Cover Sheet for

ENVIRONMENTAL CHEMISTRY METHOD

Pestcide Name: Halosulfuron

MRID #: 429763-05

Matrix: Water

Analysis: GC/NPD

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STUDY TITLE

Analytical Method for the Determination of MON 12000 in Water Matrices

DATA REQUIREMENT

Not Applicable

429763-05

AUTHORS

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STUDY COMPLETED

September, 1993

PERFORMING LABORATORIES

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MSL 13030

RD 1206

Volume 5

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Manager, New Products Registrations

DATE: 10/14/93

GLP COMPLIANCE STATEMENT

MONSANTO COMPANY - THE AGRICULTURAL GROUP

The following report on analytical methods was not conducted in accordance with the principles of 40 CFR 160, GOOD LABORATORY PRACTICE STANDARDS (FIFRA/FFDCA), as promulgated in Federal Register, 54, No. 158, 34067-34704, 1 July, 1991.

Richard C. Durbin 10/14/93

Sponsor / Submitter Date

RD 1206

MSL-13030
ANALYTICAL METHOD FOR THE DETERMINATION OF MON 12000 IN WATER MATRICES

Dennis Arras and Martha Schlicher
September 1993

I. Summary

An analytical method was developed to provide ready determination of MON 12000 in water matrices. The methodology involves solvent partitioning of a water sample with dichloromethane, separation and clean up on a Florisil Solid Phase Extraction (SPE) column, and conversion to MON 12000 rearrangement ester. Following an additional SPE clean up, the rearrangment ester is quantitated by gas chromatography with a nitrogen-phosphorous detector (GC/NPD). Utilizing readily available equipment and instrumentation, MON 12000 can be quantitated in water at levels at or above 0.5 ppb.

II. Introduction

An analytical method was developed by Monsanto Company for the analysis of MON 12000 in water matrices. This method was developed as an analytical aid for state and federal regulatory agencies and was not developed under a protocol. All aspects of the analysis, however, were conducted utilizing Good Laboratory Practices.

III. Materials and Methods

A. Test Compound

MON 12000 analytical standard was used to prepare stock and fortification solutions of MON 12000 in acetonitrile for fortifications to water matrices at levels ranging from 0.0005 to 0.0100 ppm. All stock and fortification solutions were stored between -10 and 0 °F. Characterization and stability data of the reference material are retained by Monsanto.

B. Test Diluent

The diluent utilized for fortification and stock solution preparations was acetonitrile. The test substance was added to water matrices in acetonitrile.

C. Analysis Method

1. The method validation was conducted to provide a validation of the analytical method for analyzing low levels of MON 12000 in water matrices. MON 12000 at levels of 0.0005, 0.0010, 0.0025, 0.0050, and 0.0100 ppm were evaluated. The method involves initial extraction of MON 12000 from water matrices with dichloromethane. MON 12000, **1**, is then submitted to a Florisil solid phase extraction (SPE) clean up/separation. As indicated in Scheme I, the column fraction containing MON 12000 is then stirred overnight with 0.5 M potassium carbonate to convert MON 12000 to rearrangement ester, **2**, to provide a compound suitable for gas chromatographic analysis. Final sample cleanup is performed using a silica SPE column and the final rearrangement ester GCed using a nitrogen specific detector (NPD).

The analytical method provided in Appendix A entitled, "Analytical Method for the Determination of MON 12000 in Water" contains the procedures necessary for this analysis.

IV. Results and Discussion

Results from the method validation study for MON 12000 in water matrices are presented in Table I. The average recovery was 89% with a standard deviation of 20% for ten samples analyzed.

Table I

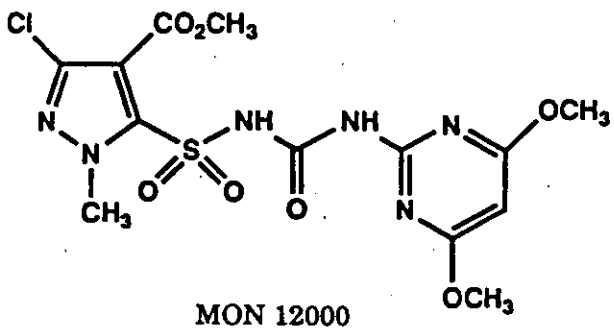
Validation Data for Water Samples Spiked with MON 12000 Quantitated as the Rearrangement Ester

Fortification Level	Recovery
0.0005 ppm	113%
0.0005 ppm	122%
0.0010 ppm	92%
0.0010 ppm	101%
0.0025 ppm	86%
0.0025 ppm	75%
0.0050 ppm	71%
0.0050 ppm	74%
0.0100 ppm	73%
0.0100 ppm	82%

V. Data Storage and Records Retention

All original raw data, or certified copies thereof, and summaries of data specific to this study will be stored in the Monsanto archives.

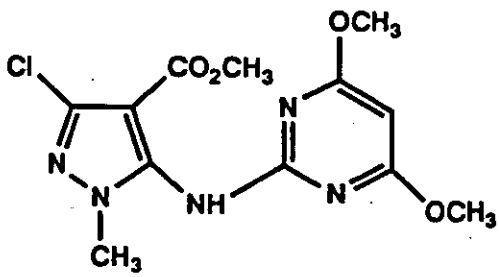
Scheme I



MON 12000

1

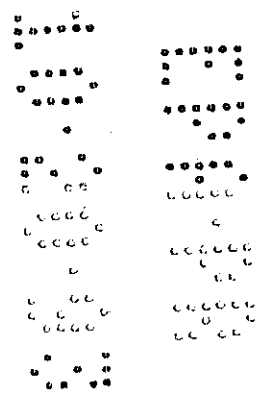
Sodium Carbonate



Rearrangement Ester

2

Appendix A



ANALYTICAL METHOD FOR THE DETERMINATION OF MON 12000 IN WATER

Author:	Dennis D. Arras
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Prepared by: Dennis D. Arras Date: 8/17/93

Approved by: Martha Schlueter Date: 8/17/93

QA Approval: Frederick M. Tiebe Date: 17 August 1993

I. SCOPE

The analytical method described here is for the determination of the MON 12000 parent compound in water matrices. This method lists the chemicals, solutions, and equipment required for this determination and gives details of the procedures required for the extraction, cleanup, and isolation of this analyte. Typical instrument conditions, example chromatograms, and validation results are also provided.

II. PRINCIPLE

The water sample is extracted twice with methylene chloride. After evaporation to dryness, a preliminary separation and cleanup is performed using a Florisil Solid Phase Extraction (SPE) column. The column fraction containing MON 12000 is stirred overnight with 0.5 M potassium carbonate to convert the MON 12000 to the Rearrangement Ester (RRE). Final sample cleanup is performed using a Silica SPE column and the sample volume adjusted by evaporation to dryness and redissolution in an isooctane/ethyl acetate mixture. Quantitation is by gas chromatography using a nitrogen specific TSD (NPD) detector.

III. EQUIPMENT

The following equipment is used in this analytical method. Specific brands are listed, but in most cases equivalent equipment obtained from other vendors can be used.

Gas Chromatograph - Varian 3700 equipped with TSD detector and Model 8000 Autosampler.

Glass Capillary Column - SPB-1, 0.75 mm ID, 1 µm film, 60 meters, Supelco catalog number 2-3720M.

Strip Chart Recorder - Recordall Series 5000, Fisher Scientific catalog number 13-939-20.

Data Aquisition System - A system having appropriate integration and graphics capability; The Monsanto Automated Chromatography System (MACS) was used.

Autosampler Vials - Varian catalog number 03-949835-01.

Analytical Balance - Sartorius AC210S.

Laboratory Balance - Sartorius LC2200S.

Rotary Evaporator System - Based upon Rinco Rotating Shaft Evaporator, Fisher Scientific catalog number 09-548-100; Kontes Vacuum Trap, Fisher K9269101000; Hot plate, Fisher HP-18325; Water Baths, Cole-Parmer L-07274-00; and Vacuum Pump, Fisher 01-096.

Bottle-Top Dispensers - Brinkmann Dispensette, Baxter Scientific catalog numbers: 10 mL, P4985-10; 50 mL, P4985-50; and 100 mL, P4985-100.

Glas-Col Benchtop Shaker - Baxter Scientific catalog number S1063-1.

Hexagonal Head, Glas-Col Shaker - Baxter Scientific catalog number S1063-10.

Separatory Funnel Holders, Glas-Col Shaker (2) - Baxter Scientific catalog number S1063-21.

Glass Powder Funnels - 80 mm Kimax, Baxter Scientific catalog number F7417-2.

Separatory Funnels - 500 mL Kimax, Baxter Scientific catalog number F7860-500.

Separatory Funnels - 250 mL Kimax, Baxter Scientific catalog number F7860-250.

Round Bottom Flasks - 500 mL Kimax, Fisher Scientific catalog number 10-067-2F.

Round Bottom Flasks - 250 mL Kimax, Fisher Scientific catalog number 10-067-2D.

Splash-Guard Adapter - Aldrich, 24/40, 250 mL, Aldrich catalog number Z14779-6.

SPE Vacuum Manifold - Supelco catalog number 5-7030M.

Teflon Solvent Guide Needles - Supelco catalog number 5-7047M.

SPE Columns - Florisil, 2 grams, P. J. Cobert catalog number 943904.

SPE Columns - Silica, 1 gram, P. J. Cobert catalog number 943900.

Magnetic Stirrers - Baxter Scientific catalog number S8275-1.

Magnetic Stirring Bars - Baxter Scientific catalog number S8311-72.

Graduated Cylinders - 10-1000 mL, Fisher Scientific catalog numbers 08-549-5B,C,D,E,G,H,J.

Erlenmeyer Flasks - 250 mL, Fisher Scientific catalog number 10-039F.

Disposable Pasteur Pipettes - 5.75 inch, Fisher Scientific catalog number 13-678-6A.

Disposable Pasteur Pipettes - 9 inch, Fisher Scientific catalog number 13-678-6B.

Volumetric Flasks - 100 mL, Fisher Scientific catalog number 10-210C.

Serological Disposable Pipettes - 0.1-10.0 mL, Fisher Scientific catalog numbers 13-676-28A,B,C,D,E,F, G.

Glass Wool - Pyrex, Fisher Scientific catalog number 11-388.

Teflon Wash Bottles - 500 mL, Baxter Scientific catalog number B7690-500.

Clamps, spatulas, tubing, and other supplies as required.

IV. REAGENTS AND SOLUTIONS

The following reagents are used in this analytical method. Specific brands are listed, but in most cases equivalent reagents from other vendors can be used. It is important to use high quality reagents to avoid chromatographic interferences.

Acetonitrile - Burdick & Jackson, High Purity, Baxter Scientific catalog number 015-4 DK.

Methylene Chloride - Burdick & Jackson, High Purity, Baxter Scientific catalog number 300-4 DK.

Methanol - Burdick & Jackson, High Purity, Baxter Scientific catalog number 230-4 DK.

Isooctane - Burdick & Jackson, High Purity, Baxter Scientific catalog number 362-4 DK.

Ethyl Acetate - Burdick & Jackson, High Purity, Baxter Scientific catalog number 100-4 DK.

Deionized Water - Burdick & Jackson, High Purity, Baxter Scientific catalog number 365-4 DK, or deionized water from Millipore Compact MQ+ system, catalog number ZD5211584.

Sodium Chloride - Certified, Fisher Scientific catalog number S271-500.

Anhydrous Sodium Sulfate - Certified, Fisher Scientific catalog number S421-500.

Potassium Carbonate - Certified, Fisher Scientific catalog number P208-500.

Sodium Phosphate (Dibasic) - AR Grade, Baxter Scientific catalog number 7914-500 NY

Sodium Phosphate (Monobasic) - AR Grade, Baster Scientific catalog number 7892-500 NY

75% Acetonitrile/25% water (V/V).

Saturated Aqueous Sodium Chloride Solution.

50% Methylene Chloride/50% Methanol (V/V).

85% Methylene Chloride/15% Methanol (V/V).

95% Methylene Chloride/5% Methanol (V/V).

Aqueous Potassium Carbonate Solution - 0.5 M.

95% Isooctane/5% Ethyl Acetate (V/V).

70% Isooctane/30% Ethyl Acetate (V/V).

1.0 M phosphate buffer pH = 7: (Dissolve 53.8 grams of sodium phosphate, monobasic, and 163.5 grams sodium phosphate, dibasic, in 1 liter of deionized water. Mix well to insure complete dissolution.)

MON 12000 - 1 H-Pyrazole-4-carboxylic acid, 3-chloro-[[[[(4,6-dimethoxy-2-pyrimidinyl-amino]carbonyl]amino]sulfonyl]-1-methyl, methyl ester of known analytical purity (If purity is less than 95%, purity correction should be made).

MON 5781 Rearrangement Ester (RRE) - 1 H Pyrazole-4-carboxylic acid, 3-chloro-5-[(4,6-dimethoxy-2-pyrimidinyl)amino]-1-methyl, methyl ester of known analytical purity (If purity is less than 95%, purity correction should be made).

V. PREPARATION OF ANALYTICAL STANDARDS

1. Fortification Spiking Solutions

MON 12000

Stock Fortification Standard - This standard is prepared by weighing 0.0100 ± 0.0003 g of MON 12000 into a 100 mL volumetric flask. The standard is diluted to the volumetric mark with acetonitrile. This stock solution contains 100 µg/mL of MON 12000.

MON 12000 Working Standards

1. Dilute 7.5 mL of the 100 µg/mL stock solution to 100 mL with acetonitrile in a volumetric flask to provide a 7.5 µg/mL solution of MON 12000.
2. Dilute 6.0 mL of the 100 µg/mL stock solution to 100 mL with acetonitrile in a volumetric flask to provide a 6.0 µg/mL solution of MON 12000.
3. Dilute 4.5 mL of the 100 µg/mL stock solution to 100 mL with acetonitrile in a volumetric flask to provide a 4.5 µg/mL solution of MON 12000.
4. Dilute 3.0 mL of the 100 µg/mL stock solution to 100 mL with acetonitrile in a volumetric flask to provide a 3.0 µg/mL solution of MON 12000.
5. Dilute 1.5 mL of the 100 µg/mL stock solution to 100 mL with acetonitrile in a volumetric flask to provide a 1.5 µg/mL solution of MON 12000.
6. Dilute 10 mL of the 1.5 µg/mL solution from 5 above to 100 mL with acetonitrile in a volumetric flask to provide a 0.15 µg/mL solution of MON 12000.

Store prepared standards, stock and working, in amber bottles at -10-0° F (See Section XI).

2. Detector Calibration Standards

Rearrangement Ester (RRE)

Stock Calibration Standard - This standard is prepared by weighing 0.01507 ± 0.0003 g of MON 5781 RRE into a 100 mL volumetric flask. The standard is diluted to the volumetric mark with isooctane. Mix well by repeated inversions and sonicate if necessary for complete dissolution of the RRE. This stock solution contains 200 µg/mL of the RRE expressed as MON 12000 equivalents. Ten mL of this solution is diluted to 100 mL with isooctane in a second volumetric flask to provide a stock solution containing 20 µg/mL in MON 12000 equivalents.

RRE Working Standards
(Concentrations expressed in MON 12000 Equivalents)

1. Dilute 5 mL of the 20 µg/mL stock solution to 100 mL with 98% isooctane/2% ethyl acetate in a volumetric flask to provide a 1.0 µg/mL solution of RRE.

2. Dilute 4 mL of the 20 µg/mL stock solution to 100 mL with 98% isooctane/2% ethyl acetate in a volumetric flask to provide a 0.8 µg/mL solution of RRE.

3. Dilute 2.5 mL of the 20 µg/mL stock solution to 100 mL with 98% isooctane/2% ethyl acetate in a volumetric flask to provide a 0.5 µg/mL solution of RRE.

4. Dilute 1.0 mL of the 20 µg/mL stock solution to 100 mL with 98% isooctane/2% ethyl acetate in a volumetric flask to provide a 0.2 µg/mL solution of RRE.

5. Dilute 0.75 mL of the 20 µg/mL stock solution to 100 mL with 98% isooctane/2% ethyl acetate in a volumetric flask to provide a 0.15 µg/mL solution of RRE.

6. Dilute 0.5 mL of the 20 µg/mL stock solution to 100 mL with 98% isooctane/2% ethyl acetate in a volumetric flask to provide a 0.10 µg/mL solution of RRE.

7. Dilute 10 mL of the 0.50 µg/mL solution (from 3 above) to 100 mL with 98% isooctane/2% ethyl acetate in a volumetric flask to provide a 0.05 µg/mL solution of RRE.

8. Dilute 10 mL of the 0.10 µg/mL solution (from 6 above) to 100 mL with 98%isooctane/2% ethyl acetate in a volumetric flask to provide a 0.01 µg/mL solution.

VI. ANALYTICAL PROCEDURE

1. Sample Preparation

Water samples are received and stored refrigerated until just prior to analysis. The water samples are thoroughly mixed by vigorous shaking before subsampling for analysis.

2. Sample Extraction

Weigh 300 ± 0.1 grams of the previously prepared water sample into a 500 mL separatory funnel. If fortification is required, pipette the appropriate fortification of MON 12000 directly into the water at this point (See Section XI). Add 5 mL of 1.0 M pH = 7 phosphate buffer plus 52 grams of soduim chloride to the sample. Stopper the funnel and shake manually to dissolve the sodium chloride. Add 150 mL of methylene chloride, stopper the funnel, and shake for five minutes at medium speed on a mechanical shaker. After allowing the phases to separate, the lower organic layer is filtered through an 80 mm powder funnel plugged with glass wool and containing 15 mL of anhydrous sodium sulfate. The dryed organic layer is collected in a 500 mL round bottom flask. The extraction is continued by shaking for five minutes with a second portion of 150 mL of methylene chloride, which is filtered through the same sodium sulfate into the same 500 mL round bottom flask. The sodium sulfate is rinsed three times with sparing amounts of methylene chloride and the rinses collected in the sample volume. The combined, dried methylene chloride solutions are evaporated to dryness at ambient temperature on the rotary evaporators and the residues each dissolved in 2.5 mL of 85% methylene chloride/15% methanol and treated as described in the following section.

3. Florisil SPE Column Treatment

Two gram Florisil SPE columns are conditioned by heating in the oven at about 110 degrees for 40 minutes. After cooling, the column is eluted (preconditioned) with 30 mL of 50%

methylene chloride/50% methanol, followed by 20 mL of methylene chloride. Ten mL of methylene chloride is added to the sample from section 3 and the sample is mixed well by swirling. The sample is placed on the column and is followed by 30 mL of methylene chloride and 15 mL of 95% methylene chloride/5% methanol rinses of the round bottom flask. All of the preceding solvents are discarded. The column is then eluted with 60 mL of 85% methylene chloride/15% methanol into a 250 mL round bottom flask and this fraction contains the analyte of interest (MON 12000). In the above column cleanup operation the column flow rates are adjusted to a reasonable rate at which drops are still visible and the solvent is allowed to just enter the column between solvent fractions (columns should not be allowed to run dry during this process). This solution is ready to be treated as described in the following section 4.

4. Conversion to Rearrangement Ester

To the solution from above (section 3) is added a magnetic stirring bar and 50 mL of 0.5 M potassium carbonate. The flask is loosely stoppered (not tightened) and the mixture is stirred briskly overnight. After stirring, the mixture is transferred to a 250 mL separatory funnel and the round bottom flask rinsed with two 20 mL portions of methylene chloride which are also added to the mixture in the separatory funnel. The lower organic layer is then drawn off into a 250 mL round bottom flask through 15 mL of anhydrous sodium sulfate in an 80 mm powder funnel plugged with glass wool. The aqueous solution is then extracted by shaking for 4 minutes with 40 mL of methylene chloride and the layers allowed to separate. The lower methylene chloride layer is filtered through the sodium sulfate into the methylene chloride solution already in the round bottom flask. The sodium sulfate is rinsed sparingly three times with methylene chloride from a wash bottle. The methylene chloride solution containing the analyte is then evaporated to dryness at ambient temperature on the rotary evaporator. The residue in the round bottom flask is dissolved in 5 mL of 95% isooctane/5% ethyl acetate and treated as described in section 5.

Stirring times from 2 hours to 64 hours have been found to give acceptable results for this conversion. Practically, this means that the samples may be stirred over a weekend without loss of recovery if efficient scheduling requires this.

5. Silica SPE Column Treatment

One gram silica SPE columns are preconditioned with solvents by eluting with 10 mL of 70% isooctane/30% ethyl acetate followed by 10 mL of isooctane. Following this, the sample from above section 5 is placed on the column and followed with 5 and 10 mL rinses of the flask with 95% isooctane/5% ethyl acetate. Solvents eluting to this point are discarded. A 250 mL round bottom flask is placed under the column and the column eluted with 40 mL of 70% isooctane/30% ethyl acetate. This fraction contains the analyte (rearrangement ester) and is prepared for gas chromatographic analysis as described in section 6.

6. Final Sample Preparation

The 70% isooctane/30% ethyl acetate fraction from above section 6 is evaporated just to dryness at ambient temperature on the rotary evaporator and the residue dissolved in 2 mL of 95% isooctane/5% ethyl acetate. Three mL of isooctane are added and the solution mixed well by swirling briskly. Portions of each sample solution are placed in autosampler vials and analyzed as described below in section 7. Dilutions are made as necessary to keep sample concentrations within the range of the detector calibration standards.

7. Sample Analysis

The samples are analyzed by gas chromatography using a nitrogen specific detector. Injections are made using an autosampler. Instrument operating parameters are provided below in section VII.

VII. INSTRUMENTATION

The analyte, RRE, is quantified by gas chromatography using a nitrogen specific detector (NPD or TSD). Details of the operating parameters are as follows:

Varian 3700 Gas Chromatograph/Model 8000 Autosampler.
Supelco SPB-1 Glass Capillary Column, 0.75 µm ID, 1 µm film, 60 meters.
Program: 150° C (1 min); 10° C/min to 280° C (15 min hold).
Injector Temperature: 250°C
Detector Temperature: 300° C

8 psi Ultra High Purity Nitrogen
4 µL Injection
10 x 16 Attenuation

These conditions are approximate and may vary from instrument to instrument. Similar, but different equipment or column conditions may be used, but may require modification of these parameters.

VIII. INTERFERENCES

Detailed interference studies have not been performed. No interference due to solvents or labware has been observed. Example chromatograms are presented in Appendix 1.

IX. CONFIRMATORY TECHNIQUES

No confirmatory method is currently available.

X. TIME REQUIRED FOR ANALYSIS

A set of 12 samples requires approximately 2.5 days from initial extraction to setting the samples up for gas chromatographic analysis.

XI. MODIFICATIONS OR POTENTIAL PROBLEMS

Control and fortified samples should be run in the same analytical set as treated samples. Fortification solutions of MON 12000 should be carefully monitored to ensure their stability and should be stored at -10 - 0° F with only small volumes removed as needed for immediate requirements. Fresh working standards should be prepared monthly. Stock solutions have been demonstrated to be stable for at least three months, when stored frozen and unopened. Since the fortification solutions are stored under freezer conditions, they must be allowed to warm to ambient temperature before fortifications are made. This may be done by pipetting an appropriate aliquot into a small glass vial and pipetting the fortification from this after temperature equilibration. The unused portion of fortification solution is discarded.

XII. METHOD OF CALCULATION

The amount of MON 5781 rearrangement ester (RRE) is determined based upon external standard calibration. A non-weighted linear least squares estimate of the calibration curve is used to calculate the amount of RRE in the unknowns. The response of any given sample must not exceed the response of the most concentrated standard. If this occurs, dilution of the sample will be necessary.

If detector calibration standard concentrations are expressed as rearrangement ester the amount of RRE determined must be converted to the equivalent amount of MON 12000 for reporting purposes. This is accomplished using the following equation:

$$\frac{\mu g \text{ RRE Found}}{\text{Sample Mass (g)}} \times 1.327 = \text{ppm MON 12000 in sample}$$

The conversion factor, 1.327, corrects for the difference in molecular weight between MON 12000, MW = 434.8, and RRE, MW = 327.7.

When the detector calibration standards are prepared as described in Section V, part 2, the result calculated directly (without use of the 1.327 factor) will be in ppm of MON 12000 parent. Results are reported as ppm of MON 12000 .

XIII. VALIDATION LIMITS OF METHOD

The limit of quantification has not been defined. The lowest level at which the method has been validated is a fortification level of 0.0005 ppm. Method validation data is presented in Appendix 2.

APPENDIX 1

Representative GC Chromatograms