QUANTITATIVE DETERMINATION OF DCPA AND ITS METABOLITES USING STONG ANION EXCHANGE SOLID-PHASE EXTRACTION WITH GAS CHROMATOGRAPHY/ELECTRON CAPTURE DETECTION

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ABSTRACT

Rapid and accurate analytical methods are needed to assess the impacts of agricultural chemicals on soil and water resources. Solid-phase extraction (SPE) was evaluated as an alternative method to conventional liquid-liquid extraction for the quantitative analysis of the herbicide DCPA and its metabolites in ground waters. Strong anion exchange (SAX) proved to be the most effective phase for isolating the highly water soluble mono- and dicarboxylic acid metabolites in addition to the hydrophobic parent compound, DCPA. Using a gas chromatograph equipped with electron capture detection, a high degree of accuracy and precision was achieved with spike recoveries of the diacid metabolite from surface waters of 80.1 ± 2.3 %. The detection limit of the method was found to be approximately 0.05 µg/L for a 100 mL water sample. The developed method was validated by comparing the measured concentrations of DCPA and its acid metabolites in ground waters to those measured using conventional liquid-liquid extraction. In a survey of wells from Malheur county of eastern Oregon, total concentrations of the acid metabolites of DCPA in ground water ranged from less than the detection limit (0.05 µg/L) to 198 µg/L.
INTRODUCTION

In the Malheur River Basin of eastern Oregon, the use of the herbicide DCPA (2,3,5,6-tetrachloroterephthalate), or Dacthal, for the production of onions has resulted in the contamination of a ground water aquifer. This is not surprising since in a recent national survey of groundwaters, DCPA was the most frequently detected pesticide (1). It is not the parent compound that is being detected in ground water, but the diacid metabolite which has a solubility in water of 5780 mg/L.

In the soil environment, DCPA is hydrolyzed to give the corresponding mono- and dicarboxylic acid metabolites. Primary degradation of DCPA to the monoacid metabolite is thought to be biologically mediated (2). Consequently, physical properties of the soil environment such as temperature, moisture content, and soil texture are variables that affect the degradation rates of DCPA (3). In a study utilizing three soil temperatures and three moisture contents, Choi et al (1988) found that soil temperatures of 25 to 30°C, and a soil moisture content of 0.2 kg H₂O kg⁻¹ soil, were the optimum conditions for degradation of DCPA. Under these favorable conditions, a half-life for the degradation of DCPA of 11 d was observed. A half-life of 105 d was estimated for the more unfavorable conditions of 10°C soil temperature and low (0.1 kg H₂O kg⁻¹ soil) soil moisture.

Hydrolysis of the monoacid metabolite occurs more rapidly than does the parent compound—a half-life of 2.8 d was observed for degradation of the monoacid in soils at room temperature and 50 % field capacity (4). While primary degradation of DCPA to the monoacid metabolite is assumed to be microbially mediated, a study performed by Wettasinghe and Tinsley (1993) showed that the monoacid metabolite was hydrolyzed at essentially the same rate in sterile and non-sterile soils, suggesting that hydrolysis of the monoacid metabolite could proceed by both biotic and abiotic processes. Because the monoacid is an anion under environmental conditions (estimated pKₐ using the Hammet equation < 3) (5,6), it is therefore more water soluble than the parent compound. It is likely that the increased solubility of the monoacid metabolite over the parent is responsible for the monoacid's ability to be hydrolyzed to the diacid in the absence of microbial activity.

To determine the occurrence and distribution of chemicals in soil and water environments, analytical methods must be accurate, precise, sensitive, selective, and rapid. Currently, concentrations of DCPA and its metabolites in ground water samples from Malheur county are determined by means of conventional liquid-liquid extraction (LLE) (EPA method 515). Unfortunately, the conventional LLE approach is a labor-intensive, time-consuming, and costly procedure that requires relatively large volumes of solvents (7,8).

Over the last seven years, solid-phase extraction (SPE) has become an established technique
for the preparation of samples for analysis (9). The modern technique of SPE began in 1978 with the commercial introduction of pre-packed cartridges (10). More recently however, a new technology of SPE has been introduced that uses the disk configuration of membrane filters (10). The SPE disks, manufactured by 3M under the trademark Empore, have high cross-sectional areas that provide advantages not found in the cartridges including decreased back pressure allowing higher flow rates, decreased plugging by particulates, improved mass transfer, and less sample channeling through the stationary phase (10).

An application that particularly concentrates on the strengths of membrane SPE, is the extraction (from water samples) for trace analytes such as pesticides. Extractions of 1 L samples in under 10 minutes, with recoveries and precision equivalent to those obtained by LLE, are possible using the SPE disks (10). The possible advantages of SPE over LLE include reduced manual labor, less time required for sample preparation, and much smaller volumes of solvent required for extraction. Another potential benefit of disk extractions is a cleaner extract which in turn may result in lower detection and quantitation limits (11,12).

This paper presents an alternative method to the conventional LLE method for the determination of DCPA and its metabolites in ground water samples. The strong anion exchange SPE combined with gas chromatography with electron capture detection methodology provides a simple, specific procedure for the quantitative determination of DCPA and its acid metabolites in ground waters at trace levels (ng/L).
EXPERIMENTAL SECTION

Apparatus. A six-port, vacuum-driven, solid-phase extraction manifold, was donated by 3M. For this study, C18 bonded phase silica, polystyrenedivinylbenzene copolymer (SDB), and strong anion exchange (SAX) disks were donated by 3M. The 47 mm disks consist of 8µm particles with 90 ± 2% adsorbent particle entrained into a matrix of inert PTFE.

Reagents. The diacid (2,3,5,6 tetrachloroterphthallic acid) standard (100% purity) was obtained from Ricera, Inc. A standard of the monoacid (1-methyl 2,3,5,6 tetrachloroterphthallic acid) (99.7% purity) was obtained from EPA Research Triangle Park, NC. A standard of DCPA (95% purity) was obtained from ChemService, West Chester, PA. The precursor reagents and procedures for making diazomethane and diazopropane were obtained from Aldrich. The dipropyl ester of DCPA (1,4-dipropyl 2,3,5,6-tetrachloroterphthallic acid), which is the internal standard, was prepared from diacid standard using diazopropane as the derivatization reagent. Derivatization of the diacid and monoacid metabolites for this study was performed using diazomethane. CAUTION: Precursors to diazomethane and diazopropane are carcinogenic. Diazomethane and diazopropane are explosive alkylationg reagents. All procedures and work using these reagents should be done while wearing proper laboratory clothing and in a laboratory hood.

Samples. Ground water samples used for the survey were collected from Malheur county of eastern Oregon by the Oregon Department of Agriculture (ODA). Samples were collected in baked glass bottles with aluminum-lined lids and neither filtered nor preserved. All samples were stored in dark refrigeration at 40 °F and analyzed within two weeks of sampling date. A surface water, known not to contain DCPA or its metabolites, was obtained as grab samples from Oak Creek in Corvallis, OR and used for spike and recovery experiments.

Strong Anion Exchange. The first step in the extraction scheme is the pre-conditioning of the solid-phase extraction resin. A 10 mL aliquot of acetone is first passed through the disk to expand the disk matrix and to elute potential interferences. The disk is then air dried using vacuum suction. Uniform wetting of the disk is accomplished by adding a 10 mL aliquot of 1 N HCl/MeOH. In addition, the 1 N HCl/MeOH ensures that the SAX sites are occupied by chloride ions which have a relatively low affinity for the SAX phase (13). At this point, the disk should be kept wet and not allowed to dry until the sample has been fully extracted. Internal drying of the hydrophobic resin
could cause inconsistent recoveries. Once the disk is wet, 2x10 mL washes of deionized water are passed through the disk to eliminate any residual methanol. The two water rinses ensure that the methanol has been thoroughly rinsed from the disk so that it does not interfere with extraction of the analyte.

After completion of the pre-conditioning steps, the sample is added to the reservoir and pulled through the disk at full vacuum (approximately 100 mL per minute). The sample eluant is discarded. The disks are then air dried for one minute using vacuum suction.

Sample elution takes place in two steps so that the parent compound, DCPA, can be fractionated from the monoacid and diacid metabolites. A 7 mL aliquot followed by a 5 mL aliquot of MeOH is used to elute DCPA into a 40 mL sample vial--using the first aliquot also as a sample bottle wash. Each aliquot of eluting solvent is allowed to soak into the disk for one minute before being pulled through by vacuum. Using MeOH alone, only the DCPA fraction is eluted since MeOH does not interfere with the electrostatic interactions between the acid metabolites and the anion exchange sites of the disk. Three grams of granular anhydrous sodium sulfate are added to the eluate (to soak up any retained water) and the eluate evaporated (not to dryness) using a heated plate at 60°C under a stream of dry nitrogen gas. The sample is then brought back to approximately 1 mL in ethyl acetate and spiked with 1.2 µg of the dipropyl ester internal standard.

The monoacid and diacid metabolites are eluted together into a 40 mL vial using 4x4 mL aliquots of 10 % by volume concentrated H₂SO₄ in MeOH. For each elution step, the eluting solvents are vacuum-pulled into the disk (until a drip can be seen at the funnel tip) and allowed to soak in the disk for one minute before being pulled through by vacuum. The collected eluate is then evaporated to approximately 3 mL using a heated plate at 70°C under a stream of dry nitrogen gas. A 10 mL aliquot of deionized water is then added to the vial. Next, a liquid-liquid micro-extraction is performed using a 6 mL aliquot of diethyl ether followed by two additional extractions, each using 4 mL of diethyl ether. Adding the water is essential so that two distinct phases form for the liquid-liquid micro-extraction. The ether fraction is transferred to a clean vial then derivatized using 1 mL of diazomethane. Once derivatized, the sample is then evaporated to near dryness using a stream of dry nitrogen gas, diluted to 1 mL in ethyl acetate, and spiked with 1.2 µg of the dipropyl ester internal standard.

**Breakthrough Experiments.** To assess the potential for analyte breakthrough during sample extraction, two types of experiments were conducted. In the first experiment, breakthrough was
determined by passing 100 mL samples spiked with the diacid metabolite through a single disk and performing LLE on the collected eluent. Sample water that had passed through the disks was collected and transferred to a 250 mL separatory funnel. A 3x40 mL diethyl ether LLE of the collected eluent was performed and the water fraction discarded. The ether fraction was concentrated to 5 mL using a steam bath, derivatized with 1 mL of diazomethane, and spiked with 1.2 µg of the dipropyl ester internal standard.

In the second type of experiment, breakthrough was determined by passing 100 mL samples, spiked with diacid metabolite, through two disks that had been stacked one on top of the other in the extraction apparatus. After sample extraction, the two disks were separated and eluted individually. The samples were then treated prior to analysis as described above.

**Spike and Recovery Procedures.** A series of spike and recovery experiments were performed, using both deionized water and Oak Creek surface water as matrices, to determine the accuracy and precision of the SAX isolation procedure. Spike recoveries of the diacid metabolite were evaluated at two concentrations (Table II). A set of five replicate samples (n=5) of 500 mL were prepared with a diacid concentration of 0.098 µg/L. Spike recoveries (n=5) at a more environmentally relevant concentration (38 µg/L in 100 mL) were also performed.

Although the diacid metabolite is the dominant species in ground water from the Malheur county survey site, the monoacid metabolite and DCPA may be present in other environmental samples. To test the potential for the method to fractionate DCPA from its acid metabolites, spike recoveries were performed using 100 mL deionized water samples containing 38 µg/L of the diacid metabolite, 29.3 µg/L of the monoacid metabolite, and 25.2 µg/L of DCPA.

**Chromatographic Analysis.** Analyses were performed using an HP 5890 series II gas chromatograph (GC). Chromatographic separation was performed using an SE 54 column (30 m x 0.2 mm internal diameter x 0.32 µm film thickness). The oven temperature program consisted of an initial temperature of 130°C held for 3 min, followed by a 10°C/min ramp to 200°C, followed by a 4°C/min ramp to 240°C, followed lastly by a 25°C/min ramp to a final temperature of 270°C, which was then held for 3 min. Injection conditions included an injector temperature of 260°C and splitless injection with an injection volume of 1 µL. Detection was performed with a 63Ni electron capture detector (ECD) at a temperature of 300°C.
**Quantitation.** Quantitation of analyte concentrations was performed using conventional internal standard gas chromatography calibration techniques. A known amount of the dipropyl ester internal standard, was spiked into each sample at the end of the isolation and concentration procedures for quantitation purposes. The dipropyl ester of DCPA was chosen as the internal standard for two reasons: (1) it does not occur in commercial products of DCPA and therefore will not be found in environmental samples, and (2) chromatographic behavior is expected to be similar to the dimethyl ester, DCPA, due to the structural similarity of the two compounds.

Calibration curves were constructed using the diacid metabolite and the dipropyl ester internal standard. Diacid metabolite was spiked into 16 mL of 10% by volume concentrated H$_2$SO$_4$ in MeOH eluting solvent and treated as described in the isolation procedure. Concentrations of DCPA and the monoacid metabolite were determined from calibration curves made from the diacid metabolite standards.

Two calibration curves were constructed for two different ranges of diacid metabolite concentrations in order to maintain detector linearity over the wide range of analyte concentrations chosen. The low concentration curve covered a diacid metabolite concentration range of 7 to 200 pg/µL injected onto the column. For the low concentration curve, the dipropyl ester internal standard was spiked at a level of 60 pg/µL. The high concentration curve covered a diacid metabolite concentration range of 200 to 38,000 pg/µL injected onto the column. The internal standard was spiked at a level of 1200 pg/µL for the high curve.
RESULTS AND DISCUSSION

Solid-Phase Extraction. Three different types of disk phases were initially evaluated to determine the most suitable phase for the efficient recovery of the diacid metabolite. The three resins examined in this study were the strong anion exchange (SAX), polystyrene-divinylbenzene copolymer (SDB), and the C18 bonded phase silica. Unlike SAX which requires no sample preparation, sample extractions using the C18 and SDB phases require that the samples be acidified to pH 2 to ensure that the acid analytes are in their non-ionic forms and thus more likely to be retained by the hydrophobic resin. For the initial experiments, the C18 and SDB resins were evaluated using a modified version of the Empore™ suggested application procedure. Deionized water samples were spiked with the diacid metabolite, acidified to pH 2 using concentrated HCl, amended with 10 g of Na₂SO₄, and extracted using 47 mm disks. Sample water that had passed through the disks was collected, and breakthrough determined by LLE. Elution of the disks was performed using 3x5 mL of dichloromethane followed by a 7 mL aliquot of ethyl acetate. The ether extract from the LLE of passed sample water and the collected eluent were derivatized with diazomethane and spiked with the dipropyl ester internal standard.

Initial experiments of extractions with the three phases indicated that the C18 and SDB disks did not quantitatively isolate the diacid metabolite (Table I). Average recovery of duplicate samples for the C18 disk was 0 % while average breakthrough, as determined by LLE, was 95%. Average recovery and breakthrough of duplicate samples for the SDB resin was determined to be 63% and 43%, respectively. The SAX resin gave 85% recovery of the diacid metabolite, with no breakthrough detected as determined by both LLE and a double disk experiment.

Data from both breakthrough experiments verified that the SAX resin had quantitatively retained all the diacid analyte, suggesting that the remaining 15% of the mass was being retained by the disk. Increasing the total volume of eluting solvent to 32 mL resulted in a recovery of 95 %, thus accounting for most of the remaining mass. The diacid metabolite's strong affinity for the SAX resin is probably due to two main factors. First, the diacid is a di-anion with two possible bonding sites per molecule. It is therefore possible that the diacid forms a bridge between two exchange sites on the SAX resin. Second, the SAX disk is composed of a highly cross-linked styrene-divinylbenzene copolymer. Retention of the diacid is therefore probably due to hydrophobic interactions with the SDB backbone as well as the ionic interactions at the exchange sites. The increased elution volume (32 mL) required to achieve 95% recovery of the diacid was considered impractical because of the increased handling difficulty, solvent volume, and time required for analysis.
Quantitation and Detection. For purposes of this study, an environmental surface water (Oak Creek) that was shown to contain no native DCPA or its metabolites was used for detection and quantitation studies. The detection limit of the method, based on a signal-to-noise ratio of three (S/N≥3), was found to be approximately 5 pg/µL injected, which corresponds to a concentration of 0.05 µg/L in a 100 mL sample. The quantitation limit (S/N≥10) was found to be approximately 0.14 µg/L for a 100 mL sample.

Recovery and Precision. Average recoveries of the diacid metabolite in samples of deionized water and Oak Creek surface water at 38 µg/L were 81.1% and 80.1%, respectively. The average relative standard deviation (n=5) for the spiked recoveries in deionized water and Oak Creek at 38 µg/L was found to be 1.1% and 2.9%, respectively. Although it was known that concentrations of the diacid metabolite of DCPA in ground waters of the Malheur sampling site were much higher, a five replicate set of spike recoveries were performed at a concentration of 0.098 µg/L in 500 mL of Oak Creek surface water. Average recovery for the set was determined to be 84.5% with a relative standard deviation of 1.5%.

Spike recoveries of the combined parent, monoacid, and diacid metabolites all spiked into single samples of deionized water were also performed (Table III). The average recovery of DCPA in the first fraction was 85.8% while recovery of the combined monoacid and diacid metabolites was 83.9%. The average relative standard deviation (n=5) for the DCPA fraction and the combined monoacid and diacid fraction was found to be 2.8% and 2.9%, respectively.

Application to Environmental Samples. The SAX/SPE with GC/ECD method described in the experimental section of this paper was used to quantitatively determine the concentrations of DCPA and its metabolites in ground water sampled from the Malheur River Basin in eastern Oregon. Shown in Figure 1 is an example of a typical chromatogram of a ground water sample, which contains 58.0 µg/L of the acid metabolite(s) of DCPA.

Eight individual ground water samples and one composite sample (prepared by combining five individual samples) from the Malheur sampling site were split and analyzed by both the SAX/SPE method and the conventional LLE method currently used by ODA. For each procedure, a set of n=4 replicates was performed on the composite sample so that variability could be assessed. The other eight samples were performed in duplicate for the SAX/SPE method and as singles for the LLE method.
In order to compare the SAX/SPE method to the LLE method, the concentrations reported (Table IV) were corrected based on spike recoveries of 80.1% for the SAX/SPE method and 88% for the LLE method. The relative standard deviations determined from the composite sample were 3.1% for the SAX/SPE method and 2.9% for LLE. Concentrations of the acid metabolites of DCPA ranged from below detection (0.05 µg/L) to 198 µg/L as determined by the SAX/SPE method and from below detection (1µg/L) to 203 µg/L by LLE. None of the parent compound (DCPA) was detected in any of the ground water samples by the SAX/SPE method.
CONCLUSIONS

The analytical method presented in this paper is capable of accurately isolating and quantitating DCPA and its metabolites from ground waters. Strong anion exchange proved to be the only phase capable of quantitatively isolating the water soluble acid metabolites of DCPA. The reproducibility for the overall isolation and chromatographic procedures, expressed in terms of the relative standard deviation was found to be 2.9%. The average recovery of the diacid metabolite was 80.1 ± 2.3%. The detection limit based on a signal-to-noise ratio of 3:1 is 5 pg per µL injected, which corresponds to a detection limit of 0.05 µg/L for a 100 mL sample.

The developed method was used to quantitatively determine the concentrations of the acid metabolites of DCPA in a survey of ground water samples. The SAX/SPE method was validated by comparing measured concentrations of the acid metabolites from ground water samples to those obtained using conventional liquid-liquid extraction. Although the method is capable of distinguishing between the parent compound and the acid metabolites, DCPA was not detected in any of the ground water samples.

While the SAX/SPE method did not reduce sample preparation time, total solvent volumes required were decreased from 450 mL of diethyl ether for the LLE method to less than 65 mL (primarily methanol) for the SAX/SPE method. Although no DCPA was detected in the ground water samples, the ability to fractionate between the parent compound and the acid metabolites may prove useful in studies of other matrices such as surface waters.

ACKNOWLEDGMENTS

The author acknowledges the Oregon Department of Agriculture for providing the ground water samples; Craig Markell, Sue Price, and Steve StMary of 3M for the extraction apparatus, the 47 mm extraction disks, and their technical support; Eugene Johnson and Bobby Loper of the OSU pesticide residue laboratory for their help and guidance.
Table I. Evaluation of C18, SDB, and SAX Solid-Phase Extraction Resins.

<table>
<thead>
<tr>
<th></th>
<th>C18&lt;sup&gt;b&lt;/sup&gt;</th>
<th>SDB&lt;sup&gt;b&lt;/sup&gt;</th>
<th>SAX</th>
</tr>
</thead>
<tbody>
<tr>
<td>Recovered from Disk (%)</td>
<td>0</td>
<td>63</td>
<td>85</td>
</tr>
<tr>
<td>Breakthrough&lt;sup&gt;a&lt;/sup&gt; (%)</td>
<td>95</td>
<td>43</td>
<td>0</td>
</tr>
</tbody>
</table>

<sup>a</sup>breakthrough measured by liquid-liquid extraction  
<sup>b</sup>duplicate samples performed
Table II. Spike Recoveries of the Diacid Metabolite.

<table>
<thead>
<tr>
<th>Spike Level (μg/L)</th>
<th>Sample size (mL)</th>
<th>Deionized water</th>
<th>Oak Creek</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.098</td>
<td>500</td>
<td>------</td>
<td>84.5 ± 1.3% (RSD=1.5%)</td>
</tr>
<tr>
<td>38</td>
<td>100</td>
<td>81.1 ± 0.9% (RSD=1.1%)</td>
<td>80.1 ± 2.3% (RSD=2.9%)</td>
</tr>
</tbody>
</table>

*recoveries and relative standard deviation based on (n=5) replicates*
Table III. Spike Recoveries for DCPA, Monoacid, and Diacid.

<table>
<thead>
<tr>
<th>Spike Level (μg/L)</th>
<th>Sample size (mL)</th>
<th>Recovery (%)(^b)</th>
</tr>
</thead>
<tbody>
<tr>
<td>DCPA</td>
<td>25.2</td>
<td>85.8 ± 2.4</td>
</tr>
<tr>
<td></td>
<td></td>
<td>(RSD=2.8%)</td>
</tr>
<tr>
<td>Mono and Diacid</td>
<td>67.3</td>
<td>83.9 ± 2.5%</td>
</tr>
<tr>
<td></td>
<td></td>
<td>(RSD=2.9%)</td>
</tr>
</tbody>
</table>

\(^a\)DCPA, monoacid, and diacid were all spiked into each sample and fractionated as described in Experimental Section

\(^b\)recoveries and relative standard deviations based on n=5 replicates
Table IV. Survey of Groundwater Wells in Malheur County of Eastern Oregon.

Acid Metabolites of DCPA (µg/L)\(^a\)

<table>
<thead>
<tr>
<th>Sample #</th>
<th>SPE</th>
<th>LLE</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>122.8 ± 3.9 (RSD=3.1%)(^b)</td>
<td>145.2 ± 4.2 (RSD=2.9%)(^b)</td>
</tr>
<tr>
<td>2</td>
<td>77.4 ± 2.4(^{cd})</td>
<td>88.1 ± 2.6(^d)</td>
</tr>
<tr>
<td>3</td>
<td>&lt; D.L.(^{cde})</td>
<td>&lt; D.L.(^{de})</td>
</tr>
<tr>
<td>4</td>
<td>197.5 ± 6.2(^{cd})</td>
<td>203.0 ± 5.9(^d)</td>
</tr>
<tr>
<td>5</td>
<td>92.4 ± 2.9(^{cd})</td>
<td>88.5 ± 2.6(^d)</td>
</tr>
<tr>
<td>6</td>
<td>3.1 ± 0.1(^{cd})</td>
<td>4.7 ± 0.1(^d)</td>
</tr>
<tr>
<td>7</td>
<td>58.0 ± 1.7(^{cd})</td>
<td>61.5 ± 1.8(^d)</td>
</tr>
<tr>
<td>8</td>
<td>5.7 ± 0.2(^{cd})</td>
<td>8.5 ± 0.2(^d)</td>
</tr>
<tr>
<td>9</td>
<td>62.7 ± 2.0(^{cd})</td>
<td>77.0 ± 2.2(^d)</td>
</tr>
</tbody>
</table>

\(^{a}\)corrected values given; determined by spike recoveries (80.1% for SPE and 88% for LLE)

\(^{b}\)composite sample with n=4 replicates performed to determine RSD

\(^{c}\)duplicate analyses

\(^{d}\)relative standard deviation of n=4 set applied to determine range of variability

\(^{e}\)< D.L. = less than detection limit (0.05 µg/L for SPE; 1 µg/L for LLE)
Figure 1. Example of a typical chromatogram of a ground water sample at 58.0 µg/L (corrected value using avg. spike recovery of 80.1%).
LITERATURE CITED


(13) Craig Markell and Steve StMary, technical support.