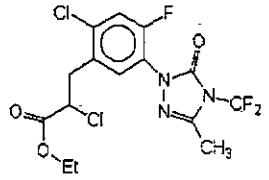
I. INTRODUCTION

F8426 is currently being developed by FMC Corporation for use in the control of broadleaf weeds, grasses and sedges in wheat, corn, and soybeans. Its common name is carfentrazone-ethyl. The chemical name of the active ingredient in F8426 is ethyl α, 2-dichloro-5-[4-(difluoro-methyl)-4,5-dihydro-3-methyl-5-oxo-1-*H*-1,2,4-triazol-1-yl]-4-fluorobenzenepropanoate. Its FMC Number is 116426, its CAS Number is 128639-02-1, and its EPA Registration Number is 128712.

F8426 has been shown to rapidly hydrolyze to F8426-chloropropionic acid (F8426-Cl-PAc) which further degrades to F8426-cinnamic acid (F8426-CAc), F8426-propionic acid (F8426-PAc), and F8426-benzoic acid (F8426-BAc) via aerobic soil metabolism. The chemical structure of F8426 is shown below:



Study Number 842E4194E1 was conducted to address the Environmental Protection Agency's (EPA) Pesticide Assessment Guidelines (Subdivision N) requirement for a Terrestrial Field Dissipation study (164-1). This is a report of the validation of the methodology used for the determination of residues of F8426 and its four major metabolites in/on soil utilizing methods reported herein.

II. SUMMARY

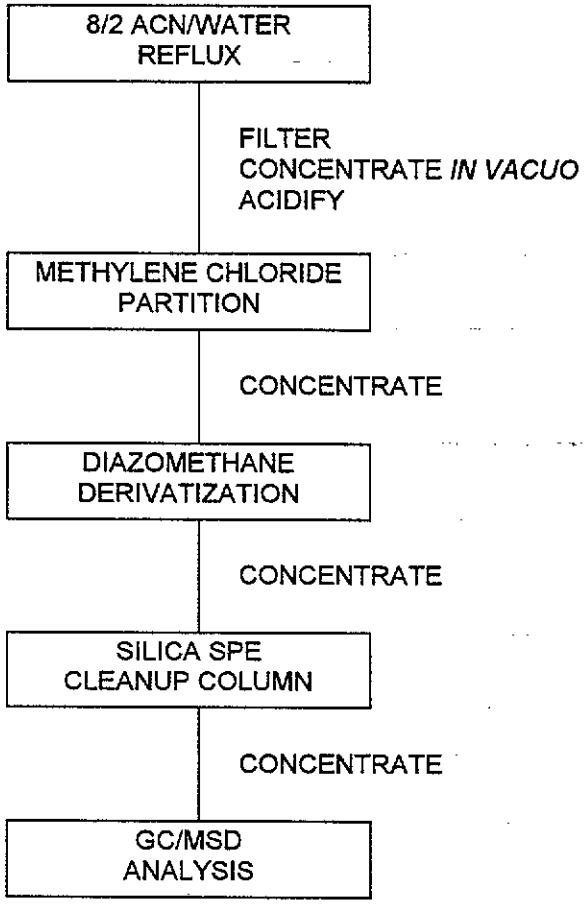
Three terrestrial field dissipation trials were conducted using F8426 50 DF in wheat, corn, and/or soybean-growing states of Kansas, Iowa and Minnesota. This report describes the analytical method developed for these studies. The analytical method has been refined and proven to be accurate and precise for the analytes of F8426 and its four major metabolites: F8426-chloropropionic acid (F8426-Cl-PAc), F8426-cinnamic acid (F8426-CAc), F8426-propionic acid (F8426-PAc), and F8426-benzoic acid (F8426-BAc).

For F8426 and its acid metabolite residue determinations, soil samples were subjected to an initial acetonitrile/water reflux extraction. The extract was concentrated *in vacuo* to remove the organic solvent. The remaining aqueous sample was acidified and partitioned with methylene chloride. An aliquot of the methylene chloride fraction was concentrated and derivatized with diazomethane/ether to methylate the acid metabolites. The derivatized sample was passed through a silica cartridge cleanup step and concentrated prior to analysis. Quantitation was achieved using capillary gas chromatography coupled with a mass selective detector (GC/MSD).

Method validation and recovery experiments were conducted with every set of samples for each of the five compounds. The analytical method was validated by fortifying and analyzing soil samples from the control plots, and monitoring the recoveries. A typical set of samples had nine samples, which included two treated plot samples in triplicate, two control plot samples, and one fortified control plot sample. Fortification levels ranged from 5 part(s) per billion (ppb) to 100 ppb for each of the five compounds. The limit of quantitation (LOQ), or lowest fortification level, for each compound was 5 ppb. The limit of detection (LOD) for each compound was estimated to be 1 ppb. Signal response less than LOD was considered non-detectable (ND).

B. Analytical Method Flow Schemes

Figure 1. Analytical Method Flow Scheme for F8426 and Metabolites.



IV. MATERIALS AND STUDY DESIGN

A. Test Substance

The active ingredient in F8426 50 DF herbicide is F8426. Its common name is carfentrazone-ethyl. The chemical name of the active ingredient in F8426 is ethyl α, 2-dichloro-5-[4-(difluoro-methyl)-4,5-dihydro-3-methyl-5-oxo-1-*H*-1,2,4-triazol-1-yl]-4-fluorobenzenepropanoate. Its FMC Number is 116426, its CAS Number is 128639-02-1, and its EPA Registration Number is 128712.

Technical standards of F8426, F8426-Cl-PAc, F8426-CAc, F8426-PAc, and F8426-BAc were used for fortification and quantitation. Refer to Table 6 for compound structures.

B. Test System

The surface soil from Trial Number 01 located in Finney County, Kansas, was classified according to USDA standards as sand. The surface soil from Trial Number 03 located in Polk County, Minnesota was likewise classified as silt loam.

C. Study Design and Procedures

1. Field Trial Design

The two field trials reported herein were conducted on no-till unplanted soil surfaces located in Kansas and Minnesota. Two test plots were used for each trial, one treated and one control.

For Trial Number 01, the top 0-10 cm depth soil cores measured 4.5 inches in diameter. Lower soil cores from 10 cm to 122 cm were 1.8 inches in diameter. For Trial Number 03, the top 0-10 cm depth soil cores measured 4.5 inches in diameter. Lower soil cores from 10 cm to 122 cm were 2 inches in diameter. For both trials, the lower cores were sectioned in 10 cm increments for the 10 cm to 90 cm depth resulting in eight 10 cm cores and a bottom 32 cm core containing the 90 cm to 122 cm section.

For the control plot, fifteen individual cores were taken and combined into a composite sample for each depth horizon.

The analytical method was validated by fortifying and analyzing soil samples from the control plots, and monitoring the recoveries. A typical set of samples had nine samples, which included two treated plot samples in triplicate, two control plot samples, and one fortified control plot sample. Fortification levels ranged from 5 ppb to 100 ppb for each of the five compounds. The limit of quantitation (LOQ), or lowest fortification level, for each compound was 5 ppb. The limit of detection (LOD) for each compound was estimated to be 1 ppb. Signal response less than LOD was considered non-detectable (ND). A summary of the method recovery is in Table 1. Individual method recovery data can be found in Table 2.

Generally, a set was considered validated when method recoveries are 60 - 120%. However, in some cases when one analyte has failed this criteria, the Study Director evaluated the circumstances, and the set was still considered acceptable.

2. Storage Interval

Soil extracts in DCM were stored at room-temperature for a maximum of 9 months, from the date of fortification and extraction to the date of analysis.

D. Dates

The dates of sampling, shipping, receipt, sample preparation, extraction, and analysis are shown in Table 5.

E. Equipment

The following equipment was utilized to conduct the methodology:

Analytical Balance, PM4800 and AT261, Mettler
Diazomethane generation apparatus, Macro Diazald® kit, Z10,025-0, Aldrich
Evaporator, N-Evap®, Organomation
Evaporator, rotary, Buchler Instruments
Evaporator, TurboVap LV, Zymark
Filter paper, GF/A 12.5 cm glass microfibre filters, Whatman
Processing equipment, food cutter and mixer, Model HCM450, Hobart
Refrigerated constant temperature circulator, Model 1155, VWR Scientific
Solid Phase Extraction (SPE) Processor, Baker SPE-12G, JT Baker
Syringes, N-series, various volumes, Hamilton
Standard laboratory glassware

F. Reagents

The following reagents (or equivalents) were used in this study. All solvents were pesticide analysis grade and/or free of contaminants:

Acetone, JT9006, JT Baker,
Acetonitrile, Omnisolv®, AX0155, EM Science
Antifoam B® Silicone Emulsion, JTB531-5, JT Baker
Carbitol® (Di-(ethylene glycol) ethyl ether), E455-0, Aldrich
Diazald®, D2,800-0, Aldrich
Dichloromethane, Omnisolv®, DX0831, EM Science
Ether, Anhydrous, JT9244, JT Baker
Ethyl Acetate, Omnisolv®, EX0241, EM Science
Hexane, Omnisolv®, HX0298, EM Science
Hydrochloric Acid, 0.25 N, JT5612, JT Baker
Potassium Hydroxide, pellets, JT3140, JT Baker
Sodium Chloride, granular, SX0420, EM Science
Sodium Sulfate, anhydrous, 12/60 mesh, JT Baker 3375-07
Solid Phase Extraction (SPE) columns, SI silica, 1g/6mL, AI-122560-08, Varian
Sulfuric Acid, concentrated, JT9681, JT Baker
Water, deionized, Barnstead PCS® System

V. ANALYTICAL PROCEDURE

A. Residue Method

The basic methodology for the analysis of F8426 and its soil metabolites was originally devised by FMC Corporation Europe (Reference 1). Slight modifications were made to the methodology and subsequently used in this study. Subsequent changes were documented in the Study File. F8426 and its soil metabolites are new compounds and therefore, throughout the laboratory experimental phase of this study, various modifications to address method problems such as low recovery of specific analytes, standards reliability, matrix enhancement, and interferences, were made.

The following is a summarized version of the analytical method utilized for the majority of the analyses, which encompasses all documented changes.

Sample Preparation:

Prior to sample workups, soil cores were prepared as follows in order to better facilitate homogeneous subsampling.

Homogenization of samples from Trial Number 01 was relatively easy due to its sandy properties. Samples were mixed by hand in its bag by shaking prior to subsampling. However, because of it larger sampling size, the 0-10 cm samples were more cumbersome to mix, and mechanical assistance was necessary. Frozen samples were homogenized by passing through a Hobart food cutter/mixer and returned to its bag. The equipment was rinsed with acetone between samples.

Homogenization of samples from Trial Number 03 was more difficult due to its clay-like properties. Samples were very cumbersome to mix, and mechanical assistance was necessary. Frozen samples were homogenized by passing through a Hobart food cutter/mixer and returned to its bag. The equipment was rinsed with acetone between samples. To prevent cross-contamination, samples

were prepared in the order of starting with the lower depths and working upwards.

Prior to any mechanical homogenization, the sample was inspected and any large stones suspected of interfering with the equipment were removed to prevent impelling.

Glassware Preparation:

Generously pre-rinse the 500 mL flat-bottom boiling flasks, condensers used for refluxing, and filtering equipment with 0.25 N HCl, followed by acetone to remove acid.

Diazomethane/Ether Preparation:

Diazomethane is not commercially available but can be generated in the laboratory. The following procedure was used to prepare diazomethane/ether utilizing the method detailed in Journal of Organic Chemistry, 1980 (Reference 2).

CAUTION: Diazomethane poses safety and health risks, and its use should be carefully evaluated (Reference 3 and 4).

Assemble the synthesis apparatus (Aldrich Macro Diazold® kit) inside the hood. Charge the reaction flask with 18 g of potassium hydroxide, 105 mL of Carbitol®, and 30 mL of ether. Fill the pocket of the condenser with dry ice and acetone. Also at this time, fill the acetone reservoir of the dry ice trap with dry ice pellets. Dissolve 64.2 g of Diazald® into 375 mL of ether. Add the resultant mixture to a separatory funnel feeding the three-necked flask.

Add a few pellets of dry ice to the water bath surrounding the 500 mL Erlenmeyer receiving flask. Allow the water bath to reach 1 - 4°F. Add several pellets of dry ice to the acetone bath surrounding the dry ice trap.

Heat the water bath beneath the reaction flask to ca. 60°C. Start stirring the mixture. Slowly introduce the Diazald® solution into the reaction flask, preferably using two hands. Open the stopcock from the condenser to the Erlenmeyer

flask. Allow the temperature of the reaction flask water bath to rise to 70 - 80°C while the Diazald® solution continues to drip.

When the condensate from the condenser is colorless, the heat source can be turned off. The baths should be allowed to return to room temperature.

The 500 mL Erlenmeyer receiving flask should be removed from the apparatus and the diazomethane mixture should be transfered to two 8 ounce glass, preferably plastic-coated, screw-capped bottles. The bottles should be stored in a freezer when not in use.

Allow any residual diazomethane left in the condenser and the reaction flask to dissipate. There should be no yellow color remaining. Disassemble the apparatus. Pour the contents of the reaction flask into the waste solvent stream. Rinse the flask thoroughly with acetone. Clean the apparatus components with soapy water. Rinse them with copious amounts of water, followed by a final rinsing with acetone. Wire brushes should not be used in cleaning because of etching and scratching. After components have dried, place them away safely where they cannot be damaged.

Sample Analysis:

REFLUX & FILTRATION

Weigh a subsample (typically 80 g) of soil into a weigh boat and transfer into a 500 mL flat-bottom boiling flask. To facilitate the transfer, the sample can be transferred to a funnel placed into a 500 mL flat-bottom boiling flask. Reflux solvent can be added to the sample to facilitate transfer into the 500 mL flat-bottom boiling flask.

For laboratory fortification sample, spike with the fortification standard by syringe directly onto the soil prior to adding reflux solvent.

For 80 g of soil, add 200 mL of acetonitrile/water 8/2 (v/v) to the boiling flask. Place on a hot plate under a cold water condenser. Manually

swirl the flask several times throughout the reflux period, or use magnetic stir bars to ensure adequate extraction. Reflux sample for one hour, and allow to cool for ca. fifteen minutes. Before removing the sample, rinse the condenser with ca. 5-10 mL of reflux solvent.

Pre-wet the vacuum filtering apparatus, including filter paper with reflux solvent. After refluxing, vacuum filter the sample through Whatman GF/A filter paper in a Buchner funnel into a vacuum flask. Rinse the 500 mL flat-bottom boiling flask and filter cake with ca. 50 mL of reflux solvent. Transfer the filtrate to a clean acid-rinsed 500 mL flat-bottom boiling flask.

CONCENTRATION (ROTOVAP)

Concentrate the sample on a roto-evaporator (water bath ca. 50 - 60°C) to ca. 30 mL to remove the acetonitrile. Monitor the evaporation and adjust the vacuum pressure carefully as flashing, or boiling over, may unexpectedly occur. Two to four drops of Antifoam B® may be adding to reduce flashing.

PARTITION

After roto-evaporation, bring the sample to ca. 50 mL using deionized water. Add 1 mL of 10% H_2SO_4. Transfer the acidified sample to a 250 mL separatory funnel, and extract the sample with 75 mL of methylene chloride (DCM). Add ca. 1-2 teaspoons of sodium chloride (NaCl) to aid in phase separation. Pass the organic phase through a bed of granular anhydrous sodium sulfate in a glass funnel with a glass wool plug into a 250 mL Phillips beaker. Extract the sample with another 75 mL of DCM, and pass the organic phase through the bed of granular anhydrous sodium sulfate combining with the solvent from the first extraction. Transfer the sample to a graduated cylinder, and adjust to final volume using DCM (typically 160 mL per 80 g soil). Transfer and store the sample in amber screw-cap bottles to prevent possible photodegradation.

CONCENTRATION

Transfer an aliquot (typically 40 mL, or 20 g soil equivalent) using a graduated cylinder to a Phillips beaker for concentration. To maximize derivatization efficiency, Phillips beakers should be 250 mL or 500 mL, using the same size glassware for all samples in the set.

Concentrate sample on a warm steam table (ca. 60 - 70°C) under a gentle stream of nitrogen *just* to dryness.

DERIVATIZATION

Add 5 mL of ethyl ether and 1 mL diazomethane/ether solution to sample and swirl the mixture. Allow sample to derivatize uncovered and undisturbed in fume hood for 30 minutes.

CONCENTRATION

Add 10 mL of hexane to sample and swirl. Concentrate sample on a steam table (ca. 60 -70°C) under a gentle stream of nitrogen to ca. 1 - 2 mL. Do not let sample go dry. Add 10 mL of hexane.

SILICA SPE CLEANUP

Using a vacuum extraction system, condition a silica gel SPE cartridge with ca. 10 mL of hexane. Do not let cartridge go dry. After attaching a 75 mL sample reservoir, pass the sample through the column. Once through, add 5 mL of 95/5 hexane/ethyl acetate (v/v) as a sample rinse. After rinsing, elute the cartridge with 10 mL 8/2 hexane/ethyl acetate (v/v) into a 13 mL graduated centrifuge tube. Flow rate should be ca. 40 mL/minute.

CONCENTRATION

Concentrate the sample under a gentle stream of nitrogen to less than 0.5 mL and bring to the appropriate final volume (typically 0.5 mL) with ethyl acetate for GC/MSD analysis.

GC ANALYSIS

Analyses for F8426, F8426-Cl-PAc, F8426-CAc, F8426-PAc, and F8426-BAc were performed on a Hewlett-Packard 5890 Series II gas chromatograph equipped with a HP 7673 Auto Sampler and a HP 5972 mass selective detector in Selected Ion Monitoring mode. A HP-5 (5% phenyl methyl crosslinked silicone) capillary column was used. Typical GC operating conditions are shown in Appendix A.

QUANTITATION STANDARDS

A control sample should be prepared with each set to serve as the "matrix standard" to reduce instrument-related enhancement.

After concentrating to *just* dryness and prior to derivatizing, spike the control sample with standard prepared in acetonitrile using a syringe (typical spiking volume should be less than 200 µL to avoid inhibiting derivatization). Carry "matrix standard" through remaining procedure. Typical final volume should be no greater than 2 mL to maintain adequate matrix effect. A "matrix standard" was typically prepared by derivatizing 160µL of a 10ng/µL standard solution. Final volume was adjusted to 2 mL to produce a 800 pg/µL standard. Serial dilutions were then made to produce standards ranging from 100 pg/µL to 400 pg/µL. To achieve greater accuracy and precision, all measurements for dilutions should be made using a pipet or syringe.

B. Instrumentation

Analyses for F8426, F8426-Cl-PAc, F8426-CAc, F8426-PAc, and F8426-BAc were performed on a Hewlett-Packard 5890 Series II gas chromatograph equipped with a HP 7673 Auto Sampler and a HP 5972 mass selective detector in Selected Ion Monitoring mode. A HP-5 (5% phenyl methyl crosslinked silicone) capillary column was used. Typical GC operating conditions are shown in Appendix A.

C. Method Validation and Quality Control

1. Experimental Design

Method validation and recovery experiments were conducted with most sets of samples for each of the five compounds. The analytical method was validated by fortifying and analyzing soil samples from the control plots, and monitoring the recoveries. A typical set of samples had nine samples, which included two treated plot samples in triplicate, two control plot samples, and one fortified control plot sample. Fortification levels ranged from 5 ppb to 100 ppb for each of the five ompounds. The limit of quantitation (LOQ), or lowest fortification level, for each compound was 5 ppb. The limit of detection (LOD) for each compound was estimated to be 1 ppb. Signal response less than LOD was considered non-detectable (ND). A summary of the method recovery is in Table 1. Individual method recovery data can be found in Table 2.

2. Preparation of Standards

Technical standards of F8426 and its metabolites are certified every two years for purity. Stock standard solutions were prepared from these technical standards by weighing a known amount of the technical material into a volumetric flask and diluting with acetonitrile. The resulting stock standard concentration was 1 µg/µL. A 10 ng/µL mixed standard was made from the stock solutions of the five compounds for the purpose of fortification and quantitation standard preparation using acetonitrile.

Quantitation standards were typically prepared with each set of samples by spiking a control sample just prior to derivatization, and carrying it through the rest of the procedure. Subsequent dilutions were made for GC analysis. The purpose of preparing the standard in soil matrix ("matrix standard") was to lessen matrix-related chromatographic enhancement. The

purpose for spiking the control sample prior to derivatization was to lessen matrix-related derivatization variability. Both phenomena were encountered during the beginning of the study. A "matrix standard" was typically prepared by derivatizing 160µL of a 10ng/µL standard solution. Final volume was adjusted to 2 mL to produce a 800 pg/µL standard. Serial dilutions were then made to produce standards ranging from 100 pg/µL to 400 pg/µL.

A complete inventory and detailed description of the technical standards and standard solutions used during this study, and pertinent information can be found in Tables 6 and 7.

3. Fortification Procedure

Control plot soil samples were fortified with an appropriate standard prior to the addition of reflux solvent. All fortifications were made using a microliter syringe (typical spiking volume was less than 1000 µL).

D. Method of Calculation

Quantitation of residues was based on a best-fit line equation generated from external standard injections at not less than three different levels.

Using a computer spreadsheet or calculator, a best-fit linear regression line was determined in the form of the standard equation: $y = mx + b$, where y is the area counts, x is the amount of analyte (pg), m is the slope, and b is the y-intercept.

Rearranging the equation gives: $(y - b)/m = x$, where x is the amount of analyte.

The residue amount can be calculated using:

amount of sample injected =

volume injected (µL) x sample concentration (mg/µL)

$$\text{residue amt (ppb)} = \frac{\text{amount of analyte (pg)}}{\text{amount of sample injected (mg)}}$$

The following example was derived from Set Number 3-28 for F8426-BAc. See Figures 4, 8, and 16 for examples of corresponding chromatograms.

Sample ID	Vol Inj'd (µL)	Amt Inj'd (pg)	Area Counts
100pg/µL STD	2	200	405765
200pg/µL STD	2	400	777534
200pg/µL STD	2	400	776024
400pg/µL STD	2	800	1445504

From the above data, a best-fit linear regression line was generated, where the independent variable (x) was the amount of standard injected, and the dependent variable (y) was the area count. The equation to the line was: $y = (1.720\times10^{3})x + 7.719\times10^{4}$.

For the fortified sample, 03-I-10C @ 20ppb for F8426-BAc, the following calculations were performed:

Sample ID	Vol Inj'd (µL)	Sample Conc (mg/µL)	Area Counts
03-I-10C @ 20ppb	2	20	1318718

$$\text{amt of analyte} = \frac{1318718 - 7.719\times10^{4}}{1.720\times10^{3}} = 721.18 \text{ pg}$$

$$\text{amt of sample inj'd} = 2\ \mu\text{L} \times 20\ \text{mg}/\mu\text{L} = 40\ \text{mg}$$

$$\text{residue amt} = \frac{721.18\ \text{pg}}{40\ \text{mg}} = 18.05\ \text{ppb}$$

To determine the percent recovery of the fortified samples, detectable residues found in the control sample (> 1 ppb) was subtracted from the residue amount found in the fortified samples, and divided by the theoretical fortification level.

The following example was derived from Set Number 3-28, fortified control sample from above and control sample 03-I-10C for F8426-BAc.

$$\%\ \text{Recovery} = \frac{18.05 - 0}{20} \times 100 = 90\%$$

E. Interferences

Interferences were encountered when analyzing F8426-CAc and F8426. Detectable residues (≥ 1 ppb) of F8426-CAc and F8426 were sporatically found in control samples. It was not found consistently upon repeated analyses and was determined to be an interfering residue. Utilizing the selectivity of the mass selective detector, a new ion was monitored (361 amu), along with the original ion (326 amu), to confirm the true presence of F8426-CAc. Using ion 361 amu showed no trace of the interfering residue. Quantitation was subsequently made using ion 361 amu. This modification led to the repeated analysis of only F8426-CAc for most upper horizon samples (0-20 cm layers), where detectable residues had been previously found.

An interfering residue for F8426 was also suspected, but was determined to be lab-incurred contamination. Detectable residues (≥ 1 ppb) of F8426 would be found in control samples and confirmed by mass selective detector. It was discovered that routine rinsing of glassware with 0.25 N HCl would remove the contamination.

F. Confirmatory Techniques

Prior to the initiation of the study, mass spectrums were obtained for the five analytes. For the study, analytes were quantitated using a selected ion based on the mass spectra. All analyses were performed using a gas chromatograph equipped with a mass selective detector in Selected Ion Mode. On some occasions, notibly during the repeated analyses for F8426-CAc, multiple ions were monitored to confirmed the true presence of the analyte.

G. Time Required for Analysis

The time required for an experienced analyst to work up a set of nine samples was about 12 hours. It took about five additional hours for the samples to be analyzed by gas chromatograph. It took about five hours to prepare ca. 500 mL of diazomethane/ether. Practical places for stopping

the sample work up are: 1) after Partition step, 2) after second Concentration step, 3) after Silica SPE Cleanup step, or 4) after third Concentration step.

H. Modifications or Potential Problems

F8426 and its soil metabolites are new compounds and therefore, throughout the laboratory experimental phase of this study, various modifications to address method problems such as low recovery of specific analytes, standards reliability, matrix enhancement, and interferences were made.

Glassware used in the Refluxing step should be rinsed with 0.25 N HCl in order to minimize the opportunity for laboratory-incurred contamination, notably F8426-CAc and F8426. Also, for certain soil types, notably the Minnesota trial, the absence of this acid pre-rinse is suspected of being linked to low F8426 recoveries.

Ensure the sample is sufficiently concentrated at the Concentration (Rotovap) step so that the acetonitrile is removed. Any residual acetonitrile will affect the extraction efficiency, most notibly F8426-BAc, in the following Partition step.

Derivatization should be done in 250 mL or 500 mL Phillips beakers. Use of smaller volume glassware was observed to decrease the derivatization efficiency of F8426-CAc. Also, typical spiking volume of the underivatized standard should be less than 200 µL to avoid inhibiting derivatization. Typical final volume should be no greater than 2 mL to maintain adequate matrix effect.

It may be necessary to change the injector liner (typically quartz 2 mm splitless packed with ca. 1 cm silanized glass wool) and/or remove a portion of the column front-end following analysis of surface (0-10cm) soils. The high organic matter content of the samples will char and coat the liner and/or column and is evident by a decrease in detector sensitivity.

Table 6. Reference Substance Table.

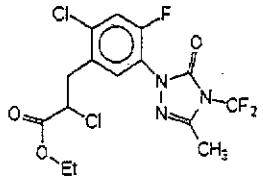
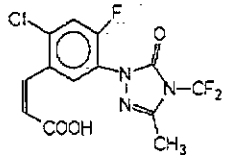
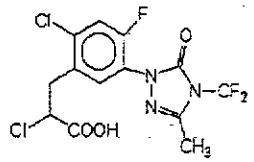
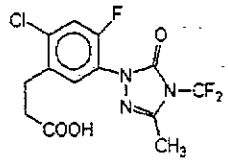
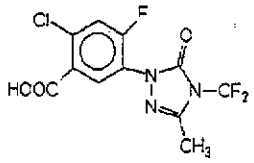
Common Name	Chemical Name/Structure	Code Number	Rich. Inv. No.	% Purity
F8426	Ethyl a, 2-dichloro-5-[4-(difluormethyl)-4,5-dihydro-3-methyl-5-oxo-1-H-1,2,4-triazol-1-yl]-4-fluorobenzene-propanoate 	FMC 116426	256 279	98.6 97.6
F8426-cinnamic acid (F8426-CAc)	2-Chloro-5-[4-(difluoromethyl)-4,5-dihydro-3-methyl-5-oxo-1-H-1,2,4-triazol-1-yl]-4-fluorobenzenepropenoic acid 	FMC 125151	258	99.0
F8426-chloroproprionic acid (F8426-Cl-PAc)	a, 2-Dichloro-5-[4-(difluoromethyl)-4,5-dihydro-3-methyl-5-oxo-1-H-1,2,4-triazol-1-yl]-4-fluorobenzenepropanoic acid 	FMC 124161	257 264	94.1 94.1
F8426-proprionic acid (F8426-PAc)	2-Chloro-5-[4-(difluoromethyl)-4,5-dihydro-3-methyl-5-oxo-1-H-1,2,4-triazol-1-yl]-4-fluorobenzenepropanoic acid 	FMC 125165	260 263	98.5 98.5

Table 6a. Reference Substance Table. (Continued)

Common Name	Chemical Name/Structure	Code Number	Rich. Inv. No.	% Purity
F8426-benzoic acid (F8426-BAc)	2-Chloro-5-[4-difluoromethyl)]-4,5-dihydro-3-methyl-5-oxo-1-H-1,2,4-triazol-1-yl]-4-fluorobenzoic acid 	FMC 97083	259 280	99.7 97

XII. APPENDICES

Appendix A. Instrument Parameters

The following are typical GC parameters and may be modified as necessary.

GC System: Hewlett-Packard 5890 Series II w/ Electronic Pressure Programming (EPP)
Hewlett-Packard 5972 Mass Selective Detector
Hewlett-Packard 7673 Automatic Liquid Sampler

Column: Hewlett-Packard HP-5, 5% Phenyl Methyl Crosslinked Silicone; 20-25 meter x 0.32 mm id x 0.52 um film thickness

Carrier Gas: Helium

Carrier Flow: 1.45 mL/min - 1.83 mL/min

EPP Pressure: 8.0 psi constant flow @ 185°C

Temperatures:
Injector: 250°C
Oven: 150°C hold 1 minute,
then increased at 25°C/min to 250°C,
then increased at 5°C/min to 260°C,
holding for 4 min,
then increased at 30°C/min to 295°C,
holding for 2.75 min.

Ions Monitored:

F8426-BAc:	335 amu	4.2-5.7 min RT
F8426-PAc:	303 amu	4.8-6.5 min RT
F8426-Cl-PAc:	326 amu	5.2-7.1 min RT
F8426-CAc:	326 or 361 amu	5.2-7.3 min RT
F8426:	312 or 330 amu	5.3-7.5 min RT