

Table 1. Distribution of FOE 5043 metabolites in plant matrices from crops grown in soil treated with [ $^{14}\text{C}$ ] FOE 5043. (Values in percent of total radioactive FOE 5043 residue).

<u>Crop Matrix</u>	<u>FOE 5043 Metabolites</u>					
	<u>Oxalate</u>	<u>Sulfonic Acid</u>	<u>Thioglycolate Sulfoxide</u>	<u>Methyl Sulfone</u>	<u>Methyl Sulfoxide</u>	<u>Thiolactate Sulfoxide</u>
Corn <sup>1</sup>						
Forage	44	7	10	1	1	10
Fodder	42	6	11	1	1	9
Soybeans						
Forage	15	40	19	5	2	0
Fresh Beans	0	0	39	4	2	0
Hay	16	40	17	5	4	0
Dry Beans	5	8	39	6	5	0

<sup>1</sup> The residues in corn kernels were too low (0.012 ppm) for identification of individual metabolites.

Table 2. Recovery of FOE 5043 and its metabolites from plant matrices.

<u>Crop Matrix</u>	<u>Compound</u>	<u>Ppm Fortification</u>	<u>Sample Recovery (%)</u>
Corn Forage <sup>1</sup>	FOE 5043	0.10	83/ 83
	FOE oxalate	0.10	72/ 78
	FOE sulfonic acid	0.10	75/ 64
	FOE thioglycolate sulfoxide	0.10	80/ 78
Corn Forage <sup>1</sup>	FOE 5043	0.05	91
	FOE oxalate	0.05	79
	FOE sulfonic acid	0.05	65
	FOE thioglycolate sulfoxide	0.05	73
Corn Grain <sup>2</sup>	FOE 5043	0.10	112/ 116
	FOE oxalate	0.10	79/ 82
	FOE sulfonic acid	0.10	78/ 91
	FOE thioglycolate sulfoxide	0.10	86/ 98
Corn Fodder <sup>3</sup>	Mixed standard	0.10	83/ 90
Corn Oil <sup>4</sup>	FOE 5043	0.10	83/ 86
	FOE oxalate	0.10	78/ 72
	FOE sulfonic acid	0.10	67/ 74
	FOE thioglycolate sulfoxide	0.10	76/ 77

<sup>1</sup> For summaries of the raw data and chromatograms, see Appendix 6. Control samples had residues of <0.05 ppm.

<sup>2</sup> For summaries of the raw data and chromatograms, see Appendix 7. Control samples had residues of <0.10 ppm.

<sup>3</sup> For summaries of the raw data and chromatograms, see Appendix 8. Control samples had residues of <0.10 ppm.

<sup>4</sup> For summaries of the raw data and chromatograms, see Appendix 9. Control samples had residues of <0.10 ppm.

<u>Crop Matrix</u>	<u>Compound</u>	<u>Ppm Fortification</u>	<u>Sample Recovery (%)</u>
Peanut Nutmeat <sup>5</sup>	Mixed Standard	0.10	78/ 72
Spinach Tops <sup>6</sup>	Mixed Standard	0.05	71/ 72
Soybean Seed <sup>7</sup>	FOE 5043	0.10	89/ 91
	FOE oxalate	0.10	89/ 80
	FOE sulfonic acid	0.10	74/ 85
	FOE thioglycolate sulfoxide	0.10	86/ 82
Soybean Forage <sup>8</sup>	FOE 5043	0.10	89/ 85
	FOE oxalate	0.10	85/ 83
	FOE sulfonic acid	0.10	74/ 74
	FOE thioglycolate sulfoxide	0.10	79/ 80
Soybean Soapstock <sup>9</sup>	Mixed standard	0.10	83/ 85
Sunflower Seed <sup>10</sup>	FOE 5043	0.10	94/ 95
	FOE oxalate	0.10	92/ 87
	FOE sulfonic acid	0.10	70/ 70
	FOE thioglycolate sulfoxide	0.10	93/ 75

<sup>5</sup> For summaries of the raw data and chromatograms, see Appendix 10. Control samples had residues of <0.10 ppm.

<sup>6</sup> For summaries of the raw data and chromatograms, see Appendix 11. Control samples had residues of <0.05 ppm.

<sup>7</sup> For summaries of the raw data and chromatograms, see Appendix 12. Control samples had residues of <0.10 ppm.

<sup>8</sup> For summaries of the raw data and chromatograms, see Appendix 13. Control samples had residues of <0.10 ppm.

<sup>9</sup> For summaries of the raw data and chromatograms, see Appendix 14. Control samples had residues of <0.10 ppm.

<sup>10</sup> For summaries of the raw data and chromatograms, see Appendix 15. Control samples had residues of <0.10 ppm.

Table 2 (cont.)

<u>Crop Matrix</u>	<u>Compound</u>	<u>Ppm Fortification</u>	<u>Sample Recovery (%)</u>
Turnip Roots <sup>11</sup>	Mixed standard	0.05	71/ 72
Wheat Grain <sup>12</sup>	FOE 5043	0.10	88/ 86
	FOE oxalate	0.10	87/ 85
	FOE sulfonic acid	0.10	73/ 65
	FOE thioglycolate sulfoxide	0.10	80/ 77
Wheat Grain <sup>12</sup>	Mixed standard	0.05	78/ 79
Wheat Straw <sup>13</sup>	Mixed standard	0.10	80/ 85

<sup>11</sup> For summaries of the raw data and chromatograms, see Appendix 16. Control samples had residues of <0.05 ppm.

<sup>12</sup> For summaries of the raw data and chromatograms, see Appendix 17. Control samples had residues of <0.05 ppm.

<sup>13</sup> For summaries of the raw data and chromatograms, see Appendix 18. Control samples had residues of <0.10 ppm.

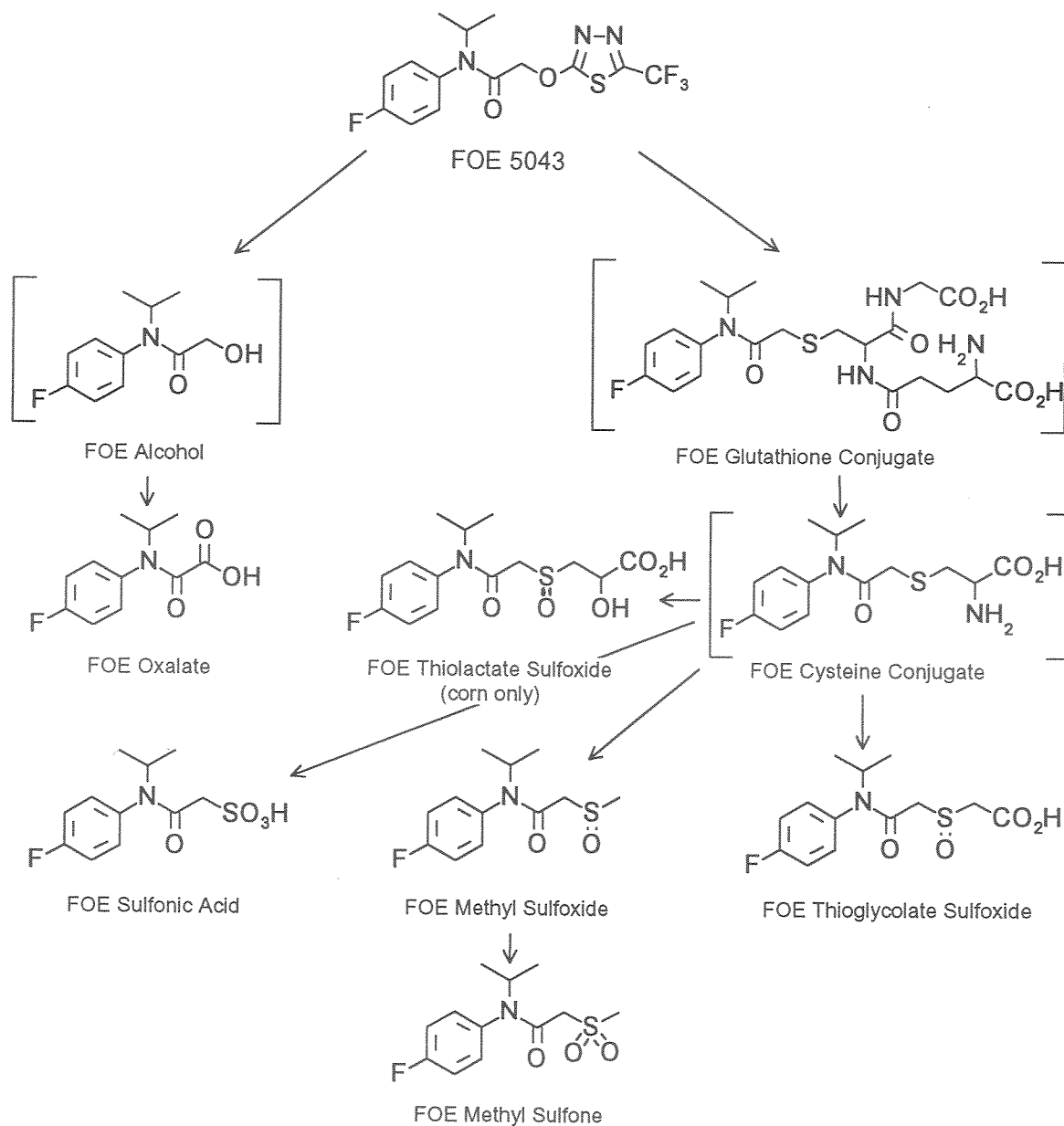


Figure 1. Plant metabolism of FOE 5043.

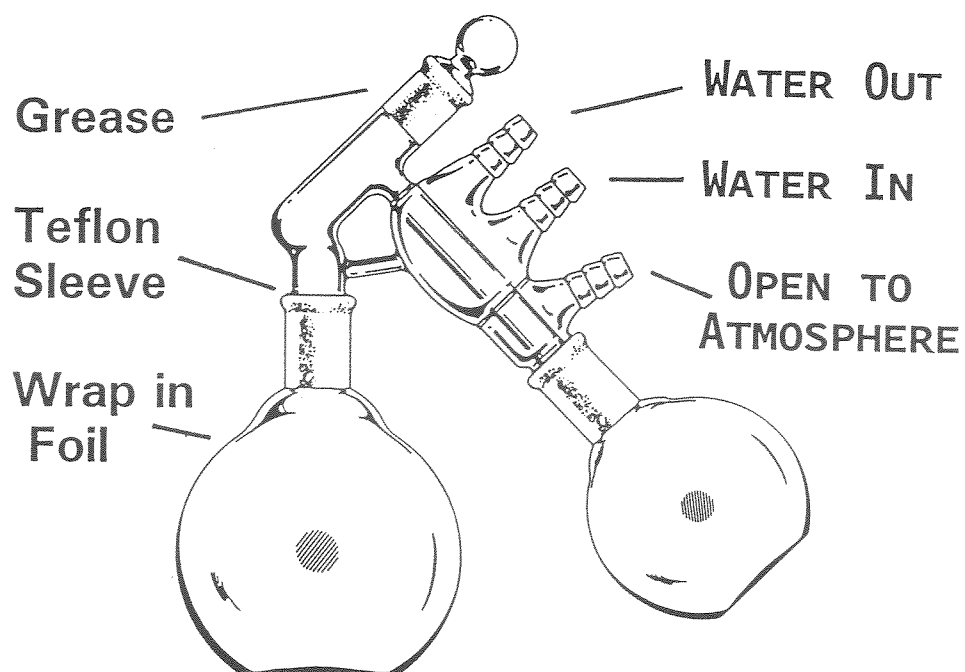


Figure 2. Short path distillation setup for steam distillation of the acid digest.

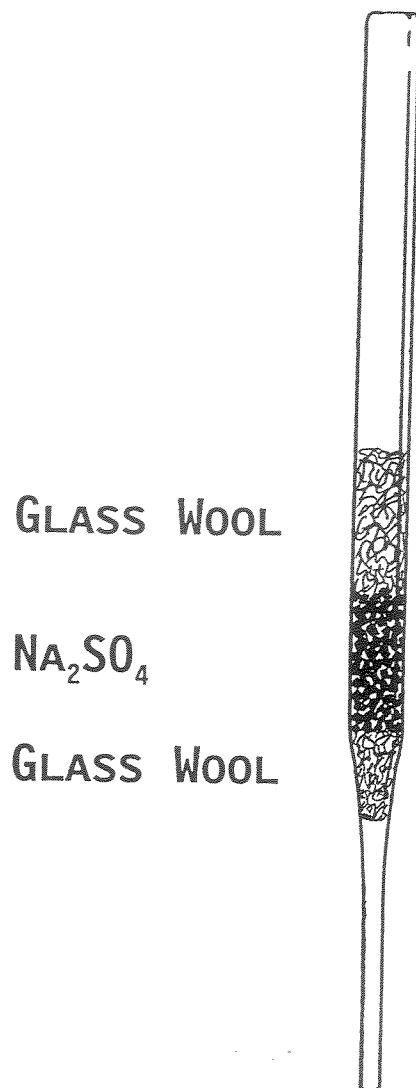


Figure 3. Drying tube setup for the drying of organic solutions. Preparation: Add a small plug of glass wool to a disposable Pasteur pipet. Add about 0.5 g of anhydrous sodium sulfate to the pipet. Add a loose plug of glass wool about 2 cm long to the pipet.

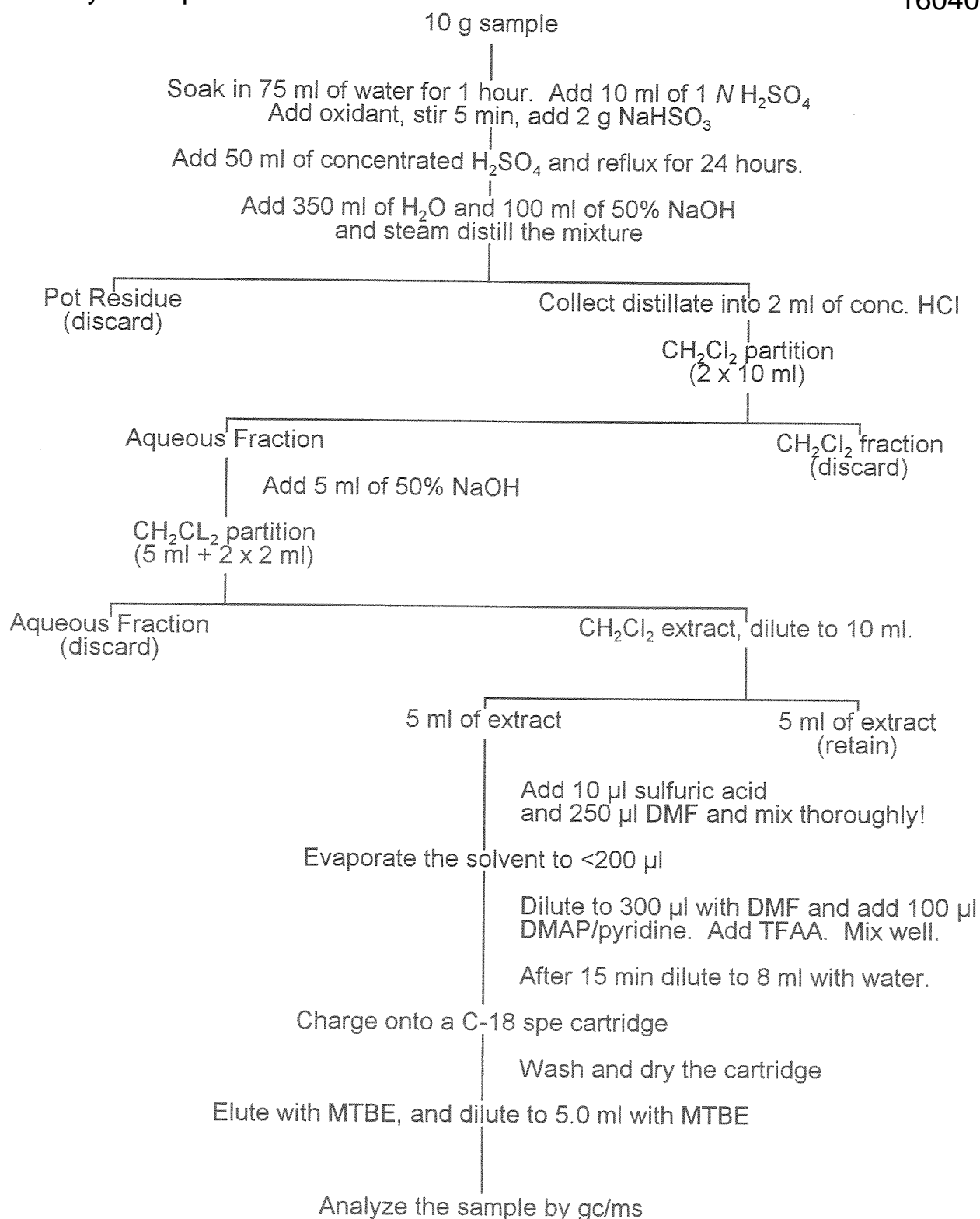


Figure 4. Flow diagram of the analytical residue method.

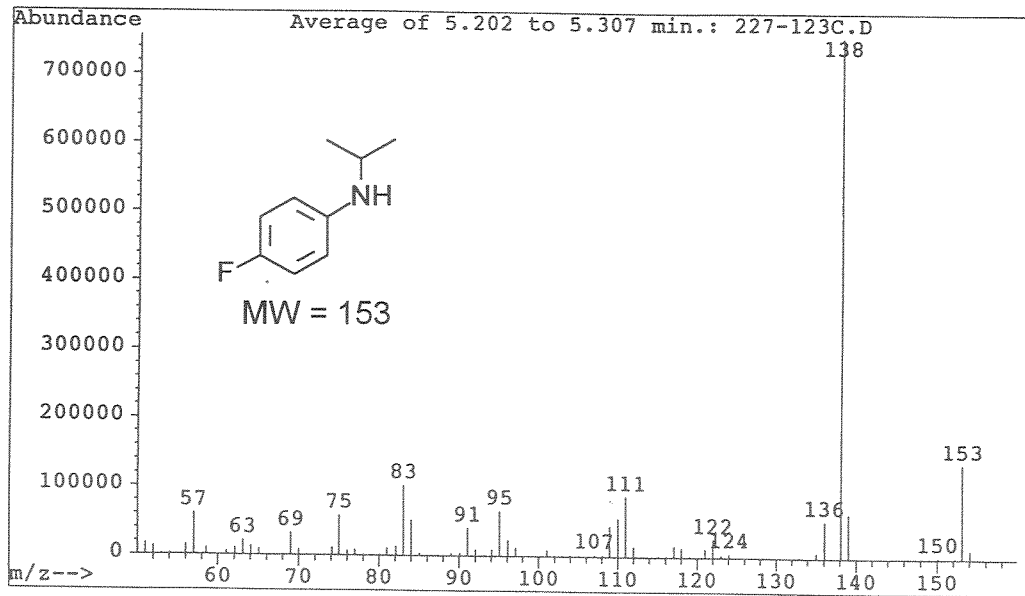
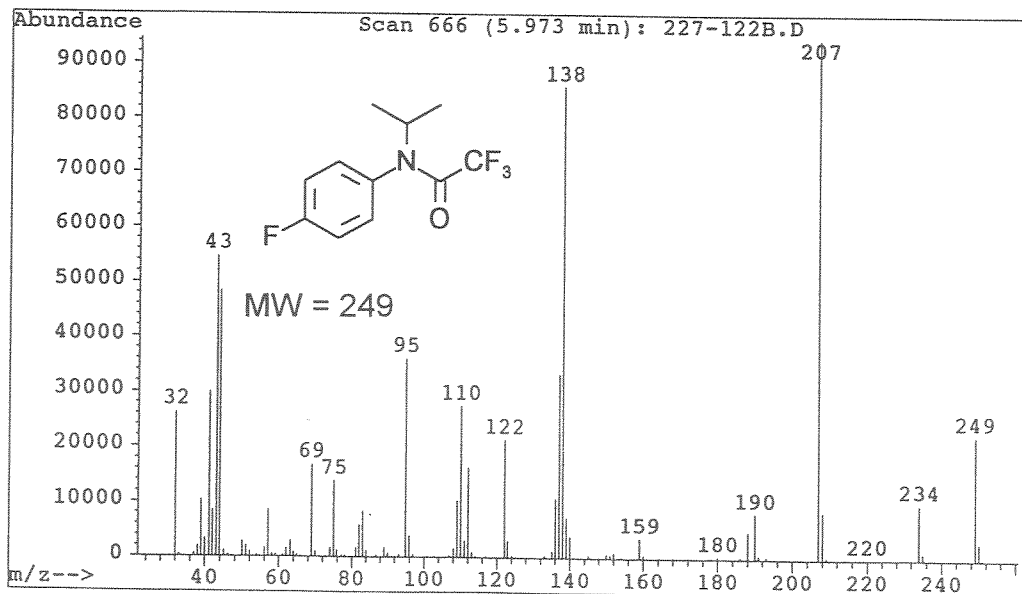
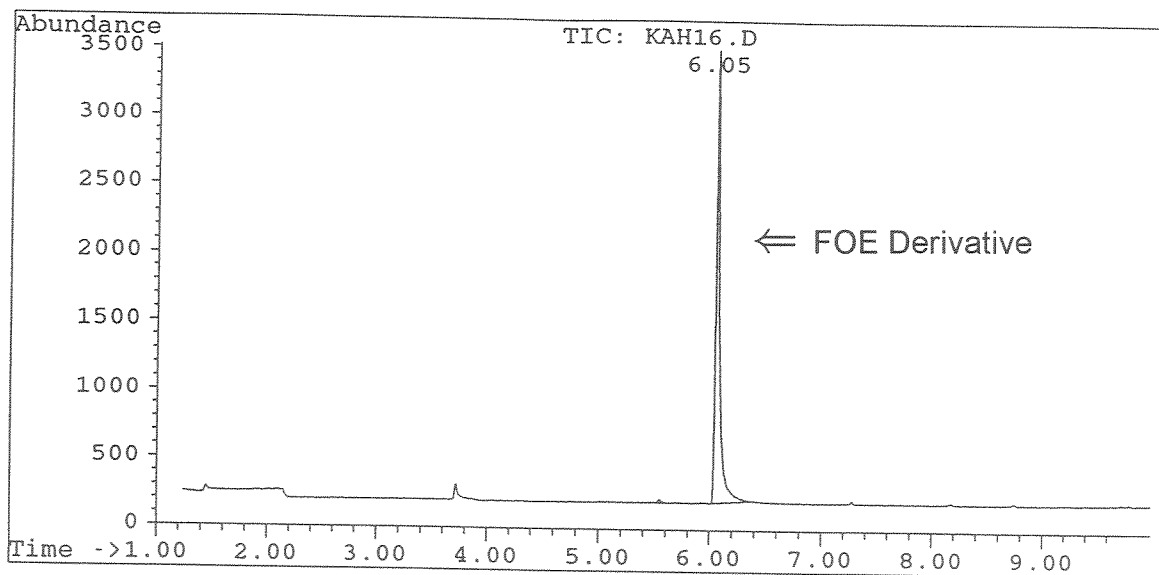
4-Fluoro-*N*-methylethyl benzenamine.4-fluoro-*N*-methylethyl benzenamine trifluoroacetamide.

Figure 5. Ei mass spectra of fluoroaniline and the trifluoroacetamide derivative.

A. Total ion chromatogram of the FOE trifluoroacetamide derivative.



B. Single ion chromatograms for m/z of 138, 207, and 249.

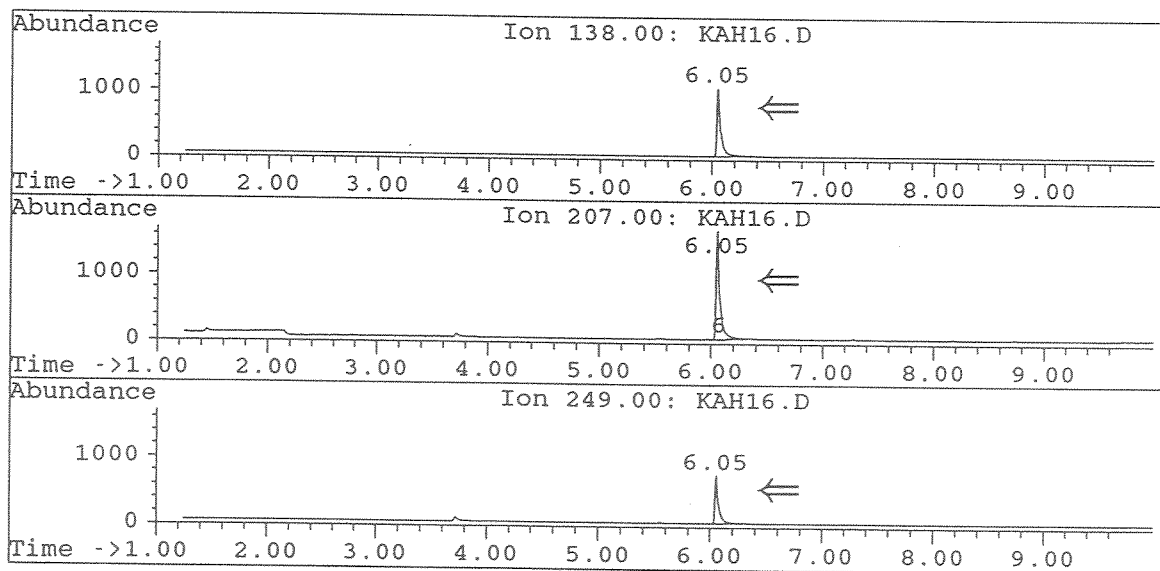


Figure 6. Gc/ms chromatogram of a 4-fluoro-N-methylethyl benzenamine trifluoroacetamide standard sample.

Appendix 1. Archive listing of notebook references and project personnel.

Notebook Reference

<u>Notebook Number</u>	<u>Name</u>	<u>Year Issued</u>	<u>Page Numbers</u>
92-B-145	V. J. Lemke T. J. Gould	1992	all pages
92-B-151	V. J. Lemke T. J. Gould K. L. Zoloty	1993	all pages
89-R-148	J. Morgan	1989	222, 271
93-B-8	J. Morgan	1993	172

Project Personnel

<u>Name</u>	<u>Duties</u>
T. J. Gould	Study director, generation and maintenance of raw data, extraction and preparation of tissues, chromatography, synthesis of trifluoroacetamide derivative, preparation of final report.
V. J. Lemke	Generation and maintenance of raw data, extraction and preparation of tissues, chromatography, generation of mass spectra, preparation of final report.
K. L. Zoloty	Generation of raw data, extraction and preparation of tissues.
J. Morgan	Synthesis of reference standards.

Appendix 2. Synthesis of 4-fluoro-*N*-methylethyl benzenamine trifluoroacetamide.

Procedure: To a solution of 4-fluoro-*N*-methylethyl benzenamine (10.0 g, 65.4 mmol) in methylene chloride (100 ml) contained in a 250 ml flask was added, with stirring, 0.2% (w/w) DMAP/pyridine solution (10.6 ml, 2.0 equiv.). The flask was cooled in an ice bath. After about 5 min, trifluoroacetic anhydride (10.2 ml, 1.1 equiv.) was added dropwise over a 15 min period. The ice was allowed to melt, and the mixture was stirred overnight at room temperature. The mixture was diluted with methylene chloride (150 ml), washed with 4 *N* HCl solution (2 x 50 ml), and then washed with saturated sodium bicarbonate solution (2 x 100 ml). The methylene chloride solution was filtered through a plug of glass wool and dried over anhydrous magnesium sulfate.

The methylene chloride solution of 4-fluoro-*N*-methylethyl benzenamine trifluoroacetamide was percolated through a bed of silica gel (6.5 cm x 3.0 cm) supported on a sintered glass frit (6.5 cm dia.). The silica gel was washed with methylene chloride (150 ml). The methylene chloride solvent was removed on the rotary vacuum evaporator, and the residual oil was distilled at reduced pressure (160-163°C at 75 torr) to give 22.7 g (91%) of a pale oil.

Spectral data: <sup>1</sup>H-NMR (300 MHz) δ 7.15-7.05 (m, 4H), 4.86 (sept, J = 6.8 Hz, 1H), 1.08 (d, J = 6.8 Hz, 6H); <sup>13</sup>C-NMR (75.4 MHz) δ 162.84 (d, J = 250 Hz), 156.47 (q, J = 34.7 Hz), 132.28 (d, J = 8.5 Hz), 130.76, 116.40 (q, J = 288.5 Hz), 115.80 (d, J = 23.2 Hz), 49.30, 20.20. <sup>19</sup>F-NMR δ (282.2 MHz) 126.63, 82.55.