml} \times 60 \text{ AF}}{5 \text{ g}} \times \frac{1 \text{ ppm}}{1 \mu\text{g/g}} = 15.89 \text{ ppm}$$

4. Results and Discussion*Method Development*

Famoxadone recoveries were low during method tryout runs. Cymoxanil recoveries were in the acceptable range in the method tryout sets. The low famoxadone recoveries were attributed to signal suppression due to co-extractants from the hop matrix during the analysis. The extract was a bright yellow color when eluted from the florasil column and concentrated.

Initially additional clean-up steps were tried with various SPE bonded phases. None of the combinations tested gave adequate recovery. Finally 2.0 ml of extract rather than 20.0 ml was loaded onto the florasil column and the interference was retained. The resulting extract had only a faint tinge of yellow color to it.

Loading only 2.0 mls of extract resulted in a corresponding rise of the LOQ to 0.5ppm for famoxadone. However, high levels of famoxadone were anticipated in the hop samples because of the way hops are dried in the field before sampling.

Method Validation

The method validation was completed before any field treated samples were analyzed. The residue raw data sheets are displayed on pages 15-20. Recoveries ranged from 75 to 115% for cymoxanil with a mean of 89% and a standard deviation of 11%. For famoxadone recoveries

ranged from 70 to 95% with a mean of 87% and a standard deviation of 8%.

Results of Field Samples

The analysis of the crop sample began on 20Mar01 and was completed on 23Mar01.

No control samples showed quantifiable residues. Residue levels at each site are listed below:

Site	Famoxadone (ppm)	Cymoxanil (ppm)
WA	14.7, 15.8	1.52, 1.13
ID	46.8, 44.5	4.07, 3.45
OR	43.9, 39.2	1.20, 1.14

The minimum level samples were fortified at was 0.05 ppm for cymoxanil and 0.5 ppm for famoxadone. These levels are slightly higher than the levels predicted from the LOQ calculations on pages 29 and 30 which predicted an LOQ of 0.04 ppm for cymoxanil and 0.3 ppm for famoxadone. Since residue levels in the treated samples were so much higher than the predicted LOQ, no further work was performed to address the difference between the predicted LOQ and the lowest fortification. Additionally, raising the LOQ to 0.5 ppm for famoxadone did not have a negative impact on this study because the levels in the treated samples ranged from 14.7 to 46.8 ppm.

Example chromatograms standards and typical standard curve are shown on pages 31 thru 33. Example of control, fortified and treated chromatograms from each site begin on the following pages:

WA-21	page 36
ID-17	page 42
OR-29	page 48

Storage Stability

The storage stability samples were established on 27-Sep-00 approximately four weeks after the samples from the Oregon test site were collected. Samples were fortified at 0.4 ppm famoxadone and 0.16 ppm cymoxanil, which is sufficiently above the LOQ for each analyte established in the method to monitor if any degradation took place.

During method development the LOQ for famoxadone was raised 0.05 to 0.5 ppm. Some minor background levels of famoxadone were observed in the chromatogram of the stored control sample equivalent to approximately 20% of the fortified samples. This was subtracted

and corrected recoveries for the freshly fortified samples were 76 and 82%. The 9 month stability samples had recoveries of 85 and 70%¹.

For cymoxanil, recoveries for the freshly fortified samples were 87 and 85%. The 9 month stability samples had recoveries of 84 and 88%.

Overall, results of 9 month storage stability interval on pages 27 and 28 showed that both cymoxanil and famoxadone were stable for the interval hop samples were stored in this study.

¹ The original analytical report contained a typographical error. This value was originally reported as 77%. The error was found during the QA audit of the final report, and revised on 29 Apr 03 by the Analytical Project Director.

PR. No.:07796 Set-MV1 Famoxadone(JE874)- Hops										
<i>Sample Number</i>	<i>Date Extracted</i>	<i>Date Analyzed</i>	<i>Fort. Level (ppm)</i>	<i>Std Conc. (ug/mL)</i>	<i>Peak Area (stdns bolded)</i>	<i>FV (ml)</i>	<i>AF</i>	<i>Smple Mass (g)</i>	<i>JE874 (ppm)</i>	<i>JE874 % Rec</i>
		March 13, 2001	x	0.100	334697	x	x	x	x	x
		March 13, 2001	x	0.100	334236	x	x	x	x	x
		March 13, 2001	Blank	x	0	x	x	x	x	x
7796.-ID17A-A	March 13, 2001	March 13, 2001	0.00	x	0	4.0	60.0	5.03	<0.5	x
		March 13, 2001	x	0.010	35683	x	x	x	x	
		March 13, 2001	x	0.010	29305	x	x	x	x	x
7796.-ID17A-B*	March 13, 2001	March 13, 2001	5.00	x	281459	4.0	60.0	5.02	4.59	92
7796.-ID17A-B*	March 13, 2001	March 13, 2001	5.00	x	287368	4.0	60.0	5.02	4.69	94
7796.-ID17A-C	March 13, 2001	March 13, 2001	5.00	x	281562	4.0	60.0	5.01	4.59	92
x	x	March 13, 2001	x	0.050	147614	x	x	x	x	x
7796.-ID17A-D	March 13, 2001	March 13, 2001	50.00	x	300820	40.0	60.0	5.00	46.00	92
x	x	March 13, 2001	x	0.250	837712	x	x	x	x	x

PPM = [peak area*Avg. response factor.] * [FV*AF/Sample Mass]

RF=Standard Concentration/Peak area of Standard

Avg Response factor=(RF before sample+RF after sample)/2

% Recovery = [(ppm) in Fort sample/Fort.Level (ppm)]*100

AF = Aliquot Factor

Sample Mass = weight of original sample (g)

x = Not Applicable

FV= Final Volume

*Two injections were made. The average, 93%, will be reported

PR. No.:07796 Set-MV1 Cymoxanil(T3217)- Hops										
<u>Sample Number</u>	<u>Date Extracted</u>	<u>Date Analyzed</u>	<u>Fort. Level (ppm)</u>	<u>Std Conc. (ug/mL)</u>	<u>Peak Area (stdns bolded)</u>	<u>FV (ml)</u>	<u>AF</u>	<u>Smple Mass (g)</u>	<u>T3217 (ppm)</u>	<u>T3217 % Rec</u>
		March 13, 2001	x	0.100	268575	x	x	x	x	x
		March 13, 2001	x	0.100	263184	x	x	x	x	x
		March 13, 2001	Blank	x	0	x	x	x	x	x
7796.-ID17A-A	March 13, 2001	March 13, 2001	0.00	x	0	2.0	16.7	5.03	<0.05	x
		March 13, 2001	x	0.010	26123	x	x	x	x	x
7796.-ID17A-B*	March 13, 2001	March 13, 2001	0.20	x	90247	2.0	16.7	5.02	0.23	115
7796.-ID17A-B*	March 13, 2001	March 13, 2001	0.20	x	77955	2.0	16.7	5.02	0.20	99
7796.-ID17A-C	March 13, 2001	March 13, 2001	0.20	x	64957	2.0	16.7	5.01	0.17	83
	x	March 13, 2001	x	0.050	129147	x	x	x	x	x
x	x	March 13, 2001	x	0.050	131897	x	x	x	x	x
7796.-ID17A-D	March 13, 2001	March 13, 2001	10.00	x	235083	30.0	16.7	5.00	8.82	88
x	x	March 13, 2001	x	0.250	675338	x	x	x	x	x

PPM = [peak area*Avg. response factor.] * [FV*AF/Sample Mass]

RF=Standard Concentration/Peak area of Standard

Avg Response factor= (RF before sample+RF after sample)/2

% Recovery = [(ppm) in Fort sample/Fort.Level (ppm)]*100

Sample Mass = weight of original sample (g)

x = Not Applicable

FV= Final Volume

*Two injections were made. The average 107% will be reported.

PR. No.:07796 Set-MV2 Famoxadone(JE874)- Hops										
Sample Number	Date Extracted	Date Analyzed	Fort. Level (ppm)	Std Conc. (ug/mL)	Peak Area (stnds bolded)	FV (ml)	AF	Smple Mass (g)	JE874 (ppm)	JE874 % Rec
		March 15, 2001	x	0.100	402213	x	x	x	x	x
		March 15, 2001	x	0.100	416655	x	x	x	x	x
		March 15, 2001	Blank	x	0	x	x	x	x	x
7796-WA21A-A	March 15, 2001	March 15, 2001	0.00	x	0	4.0	60.0	5.02	<0.5	x
		March 15, 2001	x	0.005	17341	x	x	x	x	x
		March 15, 2001	x	0.010	35436	x	x	x	x	x
7796-WA21A-B*	March 15, 2001	March 15, 2001	0.50	x	30612	4.0	60.0	5.05	0.41	82
7796-WA21A-B*	March 15, 2001	March 15, 2001	0.50	x	30914	4.0	60.0	5.05	0.42	83
7796-WA21A-C	March 15, 2001	March 15, 2001	0.50	x	26199	4.0	60.0	5.01	0.35	70
x	x	March 15, 2001	x	0.050	180356	x	x	x	x	x
7796-WA21A-D	March 15, 2001	March 15, 2001	5.00	x	**	40.0	60.0	5.00	-	-
x	x	March 15, 2001	x	0.250	982747	x	x	x	x	x

PPM = [peak area*Avg. response factor.] * [FV*AF/Sample Mass]

RF=Standard Concentration/Peak area of Standard

Avg Response factor= (RF before sample+RF after sample)/2

% Recovery = [(ppm) in Fort sample/Fort.Level (ppm)]*100

AF = Aliquot Factor

Sample Mass = weight of original sample (g)

x = Not Applicable

FV= Final Volume

*Two injections were made. The average, 82%, will be reported.

** Mass Spec not performed on sample. Analysis will be re-run and reported with method validation set 3

PR. No.:07796 Set-MV2 Cymoxanil(T3217)- Hops										
Sample Number	Date Extracted	Date Analyzed	Fort. Level (ppm)	Std Conc. (ug/mL)	Peak Area (stdns bolded)	FV (ml)	AF	Smple Mass (g)	T3217 (ppm)	T3217 % Rec
		March 15, 2001	x	0.100	336789	x	x	x	x	x
		March 15, 2001	x	0.100	334660	x	x	x	x	x
		March 15, 2001	Blank	x	0	x	x	x	x	x
7796-WA21A-A	March 15, 2001	March 15, 2001	0.00	x	0	2.0	16.7	5.02	<0.05	x
		March 15, 2001	x	0.005	15314	x	x	x	x	x
		March 15, 2001	x	0.010	33543	x	x	x	x	x
7796-WA21A-B*	March 15, 2001	March 15, 2001	0.05	x	20124	2.0	16.7	5.05	0.04	88
7796-WA21A-B*	March 15, 2001	March 15, 2001	0.05	x	18653	2.0	16.7	5.05	0.04	81
7796-WA21A-C	March 15, 2001	March 15, 2001	0.05	x	13698	2.5	16.7	5.01	0.04	75
x	x	March 15, 2001	x	0.050	141397	x	x	x	x	x
7796-WA21A-D	March 15, 2001	March 15, 2001	0.20	x	80856	2.0	16.7	5.00	0.18	90
x	x	March 15, 2001	x	0.250	798457	x	x	x	x	x

PPM = [peak area*Avg. response factor.] * [FV*AF/Sample Mass]

RF=Standard Concentration/Peak area of Standard

Avg Response factor= (RF before sample+RF after sample)/2

% Recovery = [(ppm) in Fort sample/Fort.Level (ppm)]*100

AF = Aliquot factor

Sample Mass = weight of original sample (g)

x = Not Applicable

FV= Final Volume

*Two injections were made. The average, 84%, will be reported.

PR. No.:07796 Set-MV3 Famoxadone(JE874)- Hops										
Sample Number	Date Extracted	Date Analyzed	Fort. Level (ppm)	Std Conc. (ug/mL)	Peak Area (stnds bolded)	FV (ml)	AF	Smple Mass (g)	JE874 (ppm)	JE874 % Rec
		March 16, 2001	x	0.100	220944	x	x	x	x	x
		March 16, 2001	Blank	x	0	x	x	x	x	x
7796-OR29A-A	March 16, 2001	March 16, 2001	0.00	x	660	4.0	60.0	5.00	<0.5	x
		March 16, 2001	x	0.005	11749	x	x	x	x	x
		March 16, 2001	x	0.010	20036	x	x	x	x	x
7796-OR29A-B	March 16, 2001	March 16, 2001	0.50	x	16441	4.0	60.0	5.02	0.38	76
7796-OR29A-C	March 16, 2001	March 16, 2001	50.00	x	196047	40.0	60.0	5.01	45.21	90
x	x	March 16, 2001	x	0.050	108258	x	x	x	x	x
7796-OR29A-D	March 16, 2001	March 16, 2001	50.00	x	200058	40.0	60.0	5.02	43.85	88
7796-WA21A-D*	March 16, 2001	March 16, 2001	5.00	x	216846	4.0	60.0	5.00	4.75	95
x	x	March 16, 2001	x	0.250	553724	x	x	x	x	x

PPM = [peak area*Avg. response factor.] * [FV*AF/Sample Mass]

RF=Standard Concentration/Peak area of Standard

Avg Response factor= (RF before sample+RF after sample)/2

% Recovery = [(ppm) in Fort sample/Fort.Level (ppm)]*100

AF = Aliquot Factor

Sample Mass = weight of original sample (g)

x = Not Applicable

FV= Final Volume

* Re-analysis of Sample D from method Validation Set 2

PR. No.:07796 Set-MV3 Cymoxanil(T3217)- Hops										
<i>Sample Number</i>	<i>Date Extracted</i>	<i>Date Analyzed</i>	<i>Fort. Level (ppm)</i>	<i>Std Conc. (ug/mL)</i>	<i>Peak Area (stdns bolded)</i>	<i>FV (ml)</i>	<i>AF</i>	<i>Smple Mass (g)</i>	<i>T3217 (ppm)</i>	<i>T3217 % Rec</i>
		March 16, 2001	x	0.100	334746	x	x	x	x	x
		March 16, 2001	Blank	x	0	x	x	x	x	x
7796-OR29A-A	March 16, 2001	March 16, 2001	0.00	x	0	2.0	16.7	5.00	<0.05	x
		March 16, 2001	x	0.005	14033	x	x	x	x	x
		March 16, 2001	x	0.010	35838	x	x	x	x	x
7796-OR29A-B	March 16, 2001	March 16, 2001	0.05	x	23465	2.0	16.7	5.02	0.05	91
7796-OR29A-C	March 16, 2001	March 16, 2001	10.00	x	313011	30.0	16.7	5.01	9.14	91
x	x	March 16, 2001	x	0.050	164464	x	x	x	x	x
7796-OR29A-D	March 16, 2001	March 16, 2001	10.00	x	261582	30.0	16.7	5.02	8.32	83
x	x	March 16, 2001	x	0.250	755129	x	x	x	x	x

PPM = [peak area*Avg. response factor.] * [FV*AF/Sample Mass]

RF=Standard Concentration/Peak area of Standard

Avg Response factor= (RF before sample+RF after sample)/2

% Recovery = [(ppm) in Fort sample/Fort.Level (ppm)]*100

AF = Aliquot Factor

Sample Mass = weight of original sample (g)

x = Not Applicable

FV= Final Volume

PR. No.:07796 Set-1 Famoxadone(JE874)- Hops										
<i>Sample Number</i>	<i>Date Extracted</i>	<i>Date Analyzed</i>	<i>Fort. Level (ppm)</i>	<i>Std Conc. (ug/mL)</i>	<i>Peak Area (stdns bolded)</i>	<i>FV (ml)</i>	<i>AF</i>	<i>Smple Mass (g)</i>	<i>JE874 (ppm)</i>	<i>JE874 % Rec</i>
		March 20, 2001	x	0.100	239488	x	x	x	x	x
		March 20, 2001	x	0.100	237628	x	x	x	x	x
		March 20, 2001	Blank	x	0	x	x	x	x	x
7796-WA21B-A	March 20, 2001	March 20, 2001	0.00	x	0	4.0	60.0	5.02	<0.5	x
		March 20, 2001	x	0.005	11557	x	x	x	x	x
		March 20, 2001	x	0.010	18207	x	x	x	x	x
7796-WA21B-B*	March 20, 2001	March 20, 2001	0.50	x	15919	4.0	60.0	5.01	0.39	77
7796-WA21B-B*	March 20, 2001	March 20, 2001	0.50	x	15472	4.0	60.0	5.01	0.38	75
7796-WA21C-C	March 20, 2001	March 20, 2001	x	x	654786	4.0	60.0	5.01	15.89	x
x	x	March 20, 2001	x	0.050	108234	x	x	x	x	x
7796-WA21D-D	March 20, 2001	March 20, 2001	x	x	723459	4.0	60.0	5.04	15.20	-
x	x	March 20, 2001	x	0.250	604551	x	x	x	x	x

PPM = [peak area*Avg. response factor.] * [FV*AF/Sample Mass]

RF=Standard Concentration/Peak area of Standard

Avg Response factor= (RF before sample+RF after sample)/2

% Recovery = [(ppm) in Fort sample/Fort.Level (ppm)]*100

AF = Aliquot Factor

Sample Mass = weight of original sample (g)

x = Not Applicable

FV= Final Volume

*Two injections were made. The average, 76%, will be reported.

PR. No.:07796 Set-1 Cymoxanil(T3217)- Hops										
Sample Number	Date Extracted	Date Analyzed	Fort. Level (ppm)	Std Conc. (ug/mL)	Peak Area (stnds bolded)	FV (ml)	AF	Smple Mass (g)	T3217 (ppm)	T3217 % Rec
		March 20, 2001	x	0.100	386618	x	x	x	x	x
		March 20, 2001	x	0.100	386314	x	x	x	x	x
		March 20, 2001	Blank	x	0	x	x	x	x	x
7796-WA21B-A	March 20, 2001	March 20, 2001	0.00	x	0	2.0	16.7	5.02	<0.05	x
		March 20, 2001	x	0.005	14606	x	x	x	x	x
		March 20, 2001	x	0.010	34444	x	x	x	x	x
7796-WA21B-B*	March 20, 2001	March 20, 2001	0.05	x	19504	2.0	16.7	5.01	0.04	78
7796-WA21B-B*	March 20, 2001	March 20, 2001	0.05	x	18882	2.0	16.7	5.01	0.04	76
7796-WA21C-C	March 20, 2001	March 20, 2001	x	x	606101	2.5	16.7	5.01	1.52	x
x	x	March 20, 2001	x	0.050	161036	x	x	x	x	x
7796-WA21D-D	March 20, 2001	March 20, 2001	x	x	568901	2.0	16.7	5.04	1.13	x
x	x	March 20, 2001	x	0.250	872439	x	x	x	x	x

PPM = [peak area*Avg. response factor.] * [FV*AF/Sample Mass]

RF=Standard Concentration/Peak area of Standard

Avg Response factor= (RF before sample+RF after sample)/2

% Recovery = [(ppm) in Fort sample/Fort.Level (ppm)]*100

AF = Aliquot Factor

Sample Mass = weight of original sample (g)

x = Not Applicable

AF = Aliquot Factor

FV= Final Volume

*Two injections were made. The average, 77%, will be reported.

PR. No.:07796 Set-2 Famoxadone(JE874)- Hops										
<i>Sample Number</i>	<i>Date Extracted</i>	<i>Date Analyzed</i>	<i>Fort. Level (ppm)</i>	<i>Std Conc. (ug/mL)</i>	<i>Peak Area (stdns bolded)</i>	<i>FV (ml)</i>	<i>AF</i>	<i>Smple Mass (g)</i>	<i>JE874 (ppm)</i>	<i>JE874 % Rec</i>
		March 21, 2001	x	0.100	235017	x	x	x	x	x
		March 21, 2001	x	0.100	226951	x	x	x	x	x
		March 21, 2001	Blank	x	0	x	x	x	x	x
7796-ID17B-A	March 21, 2001	March 21, 2001	0.00	x	0	8.0	60.0	5.01	<0.5	x
		March 21, 2001	x	0.005	10174	x	x	x	x	x
		March 21, 2001	x	0.010	18403	x	x	x	x	x
7796-ID17B-B*	March 21, 2001	March 21, 2001	50.00	x	199759	40.0	60.0	5.01	48.96	98
7796-ID17B-B*	March 21, 2001	March 21, 2001	50.00	x	204370	40.0	60.0	5.01	50.09	100
7796-ID17C-C	March 21, 2001	March 21, 2001	x	x	1086920	8.0	60.0	5.02	53.28	x
x	x	March 21, 2001	x	0.050	104636	x	x	x	x	x
7796-ID17D-D	March 21, 2001	March 21, 2001	x	x	1101110	8.0	60.0	5.04	47.13	-
x	x	March 21, 2001	x	0.250	604001	x	x	x	x	x
x	x	March 21, 2001	x	0.250	597130	x	x	x	x	x
7796WA21C-C**	March 21, 2001	March 21, 2001	x	x	334690	4.0	60.0	5.01	14.70	-
7796WA21D-D**	March 21, 2001	March 21, 2001	x	x	360216	4.0	60.0	5.04	15.82	-
x	x	March 21, 2001	x	0.100	201431	x	x	x	x	x

PPM = [peak area*Avg. response factor.] * [FV*AF/Sample Mass]

RF=Standard Concentration/Peak area of Standard

Avg Response factor= (RF before sample+RF after sample)/2

% Recovery = [(ppm) in Fort sample/Fort.Level (ppm)]*100

AF = Aliquot Factor

Sample Mass = weight of original sample (g)

x = Not Applicable

FV= Final Volume

**Samples from Set 2 (3/21/2001) were diluted by a factor of 2 and re-analyzed.

*Two injections were made. The average, 99%, will be reported.

PR. No.:07796 Set-2 Cymoxanil(T3217)- Hops										
<i>Sample Number</i>	<i>Date Extracted</i>	<i>Date Analyzed</i>	<i>Fort. Level (ppm)</i>	<i>Std Conc. (ug/mL)</i>	<i>Peak Area (stnds bolded)</i>	<i>FV (ml)</i>	<i>AF</i>	<i>Smple Mass (g)</i>	<i>T3217 (ppm)</i>	<i>T3217 % Rec</i>
		March 21, 2001	x	0.100	385044	x	x	x	x	x
		March 21, 2001	x	0.100	334570	x	x	x	x	x
		March 21, 2001	Blank	x	0	x	x	x	x	x
7796-ID17B-A	March 21, 2001	March 21, 2001	0.00	x	929	5.0	16.7	5.01	<0.05	x
		March 21, 2001	x	0.005	14298	x	x	x	x	x
		March 21, 2001	x	0.010	29349	x	x	x	x	x
7796-ID17B-B*	March 21, 2001	March 21, 2001	10.00	x	301526	30.0	16.7	5.01	9.26	93
7796-ID17B-B)*	March 21, 2001	March 21, 2001	10.00	x	311138	30.0	16.7	5.01	9.56	96
7796-ID17C-C	March 21, 2001	March 21, 2001	x	x	795493	5.0	16.7	5.02	4.07	x
x	x	March 21, 2001	x	0.050	183524	x	x	x	x	x
7796-ID17D-D	March 21, 2001	March 21, 2001	x	x	776119	5.0	16.7	5.04	3.45	x
x	x	March 21, 2001	x	0.250	963296	x	x	x	x	x

PPM = [peak area*Avg. response factor.] * [FV*AF/Sample Mass]

RF=Standard Concentration/Peak area of Standard

Avg Response factor=(RF before sample+RF after sample)/2

% Recovery = [(ppm) in Fort sample/Fort.Level (ppm)]*100

Sample Mass = weight of original sample (g)

x = Not Applicable

AF = Aliquot Factor

FV= Final Volume

*Two injections were made. The average, 94%, will be reported.

PR. No.:07796 Set-3 Famoxadone(JE874)- Hops										
<i>Sample Number</i>	<i>Date Extracted</i>	<i>Date Analyzed</i>	<i>Fort. Level (ppm)</i>	<i>Std Conc. (ug/mL)</i>	<i>Peak Area (stnds bolded)</i>	<i>FV (ml)</i>	<i>AF</i>	<i>Smple Mass (g)</i>	<i>JE874 (ppm)</i>	<i>JE874 % Rec</i>
		March 23, 2001	x	0.100	277296	x	x	x	x	x
		March 23, 2001	x	0.100	271601	x	x	x	x	x
		March 23, 2001	Blank	x	0	x	x	x	x	x
7796-OR29B-A	March 23, 2001	March 23, 2001	0.00	x	0	16.0	60.0	5.02	<0.5	x
		March 23, 2001	x	0.005	11002	x	x	x	x	x
		March 23, 2001	x	0.010	20007	x	x	x	x	x
7796-OR29B-B*	March 23, 2001	March 23, 2001	50.00	x	242629	40.0	60.0	5.02	50.22	100
7796-OR29B-B*	March 23, 2001	March 23, 2001	50.00	x	244966	40.0	60.0	5.02	50.70	101
7796-OR29C-C	March 23, 2000	March 23, 2001	x	x	530392	16.0	60.0	5.05	43.91	x
x	x	March 23, 2001	x	0.050	137915	x	x	x	x	x
7796-OR29D-D	March 23, 2001	March 23, 2001	x	x	581758	16.0	60.0	5.04	39.22	-
x	x	March 23, 2001	x	0.250	736002	x	x	x	x	x
x	x	March 23, 2001	x	0.250	715469	x	x	x	x	x
7796-ID17C-C**	March 23, 2001	March 23, 2001	x	x	464059	8.0	60.0	5.02	46.85	-
7796-ID17D-D**	March 23, 2001	March 23, 2001	x	x	440438	8.0	60.0	5.04	44.46	-
x	x	March 23, 2001	x	0.100	284403	x	x	x	x	x

PPM = [peak area*Avg. response factor.] * [FV*AF/Sample Mass]

RF=Standard Concentration/Peak area of Standard

Avg Response factor= (RF before sample+RF after sample)/2

% Recovery = [(ppm) in Fort sample/Fort.Level (ppm)]*100

AF = Aliquot Factor

Sample Mass = weight of original sample (g)

x = Not Applicable

FV= Final Volume

**Samples from Set 2 (3/21/2001) were diluted by a factor of 3 and re-analyzed.

*Two injections were made. The average, 100%, will be reported.

PR. No.:07796 Set-3 Cymoxanil(T3217)- Hops										
Sample Number	Date Extracted	Date Analyzed	Fort. Level (ppm)	Std Conc. (ug/mL)	Peak Area (stnds bolded)	FV (ml)	AF	Smple Mass (g)	T3217 (ppm)	T3217 % Rec
		March 23, 2001	x	0.100	368827	x	x	x	x	x
		March 23, 2001	x	0.100	374691	x	x	x	x	x
		March 23, 2001	Blank	x	0	x	x	x	x	x
7796-OR29B-A	March 23, 2001	March 23, 2001	0.00	x	0	5.0	16.7	5.02	<0.05	x
		March 23, 2001	x	0.005	15410	x	x	x	x	x
		March 23, 2001	x	0.010	30862	x	x	x	x	x
7796-OR29B-B*	March 23, 2001	March 23, 2001	10.00	x	307382	30.0	16.7	5.02	9.36	94
7796-OR29B-B*	March 23, 2001	March 23, 2001	10.00	x	302275	30.0	16.7	5.02	9.19	92
7796-OR29C-C	March 23, 2001	March 23, 2001	x	x	236176	5.0	16.7	5.05	1.20	x
x	x	March 23, 2001	x	0.050	176942	x	x	x	x	x
7796-OR29D-D	March 23, 2001	March 23, 2001	x	x	249340	5.0	16.7	5.04	1.14	x
x	x	March 23, 2001	x	0.250	938519	x	x	x	x	x

PPM = [peak area*Avg. response factor.] * [FV*AF/Sample Mass]

RF=Standard Concentration/Peak area of Standard

Avg Response factor= (RF before sample+RF after sample)/2

% Recovery = [(ppm) in Fort sample/Fort.Level (ppm)]*100

Sample Mass = weight of original sample (g)

x = Not Applicable

AF= Aliquot Factor

FV= Final Volume

*Two injections were made. The average, 93%, will be reported.

PR. No.:07796 9 Month Freezer Stability Famoxadone(JE874)- Hops										
Sample Number	Date Extracted	Date Analyzed	Fort. Level (ppm)	Std Conc. (ug/mL)	Peak Area (stnds bolded)	FV (ml)	AF	Smple Mass (g)	JE874 (ppm)	JE874 % Rec
		June 7, 2001	Blank	x	0	x	x	x	x	x
		June 7, 2001	x	0.010	52128	x	x	x	x	x
		June 7, 2001	Blank	x	0	x	x	x	x	x
7796-OR29A-1	June 6, 2001	June 7, 2001	0.00	x	8305*	4.0	60.0	5.03	<0.5	x
7796-OR29A-2	June 6,2001	June 7, 2001	0.40	x	37052	4.0	60.0	5.08	0.31	76
		June 7, 2001	x	0.050	329242	x	x	x	x	x
7796-OR29A-3	June 6, 2001	June 7, 2001	0.40	x	44786	4.0	60.0	5.04	0.33	82
7796-OR29A-13	June 6, 2001	June 7, 2001	0.40	x	46386	4.0	60.0	5.02	0.34	85**
x	x	June 7, 2001	x	0.100	645035	x	x	x	x	x
		June 7, 2001	Blank	x	0	x	x	x	x	x
7796-OR29A-14	June 6, 2001	June 7, 2001	0.40	x	38648	4.0	60.0	5.02	0.31	70**
x	x	June 7, 2001	x	0.250	1724800	x	x	x	x	x
x	x	June 7, 2001	x	0.007	39698	x	x	x	x	x

PPM = [peak area*Avg. response factor.] * [FV*AF/Sample Mass]

RF=Standard Concentration/Peak area of Standard

Avg Response factor=(RF before sample+RF after sample)/2

% Recovery = [(ppm) in Fort sample/Fort.Level (ppm)]*100

AF = Aliquot Factor

Sample Mass = weight of original sample (g)

x = Not Applicable

FV= Final Volume

AF=Aliquot Factor

* Subtracted 8305 from peak areas of fortified samples in set.

** Stability Samples

PR. No.:07796 9 Month Freezer Stability Cymoxanil(T3217)- Hops										
Sample Number	Date Extracted	Date Analyzed	Fort. Level (ppm)	Std Conc. (ug/mL)	Peak Area (stnds bolded)	FV (ml)	AF	Smple Mass (g)	T3217 (ppm)	T3217 % Rec
		June 12, 2001	Blank	x	0	x	x	x	x	x
		June 12, 2001	x	0.010	198152	x	x	x	x	x
		June 12, 2001	Blank	x	0	x	x	x	x	x
7796-OR29A-1	June 6, 2001	June 12, 2001	0.00	x	0	3.0	16.7	5.03	<0.05	x
7796-OR29A-2	June 6,2001	June 12, 2001	1.00	x	565897	10.0	16.7	5.08	0.87	87
		June 12, 2001	x	0.050	1209160	x	x	x	x	x
7796-OR29A-3	June 6, 2001	June 12, 2001	1.00	x	603960	10.0	16.7	5.04	0.85	85
7796-OR29A-13	June 6, 2001	June 12, 2001	0.16	x	317846	3.0	16.7	5.02	0.13	84*
x	x	June 12, 2001	x	0.100	2327360	x	x	x	x	x
		June 12, 2001	Blank	x	0	x	x	x	x	x
7796-OR29A-14	June 6, 2001	June 12, 2001	0.16	x	315078	3.0	16.7	5.02	0.14	88*
x	x	June 12, 2001	x	0.007	152183	x	x	x	x	x
x	x	June 12, 2001	x	0.250	5733000	x	x	x	x	x

PPM = [peak area*Avg. response factor.] * [FV*AF/Sample Mass]

RF=Standard Concentration/Peak area of Standard

Avg Response factor=(RF before sample+RF after sample)/2

% Recovery = [(ppm) in Fort sample/Fort.Level (ppm)]*100

Sample Mass = weight of original sample (g)

x = Not Applicable

FV= Final Volume

AF=Aliquot Factor

* Stability Samples

FAMOXADONE**Calculations for Determination of LOD / LOQ**Lowest Level of Method Validation (ppm) = 0.5

degrees of freedom	Number of Replicates	One-tailed t-statistic
0	1	infinity
1	2	31.821
2	3	6.965
3	4	4.541
4	5	3.747
5	6	3.365
6	7	3.143
7	8	2.998
8	9	2.896
9	10	2.821
10	11	2.764
11	12	2.718
12	13	2.681
13	14	2.65
14	15	2.624
15	16	2.602
16	17	2.583
17	18	2.567
18	19	2.552
19	20	2.539
20	21	2.528
21	22	2.518
22	23	2.508
23	24	2.500
24	25	2.492
25	26	2.485

Amount Detected
(ppm)

0.41
0.42
0.35
0.38
0.39
0.38

Avg. Recovery	0.3883 ppm	n = 6
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	Std. Dev.	One-tailed t-statistic	MDL (LOD)
For Lowest Spike Only:	0.024832774	3.365	0.083562

For (n) Replicates =
(n-1) Deg. of Freedom

= (t-statistic) X (Std. Dev.)

For Lowest Spike Only:	PQL (LOQ) 0.2506861
	= (MDL) X (3)

This method is based on the method described in Roy-Keith Smith's Handbook of Environmental Analysis, Fourth Edition, Genium Publishing Corporation.

CYMOXANIL

Calculations for Determination of LOD / LOQ

Lowest Level of Method Validation (ppm) = 0.05

degrees of freedom	Number of Replicates	One-tailed t-statistic
0	1	infinity
1	2	31.821
2	3	6.965
3	4	4.541
4	5	3.747
5	6	3.365
6	7	3.143
7	8	2.998
8	9	2.896
9	10	2.821
10	11	2.764
11	12	2.718
12	13	2.681
13	14	2.65
14	15	2.624
15	16	2.602
16	17	2.583
17	18	2.567
18	19	2.552
19	20	2.539
20	21	2.528
21	22	2.518
22	23	2.508
23	24	2.500
24	25	2.492
25	26	2.485

Amount Detected
(ppm)

0.044
0.041
0.037
0.046
0.039
0.038

Avg. Recovery	0.0408 ppm	n = 6
---------------	------------	-------

	Std. Dev.	One-tailed t-statistic	MDL (LOD)
For Lowest Spike Only:	0.003544949	3.365	0.0119286

For (n) Replicates =
(n-1) Deg. of Freedom

= (t-statistic) X (Std. Dev.)

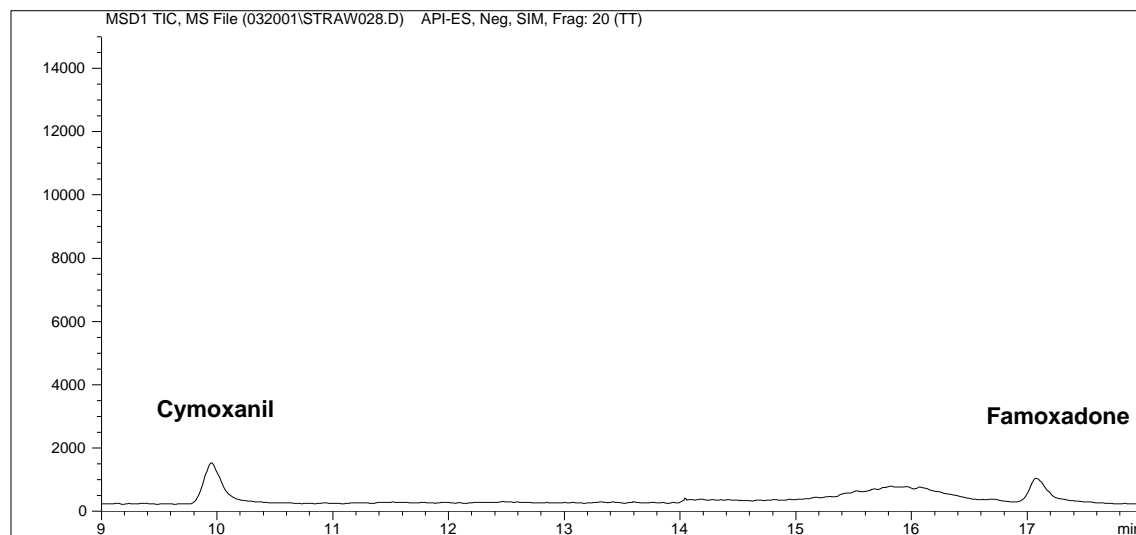
For Lowest Spike Only:

PQL (LOQ)**0.0357858**

= (MDL) X (3)

This method is based on the method described in Roy-Keith Smith's Handbook of Environmental Analysis, Fourth Edition, Genium Publishing Corporation.

REPRESENTATIVE STANDARD 0.005UG/ML



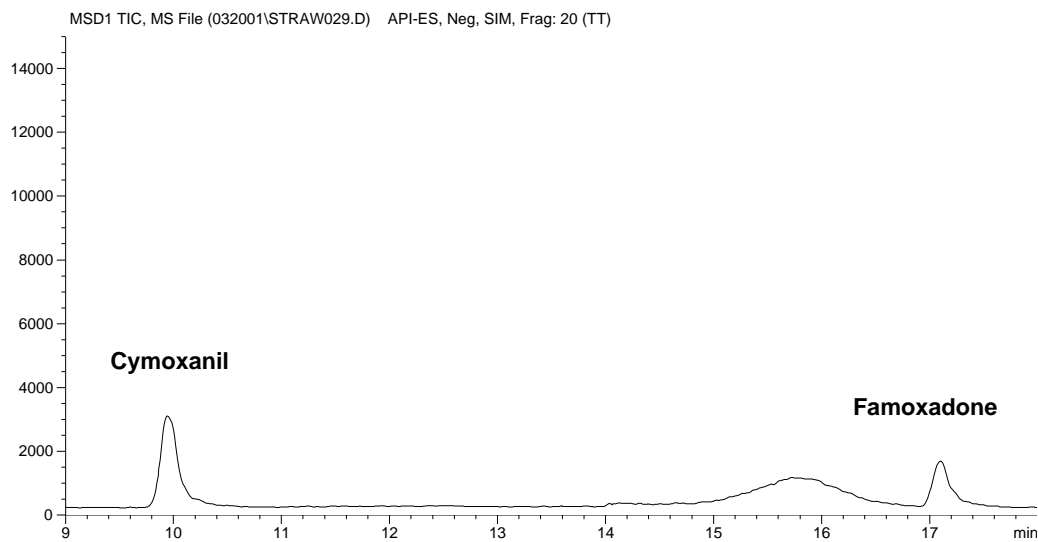
0.005 ug/ml Standard

Cymoxanil

Pk Area: 14606

Date Analyzed: 20 Mar 2001

REPRESENTATIVE STANDARD 0.010 UG/ML



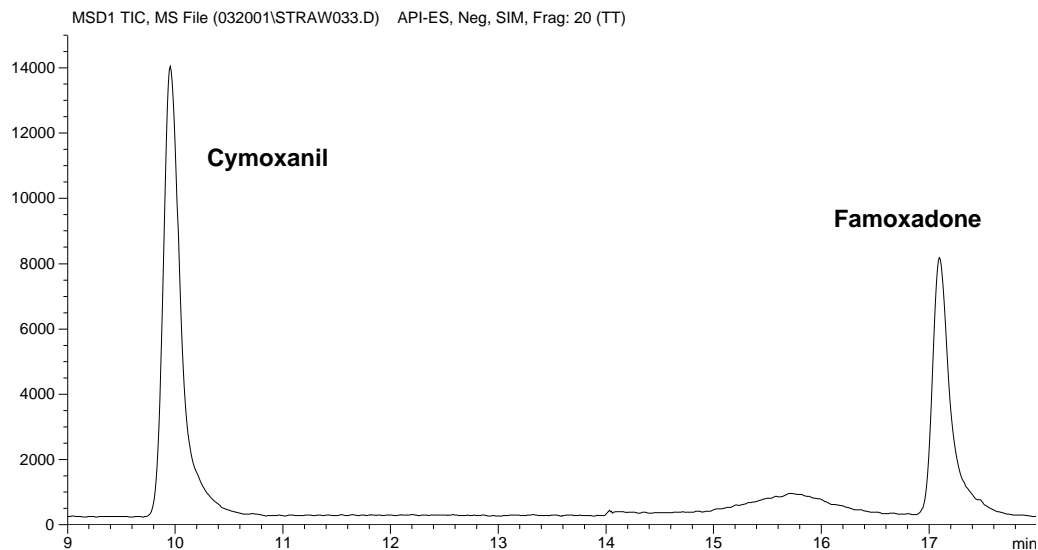
0.010 ug/ml Standard

Cymoxanil

Pk Area: 34444

Date Analyzed: 20 Mar 2001

REPRESENTATIVE STANDARD 0.050 UG/ML

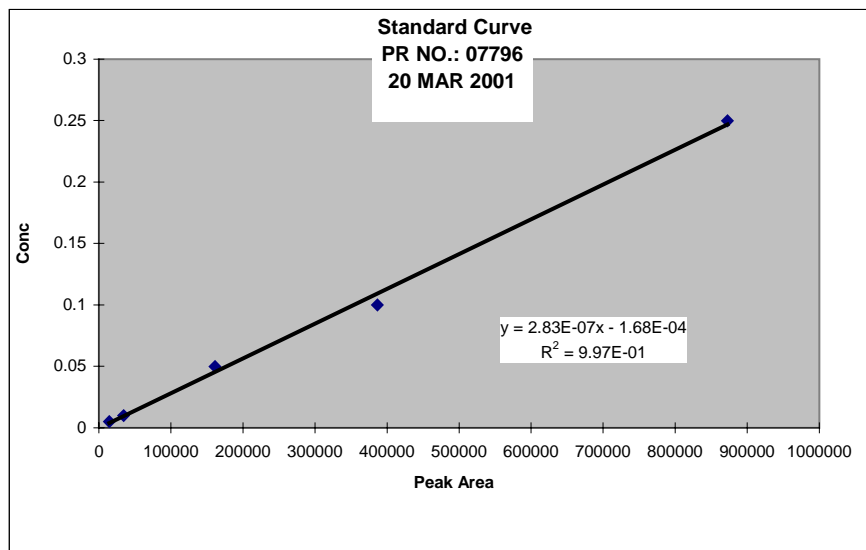


0.050 ug/ml Standard

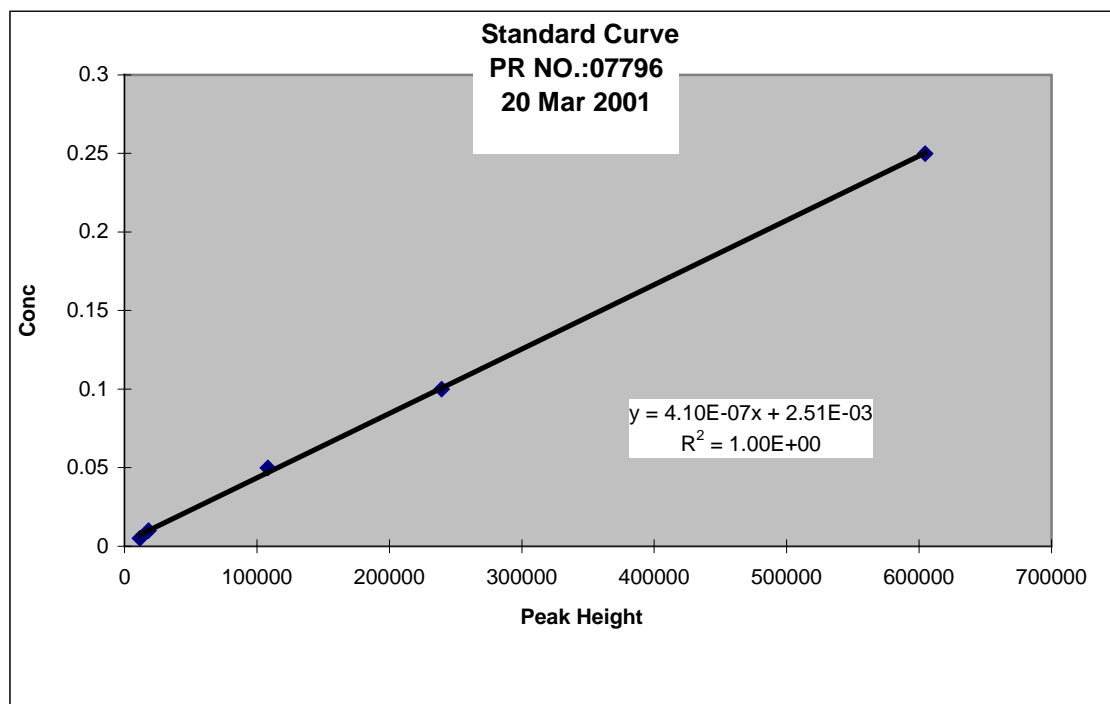
Cymoxanil

Pk Area: 161036

Date Analyzed: 20 Mar 2001

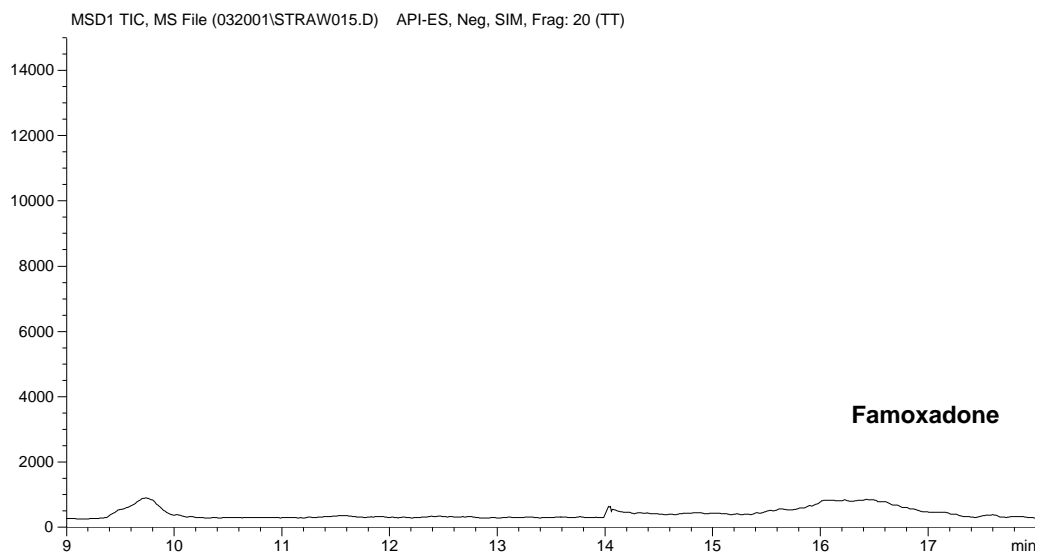
REPRESENTATIVE STANDARD CURVE FOR CYMOXANIL

Pk Area	Conc
14606	0.005
34444	0.01
161036	0.05
386618	0.1
872439	0.25

REPRESENTATIVE STANDARD CURVE FOR FAMOXADONE

Pk Ht	Conc
11557	0.005
18207	0.01
108234	0.05
239488	0.1
604451	0.25

EXAMPLE CHROMATOGRAMS OF CONTROL, FORTIFIED, AND TREATED HOP SAMPLES FROM WA-21: FAMOXADONE



Control Sample

Sample ID: 7796-WA21B-A

Test Site: Prosser WA

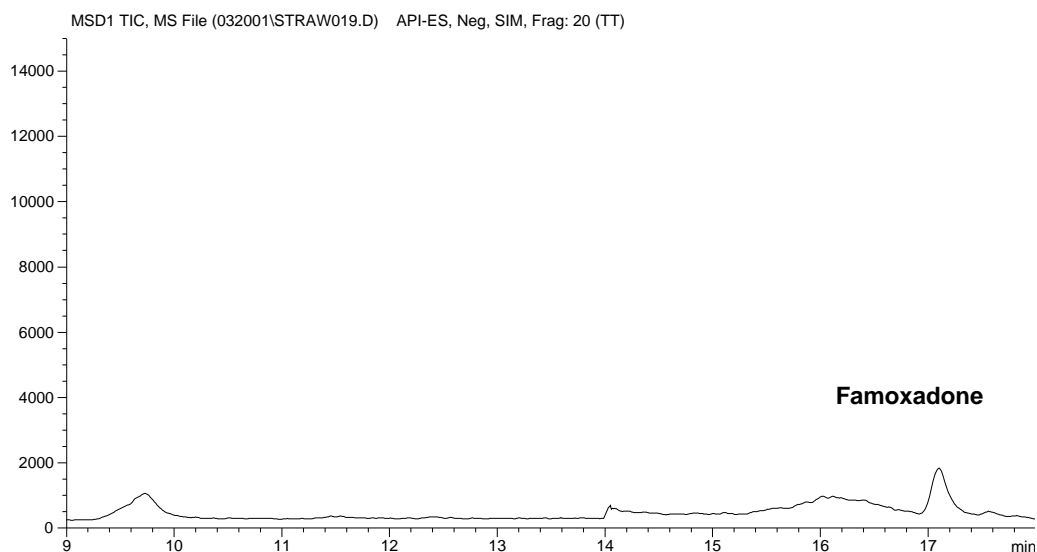
Set No.: Set 1

Analysis Date: 20 Mar 2001

Peak Area 0

Famoxadone Found (ppm): <0.5 ppm

EXAMPLE CHROMATOGRAMS OF CONTROL, FORTIFIED, AND TREATED HOP SAMPLES FROM WA-21: FAMOXADONE



Fortified Sample- 0.5 ppm

Sample ID: 7796-WA21B-B

Test Site: Prosser WA

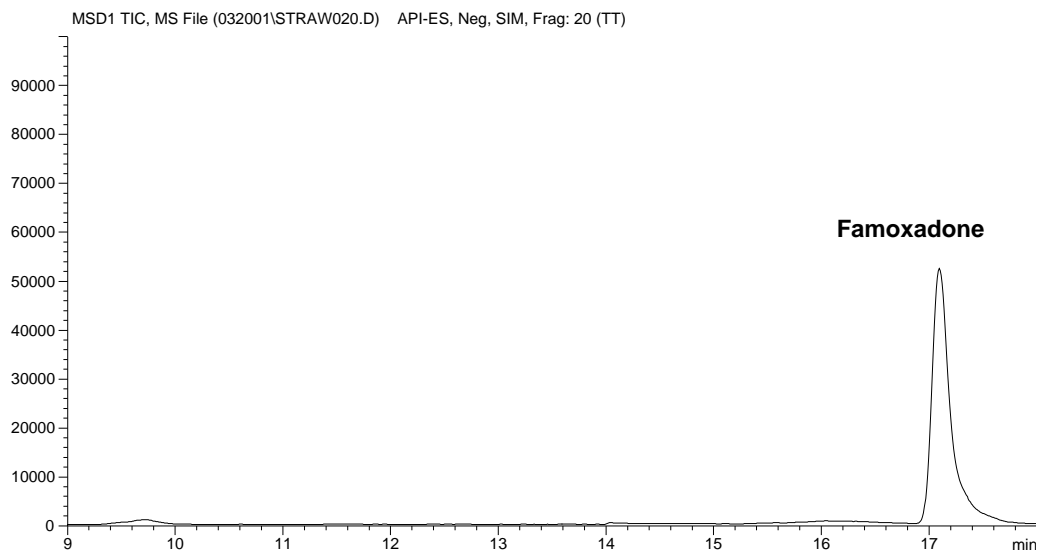
Set No.: Set 1

Analysis Date: 20 Mar 2001

Peak Area 15472

Famoxadone Found (ppm): 0.38 ppm (75% rec.)

**EXAMPLE CHROMATOGRAMS OF CONTROL, FORTIFIED, AND
TREATED HOP SAMPLES FROM WA-21:
FAMOXADONE**



Treated Sample-

Sample ID: 7796-WA21C-C

Test Site: Prosser WA

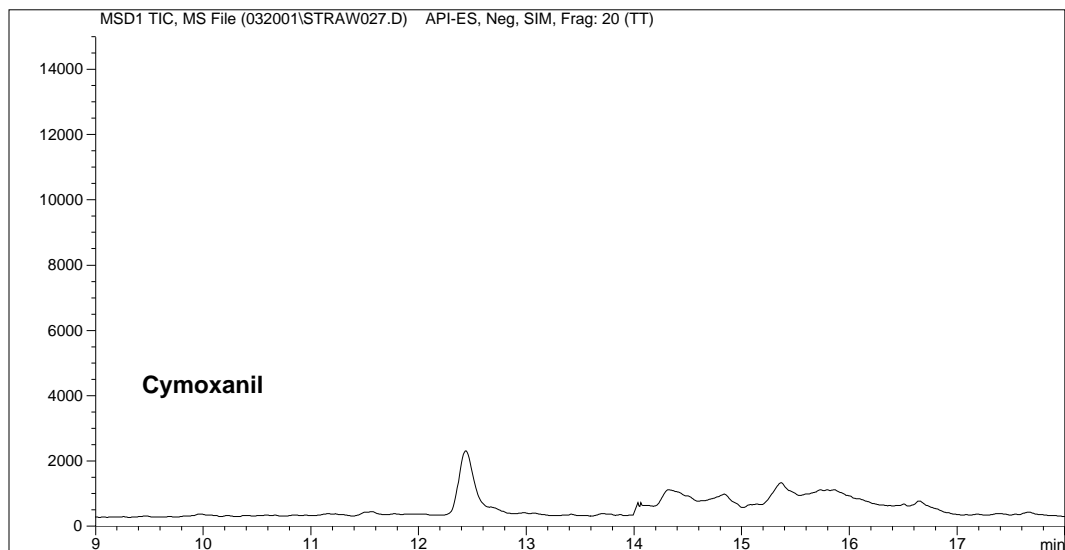
Set No.: Set 1

Analysis Date: 20 Mar 2001

Peak Area 654786

Famoxadone Found (ppm): 15.89 ppm

**EXAMPLE CHROMATOGRAMS OF CONTROL, FORTIFIED, AND
TREATED HOP SAMPLES FROM WA-21:
CYMOXANIL**



Control Sample

Sample ID: 7796-WA21B-A

Test Site: Prosser WA

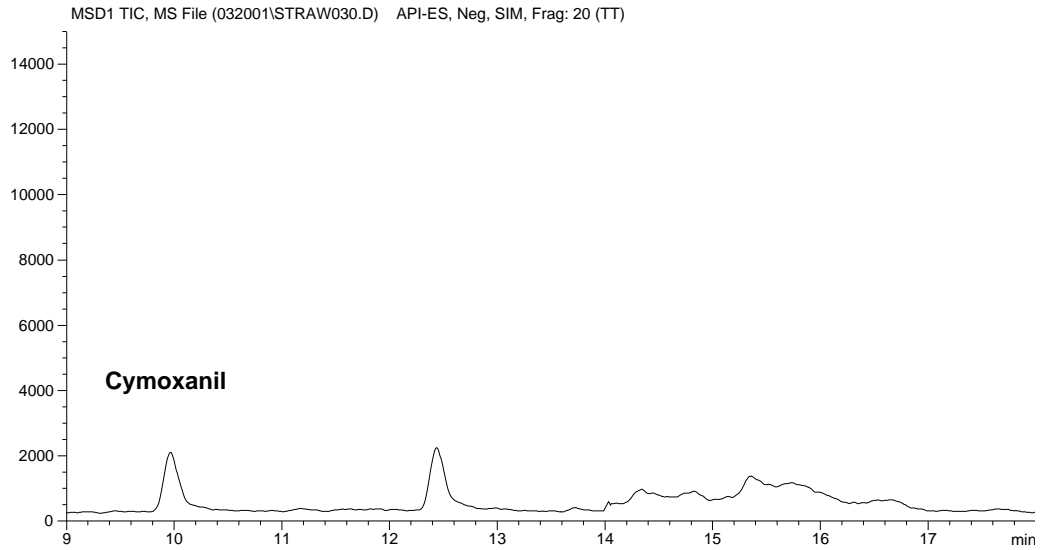
Set No.: Set 1

Analysis Date: 20 Mar 2001

Peak Area 0

Cymoxanil Found (ppm): <0.05 ppm

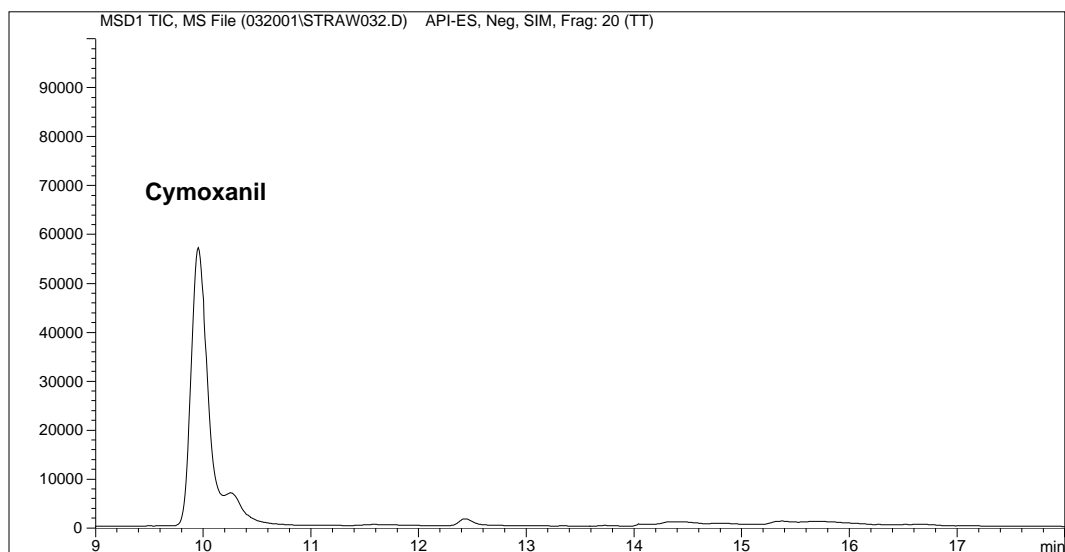
**EXAMPLE CHROMATOGRAMS OF CONTROL, FORTIFIED, AND
TREATED HOP SAMPLES FROM WA-21:
CYMOXANIL**



Fortified Sample- 0.05 ppm

Sample ID: 7796-WA21B-B
Test Site: Prosser WA
Set No.: Set 1
Analysis Date: 20 Mar 2001
Peak Area 19504
Cymoxanil Found (ppm): 0.04ppm (78% rec)

**EXAMPLE CHROMATOGRAMS OF CONTROL, FORTIFIED, AND
TREATED HOP SAMPLES FROM WA-21:
CYMOXANIL**



Treated Sample

Sample ID: 7796-WA21C-C

Test Site: Prosser WA

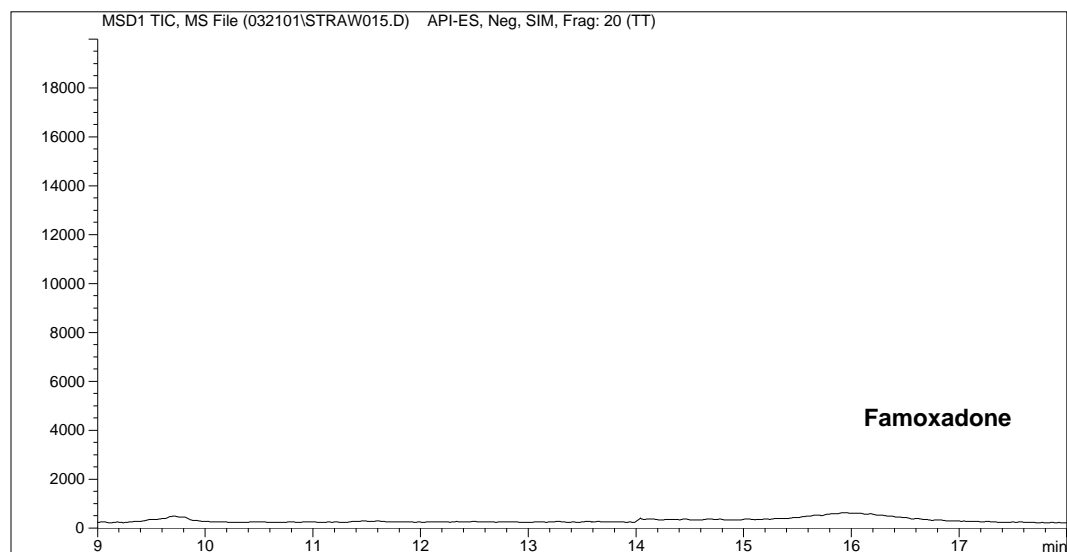
Set No.: Set 1

Analysis Date: 20 Mar 2001

Peak Area 606101

Cymoxanil Found (ppm): 1.52 ppm

**EXAMPLE CHROMATOGRAMS OF CONTROL, FORTIFIED, AND
TREATED HOP SAMPLES FROM ID-17:
FAMOXADONE**



Control Sample

Sample ID: 7796-ID17B-A

Test Site: Parma, ID

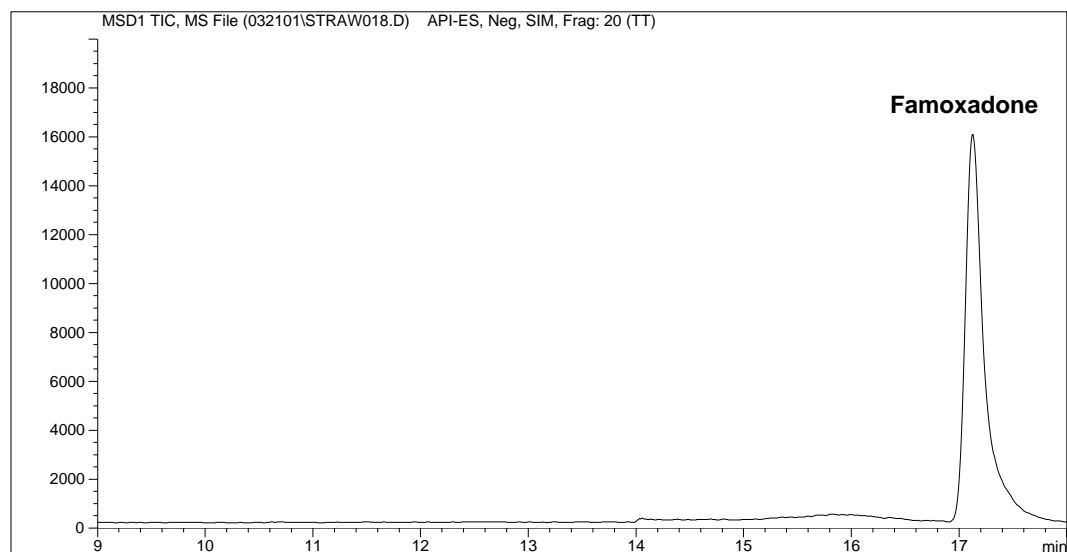
Set No.: Set 2

Analysis Date: 21 Mar 2001

Peak Area 0

Famoxadone Found (ppm): <0.5 ppm

**EXAMPLE CHROMATOGRAMS OF CONTROL, FORTIFIED, AND
TREATED HOP SAMPLES FROM ID-17:
FAMOXADONE**



Fortified Sample- 50 ppm

Sample ID: 7796-ID17B-B

Test Site: Parma, ID

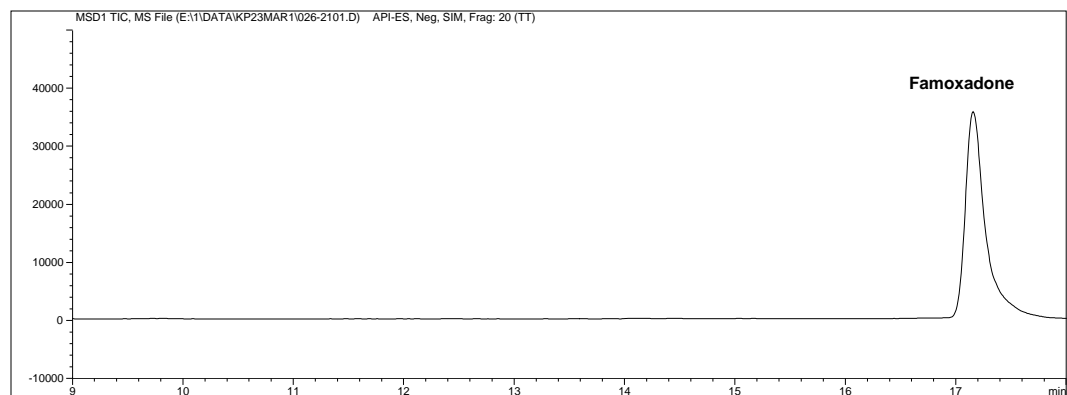
Set No.: Set 2

Analysis Date: 21 Mar 2001

Peak Area 199759

Famoxadone Found (ppm): 48.96 (98% rec)

**EXAMPLE CHROMATOGRAMS OF CONTROL, FORTIFIED, AND
TREATED HOP SAMPLES FROM ID-17:
FAMOXADONE**



Treated Sample

Sample ID: 7796-ID17C-C

Test Site: Parma, ID

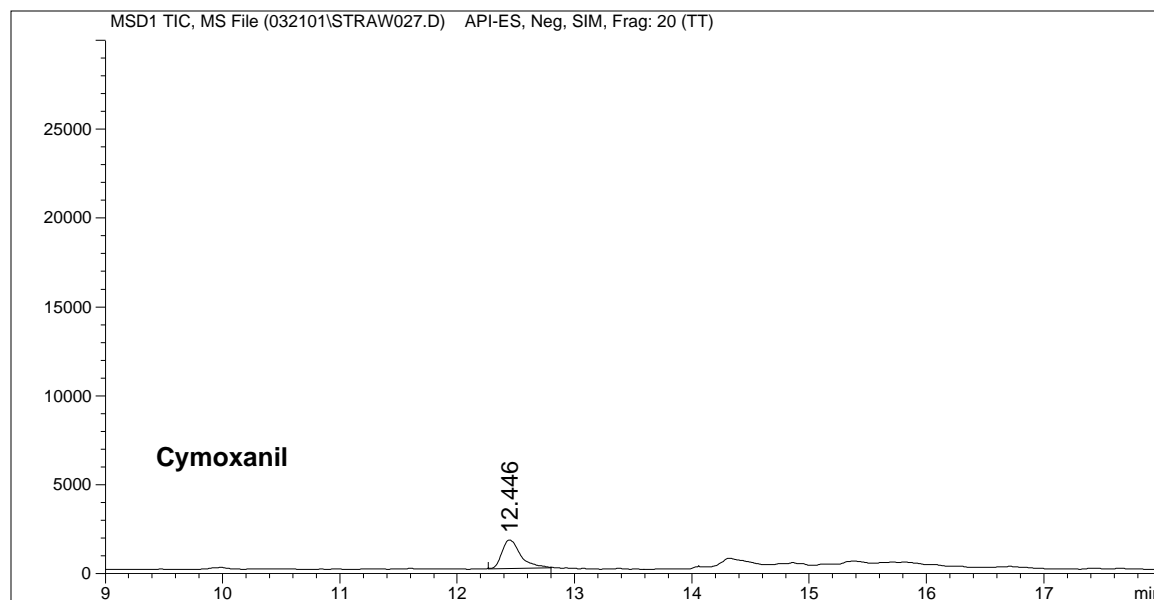
Set No.: Set 3

Analysis Date: 23 Mar 2001

Peak Area 464059

Famoxadone Found (ppm): 46.85

**EXAMPLE CHROMATOGRAMS OF CONTROL, FORTIFIED, AND
TREATED HOP SAMPLES FROM ID-17:
CYMOXANIL**



Control Sample

Sample ID: 7796-ID17B-A

Test Site: Parma, ID

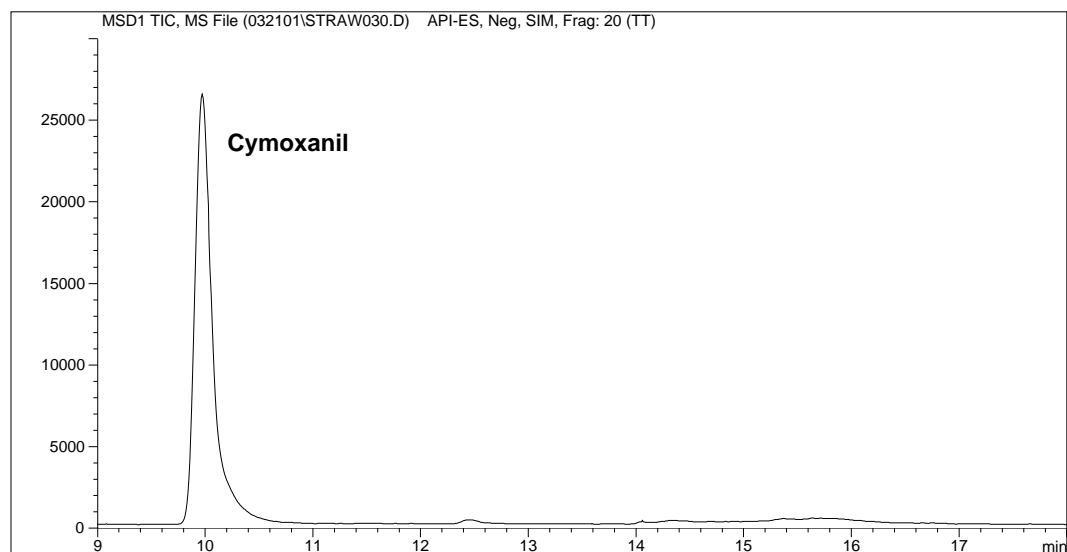
Set No.: Set 2

Analysis Date: 21 Mar 2001

Peak Area 929

Cymoxanil Found (ppm): <0.05

**EXAMPLE CHROMATOGRAMS OF CONTROL, FORTIFIED, AND
TREATED HOP SAMPLES FROM ID-17:
CYMOXANIL**



Fortified Sample- 10ppm

Sample ID: 7796-ID17B-B

Test Site: Parma, ID

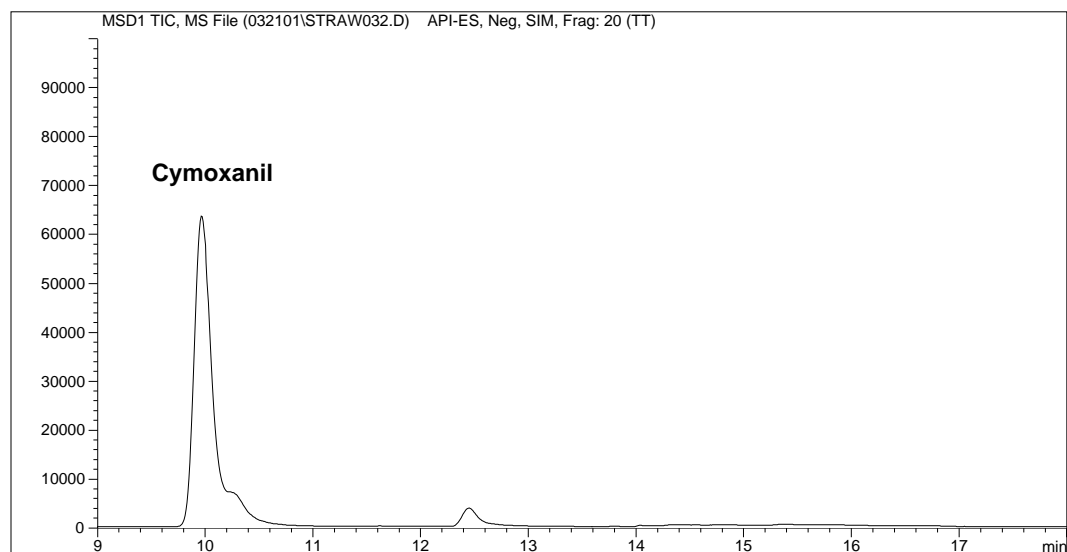
Set No.: Set 2

Analysis Date: 21 Mar 2001

Peak Area 301526

Cymoxanil Found (ppm): 9.26 ppm (93% rec)

**EXAMPLE CHROMATOGRAMS OF CONTROL, FORTIFIED, AND
TREATED HOP SAMPLES FROM ID-17:
CYMOXANIL**



Treated Sample

Sample ID: 7796-ID17C-C

Test Site: Parma, ID

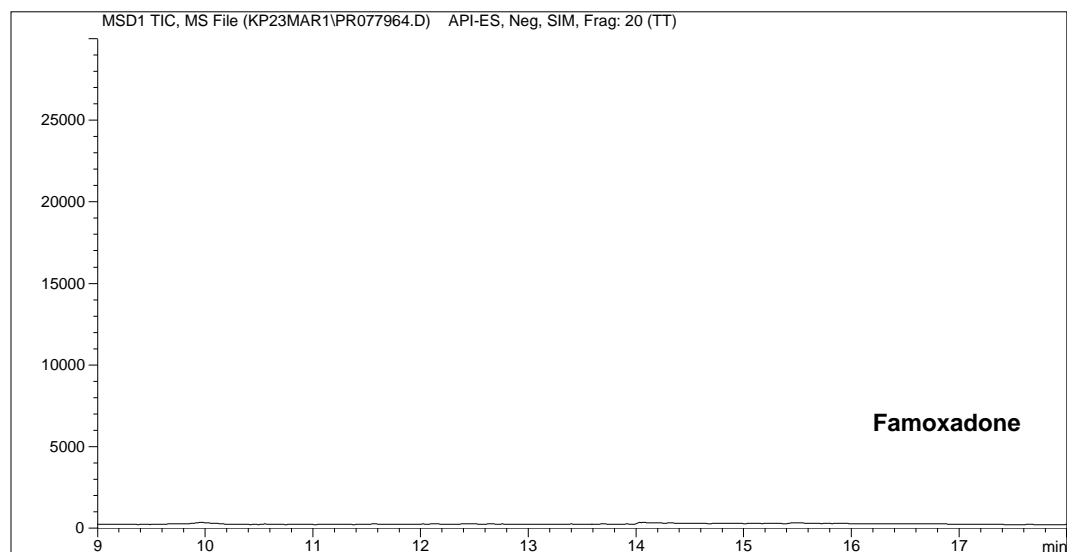
Set No.: Set 2

Analysis Date: 21 Mar 2001

Peak Area 795493

Cymoxanil Found (ppm): 4.07 ppm

**EXAMPLE CHROMATOGRAMS OF CONTROL, FORTIFIED, AND
TREATED HOP SAMPLES FROM OR-29:
FAMOXADONE**



Control Sample

Sample ID: 7796-OR29B-A

Test Site: Hubbard, OR

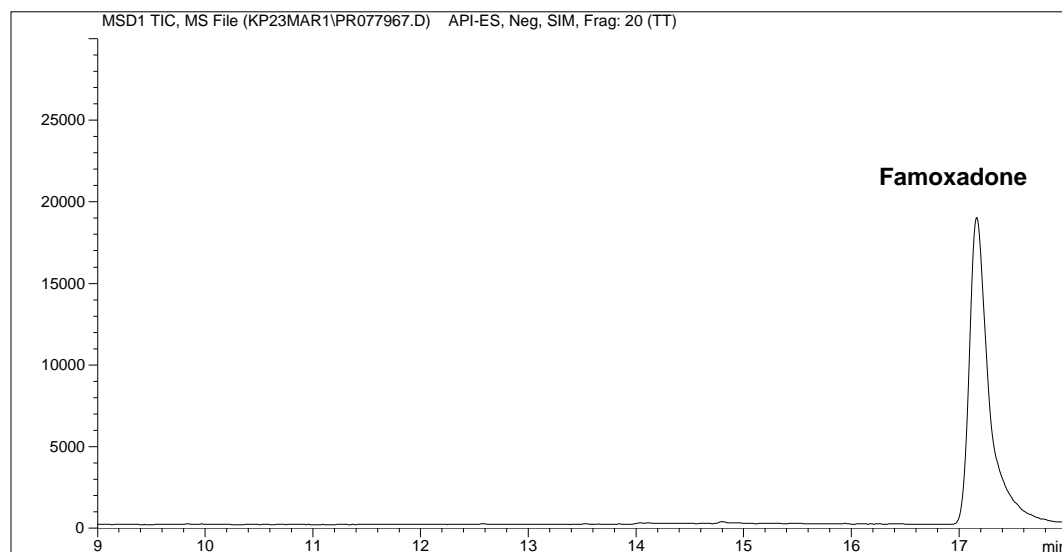
Set No.: Set 3

Analysis Date: 23 Mar 2001

Peak Area 0

Famoxadone Found (ppm): <0.50 ppm

**EXAMPLE CHROMATOGRAMS OF CONTROL, FORTIFIED, AND
TREATED HOP SAMPLES FROM OR-29:
FAMOXADONE**



Fortified Sample- 50 ppm

Sample ID: 7796-OR29B-B

Test Site: Hubbard, OR

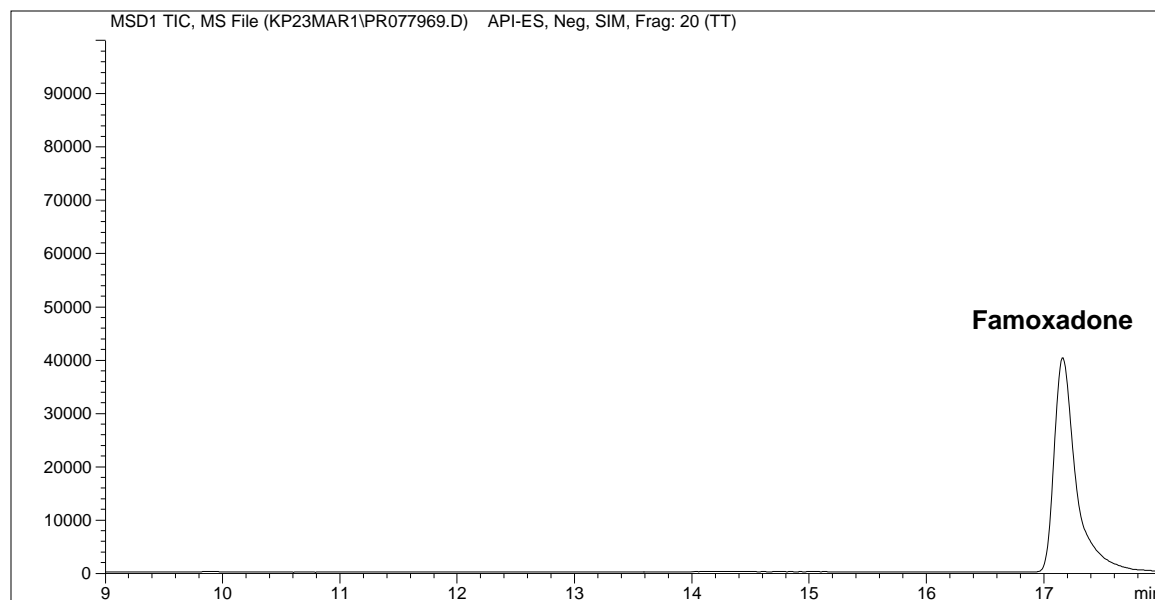
Set No.: Set 3

Analysis Date: 23 Mar 2001

Peak Area 242629

Famoxadone Found (ppm): 50.22 (100% rec)

**EXAMPLE CHROMATOGRAMS OF CONTROL, FORTIFIED, AND
TREATED HOP SAMPLES FROM OR-29:
FAMOXADONE**



Treated Sample

Sample ID: 7796-OR29C-C

Test Site: Hubbard, OR

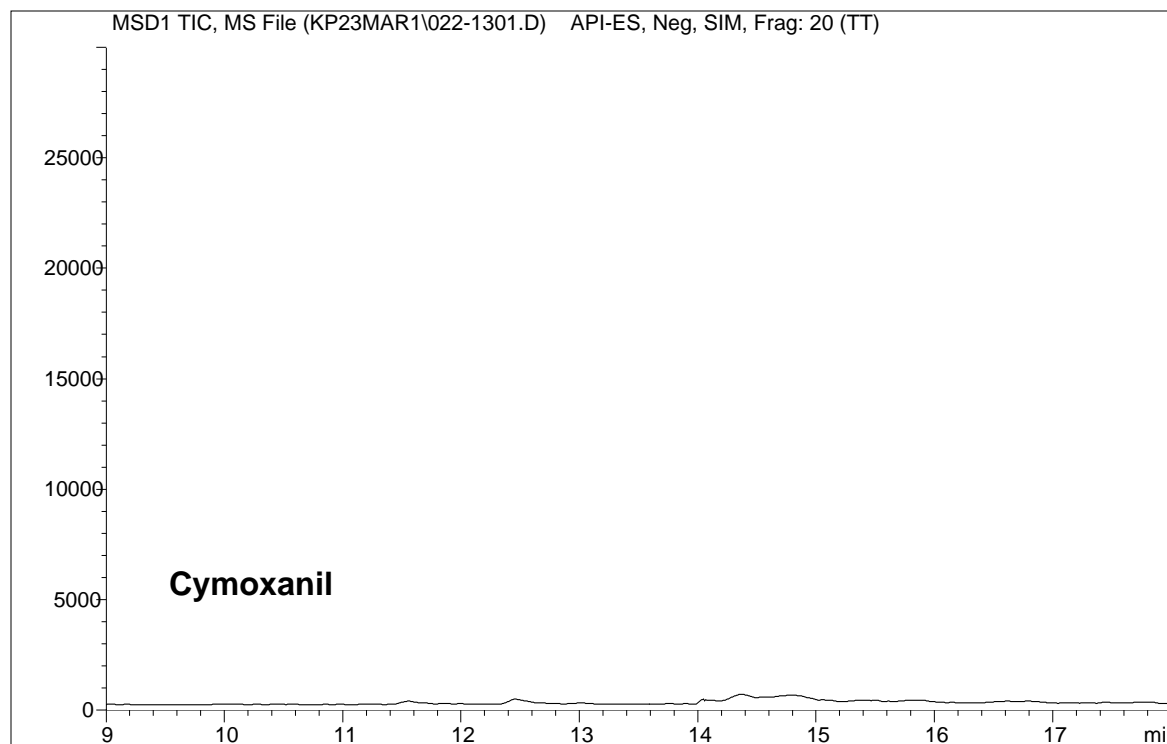
Set No.: Set 3

Analysis Date: 23 Mar 2001

Peak Area 530392

Famoxadone Found (ppm): 43.91 ppm

**EXAMPLE CHROMATOGRAMS OF CONTROL, FORTIFIED, AND
TREATED HOP SAMPLES FROM OR-29:
CYMOXANIL**



Control Sample

Sample ID: 7796-OR29B-A

Test Site: Hubbard, OR

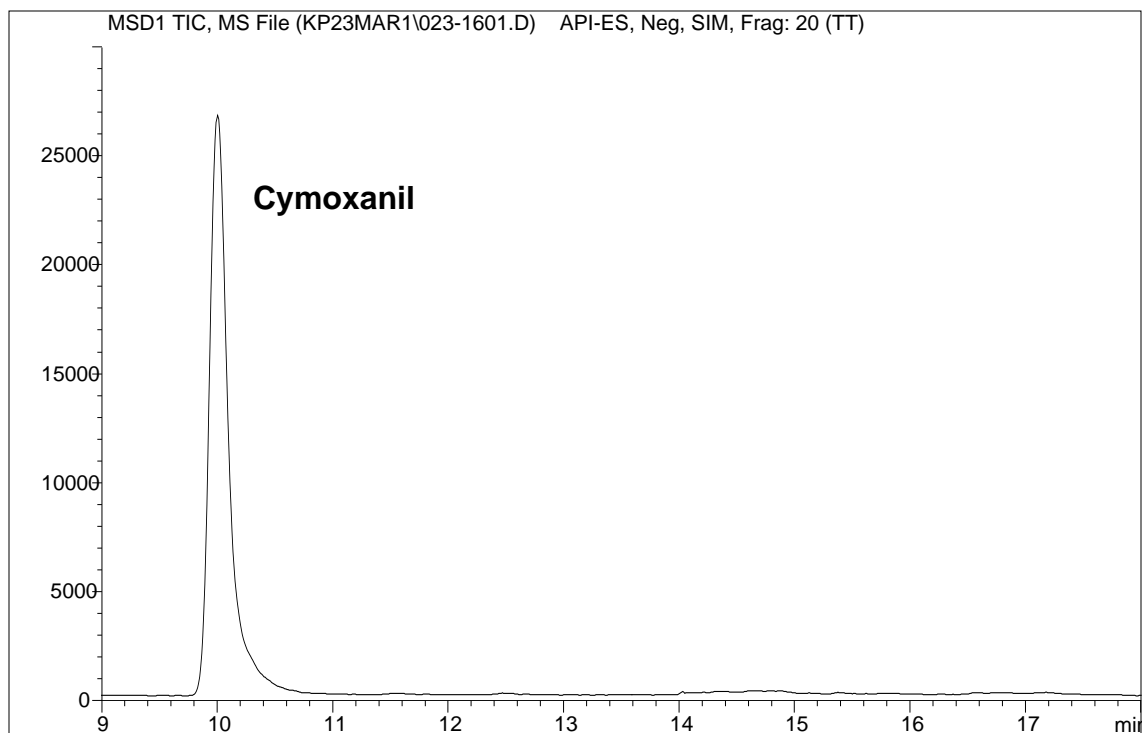
Set No.: Set 3

Analysis Date: 23 Mar 2001

Peak Area 0

Cymoxanil Found (ppm): <0.05 ppm

**EXAMPLE CHROMATOGRAMS OF CONTROL, FORTIFIED, AND
TREATED HOP SAMPLES FROM OR-29:
CYMOXANIL**



Fortified Sample- 10 ppm

Sample ID: 7796-OR29B-B

Test Site: Hubbard, OR

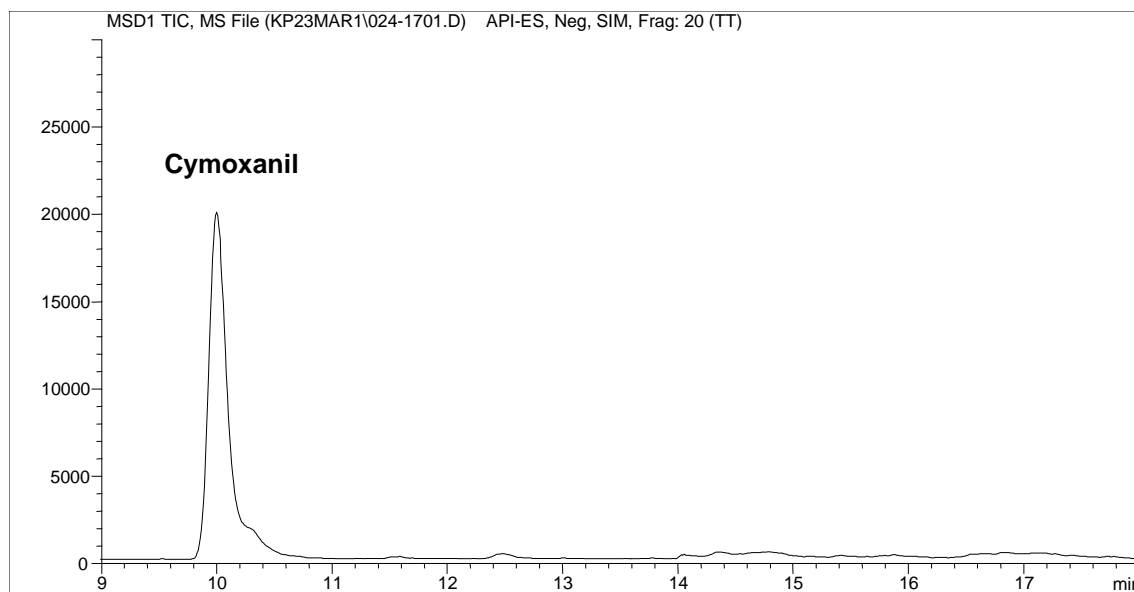
Set No.: Set 3

Analysis Date: 23 Mar 2001

Peak Area 307382

Cymoxanil Found (ppm): 9.36 ppm (94% rec)

**EXAMPLE CHROMATOGRAMS OF CONTROL, FORTIFIED, AND
TREATED HOP SAMPLES FROM OR-29:
CYMOXANIL**



Treated Sample

Sample ID: 7796-OR29C-C

Test Site: Hubbard, OR

Set No.: Set 3

Analysis Date: 23 Mar 2001

Peak Area 236176

Cymoxanil Found (ppm): 1.20 ppm

