Mobile Laboratory Support to Site Mitigation: Simplified Method for Measuring Toxaphene Contamination by Gas Chromatography/Mass Spectrometry with Selective Ion Monitoring

ECL Report 2008-01

January 2008
Environmental Chemistry Laboratory Report

Mobile Laboratory Support to Site Mitigation: Simplified Method for Measuring Toxaphene Contamination by Gas Chromatography/Mass Spectrometry with Selective Ion Monitoring

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Bruce LaBelle, Ph.D.
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Environmental Chemistry Laboratory
Department of Toxic Substances Control

2/11/09
Date
EXECUTIVE SUMMARY

Although the primary responsibility of the DTSC Mobile Laboratory (ML) is to rapidly respond to any emergency in California, the ML also supports Site Mitigation projects on request. During January 22–25, 2007, the ML team was deployed to Dos Palos, CA to help in the investigation of a site (Central Valley Fertilizer) contaminated with toxaphene. A simplified screening method was developed to identify and measure toxaphene with a Limit of Quantitation (LOQ) of 0.5 ppm. Using this method, the ML team successfully completed the analysis of 36 soil samples in less than 3 days. Toxaphene was measured in 6 of those samples above the action limit (1.8 ppm) set for this site. These on-site measurements provided project managers with timely information. An extraction step will be added to this method in the near future and a comparison against standard methods will be undertaken.
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ABBREVIATIONS AND ACRONYMS

AMDIS = Automatic Mass Spectrum Deconvolution Identification Software
GC/ECD = Gas Chromatograph/electron capture detector
GC/MS = Gas Chromatograph/Mass Spectrometer
GC/MSD = Gas Chromatograph/Mass Selective Detector
NIMS = Negative Ion Mass Spectrometry
SIM = Selective Ion Monitoring
SOP = Standard Operating Procedure
TIC = Total Ion Chromatogram
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Figure 4. Chromatogram of toxaphene found in a positive soil sample
INTRODUCTION

The Department of Toxic Substances Control's (DTSC) Mobile Laboratory (ML) was funded by the Homeland Security Grant Program (HSGP). The primary duty of the ML team is emergency response. Because of its mobile deployment capabilities, the ML team is designated to quickly identify unknown chemical releases from natural disasters, industrial spills, explosions or terrorist attacks.

Site Mitigation Projects are a major part of DTSC’s responsibilities. Ongoing projects are essential for the protection of public health and the environment within the state. One of the difficulties for most project managers is obtaining timely assessment information about the contaminated site and/or to locate on-site hot spots. The ML team can be instrumental in Site Mitigation projects (1). A core capability of the ML is to characterize site contamination with qualitative and quantitative chemical data in order to quickly supply project managers with critical information for on-site decision making and risk assessment. In response to a request from the Site Mitigation Branch Project Manager, the ML team was deployed to Dos Palos, County of Merced, California, on January 22-25, 2007.

METHODS

Site Description
The abandoned property belongs to the Central Valley Fertilizer Company and consists of a cement bed for washing pesticide spraying equipment and vehicles, a waste water collection basin and a half acre evaporation pond. The sample plan and abandoned site are presented in Figure 1. The scope of the assignment was to characterize the site for toxaphene contamination with the objective of procuring risk assessment information for future cleanup or litigation.

Approach
A simplified method to analyze soil samples for toxaphene was developed for this Site Mitigation project. The method utilized the on-board GC/MS with a SIM mode to quickly screen soil samples for toxaphene. The project manager required a limit-of-quantitation (LOQ) level of 1.8 ppm; therefore, the limit-of-detection (LOD) level was set at 3 times below that, i.e., at 0.5 ppm.

Materials and Reagents
2. Standard – Toxaphene, National Institute of Standards & Technology, Gaithersburg, MD 20899.
3. Internal Standard: 2-florobiphenyl (Supelco, 595 North Harrison Road, Bellefonte, PA).
4. Surrogate: decachlorobiphenyl (Supelco, 595 North Harrison Road, Bellefonte, PA).
Apparatus
The ML consists of one vehicle (Ford F-555), a satellite communication system and two
gas chromatograph/mass spectrometers (GC/MS) specifically designed for detecting
toxic industrial chemicals in air, soil and water. The advanced analytical instruments
are complemented by a series of field instruments for the quick detection of volatile
compounds, metals and other chemicals. The GC/MS conditions used in this study
were:

1. GC/MSD: Agilent 6850/5973 GC/MSD with ChemStation computer system.
2. Column: HP-5MS (5% phenyl methyl siloxane).
3. Temperature program: rate 20°C/min, initial temperature 80°C, final temperature
   300°C and run time 14.5 min.
4. MSD/SIM setup: auto tune, DFTPP tune, toxaphene target ion 159 m/z, reference
   ions 231 m/z and 233 m/z.
5. Internal standard: 2-florobiphenyl target ion 172 m/z, reference ions 170 m/z and 171
   m/z.
6. Surrogate: decachlorobiphenyl target ion 241 m/z, reference ions 170 m/z and 172
   m/z.

Sample and Standard Preparation
Extraction: Approximately 5g soil were weighed into a 40 mL VOA vial, and 5mL of a
hexane solution containing 2-florobiphenyl (1ug/mL) and decachlorobiphenyl (1ug/mL)
as internal standard and surrogate, respectively, were added. The vial was extracted for
approximately 3 min on a vortex mixer and approximately 1 mL of the top layer was
taken into an autosampler vial for GC/MSD injection. If the soil samples were wet,
anhydrous Na2SO4 (ca. 1.0g) was premixed before extraction.
Standards: Six level standards (0.5, 1.0, 2.0, 4.0, 8.0 and 10 ug/mL) were made from
soil matrix blanks.
Quantitation retention time: the average of four retention time peaks (9.57, 9.91, 10.65
and 11.15 min) was used.

RESULTS AND DISCUSSION

Gas chromatogram peak retention time and peak area, as well as mass spectrum and
ion abundance have been successfully used for volatile and semi-volatile organic
compound identification and quantitation. But multi-component analytes, such as
toxaphene, aroclors and chlordane present unusual difficulties due to their undefined
peaks, multi-humps, and weathering effect changes. As such, these compounds
present an interesting challenge. Furthermore, Method 8270C (2) has suggested that
the recommended method to test these analytes is Method 8081A (3), i.e., using Gas
Chromatography/electron capture detector (GC/ECD), rather than GC/MSD because of
recommended using chemical ionization technique to monitor toxaphene degradation
products by negative ion mass spectrometry (NIMS) (4). However the ML’s on-board
GC/MSDs are standard-issue instruments for homeland security rapid identification; and
a GC/MSD with Automatic Mass Spectrum Deconvolution Identification Software
(AMDIS) superbly detects chemical agents at trace levels. Furthermore, space
limitations make it infeasible to accommodate additional instruments such as a GC/ECD and GC/NIMS with their on-board computer systems. With these considerations in mind, a simplified method to analyze toxaphene by GC/SIM for site characterization was developed to respond to the site mitigation project.

Literature review (5) demonstrated successful results by averaging 4 predetermined retention time area counts for quantitation, therefore, 9.57, 9.91, 10.65 and 11.15 min were chosen after toxaphene hump shape pattern evaluation in the total ion chromatogram (TIC). To establish the quantitation table, 6 levels (0.5, 1.0, 2.0, 4.0, 8.0 and 10 ug/mL) standards were used for each retention time. All standards were made from a hexane extraction in pre-tested soil blanks to suppress soil matrix interference, increase the S/N ratio and enhance sensitivity. The correlation coefficient of the 6 level standards for 4 retention times were $r^2= 0.999$ at 9.57 min, 0.997 at 9.91 min, 0.999 at 10.65 min and 0.999 at 11.15 min. Internal standard, 2-fluorobiphenyl, was used to obtain relative response factors in quantitation and surrogate decachlorobiphenyl was added into each sample for method recovery data.

The ML staff received 36 samples from the on-site project manager which were prepared according to the SOP (6). The samples were placed on the autosampler injector for an overnight run and the data were reviewed and processed the following day. The non-detected (ND) samples were screened out by visual comparison or by overlaying the extracted ion (159 m/z) chromatogram to the chromatogram of standards, the extracted ion chromatogram of which is presented in Figure 2. Because of the unique shape of toxaphene, this is a very effective way to differentiate negative samples from positive samples. One ND sample and the 8.0 ppm toxaphene positive sample are presented in Figures 3 and 4, respectively. The results of the positive samples were calculated based on the average of 4 retention time reported values, and the results are presented in Table 1 and Table2. The average surrogate (decachlorobiphenyl) recovery %, SD and CV % were 92.3, 12.2 and 13.2 from 36 samples respectively.

Development plans for a simple extraction and cleanup procedure are being considered for the near future, and a comparison against standard methods will be undertaken.

CONCLUSIONS

Site Mitigation is a major part of DTSC’s responsibility. The ML scientists used their simplified GC/MS/SIM method to complete 36 samples in less than 3 days and to detect 6 contaminated hot spots on the site. This simplified method was developed for quick on-site screening and site characterization purposes. This method is simple, fast and easy to use as no lengthy extraction and cleanup procedures are necessary. However, this method may be biased towards false positives rather than false negatives; consequently, development plans for simple extraction and cleanup procedures are being considered for the near future.
REFERENCES

(1) DTSC, SOP 1000-S, Mobile Laboratory Activation and Deployment (2007)
(5) PerkinElmer Instruments, Environmental Application Note.
(7) Food, Testing & Analysis 6, 13-18, 46. 2000

ACKNOWLEDGMENTS

The authors thank Maria Gillette, Project Manager and her associates, Brad Parsons, Jerry Lile and Sam Martinez, for preparing on-site homogenous soil samples. The authors also wish to thank Dr. Bruce La Belle (Division Chief) and Dr. Myrto Petreas (Branch Chief) for their support and encouragement of this research project.
Table 1. Quantitative Results by Averaging Calibrated Results from 4 retention times

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<td>ALS Vial</td>
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Quant Title: TOXAPHENE 3 IONS SIM GCMS
QLast Update: Tue Jan 23 10:53:28 2007
Response via: Initial Calibration

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<th>Response</th>
<th>Conc Units</th>
<th>Dev(Min)</th>
</tr>
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<tbody>
<tr>
<td>1) Internal Std(2-FBP)</td>
<td>5.065</td>
<td>172</td>
<td>1277116</td>
<td>1.00 ppm</td>
</tr>
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</table>

System Monitoring Compounds
Spiked Amount: 1.000
Recovery: 11.00%

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<th>Target Compounds</th>
<th>R.T.</th>
<th>Conc Units</th>
<th>Dev(Min)</th>
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<tr>
<td>2) Toxaphene RT9.57</td>
<td>9.572</td>
<td>159</td>
<td>1554m</td>
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<td>9.910</td>
<td>159</td>
<td>2116</td>
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<td>4) Toxaphene RT10.65</td>
<td>10.652</td>
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<td>4481</td>
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<td>5) Toxaphene RT11.15</td>
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(#{ qualifier out of range (m) = manual integration (+) = signals summed }

TXPN3LB.M Tue Jul 24 10:05:22 2007

Average = 4.38 ppm

Table 2. Results of Toxaphene and Surrogate Recovery in soil samples
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<th>Collector's Number</th>
<th>Toxaphene, mg/Kg</th>
<th>Decachlorobiphenyl, %</th>
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<td><strong>CV</strong></td>
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<tr>
<td><strong>n</strong></td>
<td></td>
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Figure 1. Site Mitigation Sampling Plan and photograph of abandoned site
Figure 2. Chromatogram of the extracted ion 159 m/z from toxaphene standard at 4 ppm.
Figure 3. Chromatogram of a non-detected (ND) toxaphene soil sample.
Figure 4. Chromatogram of toxaphene found in a positive soil sample.
Appendix – Sample Reports
## Requestor Address

Maria Gillette  
OTSC Region 1  
Sacramento, CA

## Date Collected
1/22/2007

## Date Received by Lab
1/22/2007

## Date Extracted
1/22/2007

## Date Analyzed
1/22/2007

## Analysis Method
HPLC

## Sampling Location
Central Valley Fertilizer  
Azusa Rd  
Duarte, CA

## Extraction Methods
- EPA 3510C for aqueous samples; Separatory funnel extraction with methyl chloride. Solvent evaporation to volume.
- EPA 3516 for solid samples. Maceration extraction with hexanes/acetone, followed by solvent evaporation to volume.
- EPA 3550B for solid samples. Methanol extraction with hexane/acetone, followed by solvent evaporation to volume.
- EPA 3580A for oils & organic liquid samples; solvent dilution with hexane.
- EPA 3620F for biological samples; modified procedure by injection of 50 mL of sample into 50 mL of hexane.
- EPA 8081A Analysis by DB-1701 capillary column GC/ECD & confirmation by DB-6 capillary column.

## Analytical Method

<table>
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<td>Unisafe</td>
<td>551-24-3</td>
<td>ND</td>
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<td>ND</td>
<td>ND</td>
<td>ND</td>
</tr>
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</table>

### Note
- QL = Quantitation Limit = Lowest calibration standard x dilution factor
- ND = Not detected or detected but below QL

### Comments

- Analyzed by: Orlando Garbin
- Reviewed by: Modam Gill
- Approved by: Keh-chuh Ting

### Signature

- Orlando Garbin
- Modam Gill
- Keh-chuh Ting

### Date

- 3/6/07
- 3/7/07
**Report Details**

**Requestor Address:**
Marla Gillette
PTSC Region 7
Sacramento, CA

**Sampling Location:**
Central Valley Fertilizer
Aqua Rd
Dox, PA, CA

**Extraction Methods:**
- EPA 3510C for aqueous samples; sequentially fumigated extraction with methylene chloride, solvent exchange to hexane.
- EPA 3546 for solid samples; microwave extraction with methanol, followed by solvent exchange to hexane.
- EPA 3550B for solid, extraction with hexane/acetone; followed by solvent exchange to hexane.
- EPA 3560A for oils and organic liquid samples; solvent dilution with hexane.

**Cleanup Method:**
- EPA 1620 for solid samples cleanup; normally not performed unless otherwise mentioned in the report.
- EPA 620 for solid samples cleanup; solvent extraction, followed by solvent exchange to hexane.

**Analytical Method:**
- General analysis by GC/MS confirmatory by DB-5 capillary column.

**Sample Matrix:**
- Soil
- Water

**Compound** | **CAS #** | **EOO**: 021-02-2 | **EOO1034**: A5001034 | **EOO1037**: A5001037 | **EOO1038**: A5001038 | **EOO1039**: A5001039 | **EOO1040**: A5001040
--- | --- | --- | --- | --- | --- | --- | --- |
**Units** | **mg/kg** | **mg/kg** | **mg/kg** | **mg/kg** | **mg/kg** | **mg/kg** | **No Data** |
**Soil** | A | B | C | D | E | F | No Data |
**Water** | G | H | I | J | K | L | No Data |

**Surrogate Standard Recovery:**

| **Compound** | **EOO1034** | **EOO1037** | **EOO1038** | **EOO1039** | **EOO1040** |
--- | --- | --- | --- | --- | --- |
**EOO**: 021-02-2 | B | A | C | D | E |

**Note:**
- **QL:** Quantitation Limit - lowest calibration standard x dilution factor
- **ND:** Not detected or detected but below QL

**Comments:**

- **Analyzed by:** Orlando Garbin
- **Reviewed by:** Modan Gill
- **Approved by:** Kahl-chung Ting

**Date:**
- **Signature:**
  - **Approving:** 3/27/07
  - **Analyzing:** 3/27/07
  - **Reviewing:** 3/27/07

---

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### Analytical Method:
EPA 8010B (Analysis by ES-109 capillary column GC/FID & confirmation by MS-5 capillary column)

### Sample Matrix:
- Soil
- Sediment
- Water

### Compound Table:

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<th>mg/kg</th>
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### Surrogate Standard Recovery:

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<td>78.0</td>
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<td>041-39-4</td>
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</table>

**Note:**
- QL = Quantitation Limit = Lowest calibration standard x dilution factor
- ND = Not detected or detected below QL

**Comments:**

- Analyzed by: Orlando Garbin
- Reviewed by: Modam Gill
- Approved by: Keel-chuh Ting

*Signature Dates:*
- Orlando Garbin: 3/7/07
- Modam Gill: 2/7/07
- Keel-chuh Ting: 3/7/07
### Requestor Address

Marie Gillette  
DTSC Region I  
Sacramento, CA

### Sampling Location

Central Valley Fertilizer  
Anna Rd  
Due Palas, CA

### Extraction Methods

- **EPA 3510C** for aqueous samples: separatory funnel extraction with methylene chloride. Solvent exchange to hexane.
- **EPA 3540** for solid samples: microwave extraction with methanol/acetone, followed by solvent exchange to hexane.
- **EPA 3550** for solid samples: maceration extraction with hexane/acetone, followed by solvent exchange to hexane.

### Cleanup Method

**EPA 3520** for liquid samples; solvent dilution with hexane.

### Analytical Method

EPA 6081A Analysis by DB-1701 capillary column GC/MS & confirmation by DB-5 capillary column

<table>
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<tr>
<th>I.D. Number</th>
<th>Sample</th>
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<th>UV Path 4-2</th>
<th>IR Path 4-2</th>
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### Surveyed Standard Recovery

| Compound | 1015-24-1 | 78.0 | 81.0 | 77.0 | 99.0 | 94.0 | 101 |

**Note**:  
- GL = Quantitation Limit - Lowest calibration standard x dilution factor  
- ND = Not detected or detected below GL

### Comments

- Analyzed by: Orlando Sarbin  
  Signature: [Signature Image]  
  Date: 3/07/07

- Reviewed by: Melan Grif  
  Signature: [Signature Image]  
  Date: 7/3/07

- Approved by: Reh-ah-shuk Ting  
  Signature: [Signature Image]  
  Date: 3/07/07
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<th>POND 5-2</th>
<th>CVV 7A-6.9</th>
<th>CVV 7A-6.12</th>
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</tr>
<tr>
<td>Unit</td>
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<td>mg/Kg</td>
<td>mg/Kg</td>
<td>mg/Kg</td>
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<td>Composed CAS#</td>
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</table>

**Note:**
- **QL** = Quantitation Limit = Lowest calibration standard x dilution factor
- **ND** = Not detected or detected below QL

**Comments:**

- Analyzed by: Orlando Garbin
- Reviewed by: Meen Gill
- Approved by: Keh-Chuah Ting

[Signatures and dates for analysis and review]
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</tbody>
</table>

**Note:**
- QL = Quantitation Limit = Lowest calibration standard x dilution factor
- ND = Not detected or detected below QL

**Comments:**
* Detected value exceeded linear calibration. This is an estimate.

**Analyzed by:** Orlando Garbin
**Reviewed by:** Modan Gill
**Approved by:** Xuh-chuh Ting

**Date signed:** 3/07/07
**Date reviewed:** 3/7/07
**Date approved:** 3/7/07
Requestor Address: Maria Gillette  
ATTP Region 1  
Sacramento, CA

Sampling Location: Central Valley Fertilizer  
Aruba Rd  
Doo Pals, CA

Extraction Method: Extraction Holding Time Mat: YES  
Analysis Holding Time Mat: YES

Extraction Methods: EPA 3510C for aqueous samples; separatory funnel extraction with methyl chloride; solvent exchange to hexane.  
EPA 3546 for solid samples; microwave extraction with hexane/acetone, followed by solvent exchange to hexane.  
EPA 3550B for solid samples; partition extraction with hexane/acetone, followed by solvent exchange to hexane.  
EPA 3550A for oils & organic liquid samples; solvent dilution with hexane.

Cleanup Method: EPA 3629 Distillation column cleanup. (Manual not performed unless otherwise mentioned in the report).

Analytical Methods: EPA 8081A Analysis by DB-5GC capillary column GC/FID & confirmation by DB-5 capillary column.

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<th>Soil</th>
<th>Soil</th>
<th>Soil</th>
<th>Soil</th>
<th>Soil</th>
<th>Soil</th>
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</thead>
<tbody>
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<td>mg/kg</td>
<td>mg/kg</td>
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Note: QL = Quantitation Limit; Lowest calibration standard x dilution factor.  
ND = Not detected or detected below QL

Surrogate Standard Recovery:

| DECACHLOROBIPHENYL | 001-24-1 | 103 | 103 | 104 | 102 | 102 | 101 |

Date: 1/21/2008  
Signatures: Orlando Garbin  
Reviewed by: Modam Gill  
Approved by: Keh-ehuh Ting  
Date: 3/7/07

Comments:
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<th>ECL Number:</th>
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</tbody>
</table>

**Note:**
- QL = Quantitation Limit = Lowest calibration standard x dilution factor
- ND = Not detected or detected below QL

**Comments:**
- Analyzed by: Orlando Garbin
- Reviewed by: Modem Gill
- Approved by: Keh-chuh Ting