OFFICE OF INSPECTOR GENERAL

Catalyst for Improving the Environment

Addendum to Ombudsman Report

Appropriate Testing and Timely Reporting Are Needed at the Hercules 009 Landfill Superfund Site, Brunswick, Georgia

Report 2005-P-00022 (Addendum)

September 13, 2005

Abbreviations

µg/l	Micrograms per liter
µg/m^3	Micrograms per cubic meter
ACT	Acid-activated copper treatment
AMAP	Arctic Monitoring and Assessment Programme
CLE	Cod Liver Extract
CSF	Cancer slope factor
ECD	Electron capture detector
EPA	U. S. Environmental Protection Agency
EPD	Georgia Environmental Protection Division
FOIA	Freedom of Information Act
g/kg	Grams per kilogram
GC/ECD	Gas chromatography/electron capture detector
GC-ECNI-MS	Gas chromatography electron-capture negative ion mass spectrometry
GJIC	Gap junctional intercellular communication
GST-p	Placental form of glutathione-S-transferase
H_2SO_4	Sulfuric acid
HCH	Halogenated hydrocarbon
HCl	Hydrochloric acid
IRIS	Integrated Risk Information System
LOAEL	Lowest observed adverse effect level
LOEC	Lowest effective concentration
MATT	Investigation into the Monitoring, Analysis, and Toxicity of Toxaphene
MCL	Maximum contaminant level
mg/kg	Milligrams per kilogram
NIMS	Negative ion mass spectrometry
NOAEL	No observable adverse effect level
OEA	Office of Environmental Accountability
OIG	Office of Inspector General
PRG	Preliminary Remediation Goals
QA/QC	Quality assurance/quality control
RfD	Reference dose
SAT	Strong acid treatment
SESD	Science and Ecosystem Support Division
SRTSB/TSS	Superfund Remedial and Technical Services Branch/Technical Services Section
TDI	Tolerable daily intake
TPA	Tumor promoter 12-O-tetradecanoylphorbol-13-acetate
TT	Technical toxaphene
UCL	Upper confidence limit
UVT	Uv-treated toxaphene
WT	Weathered toxaphene, or toxaphene breakdown products

Cover photo: An aerial view of the Hercules 009 Landfill, which is in the center and left-center of this photograph, provided by Hercules Incorporated.

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UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 4
ATLANTA FEDERAL CENTER
61 FORSYTH STREET
ATLANTA, GEORGIA 30303-8960

June 29, 2005

MEMORANDUM

SUBJECT: Comments on the Draft Ombudsman Report
Appropriate Testing and Timely Reporting Are Needed at the Hercules 009 Landfill Superfund Site, Brunswick, Georgia; Assignment 2004-124

FROM: J. I. Palmer, Jr.
Regional Administrator

TO: Paul D. McKechnie
Acting Ombudsman
Office of Congressional and Public Liaison

Thank you for the opportunity to provide comments on the draft report. The following comments relate to the subject document, and are provided primarily to address the action required in EPA Manual 2750. In accordance with the memorandum transmitting the draft report, we have followed the instructions to "address the factual accuracy of the draft report and indicate concurrence or noncurrence with each finding and proposed recommendation. If you do not concur with the proposed recommendation, please provide any alternative actions you wish to be considered for the final report." The comments are a consolidation of input from Region 4's Analytical Support Branch of the Science and Ecosystem Support Division, and the Superfund Remedial and Technical Support Branch of the Waste Management Division. In addition, the Region has included comments from the Region 4 Office of Environmental Accountability (OEA) that address noted excerpts in the draft report.

Excerpt from the Draft Ombudsman Report Page 8, Recommendation 2.1:

"Use negative ion mass spectroscopy to definitely determine if toxaphene breakdown products are present in the surrounding groundwater at the Hercules 009 Landfill site, and (if so) in what amounts."

Response to Recommendation 2.1

The Region has used negative ion mass spectrometry (NIMS) to determine the presence of weathered toxaphene (WT). After the issuance of the Preliminary Technical Draft from the OIG, samples were collected from the 009 Landfill monitoring wells by Hercules, Inc., under a voluntary interim action on March 5th and analyzed by Keith Maruya at the Skidaway Institute of

Oceanography using the NIMS method from a peer reviewed scientific publication. The Hercules, Inc., March 2005 Data Report is attached as an addendum.

Region 4's laboratory is willing to participate in a multi-laboratory method validation study for toxaphene congeners in environmental samples. However, since the Agency as a whole would obviously benefit from a validated NIMS method for toxaphene congeners, we believe that a multi-laboratory method validation study should be initiated at the program level by the Office of Solid Waste and Emergency Response. A validated method will serve both the regulated community and the Agency by assuring that analytical data produced by the method are defensible, of known quality, and suitable for risk assessment decision making.

Excerpt from the Draft Ombudsman Report, Page 8 Recommendation 2.2:

"If toxaphene breakdown products are found in the groundwater, assess the resulting risk to human health and take appropriate action."

Response to Recommendation 2.2

In response to the Preliminary Technical Draft from the OIG, Region 4 conducted a thorough literature review for information on the toxicity of WT and reached the following conclusions:

Application of the MCL to Groundwater at 009 Landfill

The NIMS analytical results from groundwater at the 009 Landfill are all significantly less than the MCL for technical toxpahene (TT) of 3 ug/L. Based on the Region 4 preliminary toxicity assessment, it is reasonable to assume that this MCL is protective for WT as well as TT. The Region 4 Draft Report on WT toxicity is attached as an addendum and a short summary is provided below.

Toxicity Criterion for Weathered Toxaphene

To develop a human toxicity criterion for WT, three choices must be made: (1) the critical toxic endpoint; (2) the threshold dose value based on the critical endpoint; and (3) the uncertainty/safety factors applied to determine a reference dose.

Region 4 toxicologists believe that the most appropriate endpoint for WT is tumor promotion. This endpoint was chosen because it appears most relevant to humans, and focusing on this endpoint is also protective of other toxic endpoints, such as immunologic and developmental effects.

The Monitoring, Analysis and Toxicity of Toxaphene in Marine Foodstuffs (MATT) study from the European Union is the sole toxicological study based on WT and thus chemically is most relevant to human exposure. The critical study was performed in rats with changes in liver cells that represent precancerous changes as the endpoint. The no observable adverse effect level (NOAEL) from this study was 0.69 mg/kg-day as

calculated by MATT or 0.6 mg/kg-day in the Region 4 analysis. Note these values are very similar.

The following uncertainty factors were considered: 10 for animal-to-human extrapolation and 10 for human variability. In spite of the paucity of studies on WT, the literature is replete with studies on TT that cover a range of endpoints. Many of these studies resulted in higher and less protective NOAELs. The studies that had similar values for NOAELs were ingestion or oral gavage studies based on TT. The bulk of toxaphene administered orally is excreted or metabolized quickly; hence, the internal doses of WT and TT and the subsequent toxaphene body burdens end up being of similar magnitude. Because of the large database of toxicity studies of TT, we believe no additional database insufficiency uncertainty factor was needed. Hence, the combined uncertainty factor is 100.

This derivation of a reference dose for WT based on tumor promotion is consistent with the recently finalized EPA cancer guidelines which state that the consideration of mode of action vis-à-vis toxicity is paramount in the development of a toxicity criterion.

The Need for Peer Review

Unfortunately, the laboratory studies on WT toxicity that the MATT report relied on have not yet undergone peer review because of logistical issues (one of the authors moving to a new university). Region 4 agrees with the OIG report that additional toxicity studies may be helpful. Peer review would also be helpful in elucidating some of the apparent errors in interpretation of EPA cancer potency factor derivation identified in the MATT report.

On page 19 of the OIG report, the MATT was quoted as indicating that WT is approximately twice as carcinogenic as TT. This statement of the MATT report is incorrect and unfortunately repeated in the OIG report. It is not entirely clear on what basis the MATT report makes this statement, but there are two possibilities.

First, the MATT report presented a misunderstanding of the EPA TT slope factor in which the MATT confused the units. The upshot of this misunderstanding is that the MATT toxicity criterion appears for WT about twice as stringent at the EPA toxicity criterion for the original TT mixture. In truth, the EPA toxicity criterion is 300 times more stringent than the MATT criterion.

Second, the MATT interpreted some empirical data obtained from a cell culture system and possibly made a large and unfounded conceptual leap. In this cell culture system, the toxic endpoint was disruption of intercellular communication. Calculations indicated that the WT was twice as effective in blocking intercellular communication as TT. However, to make the leap of claiming that effects on intercellular communication in a cell culture

system is tantamount to a carcinogenic response in a whole animal is a very large leap indeed. This conceptual leap is not endorsed by Region 4 toxicologists.

Interim Strategy for Risk Assessment

In keeping with the OIG intention of using the best available science, Region 4 has two proposals for an interim strategy. The preferred approach is to use the toxicity criterion for WT developed by Region 4 Technical Services and based on the laboratory study in the MATT. Presently, the Region 4 report is still in draft, but should be finalized relatively soon. Hence, Region 4 requests that the OIG review both the MATT laboratory study and the Region 4 derivation of toxicity criteria to determine their soundness and applicability. An alternative approach is to use a toxicity criterion based on TT. The EPA toxicity criterion for TT now on IRIS was last revised in 1991. The toxicity value is based on rodent bioassays conducted in 1978 and 1979. In 2000, Goodman[1] reanalyzed these data using newer methods based on EPA guidance and concluded that TT was actually tenfold less toxic than the IRIS value. Region 4 believes that our preferred approach represents the best available science and would provide a reasonable interim approach.

Excerpt from the Draft Ombudsman Report Page 13, 3.1:

"Issue the report on the Hercules 009 Landfill 5-year review with the conclusion that the protectiveness of the groundwater cleanup cannot be determined at this time, and further evaluation is needed. A timeframe should be estimated for such an evaluation."

Response to Recommendation 3.1:

Based on the recent evidence provided by the NIMS data and the toxicity criterion developed by Region 4 based on the MATT laboratory study, Region 4 proposes that the data and toxicity review be included in the release of the 5-year review and a determination of protectiveness be issued.

Excerpt from the Draft Ombudsman Report Page 13, 3.2 :

"Ensure that restrictions are placed in the property deed to control future use of the land and groundwater."

Response to Recommendation 3.2:

When Hercules, Inc., entered into the Consent Decree for RD/RA with the U.S., it agreed to perform all operation and maintenance activities required to maintain the effectiveness of the

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[1] Goodman JI, Brusick DJ, Busey WM, Cohen SM, Lamb JC, Starr TB. (2000) Reevaluation of the cancer potency factor of toxaphene: recommendations from a peer review panel. Toxicol Sci. 2000 May;55(1):3-16.

Remedial Action. As set out in the Record of Decision, operation and maintenance of the multi-media cover was to continue for a minimum of 30 years. (See Section 9.A.1). The Consent Decree for RD/RA requires Hercules, Inc., to not only record a certified copy of the Consent Decree with the Glynn County registry of deeds, but to also include within any instrument conveying any interest in the property a notice describing the restrictions applicable to the property, the provisions of the Consent Decree with respect to institutional controls and EPA's right of access, and the obligations of successors-in-title. In addition, Hercules, Inc., and any successor-in-title must provide written notice to EPA of any proposed conveyance of any interest in the title. As part of EPA's statutory mandate for a 5-year review of a remedy's effectiveness if waste is left in place, the Consent Decree for RD/RA further obligates Hercules, Inc., to conduct any studies and investigations that EPA might request in support of its 5-year review. Moreover, no conveyance by Hercules, Inc., of any interest in property, however minor, will release or otherwise affect the liability of Hercules, Inc., to comply with the terms of the Consent Decree for RD/RA.

In light of the fact that the remedy has achieved the performance standards established in the Record of Decision and is believed to be currently performing as designed, EPA's statutory authorities and the enforcement-based tools arising from the Consent Decree for RD/RA are presently believed to be adequate institutional controls. Of course, if environmental or other conditions change, existing State-based legal authorities may in the future also be utilized to facilitate proprietary controls, such as an environmental easement designed to protect groundwater, if determined by EPA to be necessary.

Excerpt from the Draft Ombudsman Report Page 13, 3.3:

"Confirm that no one in the vicinity is using the groundwater."

Response to Recommendation 3.3 :

Since the perimeter monitoring wells are properly functioning as an intended early warning system, it becomes unnecessary to document private well water use outside the perimeter. Nonetheless, Hercules, Inc. has investigated the status of private registration of new wells in the immediate area. In checking with the Glynn County Environmental Department, Hercules was informed that the County has no record for the past several years of anyone advising them of the intent to drill a private drinking water well in the immediate area of the 009 Landfill. Hercules has also been informed by the previously affected residents that they continue to use city water for drinking purposes. Finally, Hercules has conducted a visual canvassing of the area to locate well house structures. This effort indicates that no new private drinking water wells have been installed in the immediate area surrounding the site.

The perimeter well monitoring system has been evaluated by a hydrogeologist in Region 4's Technical Services Section. The ability to capture leachate was the focus of the evaluation and the leachate capture system was found to be effective. The Final Technical Memo is attached as

an addendum. These wells are closest to the source and allow the earliest warning signal that downgradient wells may be affected. Since the recent NIMS and MATT comparison demonstrate that the perimeter wells do not pose a risk and the monitoring wells act as an effective early warning signal, it is unnecessary to obtain additional groundwater samples from locations beyond the perimeter well system.

Attachments (6)

1. Hercules, Inc., March 2005 Data Package for 009 Landfill using NIMS

2. A Re-analysis of the European MATT (2000) Toxicity Data and Development of a Reference Dose for Weathered Toxaphene (DRAFT)

3. Differences between Cancer and Non-Cancer Risk Assessment using Toxaphene as an example

4. October/December 2003 Technical Memos from SRTSB/TSS describing sufficiency of the monitoring wells to detect migration

5. Cover Memo and Complete Comments from SESD

6. Cover Memo and Complete Comments from OEA

cc w/o attachments:
Winston A. Smith, Region 4 WD
Franklin Hill, Region 4 WD
Scott Sudweeks, Region 4 WD
Ted Simon, Region 4 WD
Kay Wischkaemper, Region 4 WD
Derek Matory, Region 4 WD
Randall Chaffins, Region 4 WD
Leo Francendese, Region 4 WD
Gregory Luetscher, Region 4 WD
Gary Bennett, Region 4 SESD
Lavon Revels, Region 4 SESD
Charlie Hooper, Region 4 SESD
David Lopez, OSWER/OERR
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HERCULES

Hercules Incorporated
Hercules Research Center
500 Hercules Road
Wilmington, DE 19808-1599
Writer's Direct Dial: 302-995-3456

April 11, 2005

VIA OVERNIGHT MAIL

Mr. Leo Francendese
U.S. EPA Region IV, Waste Division
South Site Management Branch
61 Forsyth Street
Atlanta, Georgia 30303-3104

RE: Hercules 009 Landfill - Brunswick, GA

Dear Mr. Francendese:

In response to various concerns regarding the 009 Landfill, Hercules scheduled the annual groundwater monitoring and inspection earlier in the year and volunteered to use additional analytical methods to assess the most conservative degree of potential risk. The following is a summary:

During the week of March 7, 2005 RMT, Inc. mobilized to the site and initiated the inspection and collected eighteen (18) groundwater samples (an additional seven wells were sampled per EPA request). These samples were sent to EnChem Laboratories Green Bay Wisconsin who performed the extraction and then sent aliquots of the extracts to Dr. Keith Maruya – Skidaway Institute of Oceanography – Savannah Georgia. Subsequently, EnChem performed a copper clean-up procedure to remove sulfur compounds and conducted analyses of the extracts using the analytical protocol which has been used for all post remediation groundwater analyses Method 1 (August 14, 1997 Protocol). EnChem also analyzed the samples using Method 2 (Total Area Under the Curve). These methods are described in detail in Attachment 1. Concurrent with EnChem's work, Dr. Maruya conducted analyses of the extracts using Electron Capture Detector (ECD) and ECD-NCIMS. Dr. Maruya also encountered sulfur interferences and performed a copper clean up procedure as noted in his report. A summary table and a copy of all results are attached. Attachment 2.

The following are a few key observations with regard to this study: 1) The ECD-NCIMS method is essentially the same as Method 2 (Total Area Under the Curve), however non-toxaphene related compounds can be removed from the quantitation method. 2) ECD-NCIMS is also a non-standard and non-EPA approved methodology. 3) Method 2 (Total Area Under the Curve) is the most conservative assessment tool. 4) All results regardless of which method used were numerically less than Maximum Contaminant Level for toxaphene of 3.0 ug/L.

We will be sending the Annual Inspection and Groundwater Monitoring Report within the next month. Please call me if you have any questions regarding these results.

Sincerely,

Timothy D. Hassett
Staff Environmental Engineer

TDH/ijc
NCIMS.doc
Enclosures

Attachment 1

FINAL

APPENDIX 1

SUPPLEMENTAL ANALYTICAL PROTOCOLS FOR THE DETERMINATION OF TOXAPHENE IN SAMPLES FROM TERRY CREEK, BRUNSWICK, GEORGIA

SUPPLEMENTAL ANALYTICAL PROTOCOLS FOR THE DETERMINATION OF TOXAPHENE IN SAMPLES FROM TERRY CREEK, BRUNSWICK, GEORGIA

At a meeting in Athens, Georgia, on September 13, 2000, representatives from the U.S. Environmental Protection Agency Region IV (EPA), the State of Georgia Environmental Protection Division (EPD), Hercules Incorporated (Hercules), and FJC Analytical Consulting, Inc. met to discuss the analysis of samples from various environmental media for possible toxaphene components. A second meeting was held in Athens, Georgia, on June 13, 2001 to review the draft of the proposed analytical protocol document. In addition to the original group of chemists, representatives from En Chem, Inc., Madison, Wisconsin (En Chem), and from the Skidaway Institute of Oceanography (SkIO), Savannah, Georgia, participated in the review meeting.

The objective of this study is to measure the analytical precision among participating laboratories following the guidelines in this Supplemental Analytical Protocols document for the analysis of toxaphene and possible toxaphene components. This study is not designed to produce representative data, but to evaluate and compare the various quantitation guidelines presented in the study. With the objective of measuring the total array of components detected in samples collected at Terry Creek, Brunswick, Georgia, the group agreed that several supplemental calibration and calculation methods would be investigated. The results of analyses of split samples using these supplemental analytical protocols will be compared with results of analyses obtained by following the August 14, 1997, EPA-approved protocol that is currently used to detect and quantitate toxaphene in all environmental samples from Brunswick, Georgia.

As agreed at the meetings, the principles delineated in approved EPA methods will be followed in all analyses. While only Method 8081A will be followed, there will be four different procedures used to calculate the components detected in the extracts of the samples. Those calculation procedures will be:

1.) The August 14, 1997 Protocol
2.) Total Area under the Curve
3.) Toxaphene Congeners as Reference Standards
4.) Toxaphene Congeners as Guides for Peak Selection

As a separate part of this study, the extracts of the samples will also be analyzed by the experimental gas chromatography electron-capture negative ionization mass spectrometry (GC-ECNI-MS) procedure developed by Dr. K. A. Maruya at SkIO. This procedure has been published in the scientific literature [9,10], and it is included in the section on GC-ECNI-MS later in this document. While the GC-ECNI-MS analyses will be performed as part of this study, the results of analyses will be evaluated separately from the results obtained using Method 8081A because only the laboratory at SkIO is equipped for those GC-ECNI-MS analyses.

GENERAL INSTRUCTIONS:

a) Before any samples are sent to the laboratories for analyses and calculations using the four different procedures, Hercules will collect twenty sediment samples from Terry Creek. Those twenty samples will be extracted at En Chem using SW-846 Method 3550, and the extracts will be analyzed at En Chem using the August 14, 1997 protocol to determine the concentrations of toxaphene in each. Based on those preliminary analyses, seven sediment samples will be selected to provide a wide range of analyte concentrations – from "non-detect" to high concentrations. Such a wide range of concentrations is needed to test the supplemental analytical protocols most severely. The selected seven samples will be extracted according to U. S. EPA SW-846 Method 3550. The extracts will then be split, and a two-milliliter (2-mL) portion of each extract will be sent to each participating laboratory for analyses and calculations according to the four different calibration and calculation protocols.

b) The participating laboratories will be:

   i) the U.S. EPA Region IV Laboratory in Athens, Georgia;
   ii) the State of Georgia EPD Organic Laboratory in Atlanta, Georgia;
   iii) the Hercules Incorporated contractor laboratory – En Chem, Inc. in Madison, Wisconsin, and
   iv) Dr. K. A. Maruya at the Skidaway Institute of Oceanography, Savannah, Georgia.

c) All samples will be analyzed using U.S. EPA SW-846 Method 8081A[1], "Organochlorine Pesticides, Halowaxes, and PCBs as Aroclors by Gas Chromatography: Capillary Chromatography Technique," or its latest revision. All laboratories will follow the QA/QC procedures required by the method. The QA/QC measurements on the Blank, LCS/LCSD, and MS/MSD using technical toxaphene will be performed by the "August 14, 1997 Protocol," the "Total Area Under the Curve," and the "Toxaphene Congeners as Guides for Peak Selection" procedures.

d) All laboratories will use a toxaphene reference standard that matches the GC profile of the Hercules product standard, that is, Hercules technical toxaphene X16189-49. Experience has shown that a suitable reference standard for toxaphene may be purchased from Restek, Inc.[3] (Catalog No. 32005). Therefore, all participants will purchase the toxaphene reference standard from Restek, Inc. The match with this specific technical toxaphene must be established each time

that a new lot of standard is purchased. Samples of Hercules technical toxaphene X16189-49 have been furnished to the four participating laboratories.

1.) SEDIMENT SAMPLES

The sediment samples (20 grams in size) will be extracted at En Chem according to U.S. EPA SW-846 Method 3550[2], "Ultrasonic Extraction," using a 1:1 (volume/volume) mixture of hexane and acetone as the extraction solvent. All sample extracts will be exchanged to hexane and adjusted to a final volume of twenty milliliters (20 mL). The final volumes of the sample extracts may be adjusted according to the detection limit required by the data quality objectives specified in the project plan. The hexane solutions will be cleaned up by contact with sulfuric acid before analyses by gas chromatography using an electron capture detector (GC-ECD). If necessary, En Chem will also perform a cleanup to remove interference from sulfur. A two-milliliter (2-mL) portion of each extract will be distributed to each of the four laboratories. The remainder of each clean extract will be stored at 4 °C for future use, if necessary. The remainder of all sediment samples will be stored in a freezer. En Chem will also furnish to each participating laboratory the per cent solids for each sediment sample.

2.) SULFURIC ACID CLEANUP:

After the sample extract is adjusted to final volume in hexane, add, in a glass vial with a Teflon-lined screw cap, 10 mL of concentrated sulfuric acid for each 5 mL of sample extract. Shake the tube vigorously for one minute. Vent the vial carefully to relieve any pressure that may build up in the vial. Allow the layers to separate. (If excessive heat is generated during the extraction, the sample extract should be discarded and steps taken to eliminate the source of the heat generation.) If the layers are not clearly separated, centrifuge the mixture. After a clear separation is obtained, transfer the hexane layer to a GC injection vial for analysis. Store the excess extract in a clean vial with a Teflon-lined screw cap in a refrigerator at 4 °C, or less.

3.) REMOVAL OF SULFUR:

If necessary, sulfur interference will be removed from the hexane extracts according to the En Chem SOP: 3-SVO-27. Elemental mercury is introduced to the sample extracts, and the extract is then vortexed. The precipitate is allowed to settle, and the extract removed to a new tube. If performed on any sample, this treatment shall also be performed on the procedural blank.

4.) ELECTRON CAPTURE GAS CHROMATOGRAPHY PARAMETERS:

For consistent application of the method among the laboratories, the following steps are specified in more detail. Duplicate injections of each sample extract will be made. The sample extracts will be injected into two GC columns with stationary phases of different polarity. The recommended columns are a 30-meter DB-1701 (1.0-µm film thickness) Megabore column (J&W Scientific) and a 30-meter DB-5 (1.5-µm film thickness) Megabore column. Experience in the EPA Region IV laboratory has demonstrated fewer co-eluted toxaphene congener peaks on the DB-5 column. Therefore, the DB-5 column will be used for quantitation where possible, that is, when there are fewer interfering peaks on the DB-5 column than on the DB-1701 column. The Georgia EPD laboratory will use a 30-meter Megabore Restek Rtx-CLP1 (0.5 µm film thickness) column as its primary analytical column and a 30-meter Megabore Restek Rtx-CLP2 (0.42 µm film thickness) column as the confirmatory column. The laboratory at SkIO will use a DB-XLB column for quantitation and a DB-5 column for confirmation. Because En Chem will use a Hewlett-Packard 6890 GC equipped with a micro-electron capture detector (&-ECD), DB-5 and DB-1701 narrow-bore capillary (0.32-mm diameter) GC columns will be used in their laboratory. Based on work performed in the EPA Region IV Laboratory, the following GC oven temperature program is suggested as the starting point: 180 °C for 2 minutes, program to 260 °C at 6 °C/minute, maintain 260 °C for 5 to 10 minutes. Each laboratory is then free to adjust this basic profile to optimize the separation of the toxaphene congeners on their particular GC instruments. The injector will be operated at 220 °C to 250 °C, and the electron capture detector will be maintained at 300 °C to 350 °C. Tetrachloro-m-xylene (TCMX) and decachlorobiphenyl (DCB) will be used as surrogates. The surrogates will be added to all calibration solutions and to all sample extracts.

5.) DETECTION AND MEASUREMENT OF TOXAPHENE:

Five-point calibration curves will be used. Continuing calibration verification samples of technical toxaphene and of the 22-component toxaphene congener mixture from Dr. Ehrenstorfer Laboratories, available from EQ Laboratories, Inc.[7], the U. S. Distributor, will be injected at a frequency of every twenty (20) samples or after twelve (12) hours, whichever comes first. It is recommended that, once the 20-sample/12-hour calibration verification has been met, a technical toxaphene standard and a 22-component toxaphene congener standard be included after approximately every ten samples; and as the final injections at the end of each injection sequence, to minimize the number of repeat injections, in the event of the failure to meet the acceptance criteria. The Perkin-Elmer TurboChrom, the Hewlett-Packard ChemStation, or the Varian Star chromatography data systems will be used for the measurement of peak areas and peak heights.

6.) METHODS OF CALCULATION OF RESULTS OF ANALYSES:

A.) AUGUST 14, 1997 PROTOCOL

As a means of comparison, all samples will be calculated by the principles described in the August 14, 1997 analytical protocol that has been adopted for use in the analyses of all samples from Terry Creek, Brunswick, Georgia. That protocol is included as part of the Quality Assurance Project Plan (QAPP)[4] for the programs in progress. A description of the protocol has also been published in the open literature[5]. A copy of the manuscript for that publication has been given to all participants.

B.) TOTAL AREA UNDER THE CURVE

In this calculation procedure, a five-point calibration curve will be established using the technical toxaphene reference standard. The isolated and identified toxaphene congeners will be used to establish the toxaphene retention time (R_t) window. In a recent publication[6], McDonald, Vetter, and Hites reported the calculation of methylene retention indices for the isolated congeners of toxaphene. In that technical note, data on a DB-5 GC column were presented for three congeners that do not have Parlar numbers. Those compounds are: 2-endo,3-exo,5-endo,6-exo,8,8,10-heptachlorobornane (B7-1000); 2-exo,3-endo,6-exo,8,9,10-hexachlorobornane (B6-923, or Hx-Sed); and 2-endo,3-exo,5-endo,6-exo,8,9,10-heptachlorobornane (B7-1001, or Hp-Sed). Because of this recent information, it is important to include those compounds in the measurements in this procedure.

The twenty-two-component mixture of toxaphene congeners (Part No. ZA221002) and solutions of the individual congeners from Dr. Ehrenstorfer Laboratories in Augsburg, Germany, are available through the U.S. distributor, EQ Laboratories, Inc.[7] in Atlanta, Georgia. Solutions of Hx-Sed (TOX 441) and Hp-Sed (TOX 442), which are available from Promochem GmbH, Wesel, Germany, through its U.S. distributor[8], will be used to establish the R_t of those two congeners. There are no commercial sources for B7-1000; however, Dr. Walter Vetter of the University of Jena, Germany, has provided a solution of B7-1000 that will be suitable for the determination of R_t data only. The solution of B7-1000 cannot be used for quantitative measurements.

A five-point toxaphene calibration curve will be constructed for each quantitation and confirmation GC column to encompass the linear range of the ECD in each laboratory according to the principles of U.S. EPA SW-846 Method 8081A. Subsequently, chromatograms of the 22-congener mixture from Dr. Ehrenstorfer Laboratories, Hx-Sed and Hp-Sed from Promochem, and B7-1000 will be used to establish the R_t of each individual congener. The R_t window for toxaphene will be defined as beginning at the R_t of the first identified congener peak (Parlar No. 11) and ending at the R_t of the last congener peak (Parlar No. 69) in the mixture. The data system will be programmed to construct a baseline between those two retention times. The total area of all component peaks within that time interval will be used to calculate "Chlorinated Camphene" by using the technical toxaphene calibration curve. The results of analyses will be reported in units of micrograms per kilogram, dry weight (&g/kg, d.w.). If baselines to peak shoulders, or other incorrect baselines, are drawn by the data system, the analyst is directed to ignore these incorrect baselines and to assign baselines manually. The manual assignment of baselines must be documented on the chromatogram and in the report of analysis. The Georgia EPD laboratory will print a "before" and an "after" chromatogram to document the manual assignment of baselines.

Because the retention time window for toxaphene is so wide, there exists a great potential for the co-elution of other chlorinated organic compounds with the components of toxaphene. The electron capture detector provides a non-specific response and cannot differentiate those other chlorinated organic compounds from components of toxaphene. Such co-elution of components will result in an overestimation of the amount of chlorinated camphene calculated as present in the samples. Therefore, the results of analyses will be corrected for the presence of chlorinated pesticides, other than components of toxaphene, that elute within the toxaphene retention time window. The peak areas of all obvious and confirmed interfering peaks whose retention times match the retention times of other chlorinated pesticides will be subtracted from the total area under the curve (measured as described above). If there are confirmed hits (within 40% RPD) for:

8081A TCL compounds

aldrin	dieldrin
alpha-BHC	endosulfan I
beta-BHC	endosulfan II
belta-BHC	endosulfan sulfate
gamma-BHC	endrin
alpha-chlordane	endrin aldehyde
gamma-chlordane	endrin ketone
4,4'-DDD	heptachlor
4,4'-DDE	heptachlor epoxide (B)
4,4'-DDT	methoxychlor
plus technical chlordane	

8082A PCBs

Aroclor 1016	Aroclor 1248
Aroclor 1221	Aroclor 1254
Aroclor 1232	Aroclor 1260
Aroclor 1242	
plus Aroclor 1268	

the areas of the confirmed peaks will be deducted from the total toxaphene area. The corrected total area under the curve will then be used to calculate the results of analyses for the samples.

All results of analyses will be reported as "Chlorinated Camphene," and the results will be qualified as "**JN**." The "**J**" qualifier indicates an estimated value, and the "**N**" qualifier indicates presumptive evidence for the presence of chlorinated camphene.

C.) TOXAPHENE CONGENERS AS REFERENCE STANDARDS

In this calculation procedure, a five-point calibration curve will be constructed by using the 22-component toxaphene congener mixture from Dr. Ehrenstorfer Laboratories, and the Hx-Sed and Hp-Sed solutions from Promochem. Based on experience at the EPA Region IV Laboratory, the suggested concentrations for the calibration solutions are 0.001, 0.002, 0.004, 0.008, and 0.016 &g/mL. However, each laboratory must demonstrate that its GC system can attain similar sensitivity levels. The reference solution of B7-1000 cannot be used for quantitative measurements.

The GC component peaks will be identified by the laboratory data system based on R_t data. The R_t windows for each congener peak should be within ± 0.03 minutes of the average R_t of the initial calibration run for each congener peak. Baselines will be constructed under the component peaks using the "valley-to-valley" procedure that is normally used in the quantitation of individual pesticides *via* EPA SW-846 Method 8081A. If baselines to peak shoulders, or other incorrect baselines, are drawn by the data system, the analyst is directed to ignore these incorrect baselines and to assign baselines manually. The manual assignment of baselines must be documented on each chromatogram and in the report of analysis. The Georgia EPD laboratory will print a "before" and an "after" chromatogram to document the manual assignment of baselines. Peak heights will be used for all calculations.

The results of analyses will be reported for each individual congener in units of micrograms per kilogram, dry weight (&rg/kg, d.w.). The congeners will be identified and reported using their shorthand notations, for example, Hx-Sed, Parlar 11, Parlar 44, and Parlar 69. No total, or sum of the congeners, results will be reported. A congener that is detected on the quantitation column, but whose identity cannot be confirmed on the confirmation column, will be reported as "U," or Not Detected.

D.) TOXAPHENE CONGENERS AS GUIDES FOR PEAK SELECTION

In this calculation procedure, a five-point toxaphene calibration curve will be constructed using a technical toxaphene standard. This calibration curve will be used to calculate the results of analyses. However, the 22-component toxaphene congener mixture from Dr. Ehrenstorfer Laboratories, Hx-Sed and Hp-Sed from Promochem, and B7-1000 will be used for the identification and the selection of the component peaks that will be used for the calculation of the results of analyses in the sample extracts.

The baselines under the chromatograms are to be drawn from valley-to-valley under the peaks to follow the "hump" that is typical of a toxaphene chromatogram. If baselines to peak shoulders, or other incorrect baselines, are drawn, the analyst is directed to ignore these incorrect baselines and to assign baselines manually to follow the "hump." The baselines in the chromatograms of the samples must be placed under the peaks exactly as in the calibration standard. The manual assignment of baselines must be documented on each chromatogram and in the report of analysis. The Georgia EPD laboratory will print a "before" and an "after" chromatogram to document the manual assignment of baselines.

For the identification of the peaks detected, the R_t of the component peaks in the chromatograms of the sample extracts will be compared with the R_t of the individual congeners by using the entire chromatogram of each sample. Those peaks detected in the samples that match the R_t of the congeners will be selected for the calculation of the results of analyses. However, the technical toxaphene calibration curve will be used for converting the GC peak heights to microgram per kilogram (&rg/kg) units. The R_t of the peaks selected as described above will be used to identify the matching R_t peaks in the technical toxaphene calibration solutions.

Each peak selected for calibration will be calculated as if it reflected the total concentration of the standard (i.e. for a 500 µg/kg standard, each peak corresponding to a congener will be calibrated and calculated as if it were 500 µg/kg itself). Results for the samples will be calculated by averaging the results of confirmed congener peaks.

Before reporting the results of analyses for samples calculated by this procedure, the analyst must be certain that the chromatogram would be interpreted as containing toxaphene. There must be a minimum of four peaks identified as toxaphene components. If four or more peaks are detected and identified as matches for the R_t of the toxaphene congeners, then the height of each corresponding peak in the technical toxaphene standard shall be used for the calculation of the results of analyses. If fewer than four peaks are detected and identified as matches for the toxaphene congeners, additional peaks from the back half of the technical toxaphene reference standard chromatogram may be selected for use in the calculating the results of analyses. However, at least one of the four peaks used for the calculation must match the R_t of one of the congeners; and no more than three peaks from the back half of the technical toxaphene chromatogram may be used. If fewer than four components can be identified as described above, the result of analysis for that particular sample will be reported as "**U**," or Not Detected.

The response factors for the corresponding technical toxaphene component peaks are then used to convert the sample component peak heights to numerical values. These microgram per kilogram, dry weight (&g/kg, d.w.) values will be reported as "Chlorobornanes," and all reported results of analyses will be qualified as "**J**," or estimated values.

If, after calculating a result of analysis for a particular sample, the relative percent difference (RPD) between the calculated results from the quantitation column and from the confirmation column differ by more than forty percent (40%), the result will be qualified as "**N**." This is consistent with the guidelines provided by U.S. EPA Method 8081A.

7.) REPORTING OF RESULTS OF ANALYSES

The results of analyses from each laboratory will be reported to Dr. Randall Manning of the State of Georgia EPD in Atlanta, Georgia. He will tabulate the results in DRAFT form. The GC-ECD results from all participating laboratories will be grouped for comparison in one table. To preserve anonymity, letters (A, B, C, and D) will identify the results from the participating laboratories in the draft data table. The data in the report produced by Dr. Manning should include reported qualifiers to reinforce the idea that these data are not actionable data. In table footnotes defining the qualifiers, it is also recommended that mention be made that these data are not suitable for risk assessment or any other regulatory purposes.

The GC-ECNI-MS results of analyses will be presented in a separate table.

Copies of the DRAFT data tables will be sent to each participant: the EPA, EPD, EnChem, SkIO, Hercules, and FJC Analytical Consulting, Inc. Subsequent to receipt of those results, representatives from each participant (above) will meet to review in detail their experiences with the calculation procedures and to discuss the results of analyses. Each participating laboratory representative will bring to the meeting:

1.) copies of the chromatograms of all sample extracts,
2.) copies of the chromatograms of all calibration solutions,
3.) all QA/QC data (including all chromatograms),
4.) tabulated results of analyses, and
5.) case narratives for the work performed in the laboratory.

At the conclusion of the review meeting, a future course of action, including the modification or refinement of the above-described procedures, if needed, and a list of recommendations, if any, will be published.

7. REFERENCES

1.) United States Environmental Protection Agency, "Test Methods for Evaluation of Solid Waste, Volume 1B: Laboratory Manual, Physical/Chemical Methods, (SW-846)," Method 8081A, Organochlorine Pesticides and Polychlorinated Biphenyls by Gas Chromatography, Revision 1, December 1996, or the latest approved, published Revision.

2.) United States Environmental Protection Agency, "Test Methods for Evaluation of Solid Waste, Volume 1B: Laboratory Manual, Physical/Chemical Methods, (SW-846)," Method 3550A, Ultrasonic Extraction, Revision 1, November 1992, or the latest approved, published Revision.

3.) Restek, Inc., 110 Banner Circle, Bellefonte, Pennsylvania 16823.

4.) Revision O, Sampling and Analysis Plan, Quality Assurance Project Plan, Health & Safety Plan; Terry Creek Site, Brunswick, Georgia; Prepared for Hercules Incorporated by GeoSyntec Consultants, August 1997. Submitted to EPA on August 15, 1997.

5.) Carlin, F.J.; Revells, H.L.; and Reed, D.L., Chemosphere, **41** (2000) 481-486.

6.) McDonald, J. G., Vetter, W., and Hites, R. A., Anal. Chem. **2001**, 73, 1374-1376.

7.) EQ Laboratories Inc., 225 Peachtree Street N.E., Suite 506, Atlanta, Georgia 30303.

8.) Promochem LLC, P.O. Box 1126, 2931 Soldier Springs Road, Laramie, Wyoming 82070.

9.) Maruya, K. A., Vetter, W., Wakeham, S. G., and Francendese, L. Environ. Toxicol. Chem. 2000, 19, 2198-2203.

10.) Maruya, K. A., Walters, T. L., and Manning, R. O. Estuaries in press.

8.) ANALYSES OF EXTRACTS USING GC-ECNI-MS

The following section describes the procedure to be used by Dr. Keith A. Maruya at the Skidaway Institute of Oceanography (SkIO) to perform the analyses of sediment extracts by his gas chromatography-electron capture negative ionization-mass spectrometry (GC-ECNI-MS) procedure. This approach has been adapted from a previous exercise in which toxaphene residue levels were estimated in tissue samples[10]. The gas chromatography-electron capture detector (GC-ECD) analyses at SkIO will be performed using the four calibration and calculation protocols described earlier in this document. The GC-ECD and GC-ECNI-MS analyses will be performed on the sediment extracts, as received, without additional cleanup steps. If necessary, the internal standard compounds required for ECNI-MS analyses will be added to the extracts at SkIO before analyses are performed. The results of analyses from the GC-ECNI-MS procedure will be reported in a separate table from the GC-ECD results reported by the four participating laboratories.

Gas Chromatography. The sediment extracts (1 µl) will be injected into a Hewlett Packard 6890 Series II GC coupled to a 5973 mass selective detector operating in the ECNI mode. A fused silica column [30 m (L) x 0.25 mm (OD)] coated with either 0.25 µm of DB-5 or DB-XLB will be used for this analysis. The GC oven will be programmed using a method that is similar to that used for GC-ECD analyses, e.g.: 120°C (hold 1 min); ramp to 200°C @ 10°C min^{-1} (hold 1 min); ramp to 280°C @ 2°C/min (hold 11 min) for a total run time of 60 min. The injector will be maintained isothermal at between 220°C and 250°C. Methane at a pressure of ~1 torr will be used as the moderating gas. The quadrupole MS and ion source will be maintained at 106 °C and 150°C, respectively. In the full scan mode, the MS will be scanned between 200-500 daltons at 1.3 cycles s^{-1}. In the single ion monitoring (SIM) mode, the following ions will be monitored (with the corresponding homolog given in parentheses): 273/275 (Cl_5); 307/309 (Cl_6); 343/345 (Cl_7); 377/379 (Cl_8); 411/413 (Cl_9); and 445/447 (Cl_{10}) in accordance with Vetter and Maruya (2000).

Concentrations of individual toxaphene congeners will be based on a 22-component mixture of chlorinated monoterpenes ("TM2", Dr. Ehrenstorfer, Augsburg, Germany) and solutions of Hx- and Hp-Sed (Promochem LLC, Wesel, Germany). The 17 chlorobornane and 5 chlorocamphene congeners in TM2 represent Cl_6-Cl_{10} homologs (Table 1). Serial dilutions of TM2 and Hx-/Hp-Sed ranging from 2-100 pg in hexane will be used to generate a 6-point calibration curve and to compute mean response factors. The retention times for Hx- and Hp-Sed purchased from Promochem will be compared with that obtained from a solution provided by Dr. G. Fingerling (Technical University Munich, Germany

Total toxaphene concentrations (❹TOX) will be estimated by calibrating the GC-ECNI-MS with a technical toxaphene product standard ("TTX") provided by J. Hoffman of Hercules Inc, or alternatively, by a toxaphene reference standard purchased from Restek (F. Carlin pers. comm.). A known mass of TTX will be diluted in CH_2Cl_2 to create a concentrated stock (~2800 $\mu g\ ml^{-1}$). Serial dilutions will then be created in hexane at concentrations between 0.28 – 55 $\mu g\ ml^{-1}$. An average response factor for TTX will be computed by summing the areas of all peaks in the unresolved "hump" of unmodified toxaphene (Fig. 1), obtained by full scan ECNI-MS, and dividing by the known standard mass. The TTX response will then applied to the summed area of peaks eluting within a specified retention time for each sample extract. Areas for peaks corresponding to non-toxaphene compounds eluting within this time window (e.g. Cl_4-Cl_7 PCBs, and organochlorine pesticides such as chlordanes and DDTs) will be subtracted from estimates of ❹TOX. Retention times and mass spectra for PCB and pesticide analytes subtracted in this fashion will be recorded from standard reference mixtures SRM2262 and 2261, respectively (NIST, Gaithersburg, MD, USA).

Quality Assurance/Quality Control. Individual congener and total toxaphene concentrations will be validated against a comprehensive, performance based set of quality assurance/quality control (QA/QC) criteria. Surrogate compounds (dibromooctafluorobiphenyl or DBOFB and α-HCH) will be added to each sample extract prior to GC analysis to monitor post-delivery target analyte recovery. Initial GC-ECNI-MS calibration with the TM2 and Hx-/Hp-Sed standards shall result in R^2 values exceeding 0.99 for all target components. Continuing instrument calibration shall be monitored using mid-level toxaphene congener standard and TTX solutions injected every 10-12 samples; the mean relative percent deviation from the initial calibration response for TM2 analytes shall be maintained at or below 15% for the duration of the study. Mass calibration of the GC-MS system used for analyte confirmation shall be performed daily.

The presence of a toxaphene congener shall be considered confirmed using GC-ECNI-MS if the retention time ($\pm$ 0.1 s) and mass spectrum ($\geq$70%) matched that of a target analyte in the toxaphene congener standard solutions (i.e. TM2 or Hx-/Hp-Sed) (Table 1). Since $[M\text{-}Cl]^-$ fragment ions are typically predominant for toxaphene congeners using ECNI-MS (Jansson and Wideqvist 1983), the degree of chlorination for any unidentified chlorocamphene congeners shall be tentatively assigned. Several Cl_7-Cl_{10} PCB congeners, the majority of which eluted outside the expected ❹TOX retention time window, may be present. These PCBs are attributable to Aroclor 1268 contamination originating from a different tidal creek system in the Turtle/Brunswick river estuary (Maruya and Lee 1998). Peaks corresponding to PCB congeners in Aroclor 1268 that elute within the ❹TOX retention time window (e.g. IUPAC numbers 153, 187, 202 and 201) will not be included in estimated ❹TOX calculations. Pesticide analytes that are potential interferences (e.g. oxychlordane; 4,4'-DDE; 4,4'-DDT) shall be analyzed for using GC-ECNI- or electron impact MS. Peaks in the ECD chromatogram that are within the specified ❹TOX retention time window and that correspond with pesticide analytes confirmed by GC-MS will be subtracted out of ❹TOX estimates.

Data and Statistical Analyses. All chromatographic data, including peak retention times, heights, areas, and mass spectra will be obtained using HP ChemStation software. All instrument calibration and sample concentration data shall be compiled and analyzed using Microsoft Excel 97 SR-2 spreadsheet software. Statistical evaluation of the data (e.g. linear regression and correlational analyses) will be performed using the data analysis tool in Excel.

Literature Cited

JANSSON, B., AND U. WIDEQVIST. 1983. Analysis of toxaphene (PCC) and chlordane in biological samples by NCI mass spectrometry. *International Journal of Environmental Analytical Chemistry* 13:309-321.

MARUYA, K. A., AND R. F. LEE. 1998. Aroclor 1268 and toxaphene in fish from a southeastern U.S. estuary. *Environmental Science and Technology* 32:1069-1075.

MARUYA, K. A., T. L. WALTERS, AND R. O. MANNING. Residues of toxaphene in finfish and shellfish from Terry and Dupree Creeks, Georgia, U.S.A. *Estuaries* in press.

SMALLING, K. L., AND K. A. MARUYA. 2001. Gas chromatographic separation of toxaphene residues by DB-XLB. *Journal of Separation Science* 24:104-108.

VETTER, W., AND K. A. MARUYA. 2000. Congener and enantioselective analysis of toxaphene residues in sediment and biota from a contaminated estuarine wetland. *Environmental Science and Technology* 34:1627-1635.

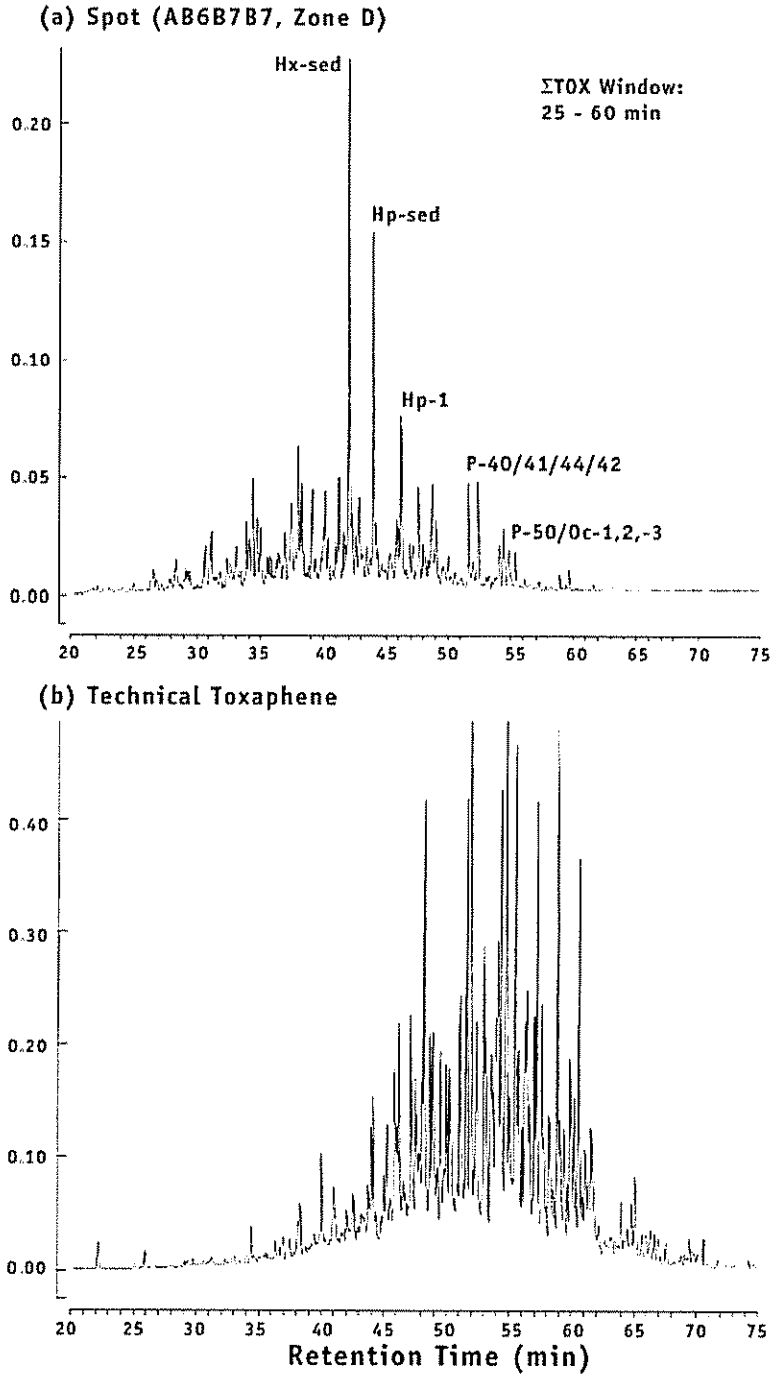
TABLE 1. Structure, homolog and chromatographic properties of individual chlorinated monoterpene ("toxaphene") target analytes. Structural formulas are for chlorobornanes except for P11, 12, 15, 25 and 31, which are chlorocamphenes (CC). Relative retention time (RRT) data from Smalling and Maruya (2001).

Parlar No.	Homolog	Structure	RRT (DB-5)	RRT (DB-XLB)
11	6	5,5,6-exo,8,9,10-hexaCC	0.585	0.599
12	6	5-exo,6-endo,8,9,9,10-hexaCC	0.591	0.603
15	6	5-exo,6-endo,7-anti,8,9,10-hexaCC	0.619	0.636
Hx-Sed*	6	2-exo,3-endo,6-exo,8,9,10	0.621	0.653
21	7	2,2,5,5,9,10,10	0.661	0.675
Hp-Sed*	7	2-endo,3-exo,5-endo,6-exo,8,9,10	0.662	0.669
25	7	5,5,6-exo,8,9,9,10-heptaCC	0.695	0.707
26	8	2-endo,3-exo,5-endo,6-exo,8,8,10,10	0.708	0.687
31	8	5,5,6-exo,8,8,9,9,10-octaCC	0.748	0.738
32	7	2,2,5-endo,6-exo,8,9,10	0.748	0.777
38	8	2,2,5,5,9,9,10,10	0.792	0.801
39	8	2,2,3-exo,5-endo,6-exo,8,9,10	0.813	0.812
40	8	2-endo,3-exo,5-endo,6-exo,8,9,10,10	0.824	0.828
41	8	2-exo,3-endo,5-exo,8,9,9,10,10	0.824	0.820
42a/b	8	2,2,5-endo,6-exo,8,8,9,10 (or 8,9,9,10)	0.832	0.839
44	8	2-exo,5,5,8,9,9,10,10	0.844	0.844
50	9	2-endo,3-exo,5-endo,6-exo,8,8,9,10,10	0.888	0.860
51	8	2,2,5,5,8,9,10,10	0.900	n/a
56	9	2,2,5-endo,6-exo,8,8,9,10,10	0.945	0.928
58	9	2,2,3-exo,5,5,8,9,10,10	0.959	0.947
59	9	2,2,5-endo,6-exo,8,9,9,10,10	0.968	0.955
62	9	2,2,5,5,8,9,9,10,10	1.000	1.000
63	9	2-exo,3-endo,5-exo,6-exo,8,8,9,10,10	1.016	1.008
69	10	2,2,5,5,6-exo,8,9,9,10,10	1.151	1.127

* not present in the 22-component "TM2" mixture (Dr. Ehrenstorfer, Augsburg, Germany)

n/a not available

Figure 1. GC-ECD F2 chromatogram of (a) edible tissue of spot (*Leiostomus xanthurus*) from Dupree Creek and (b) unmodified technical toxaphene product standard (supplied by J. Hoffman, Hercules Inc.).



Attachment 2

Attachment 2
Summary of Groundwater Results

Well	Parameter	Extract ID	Analysis Method	EnChem	Flag	Result	EQL*	MDL	Units
N-01	Toxaphene	856921-001	SW846 8081A	EnChem	<	3.0	3.0		ug/L
N-01	Chlorinated Camphene	856921-001	SW846 8081A	EnChem	<	3.0	3.0		ug/L
N-01	Chlorinated Camphene	856921-001	ECNI-MS (RT= 30-62 min)	SKIO	<=	0.00584	2.22000	0.22200	ug/L
N-01	Chlorinated Camphene	856921-001	ECNI-MS (RT= 35-62 min)	SKIO	<=	0.00584	2.22000	0.22200	ug/L
N-01	Chlorinated Camphene	856921-001	ECD (RT= 24-49 min)	SKIO	<=	0.03260	2.22000	0.22200	ug/L
N-02	Toxaphene	856921-002	SW846 8081A	EnChem	<	3.0	3.0		ug/L
N-02	Chlorinated Camphene	856921-002	SW846 8081A	EnChem	<	3.0	3.0		ug/L
N-02	Chlorinated Camphene	856921-002	ECNI-MS (RT= 30-62 min)	SKIO	<=	0.02130	2.22000	0.22200	ug/L
N-02	Chlorinated Camphene	856921-002	ECNI-MS (RT= 35-62 min)	SKIO	<=	0.02480	2.22000	0.22200	ug/L
N-02	Chlorinated Camphene	856921-002	ECD (RT= 24-49 min)	SKIO	<=	0.03450	2.22000	0.22200	ug/L
N-9S	Toxaphene	856921-003	SW846 8081A	EnChem	<	3.0	3.0		ug/L
N-9S	Chlorinated Camphene	856921-003	SW846 8081A	EnChem	<	3.0	3.0		ug/L
N-9S	Chlorinated Camphene	856921-003	ECNI-MS (RT= 30-62 min)	SKIO	<=	0.04300	2.22000	0.22200	ug/L
N-9S	Chlorinated Camphene	856921-003	ECNI-MS (RT= 35-62 min)	SKIO	<=	0.04630	2.22000	0.22200	ug/L
N-9S	Chlorinated Camphene	856921-003	ECD (RT= 24-49 min)	SKIO	<=	0.02020	2.22000	0.22200	ug/L
N-9D	Toxaphene	856921-004	SW846 8081A	EnChem	<	3.0	3.0		ug/L
N-9D	Chlorinated Camphene	856921-004	SW846 8081A	EnChem	<	3.0	3.0		ug/L
N-9D	Chlorinated Camphene	856921-004	ECNI-MS (RT= 30-62 min)	SKIO	<=	0.01440	2.22000	0.22200	ug/L
N-9D	Chlorinated Camphene	856921-004	ECNI-MS (RT= 35-62 min)	SKIO	<=	0.01440	2.22000	0.22200	ug/L
N-9D	Chlorinated Camphene	856921-004	ECD (RT= 24-49 min)	SKIO	<=	0.03400	2.22000	0.22200	ug/L
N-6SR	Toxaphene	856921-005	SW846 8081A	EnChem	<	3.0	3.0		ug/L
N-6SR	Chlorinated Camphene	856921-005	SW846 8081A	EnChem	J	1.6	3.0		ug/L
N-6SR	Chlorinated Camphene	856921-005	ECNI-MS (RT= 30-62 min)	SKIO		1.38000	2.22000	0.22200	ug/L
N-6SR	Chlorinated Camphene	856921-005	ECNI-MS (RT= 35-62 min)	SKIO		1.55000	2.22000	0.22200	ug/L
N-6SR	Chlorinated Camphene	856921-005	ECD (RT= 24-49 min)	SKIO		1.02000	2.22000	0.22200	ug/L
N-6DR	Toxaphene	856921-006	SW846 8081A	EnChem	<	3.0	3.0		ug/L
N-6DR	Chlorinated Camphene	856921-006	SW846 8081A	EnChem	<	3.0	3.0		ug/L
N-6DR	Chlorinated Camphene	856921-006	ECNI-MS (RT= 30-62 min)	SKIO	<=	0.00258	2.22000	0.22200	ug/L
N-6DR	Chlorinated Camphene	856921-006	ECNI-MS (RT= 35-62 min)	SKIO	<=	0.00258	2.22000	0.22200	ug/L
N-6DR	Chlorinated Camphene	856921-006	ECD (RT= 24-49 min)	SKIO	<=	0.00513	2.22000	0.22200	ug/L
N-11	Toxaphene	856921-007	SW846 8081A	EnChem	<	3.0	3.0		ug/L
N-11	Chlorinated Camphene	856921-006	SW846 8081A	EnChem	<	3.0	3.0		ug/L
N-11	Chlorinated Camphene	856921-007	ECNI-MS (RT= 30-62 min)	SKIO		0.67100	2.22000	0.22200	ug/L
N-11	Chlorinated Camphene	856921-007	ECNI-MS (RT= 35-62 min)	SKIO		0.74000	2.22000	0.22200	ug/L
N-11	Chlorinated Camphene	856921-007	ECD (RT= 24-49 min)	SKIO		0.59900	2.22000	0.22200	ug/L
N-14S	Toxaphene	856996-001	SW846 8081A	EnChem	<	3.0	3.0		ug/L
N-14S	Chlorinated Camphene	856996-001	SW846 8081A	EnChem	<	3.0	3.0		ug/L

Attachment 2
Summary of Groundwater Results

Well	Parameter	Extract ID	Analysis Method	EnChem	Flag	Result	EQL*	MDL	Units
N-14S	Chlorinated Camphene	856996-001	ECNI-MS (RT= 30-62 min)	SKIO	<=	0.02630	2.22000	0.22200	ug/L
N-14S	Chlorinated Camphene	856996-001	ECNI-MS (RT= 35-62 min)	SKIO	<=	0.02630	2.22000	0.22200	ug/L
N-14S	Chlorinated Camphene	856996-001	ECD (RT= 24-49 min)	SKIO	<=	0.02900	2.22000	0.22200	ug/L
N-14D	Toxaphene	856996-002	SW846 8081A	EnChem	<	3.0	3.0		ug/L
N-14D	Chlorinated Camphene	856996-002	SW846 8081A	EnChem	<	3.0	3.0		ug/L
N-14D	Chlorinated Camphene	856996-002	ECNI-MS (RT= 30-62 min)	SKIO	<=	0.00091	2.22000	0.22200	ug/L
N-14D	Chlorinated Camphene	856996-002	ECNI-MS (RT= 35-62 min)	SKIO	<=	0.00091	2.22000	0.22200	ug/L
N-14D	Chlorinated Camphene	856996-002	ECD (RT= 24-49 min)	SKIO	<=	0.03700	2.22000	0.22200	ug/L
N-10	Toxaphene	856996-003	SW846 8081A	EnChem	<	3.0	3.0		ug/L
N-10	Chlorinated Camphene	856996-003	SW846 8081A	EnChem	<	3.0	3.0		ug/L
N-10	Chlorinated Camphene	856996-003	ECNI-MS (RT= 30-62 min)	SKIO	<=	0.00687	2.22000	0.22200	ug/L
N-10	Chlorinated Camphene	856996-003	ECNI-MS (RT= 35-62 min)	SKIO	<=	0.00687	2.22000	0.22200	ug/L
N-10	Chlorinated Camphene	856996-003	ECD (RT= 24-49 min)	SKIO	<=	0.07390	2.22000	0.22200	ug/L
N-12	Toxaphene	856996-004	SW846 8081A	EnChem	<	3.0	3.0		ug/L
N-12	Chlorinated Camphene	856996-004	SW846 8081A	EnChem	<	3.0	3.0		ug/L
N-12	Chlorinated Camphene	856996-004	ECNI-MS (RT= 30-62 min)	SKIO	<=	0.01690	2.22000	0.22200	ug/L
N-12	Chlorinated Camphene	856996-004	ECNI-MS (RT= 35-62 min)	SKIO	<=	0.02620	2.22000	0.22200	ug/L
N-12	Chlorinated Camphene	856996-004	ECD (RT= 24-49 min)	SKIO	<=	0.14100	2.22000	0.22200	ug/L
N-08	Toxaphene	856996-005	SW846 8081A	EnChem	<	3.0	3.0		ug/L
N-08	Chlorinated Camphene	856996-005	SW846 8081A	EnChem	<	3.0	3.0		ug/L
N-08	Chlorinated Camphene	856996-005	ECNI-MS (RT= 30-62 min)	SKIO	<=	0.00547	2.22000	0.22200	ug/L
N-08	Chlorinated Camphene	856996-005	ECNI-MS (RT= 35-62 min)	SKIO	<=	0.00547	2.22000	0.22200	ug/L
N-08	Chlorinated Camphene	856996-005	ECD (RT= 24-49 min)	SKIO	<=	0.03570	2.22000	0.22200	ug/L
N-03	Toxaphene	856996-006	SW846 8081A	EnChem	<	3.0	3.0		ug/L
N-03	Chlorinated Camphene	856996-006	SW846 8081A	EnChem	<	3.0	3.0		ug/L
N-03	Chlorinated Camphene	856996-006	ECNI-MS (RT= 30-62 min)	SKIO	<=	0.22200	2.22000	0.22200	ug/L
N-03	Chlorinated Camphene	856996-006	ECNI-MS (RT= 35-62 min)	SKIO	<=	0.22200	2.22000	0.22200	ug/L
N-03	Chlorinated Camphene	856996-006	ECD (RT= 24-49 min)	SKIO	<=	0.06080	2.22000	0.22200	ug/L
N-05	Chlorinated Camphene	857028-001	SW846 8081A	EnChem	<	3.0	3.0		ug/L
N-05	Toxaphene	857028-001	SW846 8081A	EnChem	<	3.0	3.0		ug/L
N-05	Chlorinated Camphene	857028-001	ECNI-MS (RT= 30-62 min)	SKIO	<=	0.01140	2.22000	0.22200	ug/L
N-05	Chlorinated Camphene	857028-001	ECNI-MS (RT= 35-62 min)	SKIO	<=	0.01140	2.22000	0.22200	ug/L
N-05	Chlorinated Camphene	857028-001	ECD (RT= 24-49 min)	SKIO	<=	0.04420	2.22000	0.22200	ug/L
N-07	Toxaphene	857028-002	SW846 8081A	EnChem	<	3.0	3.0		ug/L
N-07	Chlorinated Camphene	857028-002	SW846 8081A	EnChem	<	3.0	3.0		ug/L
N-07	Chlorinated Camphene	857028-002	ECNI-MS (RT= 30-62 min)	SKIO	<=	0.22200	2.22000	0.22200	ug/L
N-07	Chlorinated Camphene	857028-002	ECNI-MS (RT= 35-62 min)	SKIO	<=	0.22200	2.22000	0.22200	ug/L
N-07	Chlorinated Camphene	857028-002	ECD (RT= 24-49 min)	SKIO	<=	0.03280	2.22000	0.22200	ug/L

Attachment 2
Summary of Groundwater Results

Well	Parameter	Extract ID	Analysis Method	EnChem	Flag	Result	EQL*	MDL	Units
N-13	Toxaphene	857028-003	SW846 8081A	EnChem	<	3.0	3.0		ug/L
N-13	Chlorinated Camphenes	857028-003	SW846 8081A	EnChem	<	3.0	3.0		ug/L
N-13	Chlorinated Camphenes	857028-003	ECNI-MS (RT= 30-62 min)	SKIO	<=	0.22200	2.22000	0.22200	ug/L
N-13	Chlorinated Camphenes	857028-003	ECNI-MS (RT= 35-62 min)	SKIO	<=	0.22200	2.22000	0.22200	ug/L
N-13	Chlorinated Camphenes	857028-003	ECD (RT= 24-49 min)	SKIO	<=	0.02930	2.22000	0.22200	ug/L
N-15S	Toxaphene	857028-004	SW846 8081A	EnChem	<	3.0	3.0		ug/L
N-15S	Chlorinated Camphenes	857028-004	SW846 8081A	EnChem	<	3.0	3.0		ug/L
N-15S	Chlorinated Camphenes	857028-004	ECNI-MS (RT= 30-62 min)	SKIO	<=	0.00413	2.22000	0.22200	ug/L
N-15S	Chlorinated Camphenes	857028-004	ECNI-MS (RT= 35-62 min)	SKIO	<=	0.00413	2.22000	0.22200	ug/L
N-15S	Chlorinated Camphenes	857028-004	ECD (RT= 24-49 min)	SKIO	<=	0.03760	2.22000	0.22200	ug/L
N-15D	Toxaphene	857028-005	SW846 8081A	EnChem	<	3.0	3.0		ug/L
N-15D	Chlorinated Camphenes	857028-005	SW846 8081A	EnChem	<	3.0	3.0		ug/L
N-15D	Chlorinated Camphenes	857028-005	ECNI-MS (RT= 30-62 min)	SKIO	<=	0.22200	2.22000	0.22200	ug/L
N-15D	Chlorinated Camphenes	857028-005	ECNI-MS (RT= 35-62 min)	SKIO	<=	0.22200	2.22000	0.22200	ug/L
N-15D	Chlorinated Camphenes	857028-005	ECD (RT= 24-49 min)	SKIO	<=	0.01710	2.22000	0.22200	ug/L
RBLK-05101	Chlorinated Camphenes	857028-006	SW846 8081A	EnChem	<	3.0	3.0		ug/L
RBLK-05101	Toxaphene	857028-006	SW846 8081A	EnChem	<	3.0	3.0		ug/L
RBLK-05101	Chlorinated Camphenes	857028-006	ECNI-MS (RT= 30-62 min)	SKIO	<=	0.22200	2.22000	0.22200	ug/L
RBLK-05101	Chlorinated Camphenes	857028-006	ECNI-MS (RT= 35-62 min)	SKIO	<=	0.22200	2.22000	0.22200	ug/L
RBLK-05101	Chlorinated Camphenes	857028-006	ECD (RT= 24-49 min)	SKIO	<=	0.05460	2.22000	0.22200	ug/L

* EQL = Reported by EnChem and MDL X 10 for SKIO

Skidaway Institute of Oceanography®
University System of Georgia

5 April 2005

Mr. Tim Hassett
Hercules Incorporated
Hercules Research Center
500 Hercules Road
Wilmington, Delaware 19808-1599
(302) 995-3456 phone

Dear Tim:

Enclosed is our summary report for the chlorocamphene analysis of 009 Landfill groundwater samples (n=23) supplied by ENCHEM on March 18, 2005. Please note that the concentrations in Table 1 have been updated to include the post-Cu treatment analyses undertaken in an attempt to remove ECD interferences. Also, please note my contact information (below) has changed as of April 1, 2005.

Sincerely yours,

K.A. Maruya

Keith A. Maruya
Associate Professor

Southern California Coastal Water Research Project (SCCWRP)
7171 Fenwick Lane
Westminster, CA 92683
714-372-9214 (phone); 714-894-9699 (fax)
email address: keithm@sccwrp.org

Summary of Methods

Twenty three (23) hexane extracts in glass vials packed on ice were received via overnight courier from Tod Noltemeyer of EN CHEM (Green Bay, WI) on 3/16/05 (see Table 1 for sample ID). Extracts were reduced to 1.0 mL using a gentle stream of nitrogen (HP, >99.99%) and transferred into 2.0 mL clear GC autosampler vials with Teflon-lined silicone septa. The extracts of 856996-007MS and -008MSD were not concentrated to 1.0 mL. One µL of each extract was analyzed using a (1) Varian 3400CX gas chromatograph with electron capture detection (GC-ECD) and (2) an Agilent 6890/5973 GC-mass selective detector operating in the electron capture negative ionization and selected ion monitoring mode (ECNI-MS-SIM). The GC stationary phase for each instrument was DB-XLB (J&W Scientific/Agilent, Folsom, CA). Detailed conditions for GC-ECD and GC-ECNI-MS-SIM are available upon request. Total chlorocamphene concentrations (ΣCC) were estimated the "total area under the curve" method (TAUC or Method 2 of the Toxaphene Working Group) using the mean response factor from an eight point calibration curve (0.111 to 55.4 ppm) of technical toxaphene product standard (TTX). Initial and final retention times for TAUC computations corresponded to B7-1000 and Parlar 69, respectively. Solutions of B7-1000 and the 22-component mixture containing Parlar 69 ("TM2") were obtained from Dr. Walter Vetter (University of Hohenheim, Germany) and Dr. Ehrenstorfer (Augsburg, Germany), respectively. Initial GC-ECD and GC-MS analyses revealed that a significant (likely sulfur containing) interference eluting early in the ΣCC retention time window was present in several extracts (Table 1), which were subsequently treated with acid-activated copper granules and reanalyzed by GC-ECD. Post-Cu treatment concentrations ranged from ~44 to 190% of pre-treatment results (Table 1).

Results and Discussion

Calibration curves for TTX were highly linear (R^2 of 0.998 and 0.999 for ECNI-MS and ECD, respectively). Based on a limit of quantitation (LOQ) equaling the lowest calibration standard (0.111 µg/mL) and an effective sample volume of 500 mL, the method detection limit (MDL) was for both techniques was 0.222 µg/L (=ppb). Thus, if chlorocamphenes were detected but at an estimated concentration that was less than the MDL, values are qualified by "<=" (less than or equal to). If no chlorocamphenes were detected, the concentration was reported as "<0.222 µg/L" (less than the MDL). In addition, two estimated concentrations based on ECNI-MS-SIM are reported (Table 1): the results in the first column reflect the retention time window defined above (i.e. 35-62 min) and the second column includes suspected lower chlorinated (e.g. pentachlorinated monoterpenes) eluting before B7-1000 (i.e. between 30-35 min). Estimated ΣCC ranged from nondetect (<0.222 µg/L) to 1.55 µg/L for sample 856921-005. The only other sample with ΣCC above the MDL (0.740 µg/L) was 856921-007 (0.740 µg/L). It was suspected that ECD would give intrinsically greater concentrations because of the greater selectivity of ECNI-MS; however, a significant interference in ECD chromatograms in more than half of the extracts precluded accurate integration of low level peaks in the pre-defined ΣCC retention time window. This large interference appeared to be related to sulfur based on full scan ECNI-MS analysis. Future analyses of 009 Landfill groundwater samples should include a step to remove sulfur (e.g. using activated copper granules) prior to ECD/TAUC analyses. Because ECNI-MS-SIM as programmed was immune to this interference, the results using this method are considered to be more accurate than those reported for ECD.

Table 1. Estimated total chlorocamphene concentration (ΣCC) in 009 Landfill (Brunswick, GA) groundwater samples.

Sample ID		Conc (ppb)		Comments
Method	ECNI-MS	ECNI-MS	ECD	
Ret Time Window	35-62 min	30-62 min	24-49 min	
856921-001	<=0.00584	<=0.00584	<=0.0326	
856921-002	<=0.0213	<=0.0248	<=0.0616	Post-Cu: <=0.0345 ppb
856921-003	<=0.0430	<=0.0463	<=0.0219	Post-Cu: <=0.0202 ppb
856921-004	<=0.0144	<=0.0144	<=0.0209	Post-Cu: <=0.0340 ppb
856921-005	1.38	1.55	0.722	Post-Cu: 1.02 ppb
856921-006	<=0.00258	<=0.00258	<=0.00513	
856921-007	0.671	0.740	0.547	Post-Cu: 0.599 ppb
856996-001	<=0.0263	<=0.0263	<=0.0347	Post-Cu: <=0.0290 ppb
856996-002	<=0.000914	<=0.000914	<=0.0370	
856996-003	<=0.00687	<=0.00687	<=0.0739	
856996-004	<=0.0169	<=0.0262	<=0.141	
856996-005	<=0.00547	<=0.00547	<=0.0357	
856996-006	<0.222	<0.222	<=0.0222	Post-Cu: <=0.0608 ppb
856996-007MS	25.5	n/a	30.8	
856996-008MSD	24.5	n/a	28.7	
857028-001	<=0.0114	<=0.0114	<=0.0154	Post-Cu: <=0.0442 ppb
857028-002	<0.222	<0.222	<=0.0164	Post-Cu: <=0.0328 ppb
857028-003	<0.222	<0.222	<=0.0293	
857028-004	<=0.00413	<=0.00413	<=0.0443	Post-Cu: <=0.0376 ppb
857028-005	<0.222	<0.222	<=0.0171	
857028-006	<0.222	<0.222	<=0.0546	
svk1082-075mb	<=0.000553	<=0.000553	<=0.00310	
svk1082-075mbLCS	35.0	n/a	26.9	

EN CHEM

A Division of Pace Analytical Services, Inc.

1241 Bellevue Street, Suite 9
Green Bay, WI 54302
920-469-2436, Fax: 920-469-8827

Analytical Report Number: 856921

Client: HERCULES, INC - DE
Project Name: HERCULES/RMT INC.
Project Number: 70102 61

Lab Contact: Tod Noltemeyer
Collected By:
Report Serial No: 856921032120051706

Lab Sample Number	Field ID	Matrix	Collection Date
856921-001	N-01	GW	03/08/05
856921-002	N-02	GW	03/08/05
856921-003	N-9S	GW	03/08/05
856921-004	N-9D	GW	03/08/05
856921-005	N-6SR	GW	03/08/05
856921-006	N-6DR	GW	03/08/05
856921-007	N-11	GW	03/08/05
856996-001	N-14S	GW	03/09/05
856996-002	N-14D	GW	03/09/05
856996-003	N-10	GW	03/09/05
856996-004	N-12	GW	03/09/05
856996-005	N-08	GW	03/09/05
856996-006	N-03	GW	03/09/05
857028-001	N-05	GW	03/10/05
857028-002	N-07	GW	03/10/05
857028-003	N-13	GW	03/10/05
857028-004	N-15S	GW	03/10/05
857028-005	N-15D	GW	03/10/05
857028-006	RBLK-05101	GW	03/10/05
857028-007	TBLK-05101	GW	03/10/05

I certify that the data contained in this Final Report has been generated and reviewed in accordance with approved methods and Laboratory Standard Operating Procedure. Exceptions, if any, are discussed in the accompanying sample comments. Release of this final report is authorized by Laboratory management, as is verified by the following signature. This report shall not be reproduced, except in full, without the written consent of Pace Analytical Services, Inc. The sample results relate only to the analytes of interest tested.

Approval Signature

3/30/05
Date

En Chem

A Division of Pace Analytical Services, Inc.

1241 Bellevue Street
Green Bay, WI 54302
920-469-2436

Analytical Report Number: 856921

Client : HERCULES, INC - DE
Project Name HERCULES/RMT INC
Project Number 70102.61
Field ID : N-01

Matrix Type GROUNDWATER
Collection Date 03/08/05
Report Date : 03/21/05
Lab Sample Number 856921-001

INORGANICS

Test	Result		EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Total Suspended Solids	1.8	B	2.0	1	mg/L		03/15/05	EPA 160.2	EPA 160.2

TOXAPHENE Prep Date: 03/14/05

Analyte	Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Toxaphene	< 3.0	3.0	1	ug/L		03/16/05	SW846 3510C	SW846 8081A

Analyzed by:

Dawn J. Kearns, Pesticide/PCB Analyst

En Chem
A Division of Pace Analytical Services, Inc.

Analytical Report Number: 856921

1241 Bellevue Street
Green Bay, WI 54302
920-469-2436

Client : HERCULES, INC - DE
Project Name HERCULES/RMT INC
Project Number 70102 61
Field ID : N-02

Matrix Type GROUNDWATER
Collection Date 03/08/05
Report Date : 03/21/05
Lab Sample Number 856921-002

INORGANICS

Test	Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Total Suspended Solids	6.8	2.0	1	mg/L		03/15/05	EPA 160.2	EPA 160.2

TOXAPHENE Prep Date: 03/14/05

Analyte	Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Toxaphene	< 3.0	3.0	1	ug/L		03/16/05	SW846 3510C	SW846 8081A

Analyzed by:

Dawn J. Kearns, Pesticide/PCB Analyst

En Chem
A Division of Pace Analytical Services, Inc

1241 Bellevue Street
Green Bay, WI 54302
920-469-2436

Analytical Report Number: 856921

Client : HERCULES, INC. - DE
Project Name HERCULES/RMT INC
Project Number 70102.61
Field ID : N-9S

Matrix Type GROUNDWATER
Collection Date 03/08/05
Report Date : 03/21/05
Lab Sample Number 856921-003

INORGANICS

Test		Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Total Suspended Solids	<	2.0	2.0	1	mg/L		03/15/05	EPA 160.2	EPA 160.2

TOXAPHENE — Prep Date: 03/14/05

Analyte		Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Toxaphene	<	3.0	3.0	1	ug/L		03/16/05	SW846 3510C	SW846 8081A

Analyzed by:

Dawn J. Kearns, Pesticide/PCB Analyst

En Chem
A Division of Pace Analytical Services. Inc.

Analytical Report Number: 856921

1241 Bellevue Street
Green Bay, WI 54302
920-469-2436

Client : HERCULES. INC - DE
Project Name HERCULES/RMT INC
Project Number 70102.61
Field ID : N-9D

Matrix Type GROUNDWATER
Collection Date 03/08/05
Report Date : 03/21/05
Lab Sample Number 856921-004

INORGANICS

Test		Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Total Suspended Solids	<	2.0	2.0	1	mg/L		03/15/05	EPA 160.2	EPA 160.2

TOXAPHENE — Prep Date: 03/14/05

Analyte		Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Toxaphene	<	3 0	3 0	1	ug/L		03/16/05	SW846 3510C	SW846 8081A

Analyzed by:

Dawn J. Kearns, Pesticide/PCB Analyst

En Chem
A Division of Pace Analytical Services, Inc

1241 Bellevue Street
Green Bay, WI 54302
920-469-2436

Analytical Report Number: 856921

Client : HERCULES, INC. - DE
Project Name HERCULES/RMT INC.
Project Number 70102 61
Field ID : N-6SR

Matrix Type GROUNDWATER
Collection Date 03/08/05
Report Date : 03/21/05
Lab Sample Number 856921-005

INORGANICS

Test		Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Total Suspended Solids	<	2.0	2.0	1	mg/L		03/15/05	EPA 160.2	EPA 160.2

TOXAPHENE **Prep Date:** 03/14/05

Analyte		Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Toxaphene	<	3.0	3.0	1	ug/L		03/16/05	SW846 3510C	SW846 8081A

Analyzed by:

Dawn J. Kearns, Pesticide/PCB Analyst

En Chem
A Division of Pace Analytical Services, Inc.

Analytical Report Number: 856921

1241 Bellevue Street
Green Bay, WI 54302
920-469-2436

Client : HERCULES, INC - DE
Project Name HERCULES/RMT INC
Project Number 70102 61
Field ID : N-6DR

Matrix Type GROUNDWATER
Collection Date 03/08/05
Report Date : 03/21/05
Lab Sample Number 856921-006

INORGANICS

Test	Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Total Suspended Solids	2.8	2.0	1	mg/L		03/15/05	EPA 160.2	EPA 160.2

TOXAPHENE Prep Date: 03/14/05

Analyte	Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Toxaphene	< 3.0	3.0	1	ug/L		03/16/05	SW846 3510C	SW846 8081A

Analyzed by:

Dawn J. Kearns, Pesticide/PCB Analyst

En Chem
A Division of Pace Analytical Services, Inc

1241 Bellevue Street
Green Bay, WI 54302
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Analytical Report Number: 856921

Client : HERCULES, INC. - DE
Project Name HERCULES/RMT INC
Project Number 70102.61
Field ID : N-11

Matrix Type GROUNDWATER
Collection Date 03/08/05
Report Date : 03/21/05
Lab Sample Number 856921-007

INORGANICS

Test	Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Total Suspended Solids	13	2.0	1	mg/L		03/15/05	EPA 160.2	EPA 160.2

TOXAPHENE

Prep Date: 03/14/05

Analyte	Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Toxaphene	< 3.0	3.0	1	ug/L		03/16/05	SW846 3510C	SW846 8081A

Analyzed by:

Dawn J. Kearns, Pesticide/PCB Analyst

En Chem
A Division of Pace Analytical Services, Inc.

Analytical Report Number: 856921

1241 Bellevue Street
Green Bay, WI 54302
920-469-2436

Client : HERCULES, INC. - DE
Project Name HERCULES/RMT INC.
Project Number 70102 61
Field ID : N-14S

Matrix Type GROUNDWATER
Collection Date 03/09/05
Report Date : 03/21/05
Lab Sample Number 856996-001

INORGANICS

Test	Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Total Suspended Solids	5.0	2.0	1	mg/L		03/16/05	EPA 160.2	EPA 160.2

TOXAPHENE — Prep Date: 03/14/05

Analyte	Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Toxaphene	< 3.0	3.0	1	ug/L		03/16/05	SW846 3510C	SW846 8081A

Analyzed by:

Dawn J. Kearns, Pesticide/PCB Analyst

En Chem
A Division of Pace Analytical Services, Inc.

Analytical Report Number: 856921

1241 Bellevue Street
Green Bay, WI 54302
920-469-2436

Client : HERCULES, INC. - DE
Project Name HERCULES/RMT INC.
Project Number 70102 61
Field ID : N-14D

Matrix Type GROUNDWATER
Collection Date 03/09/05
Report Date : 03/21/05
Lab Sample Number 856996-002

INORGANICS

Test	Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Total Suspended Solids	2.3	1.1	1	mg/L		03/16/05	EPA 160.2	EPA 160.2

TOXAPHENE — Prep Date: 03/14/05

Analyte	Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Toxaphene	< 3.0	3.0	1	ug/L		03/16/05	SW846 3510C	SW846 8081A

Analyzed by:

Dawn J. Kearns, Pesticide/PCB Analyst

En Chem
A Division of Pace Analytical Services, Inc

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Green Bay, WI 54302
920-469-2436

Analytical Report Number: 856921

Client : HERCULES, INC - DE
Project Name HERCULES/RMT INC.
Project Number 70102 61
Field ID : N-10

Matrix Type GROUNDWATER
Collection Date 03/09/05
Report Date : 03/21/05
Lab Sample Number 856996-003

INORGANICS

Test	Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Total Suspended Solids	3.3	1.1	1	mg/L		03/16/05	EPA 160.2	EPA 160.2

TOXAPHENE — Prep Date: 03/14/05

Analyte	Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Toxaphene	< 3.0	3.0	1	ug/L		03/17/05	SW846 3510C	SW846 8081A

Analyzed by:

Dawn J. Kearns, Pesticide/PCB Analyst

En Chem
A Division of Pace Analytical Services, Inc

1241 Bellevue Street
Green Bay, WI 54302
920-469-2436

Analytical Report Number: 856921

Client : HERCULES. INC - DE
Project Name HERCULES/RMT INC.
Project Number 70102 61
Field ID : N-12

Matrix Type GROUNDWATER
Collection Date 03/09/05
Report Date : 03/21/05
Lab Sample Number 856996-004

INORGANICS

Test	Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Total Suspended Solids	4.4	2.0	1	mg/L		03/16/05	EPA 160.2	EPA 160.2

TOXAPHENE — Prep Date: 03/14/05

Analyte	Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Toxaphene	< 3 0	3 0	1	ug/L		03/17/05	SW846 3510C	SW846 8081A

Analyzed by:

Dawn J. Kearns, Pesticide/PCB Analyst

En Chem
A Division of Pace Analytical Services. Inc

1241 Bellevue Street
Green Bay, WI 54302
920-469-2436

Analytical Report Number: 856921

Client : HERCULES, INC. - DE
Project Name HERCULES/RMT INC.
Project Number 70102 61
Field ID : N-08

Matrix Type GROUNDWATER
Collection Date 03/09/05
Report Date : 03/21/05
Lab Sample Number 856996-005

INORGANICS

Test	Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Total Suspended Solids	5.8	2.0	1	mg/L		03/16/05	EPA 160.2	EPA 160.2

TOXAPHENE **Prep Date:** 03/14/05

Analyte	Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Toxaphene	< 3.0	3.0	1	ug/L		03/17/05	SW846 3510C	SW846 8081A

Analyzed by:

Dawn J. Kearns, Pesticide/PCB Analyst

En Chem
A Division of Pace Analytical Services. Inc

1241 Bellevue Street
Green Bay, WI 54302
920-469-2436

Analytical Report Number: 856921

Client : HERCULES, INC. - DE
Project Name HERCULES/RMT INC.
Project Number 70102.61
Field ID : N-03

Matrix Type GROUNDWATER
Collection Date 03/09/05
Report Date : 03/21/05
Lab Sample Number 856996-006

INORGANICS

Test	Result		EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Total Suspended Solids	0.33	B	1.1	1	mg/L		03/16/05	EPA 160.2	EPA 160.2

TOXAPHENE Prep Date: 03/14/05

Analyte	Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Toxaphene	< 3.0	3.0	1	ug/L		03/17/05	SW846 3510C	SW846 8081A

Analyzed by:

Dawn J. Kearns, Pesticide/PCB Analyst

En Chem
A Division of Pace Analytical Services, Inc

1241 Bellevue Street
Green Bay, WI 54302
920-469-2436

Analytical Report Number: 856921

Client : HERCULES, INC - DE
Project Name HERCULES/RMT INC.
Project Number 70102 61
Field ID : N-05

Matrix Type GROUNDWATER
Collection Date 03/10/05
Report Date : 03/21/05
Lab Sample Number 857028-001

INORGANICS

Test	Result		EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Total Suspended Solids	2.2		1.0	1	mg/L		03/16/05	EPA 160.2	EPA 160.2

BENZENE — Prep Date: 03/14/05

Analyte	Result		EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Benzene	0.65	J	1.0	1	ug/L		03/14/05	SW846 5030B	SW846 8260B

TOXAPHENE — Prep Date: 03/14/05

Analyte		Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Toxaphene	<	3.0	3.0	1	ug/L		03/17/05	SW846 3510C	SW846 8081A

Analyzed by:

Dawn J. Keams, Pesticide/PCB Analyst

En Chem
A Division of Pace Analytical Services, Inc

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Green Bay, WI 54302
920-469-2436

Analytical Report Number: 856921

Client : HERCULES, INC - DE
Project Name HERCULES/RMT INC
Project Number 70102.61
Field ID : N-07

Matrix Type GROUNDWATER
Collection Date 03/10/05
Report Date : 03/21/05
Lab Sample Number 857028-002

INORGANICS

Test	Result		EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Total Suspended Solids	0.50	B	1.0	1	mg/L		03/16/05	EPA 160.2	EPA 160.2

TOXAPHENE *Prep Date: 03/14/05*

Analyte	Result		EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Toxaphene	< 3.0		3.0	1	ug/L		03/17/05	SW846 3510C	SW846 8081A

Analyzed by:

Dawn J. Kearns, Pesticide/PCB Analyst

En Chem
A Division of Pace Analytical Services. Inc
1241 Bellevue Street
Green Bay, WI 54302
920-469-2436

Analytical Report Number: 856921

Client : HERCULES, INC - DE
Project Name HERCULES/RMT INC
Project Number 70102.61
Field ID : N-13

Matrix Type GROUNDWATER
Collection Date 03/10/05
Report Date : 03/21/05
Lab Sample Number 857028-003

INORGANICS

Test	Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Total Suspended Solids	2.8	1.0	1	mg/L		03/16/05	EPA 160.2	EPA 160.2

TOXAPHENE

Prep Date: 03/14/05

Analyte	Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Toxaphene	< 3.0	3.0	1	ug/L		03/17/05	SW846 3510C	SW846 8081A

Analyzed by:

Dawn J. Kearns, Pesticide/PCB Analyst

En Chem
A Division of Pace Analytical Services, Inc

1241 Bellevue Street
Green Bay, WI 54302
920-469-2436

Analytical Report Number: 856921

Client : HERCULES, INC. - DE
Project Name HERCULES/RMT INC
Project Number 70102 61
Field ID : N-15S

Matrix Type GROUNDWATER
Collection Date 03/10/05
Report Date : 03/21/05
Lab Sample Number 857028-004

INORGANICS

Test	Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Total Suspended Solids	1.9	1.0	1	mg/L		03/16/05	EPA 160.2	EPA 160.2

TOXAPHENE Prep Date: 03/14/05

Analyte	Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Toxaphene	< 3.0	3.0	1	ug/L		03/17/05	SW846 3510C	SW846 8081A

Analyzed by:

Dawn J. Kearns, Pesticide/PCB Analyst

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A Division of Pace Analytical Services. Inc

1241 Bellevue Street
Green Bay, WI 54302
920-469-2436

Analytical Report Number: 856921

Client : HERCULES, INC. - DE
Project Name HERCULES/RMT INC
Project Number 70102.61
Field ID : N-15D

Matrix Type GROUNDWATER
Collection Date 03/10/05
Report Date : 03/21/05
Lab Sample Number 857028-005

INORGANICS

Test	Result		EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Total Suspended Solids	0.80	B	1.0	1	mg/L		03/16/05	EPA 160.2	EPA 160.2

TOXAPHENE *Prep Date: 03/14/05*

Analyte		Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Toxaphene	<	3.0	3.0	1	ug/L		03/17/05	SW846 3510C	SW846 8081A

Analyzed by:

Dawn J. Kearns, Pesticide/PCB Analyst

En Chem
A Division of Pace Analytical Services, Inc.
1241 Bellevue Street
Green Bay, WI 54302
920-469-2436

Analytical Report Number: 856921

Client : HERCULES, INC - DE
Project Name HERCULES/RMT INC.
Project Number 70102 61
Field ID : RBLK-05101

Matrix Type GROUNDWATER
Collection Date 03/10/05
Report Date : 03/21/05
Lab Sample Number 857028-006

BENZENE *Prep Date:* 03/14/05

Analyte		Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Benzene	<	1.0	1.0	1	ug/L		03/14/05	SW846 5030B	SW846 8260B

TOXAPHENE *Prep Date:* 03/14/05

Analyte		Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Toxaphene	<	3.0	3.0	1	ug/L		03/17/05	SW846 3510C	SW846 8081A

Analyzed by:

Dawn J. Kearns, Pesticide/PCB Analyst

En Chem
A Division of Pace Analytical Services, Inc

1241 Bellevue Street
Green Bay, WI 54302
920-469-2436

Analytical Report Number: 856921

Client : HERCULES, INC - DE
Project Name HERCULES/RMT INC
Project Number 70102.61
Field ID : TBLK-05101

Matrix Type GROUNDWATER
Collection Date 03/10/05
Report Date : 03/21/05
Lab Sample Number *857028-007*

BENZENE **Prep Date:** 03/14/05

Analyte		Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Benzene	<	1.0	1.0	1	ug/L		03/14/05	SW846 5030B	SW846 8260B

Analyzed by:

Dawn J. Kearns, Pesticide/PCB Analyst

FORM 3
WATER 8081 MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY

Lab Name: EN CHEM Contract: HERCULES LF 009

Lab Code: EN CHEM Case No.: SAS No.: SDG No.: 856921

Matrix Spike - CLIENT Sample No.: N-14S

COMPOUND	SPIKE ADDED (ug/L)	SAMPLE CONCENTRATION (ug/L)	MS CONCENTRATION (ug/L)	MS % REC #	QC. LIMITS REC.
Toxaphene	40	0.00	42	105	52-157

COMPOUND	SPIKE ADDED (ug/L)	MSD CONCENTRATION (ug/L)	MSD % REC #	% RPD #	QC LIMITS RPD	QC LIMITS REC.
Toxaphene	40	42	105	0	28	52-157

Column to be used to flag recovery and RPD values with an asterisk
* Values outside of QC limits

RPD: 0 out of 1 outside limits
Spike Recovery: 0 out of 2 outside limits

COMMENTS: _ Analyzed by: _____

_ _____

Dawn J. Kearns, Pesticide/PCB Analyst

FORM 1
VOLATILE ORGANICS ANALYSIS DATA SHEET

CLIENT SAMPLE NO.

VOG1704-01MB

Lab Name: EN CHEM - GREEN BAY
Lab Code: ENCHEMGB
Matrix: (soil/water) WATER
Sample wt/vol: 5.000 (g/mL) ML
Level: (low/med) LOW
% Moisture: not dec. _____
GC Column: DB-624 ID: 0.18 (mm)
Soil Extract Volume: _____(uL)

Contract:
SDG No.: 856921
Lab Sample ID: VOG1704-01MB
Lab File ID: 03140504
Date Received: _____
Date Analyzed: 03/14/05
Dilution Factor: 1.0
Soil Aliquot Volume: _____(uL)

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/Kg) UG/L	Q
71-43-2	BENZENE	1.00	U

FORM 2
WATER VOLATILE SYSTEM MONITORING COMPOUND RECOVERY

Lab Name: EN CHEM - GREEN BAY Contract:

Lab Code: ENCHEMGB Case No.: SAS No.: SDG No.: 856921

	CLIENT SAMPLE NO.	SMC1 #	SMC2 (TOL) #	SMC3 #	OTHER	TOT OUT
01	VOG1704-01MB	100	110	95		0
02	VOG1704-01MBLCS	99	111	100		0
03	VOG1704-01MBLCSD	101	110	99		0
04	RBLK-05101	103	110	95		0
05	TBLK-05101	103	111	93		0
06	N-05	106	108	94		0
07						
08						
09						
10						
11						
12						
13						
14						
15						
16						
17						
18						
19						
20						
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22						
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25						
26						
27						
28						
29						
30						

		QC LIMITS
SMC1	= DIBROMOFLUOROMETHANE	(69-140)
SMC2 (TOL)	= TOLUENE-D8	(72-137)
SMC3	= 4-BROMOFLUOROBENZENE	(65-133)

Column to be used to flag recovery values

* Values outside of contract required QC limits

D System Monitoring Compound diluted out

FORM 3
WATER VOLATILE LAB CONTROL SAMPLE

Lab Name: EN CHEM - GREEN BAY Contract:
Lab Code: ENCHEMGB Case No.: SAS No.: SDG No.: 856921
MS Sample No.: VOG1704-01MB

COMPOUND	SPIKE ADDED (ug/L)	SAMPLE AMOUNT (ug/L)	LCS AMOUNT (ug/L)	LCS % REC #	QC. LIMITS REC.
BENZENE	50.00	0.00	41.13	82	75-125

COMPOUND	SPIKE ADDED (ug/L)	LCSD AMOUNT (ug/L)	LCSD % REC #	% RPD #	QC LIMITS RPD	QC LIMITS REC.
BENZENE	50.00	41.29	82	0	20	75-125

Column to be used to flag recovery and RPD values with an asterisk

* Values outside of QC limits

RPD: 0 out of 1 outside limits
Spike Recovery: 0 out of 2 outside limits

COMMENTS: _____

FORM 4
VOLATILE METHOD BLANK SUMMARY

CLIENT SAMPLE NO.

VOG1704-01MB

Lab Name: EN CHEM - GREEN BAY Contract:

Lab Code: ENCHEMGB Case No.: SAS No.: SDG No.: 856921

Lab File ID: 03140504 Lab Sample ID: VOG1704-01MB

Date Analyzed: 03/14/05 Time Analyzed: 1350

GC Column: DB-624 ID: 0.18 (mm) Heated Purge: (Y/N) N

Instrument ID: HPVOA9

THIS METHOD BLANK APPLIES TO THE FOLLOWING SAMPLES, MS and MSD:

	SAMPLE NO.	LAB SAMPLE ID	LAB FILE ID	TIME ANALYZED
01	VOG1704-01MBLCS	VOG1704-01MBLCS	03140505	1415
02	VOG1704-01MBLCSD	VOG1704-01MBLCSD	03140506	1440
03	RBLK-05101	857028-006	03140514	1758
04	TBLK-05101	857028-007	03140515	1822
05	N-05	857028-001	03140516	1847
06				
07				
08				
09				
10				
11				
12				
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22				
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25				
26				
27				
28				
29				
30				

COMMENTS:

En Chem

A Division of Pace Analytical Services. Inc

Analysis Summary by Laboratory

1241 Bellevue Street
Green Bay. WI 54302

1090 Kennedy Avenue
Kimberly, WI 54136

Test Group Name	856921-001	856921-002	856921-003	856921-004	856921-005	856921-006	856921-007	856996-001	856996-002	856996-003	856996-004	856996-005	856996-006	857028-001	857028-002	857028-003	857028-004	857028-005	857028-006	857028-007
BENZENE														G					G	G
SOLIDS, TOTAL SUSPENDED	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G		
TOXAPHENE	K	K	K	K	K	K	K	K	K	K	K	K	K	K	K	K	K	K	K	

Georgia Certification	
G = En Chem Green Bay	IL-100431
K = En Chem Kimberly	E87855
S = En Chem Superior	Not Applicable
C = Subcontracted Analysis	
I = Other Pace Lab Analysis	
G = En Chem Green Bay	83006001
K = En Chem Kimberly	83001001
S = En Chem Superior	Not Applicable
C = Subcontracted Analysis	
I = Other Pace Lab Analysis	

EN CHEM

A Division of Pace Analytical Services, Inc.

1241 Bellevue Street, Suite 9
Green Bay, WI 54302
920-469-2436, Fax: 920-469-8827

Analytical Report Number: 856921

Client: HERCULES, INC - DE
Project Name: HERCULES/RMT INC.
Project Number: 70102 61

Lab Contact: Tod Noltemeyer
Collected By:
Report Serial No: 856921033020051122

Lab Sample Number	Field ID	Matrix	Collection Date
856921-001	N-01	GW	03/08/05
856921-002	N-02	GW	03/08/05
856921-003	N-9S	GW	03/08/05
856921-004	N-9D	GW	03/08/05
856921-005	N-6SR	GW	03/08/05
856921-006	N-6DR	GW	03/08/05
856921-007	N-11	GW	03/08/05
856996-001	N-14S	GW	03/09/05
856996-002	N-14D	GW	03/09/05
856996-003	N-10	GW	03/09/05
856996-004	N-12	GW	03/09/05
856996-005	N-08	GW	03/09/05
856996-006	N-03	GW	03/09/05
857028-001	N-05	GW	03/10/05
857028-002	N-07	GW	03/10/05
857028-003	N-13	GW	03/10/05
857028-004	N-15S	GW	03/10/05
857028-005	N-15D	GW	03/10/05
857028-006	RBLK-05101	GW	03/10/05

I certify that the data contained in this Final Report has been generated and reviewed in accordance with approved methods and Laboratory Standard Operating Procedure. Exceptions, if any, are discussed in the accompanying sample comments. Release of this final report is authorized by Laboratory management, as is verified by the following signature. *This report shall not be reproduced, except in full, without the written consent of Pace Analytical Services, Inc.* The sample results relate only to the analytes of interest tested.

Approval Signature

3/30/05
Date

En Chem
A Division of Pace Analytical Services, Inc

1241 Bellevue Street
Green Bay, WI 54302
920-469-2436

Analytical Report Number: 856921

Client : HERCULES, INC. - DE
Project Name *HERCULES/RMT INC.*
Project Number 70102.61
Field ID : N-01

Matrix Type GROUNDWATER
Collection Date 03/08/05
Report Date : 03/21/05
Lab Sample Number 856921-001

TOXAPHENE - TOTAL AREA UNDER CURVE — Prep Date: 03/14/05

Analyte		Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Chlorinated Camphenes	<	3.0	3.0	1	ug/L		03/16/05	SW846 3510C	SW846 8081A

Analyzed by:

Dawn J. Kearns, Pesticide/PCB Analyst

En Chem
A Division of Pace Analytical Services, Inc.

Analytical Report Number: 856921

1241 Bellevue Street
Green Bay, WI 54302
920-469-2436

Client : HERCULES, INC. - DE
Project Name HERCULES/RMT INC
Project Number 70102 61
Field ID : N-02

Matrix Type GROUNDWATER
Collection Date 03/08/05
Report Date : 03/21/05
Lab Sample Number 856921-002

TOXAPHENE - TOTAL AREA UNDER CURVE — Prep Date: 03/14/05

Analyte		Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Chlorinated Camphenes	<	3 0	3 0	1	ug/L		03/16/05	SW846 3510C	SW846 8081A

Analyzed by:

Dawn J. Keams, Pesticide/PCB Analyst

En Chem
A Division of Pace Analytical Services, Inc

Analytical Report Number: 856921

1241 Bellevue Street
Green Bay, WI 54302
920-469-2436

Client : HERCULES, INC. - DE
Project Name HERCULES/RMT INC.
Project Number 70102 61
Field ID : N-9S

Matrix Type GROUNDWATER
Collection Date 03/08/05
Report Date : *03/21/05*
Lab Sample Number 856921-003

TOXAPHENE - TOTAL AREA UNDER CURVE **Prep Date:** 03/14/05

Analyte		Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Chlorinated Camphenes	<	3.0	3.0	1	ug/L		03/16/05	SW846 3510C	SW846 8081A

Analyzed by:

Dawn J. Keams, Pesticide/PCB Analyst

En Chem
A Division of Pace Analytical Services, Inc
1241 Bellevue Street
Green Bay, WI 54302
920-469-2436

Analytical Report Number: 856921

Client : HERCULES, INC - DE
Project Name HERCULES/RMT INC
Project Number 70102 61
Field ID : N-9D

Matrix Type GROUNDWATER
Collection Date 03/08/05
Report Date : 03/21/05
Lab Sample Number 856921-004

TOXAPHENE - TOTAL AREA UNDER CURVE **Prep Date:** 03/14/05

Analyte		Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Chlorinated Camphenes	<	3 0	3.0	1	ug/L		03/16/05	SW846 3510C	SW846 8081A

Analyzed by:

Dawn J. Kearns, Pesticide/PCB Analyst

En Chem
A Division of Pace Analytical Services, Inc

Analytical Report Number: 856921

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Green Bay, WI 54302
920-469-2436

Client : HERCULES, INC - DE
Project Name HERCULES/RMT INC
Project Number 70102.61
Field ID : N-6SR

Matrix Type GROUNDWATER
Collection Date 03/08/05
Report Date : 03/21/05
Lab Sample Number *856921-005*

TOXAPHENE - TOTAL AREA UNDER CURVE **Prep Date:** 03/14/05

Analyte	Result		EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Chlorinated Camphenes	1 6	J	3.0	1	ug/L		03/16/05	SW846 3510C	SW846 8081A

Analyzed by:

Dawn J. Kearns, Pesticide/PCB Analyst

En Chem
A Division of Pace Analytical Services, Inc.

Analytical Report Number: 856921

1241 Bellevue Street
Green Bay, WI 54302
920-469-2436

Client : HERCULES, INC - DE
Project Name HERCULES/RMT INC
Project Number 70102 61
Field ID : N-6DR

Matrix Type GROUNDWATER
Collection Date 03/08/05
Report Date : 03/21/05
Lab Sample Number 856921-006

TOXAPHENE - TOTAL AREA UNDER CURVE **Prep Date:** 03/14/05

Analyte		Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Chlorinated Camphenes	<	3 0	3 0	1	ug/L		03/16/05	SW846 3510C	SW846 8081A

Analyzed by:

Dawn J. Kearns, Pesticide/PCB Analyst

En Chem
A Division of Pace Analytical Services. Inc.

Analytical Report Number: 856921

1241 Bellevue Street
Green Bay, WI 54302
920-469-2436

Client : HERCULES, INC. - DE
Project Name HERCULES/RMT INC
Project Number *70102.61*
Field ID : N-11

Matrix Type GROUNDWATER
Collection Date 03/08/05
Report Date : 03/21/05
Lab Sample Number 856921-007

TOXAPHENE - TOTAL AREA UNDER CURVE **Prep Date:** 03/14/05

Analyte		Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Chlorinated Camphenes	<	3.0	3.0	1	ug/L		03/16/05	SW846 3510C	SW846 8081A

Analyzed by:

Dawn J. Kearns, Pesticide/PCB Analyst

En Chem
A Division of Pace Analytical Services, Inc.

Analytical Report Number: 856921

1241 Bellevue Street
Green Bay, WI 54302
920-469-2436

Client : HERCULES, INC. - DE
Project Name HERCULES/RMT INC.
Project Number 70102.61
Field ID : N-14S

Matrix Type GROUNDWATER
Collection Date 03/09/05
Report Date : 03/21/05
Lab Sample Number 856996-001

TOXAPHENE - TOTAL AREA UNDER CURVE **Prep Date:** 03/14/05

Analyte		Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Chlorinated Camphenes	<	3.0	3.0	1	ug/L		03/16/05	SW846 3510C	SW846 8081A

Analyzed by:

Dawn J. Kearns, Pesticide/PCB Analyst

En Chem
A Division of Pace Analytical Services, Inc.

Analytical Report Number: 856921

1241 Bellevue Street
Green Bay, WI 54302
920-469-2436

Client : HERCULES, INC. - DE
Project Name HERCULES/RMT INC
Project Number *70102 61*
Field ID : N-14D

Matrix Type GROUNDWATER
Collection Date 03/09/05
Report Date : 03/21/05
Lab Sample Number 856996-002

TOXAPHENE - TOTAL AREA UNDER CURVE **Prep Date:** 03/14/05

Analyte		Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Chlorinated Camphenes	<	3 0	3 0	1	ug/L		03/16/05	SW846 3510C	SW846 8081A

Analyzed by:

Dawn J. Kearns, Pesticide/PCB Analyst

En Chem
A Division of Pace Analytical Services, Inc

1241 Bellevue Street
Green Bay, WI 54302
920-469-2436

Analytical Report Number: 856921

Client : HERCULES, INC. - DE
Project Name *HERCULES/RMT INC.*
Project Number 70102.61
Field ID : N-10

Matrix Type GROUNDWATER
Collection Date 03/09/05
Report Date : 03/21/05
Lab Sample Number 856996-003

TOXAPHENE - TOTAL AREA UNDER CURVE **Prep Date:** 03/14/05

Analyte		Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Chlorinated Camphenes	<	3.0	3.0	1	ug/L		03/17/05	SW846 3510C	SW846 8081A

Analyzed by:

Dawn J. Kearns, Pesticide/PCB Analyst

En Chem
A Division of Pace Analytical Services, Inc

Analytical Report Number: 856921

1241 Bellevue Street
Green Bay, WI 54302
920-469-2436

Client : HERCULES. INC. - DE
Project Name HERCULES/RMT INC.
Project Number 70102.61
Field ID : N-12

Matrix Type GROUNDWATER
Collection Date 03/09/05
Report Date : 03/21/05
Lab Sample Number 856996-004

TOXAPHENE - TOTAL AREA UNDER CURVE **Prep Date:** 03/14/05

Analyte		Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Chlorinated Camphenes	<	3.0	3.0	1	ug/L		03/17/05	SW846 3510C	SW846 8081A

Analyzed by:

Dawn J. Kearns, Pesticide/PCB Analyst

En Chem
A Division of Pace Analytical Services, Inc.
1241 Bellevue Street
Green Bay, WI 54302
920-469-2436

Analytical Report Number: 856921

Client : HERCULES. INC - DE
Project Name HERCULES/RMT INC
Project Number 70102 61
Field ID : N-08

Matrix Type GROUNDWATER
Collection Date 03/09/05
Report Date : 03/21/05
Lab Sample Number 856996-005

TOXAPHENE - TOTAL AREA UNDER CURVE **Prep Date:** 03/14/05

Analyte		Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Chlorinated Camphenes	<	3.0	3.0	1	ug/L		03/17/05	SW846 3510C	SW846 8081A

Analyzed by:

Dawn J. Kearns, Pesticide/PCB Analyst

En Chem
A Division of Pace Analytical Services, Inc
1241 Bellevue Street
Green Bay, WI 54302
920-469-2436

Analytical Report Number: 856921

Client : HERCULES, INC. - DE
Project Name HERCULES/RMT INC
Project Number 70102.61
Field ID : N-03

Matrix Type GROUNDWATER
Collection Date 03/09/05
Report Date : 03/21/05
Lab Sample Number 856996-006

TOXAPHENE - TOTAL AREA UNDER CURVE *Prep Date:* 03/14/05

Analyte		Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Chlorinated Camphenes	<	3.0	3.0	1	ug/L		03/17/05	SW846 3510C	SW846 8081A

Analyzed by:

Dawn J. Kearns, Pesticide/PCB Analyst

En Chem
A Division of Pace Analytical Services, Inc.

Analytical Report Number: 856921

1241 Bellevue Street
Green Bay, WI 54302
920-469-2436

Client : HERCULES. INC. - DE
Project Name HERCULES/RMT INC
Project Number 70102.61
Field ID : N-05

Matrix Type GROUNDWATER
Collection Date 03/10/05
Report Date : 03/21/05
Lab Sample Number 857028-001

TOXAPHENE - TOTAL AREA UNDER CURVE **Prep Date:** 03/14/05

Analyte		Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Chlorinated Camphenes	<	3.0	3.0	1	ug/L		03/17/05	SW846 3510C	SW846 8081A

Analyzed by:

Dawn J. Keams, Pesticide/PCB Analyst

En Chem
A Division of Pace Analytical Services, Inc.

Analytical Report Number: 856921

1241 Bellevue Street
Green Bay, WI 54302
920-469-2436

Client : HERCULES, INC. - DE
Project Name *HERCULES/RMT INC.*
Project Number 70102 61
Field ID : N-07

Matrix Type GROUNDWATER
Collection Date 03/10/05
Report Date : 03/21/05
Lab Sample Number 857028-002

TOXAPHENE - TOTAL AREA UNDER CURVE **Prep Date:** 03/14/05

Analyte		Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Chlorinated Camphenes	<	3 0	3 0	1	ug/L		03/17/05	SW846 3510C	SW846 8081A

Analyzed by:

Dawn J. Kearns, Pesticide/PCB Analyst

En Chem
A Division of Pace Analytical Services, Inc.

Analytical Report Number: 856921

1241 Bellevue Street
Green Bay, WI 54302
920-469-2436

Client : HERCULES, INC. - DE
Project Name HERCULES/RMT INC
Project Number 70102.61
Field ID : N-13

Matrix Type GROUNDWATER
Collection Date 03/10/05
Report Date : 03/21/05
Lab Sample Number 857028-003

TOXAPHENE - TOTAL AREA UNDER CURVE *Prep Date:* 03/14/05

Analyte		Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Chlorinated Camphenes	<	3.0	3.0	1	ug/L		03/17/05	SW846 3510C	SW846 8081A

Analyzed by:

Dawn J. Kearns, Pesticide/PCB Analyst

En Chem
A Division of Pace Analytical Services, Inc.

Analytical Report Number: 856921

1241 Bellevue Street
Green Bay, WI 54302
920-469-2436

Client : HERCULES, INC - DE
Project Name *HERCULES/RMT INC.*
Project Number 70102 61
Field ID : N-15S

Matrix Type GROUNDWATER
Collection Date 03/10/05
Report Date : 03/21/05
Lab Sample Number 857028-004

TOXAPHENE - TOTAL AREA UNDER CURVE *Prep Date: 03/14/05*

Analyte		Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Chlorinated Camphenes	<	3 0	3 0	1	ug/L		03/17/05	SW846 3510C	SW846 8081A

Analyzed by:

Dawn J. Kearns, Pesticide/PCB Analyst

En Chem
A Division of Pace Analytical Services, Inc.

Analytical Report Number: 856921

1241 Bellevue Street
Green Bay, WI 54302
920-469-2436

Client : HERCULES, INC - DE
Project Name HERCULES/RMT INC
Project Number 70102 61
Field ID : N-15D

Matrix Type GROUNDWATER
Collection Date 03/10/05
Report Date : 03/21/05
Lab Sample Number 857028-005

TOXAPHENE - TOTAL AREA UNDER CURVE **Prep Date:** 03/14/05

Analyte		Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Chlorinated Camphenes	<	3 0	3 0	1	ug/L		03/17/05	SW846 3510C	SW846 8081A

Analyzed by:

Dawn J. Keams, Pesticide/PCB Analyst

En Chem
A Division of Pace Analytical Services, Inc

1241 Bellevue Street
Green Bay, WI 54302
920-469-2436

Analytical Report Number: 856921

Client : HERCULES, INC. - DE
Project Name HERCULES/RMT INC.
Project Number 70102 61
Field ID : RBLK-05101

Matrix Type GROUNDWATER
Collection Date 03/10/05
Report Date : 03/21/05
Lab Sample Number 857028-006

TOXAPHENE - TOTAL AREA UNDER CURVE **Prep Date:** 03/14/05

Analyte		Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Chlorinated Camphenes	<	3 0	3.0	1	ug/L		03/17/05	SW846 3510C	SW846 8081A

Analyzed by:

Dawn J. Kearns, Pesticide/PCB Analyst

FORM 3
WATER 8081 MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY

Lab Name: EN CHEM Contract: HERCULES LF 009

Lab Code: EN CHEM Case No.: SAS No.: SDG No.: 856921

Matrix Spike - CLIENT Sample No.: N-14S

COMPOUND	SPIKE ADDED (ug/L)	SAMPLE CONCENTRATION (ug/L)	MS CONCENTRATION (ug/L)	MS % REC #	QC. LIMITS REC.
Chlorinated Camphene	40	0.00	41	102	52-157

COMPOUND	SPIKE ADDED (ug/L)	MSD CONCENTRATION (ug/L)	MSD % REC #	% RPD #	QC LIMITS RPD	QC LIMITS REC.
Chlorinated Camphene	40	40	100	2	28	52-157

Column to be used to flag recovery and RPD values with an asterisk
* Values outside of QC limits

RPD: 0 out of 1 outside limits
Spike Recovery: 0 out of 2 outside limits

COMMENTS: Analyzed by:

Dawn J. Kearns, Pesticide/PCB Analyst

En Chem
A Division of Pace Analytical Services, Inc.

Analytical Report Number: 856921

1241 Bellevue Street
Green Bay, WI 54302
920-469-2436

Client : HERCULES, INC. - DE
Project Name : HERCULES/RMT INC
Project Number : 70102.61
Field ID : svk1082-075mb

Matrix Type : GROUNDWATER
Collection Date : NA
Report Date : 04/05/05
Lab Sample Number : svk1082-075mb

TOXAPHENE - TOTAL AREA UNDER CURVE

Prep Date: 03/14/05

Analyte		Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Chlorinated Camphenes	<	3.0	3.0	1	ug/L		03/16/05	SW846 3510C	SW846 8081A

Analyzed by:

Dawn J. Kearns, Pesticide/PCB Analyst

En Chem
A Division of Pace Analytical Services, Inc.

Analytical Report Number: 856921

1241 Bellevue Street
Green Bay, WI 54302
920-469-2436

Client : HERCULES, INC. - DE
Project Name : HERCULES/RMT INC.
Project Number : 70102.61
Field ID : svk1082-075mb

Matrix Type : GROUNDWATER
Collection Date : NA
Report Date : 04/05/05
Lab Sample Number : svk1082-075mb

TOXAPHENE **Prep Date:** 03/14/05

Analyte		Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Toxaphene	<	3.0	3.0	1	ug/L		03/16/05	SW846 3510C	SW846 8081A

Analyzed by:

Dawn J. Kearns, Pesticide/PCB Analyst

FORM 2
WATER 8081 SURROGATE RECOVERY

Lab Name: EN CHEM Contract: HERCULES LF 009 (TAUC)

Lab Code: EN CHEM Case No.: SAS No.: SDG No.: 856921

GC Column(1): DB-1701 ID: 0.32 (mm) GC Column(2): DB-5 ID: 0.32 (mm)

	EPA SAMPLE NO.	S1 1 (TCX) #	S1 2 (TCX) #	S2 1 (DCB) #	S2 2 (DCB) #	S3 1 #	S3 2 #	S4 1 #	S4 2 #	TOT OUT
01	SVK1082-075MB	96	90	85	82					0
02	SVK1082-075MBLCS	100	96	91	88					0
03	N-01	97	94	79	77					0
04	N-02	99	97	91	88					0
05	N-9S	85	84	50	49					0
06	N-9D	98	96	73	70					0
07	N-6SR	88	86	72	70					0
08	N-6DR	95	92	83	80					0
09	N-11	84	82	60	58					0
10	N-14S	102	99	85	82					0
11	N-14D	100	96	88	85					0
12	N-10	98	95	65	63					0
13	N-12	93	90	59	57					0
14	N-08	86	84	76	72					0
15	N-03	104	99	96	92					0
16	N-14SMS	98	96	89	84					0
17	N-14SMSD	93	90	83	80					0
18	N-05	92	90	73	70					0
19	N-07	102	100	59	57					0
20	N-13	99	97	94	90					0
21	N-15S	99	97	83	79					0
22	N-15D	90	87	61	59					0
23	RBLK-05101	98	94	79	76					0
24										
25										
26										
27										
28										
29										
30										

	QC LIMITS
S1 (TCX) = Tetrachloro-m-xylene	(42-122)
S2 (DCB) = Decachlorobiphenyl	(10-128)

Column to be used to flag recovery values
* Values outside of contract required QC limits
D Surrogate diluted out

FORM II 8081

FORM 2
WATER 8081 SURROGATE RECOVERY

Lab Name: EN CHEM Contract: HERCULES LF 009 (Herc '97)

Lab Code: EN CHEM Case No.: SAS No.: SDG No.: 856921

GC Column(1): DB-1701 ID: 0.32 (mm) GC Column(2): DB-5 ID: 0.32 (mm)

	EPA SAMPLE NO.	S1 1 (TCX)#	S1 2 (TCX)#	S2 1 (DCB)#	S2 2 (DCB)#	S3 1 #	S3 2 #	S4 1 #	S4 2 #	TOT OUT
01	SVK1082-075MB	99	96	86	81					0
02	SVK1082-075MBLCS	103	101	91	90					0
03	N-01	100	96	81	76					0
04	N-02	104	100	92	88					0
05	N-9S	88	87	52	49					0
06	N-9D	101	98	73	70					0
07	N-6SR	89	87	72	72					0
08	N-6DR	100	97	85	81					0
09	N-11	86	85	60	59					0
10	N-14S	106	103	85	83					0
11	N-14D	100	98	91	86					0
12	N-10	97	98	66	64					0
13	N-12	95	92	60	57					0
14	N-08	89	85	76	74					0
15	N-03	108	106	98	93					0
16	N-14SMS	100	99	88	84					0
17	N-14SMSD	94	93	82	82					0
18	N-05	95	92	75	72					0
19	N-07	107	105	61	57					0
20	N-13	102	103	96	90					0
21	N-15S	103	100	84	81					0
22	N-15D	94	91	62	61					0
23	RBLK-05101	102	98	80	77					0
24										
25										
26										
27										
28										
29										
30										

	QC LIMITS
S1 (TCX) = Tetrachloro-m-xylene	(42-122)
S2 (DCB) = Decachlorobiphenyl	(10-128)

Column to be used to flag recovery values
* Values outside of contract required QC limits
D Surrogate diluted out

En Chem
A Division of Pace Analytical Services, Inc.

QC Summary

1241 Bellevue Street
Green Bay, WI 54302
920-469-2436
Fax: 920-469-8827

SDG: 856921
Lab Section: TOX-K
QC Batch Number: 4276
Prep Method: SW846 3510C
Analytical Method: SW846 8081A

QC Type	Client Sample ID	Lab Sample ID
MB	svk1082-075mb	svk1082-075mb
LCS	svk1082-075mbLCS	svk1082-075mbLCS

Client Sample ID	Lab Sample ID
N-01	856921-001
N-02	856921-002
N-9S	856921-003
N-9D	856921-004
N-6SR	856921-005
N-6DR	856921-006
N-11	856921-007
N-14S	856996-001
N-14D	856996-002
N-10	856996-003
N-12	856996-004
N-08	856996-005
N-03	856996-006
N-14S MS	856996-007
N-14S MSD	856996-008
N-05	857028-001
N-07	857028-002
N-13	857028-003
N-15S	857028-004
N-15D	857028-005
RBLK-05101	857028-006

Client Sample ID	Lab Sample ID
N-01	856921-001
N-02	856921-002
N-9S	856921-003
N-9D	856921-004
N-6SR	856921-005
N-6DR	856921-006
N-11	856921-007
N-14S	856996-001
N-14D	856996-002
N-10	856996-003
N-12	856996-004
N-08	856996-005
N-03	856996-006
N-14S MS	856996-007
N-14S MSD	856996-008
N-05	857028-001
N-07	857028-002
N-13	857028-003
N-15S	857028-004
N-15D	857028-005
RBLK-05101	857028-006

Test Name	Method Blank Result Conc	LCS Spiked Conc	LCS Recovery Conc	%	C	LCSD Spiked Conc	LCSD Recovery Conc	%	C	LCS/LCSD RPD %	C	LCS/LCSD Control Limits LCL %	UCL %	RPD %	Parent Sample Number	Parent Result Conc	MS Spiked Conc	MS Recovery Conc	%	C	MSD Spiked Conc	MSD Recovery Conc	%	C	MS/MSD RPD %	C	MS/MSD Control Limits LCL %	UCL %	RPD %
Toxaphene	< 1.1	40	41	104		---	---	---		---		77	126	---	---	---	---	---	---		---	---	---		---		---	---	---
Tetrachloro-m-xylene	99%	---	---	103		---	---	---		---		42	122	---	---	---	---	---	---		---	---	---		---		---	---	---
Tetrachloro-m-xylene	96%	---	---	100		---	---	---		---		42	122	---	---	---	---	---	---		---	---	---		---		---	---	---
Decachlorobiphenyl	86%	---	---	91		---	---	---		---		10	128	---	---	---	---	---	---		---	---	---		---		---	---	---
Decachlorobiphenyl	85%	---	---	91		---	---	---		---		10	128	---	---	---	---	---	---		---	---	---		---		---	---	---

Conc = ug/L unless otherwise noted
C = QC Code, see Qualifer Sheet
Parent Result is reported down to MDL in order to allow Validation of this worksheet
The %R and RPD results are calculated from raw data values with more significant figures than are reported on this form.

Report Date: 4/4/2005

En Chem

A Division of Pace Analytical Services, Inc

Analysis Summary by Laboratory

1241 Bellevue Street
Green Bay, WI 54302

1090 Kennedy Avenue
Kimberly, WI 54136

Test Group Name	856921-001	856921-002	856921-003	856921-004	856921-005	856921-006	856921-007	856996-001	856996-002	856996-003	856996-004	856996-005	856996-006	857028-001	857028-002	857028-003	857028-004	857028-005	857028-006
TOXAPHENE - TOTAL AREA UNDER	K	K	K	K	K	K	K	K	K	K	K	K	K	K	K	K	K	K	K

Georgia Certification	
G = En Chem Green Bay	IL-100431
K = En Chem Kimberly	E87855
S = En Chem Superior	Not Applicable
C = Subcontracted Analysis	
I = Other Pace Lab Analysis	
G = En Chem Green Bay	83006001
K = En Chem Kimberly	83001001
S = En Chem Superior	Not Applicable
C = Subcontracted Analysis	
I = Other Pace Lab Analysis	

En Chem
A Division of Pace Analytical Services, Inc.

QC Summary

1241 Bellevue Street
Green Bay, WI 54302
920-469-2436
Fax: 920-469-8827

SDG: 856921
Lab Section: WETCHEM
QC Batch Number: 4328
Prep Method: EPA 160.2

QC Type	Client Sample ID	Lab Sample ID
MB	WCG1705-004MB	WCG1705-004MB
LCS	WCG1705-004MBLCS	WCG1705-004MBLCS
LCSD	WCG1705-004MBLCSD	WCG1705-004MBLCSD
DUP	856973-009DUP	856973-009DUP
DUP	856948-001DUP	856948-001DUP

Analytical Method: EPA 160.2

Client Sample ID	Lab Sample ID	Client Sample ID	Lab Sample ID
N-01	856921-001	N-02	856921-002
N-9S	856921-003	N-9D	856921-004
N-6SR	856921-005	N-6DR	856921-006
N-11	856921-007		

Test Name		Method Blank Result Conc	LCS Spiked Conc	LCS Recovery Conc	%	C	LCSD Spiked Conc	LCSD Recovery Conc	%	C	LCS/LCSD RPD %	C	LCS/LCSD Control Limits LCL %	UCL %	RPD %	Parent Sample Number		Parent Result Conc		Lab Dup Conc	Lab Dup RPD %	C	Lab Dup RPD Limit %
Total Suspended Solids	<	0.25	354.0	334	94.4		354.0	336	94.9		0.6		80	120	10	856948-001		47.00		49.5	5.2		10
Total Suspended Solids	<	0.25	354.0	334	94.4		354.0	336	94.9		0.6		80	120	10	856973-009		7.000		7.2	2.8		10

Conc = mg/L unless otherwise noted
C = QC Code, see Qualifer Sheet
Parent Result is reported down to MDL in order to allow Validation of this worksheet
The %R and RPD results are calculated from raw data values with more significant figures than are reported on this form.

Report Date: 3/27/2005

En Chem

A Division of Pace Analytical Services, Inc.

QC Summary

1241 Bellevue Street
Green Bay, WI 54302
920-469-2436
Fax: 920-469-8827

SDG: 856921
Lab Section: WETCHEM
QC Batch Number: 4329
Prep Method: EPA 160.2

QC Type	Client Sample ID	Lab Sample ID
MB	WCG1705-006MB	WCG1705-006MB
LCS	WCG1705-006MBLCS	WCG1705-006MBLCS
LCSD	WCG1705-006MBLCSD	WCG1705-006MBLCSD
DUP	N-08DUP	856996-005DUP
DUP	856954-001DUP	856954-001DUP

Analytical Method: EPA 160.2

Client Sample ID	Lab Sample ID	Client Sample ID	Lab Sample ID
N-14S	856996-001	N-14D	856996-002
N-10	856996-003	N-12	856996-004
N-08	856996-005	N-03	856996-006
N-05	857028-001	N-07	857028-002
N-13	857028-003	N-15S	857028-004
N-15D	857028-005		

Test Name		Method Blank Result Conc	LCS Spiked Conc	LCS Recovery Conc	%	C	LCSD Spiked Conc	LCSD Recovery Conc	%	C	LCS/LCSD RPD %	C	LCS/LCSD Control Limits LCL %	UCL %	RPD %	Parent Sample Number	Parent Result Conc		Lab Dup Conc	Lab Dup RPD %	C	Lab Dup RPD Limit %
Total Suspended Solids	<	0.25	354.0	338	95.5		354.0	328	92.7		3.0		80	120	10	856954-001	37.33		39.33	5.2		10
Total Suspended Solids	<	0.25	354.0	338	95.5		354.0	328	92.7		3.0		80	120	10	856996-005	5.800		6	3.4		10

Conc = mg/L unless otherwise noted
C = QC Code, see Qualifer Sheet
Parent Result is reported down to MDL in order to allow Validation of this worksheet
The %R and RPD results are calculated from raw data values with more significant figures than are reported on this form.

Report Date: 3/27/2005

NELAP STIPULATION

LABORATORY:	EN CHEM – Green Bay Lab, A Division of Pace Analytical Services, Inc.
ACCREDITOR:	NELAP – ILLINOIS
ACCREDITATION ID:	100313
SCOPE:	Wastewater, HW & SW
EFFECTIVE:	March 8, 2004
EXPIRES:	January 31, 2005, Interim Accreditation Granted (IL ELAP is currently backlogged on issuing renewal certificates)

NELAP STIPULATION

LABORATORY:	*EN CHEM* – Kimberly Lab, A Division of Pace Analytical Services, Inc.
ACCREDITOR:	NELAP – FLORIDA
ACCREDITATION ID:	E87855
SCOPE:	Non-Potable Water, Solid and Chemical Materials
EFFECTIVE:	October 1, 2004
EXPIRES:	June 30, 2005

En Chem, Inc. Cooler Receipt Log

Batch No. 856921

Project Name or ID Hercules No. of Coolers: 2 Temps: 0C, 0C

A. Receipt Phase: Date cooler was opened: 3/9/05 By: RJ.

1: Were samples *received on ice? (Must be ≤ 6 C)*	YES	NO[2]	NA
2. Was there a Temperature Blank?	YES	NO	
3: Were custody seals present and intact on cooler? (Record on COC)	YES	NO	
4: Are COC documents present?	YES	NO[2]	
5: Does this Project require quick turn around analysis?	YES	NO	
6: Is there any sub-work?	YES	NO	
7: Are there any short hold time tests?	YES	NO	
8: Are any samples nearing expiration of *hold-time? (Within 2 days)*	YES[1]	NO	Contacted by/Who_____
9: Do any samples need to be Filtered or Preserved in the lab?	YES[1]	NO	Contacted by/Who_____

B. Check-in Phase: Date samples were Checked-in: 3/9/05 By: RJ.

1: Were all sample containers listed on the COC received and intact?	YES	NO[2]	NA
2: Sign the COC as received by En Chem. Completed	YES	NO	
3: *Do sample labels match the COC?*	YES	NO[2]	
4: Completed pH check on preserved samples. *(This statement does not apply to water: VOC, O&G, TOC, DRO, Total Rec Phenolics)*	YES	NO	NA
5: Do samples have correct chemical preservation? *(This statement does not apply to water: VOC, O&G, TOC, DRO, Total Rec Phenolics)*	YES	NO[2]	NA
6: Are dissolved parameters field filtered?	YES	NO[2]	NA
7: Are sample volumes adequate for tests requested?	YES	NO[2]	
8: Are VOC samples free of bubbles >6mm	YES	NO[2]	NA
9: Enter samples into logbook. Completed	YES	NO	
10: Place laboratory sample number on all containers and *COC. Completed*	YES	NO	
11: Complete Laboratory Tracking Sheet (LTS). Completed	YES	NO	NA
12: Start Nonconformance form	YES	NO	NA
13: Initiate Subcontracting procedure. Completed	YES	NO	NA
14: Check laboratory sample number on all containers and COC. 3/9/05	YES	NO	NA

Short Hold-time tests:

24 Hours or less	48 Hours	7 days	Footnotes
Coliform	*BOD*	Ash	1 Notify proper lab group immediately.
Corrosivity = pH	Color	Aqueous Extractable Organics- ALL	2 Complete nonconformance memo.
Dissolved Oxygen	Nitrite or Nitrate	Flashpoint	
Hexavalent Chromium	Ortho Phosphorus	Free Liquids	
HPC	Surfactants	Sulfide	
Ferrous Iron	Turbidity	TDS	
Eh	En Core Preservation	TSS	
Odor	*Power stop preservation*	*Total Solids*	
Residual Chlorine		TVS	
Sulfite		TVSS	
		Unpreserved VOC's	

Rev. 2/05/04, Attachment to 1-REC-5
Subject to QA Audit.

Reviewed by/date 3/30/05

p:/everyone/forms/samplereceiving/crl.doc

PROJECT WORK ORDER

856921

Project: Hercules Landfill 009
Project Number: 70102.61
Sample Date: Week of 03/07/05
Project Manager: Steve Webb
Project Contact: Mark Miesfeldt/Charity Teamer

Enchem
1241 Bellevue St., Suite 9
Green Bay, WI 54302
Contact: Tom Trainor 800-736-2436
Fax: 920-469-8827

Annual Sample Collection Checklist for Hercules
March 2005 Sampling Event

LOCATION	Benzene Method 8260	Toxaphene Method SW-846	Total Suspended Solids	Field Ph, Temp, Spec Cond	Comments/Instructions
	A	B	C		
N-01					Collect full round water levels.
N-02					Note visual observation of turbidity
N-03					
N-05					
N-06SR					
N-06DR					
N-07					
N-08					
N-09S					
N-09D					
N-10					
N-11					
N-12					
N-13					
N-14S					
N-14D					
N-15S					
N-15D					
RBLK-05101					
TBLK-05101					

A = 40mL Glass Container, Teflon Lined Septum\14 Day HT; HCL preserved.
B = 2 - 1 liter ambers for each location.
C = 1L nalgene w/no preservatives for each location.
Collect enough of one well for MS/MSD on Toxaphene (ie; 3 extra liters).

RMT Sampling Procedure
Please refer to the sampling procedure provided in the front pocket of the health and safety plan

RMT NOTES:
Conduct the landfill cap site inspection (include photographs)

CHAIN OF CUSTODY RECORD

75233

30 Patewood Drive, Suite 100, Patewood Plaza One, Greenville, SC 29615-3535
Phone 864/281-0030 • Fax 864/281-0288

Project No.	Project/Client:
70102.61	Hercules / RMT Inc.

Project Manager/Contact Person: Steve Webb / Charity Teamer

Lab No.	Yr. 05 Date	Time	Sample Station ID	Total Number of Containers	MATRIX	Analyses Requested: Toxaphene/Method SW-8081A	Toxaphene/Method SW-8081A	T-55						Comments:
						Filtered (Yes/No)	No	No						
						Preserved (Code)	A	A						
001	3/4	0925	N-01	3	GW	X	X							2-Hand, 1-Kelly Workorder
002	3/4	0945	N-02	3	GW	X	X							
003	3/4	1140	N-9S	3	GW	X	X							
004	3/4	1300	N-9D	3	GW	X	X							
005	3/4	1415	N-6SR	3	GW	X	X							
006	3/4	1450	N-6DR	3	GW	X	X							
007	3/4	1540	N-11	3	GW	X	X							

PRESERVED CODES
A — NONE
B — HNO_3
C — H_2SO_4
D — NaOH
E — HCl
F — METHANOL
G — _____

SPECIAL INSTRUCTIONS: E-Mail Results to Steve Webb and Charity Teamer @ RMT Greenville SC

SAMPLER Relinquished by (Signature)	Date/Time	Received by (Signature)	Date/Time
Joe D Kim	3/6/05 1700		
Relinquished by (Signature)	Date/Time	Received by (Signature)	Date/Time
Fed Ex 847369845134		R Jacobs	3/9/05 945
Relinquished by (Signature)	Date/Time	Received by (Signature)	Date/Time

HAZARDS ASSOCIATED WITH SAMPLES
- ☐ Flammable
- ☐ Corrosive
- ☐ Highly Toxic
- ☐ Other (list) _____

Turn Around (circle one): (Normal) Rush
Report Due _____

(For Lab Use Only)
Receipt Temp: 0°C, 0°C
Temp Blank Y (N)
Receipt pH (Wet/Metals)

Custody Seal: Present/Absent Intact/Not Intact Seal #s

F-268 (6/04)

WHITE—LABORATORY COPY YELLOW—REPORT APPENDIX PINK—SAMPLER/SUBMITTER

851-921

En Chem, Inc. Cooler Receipt Log

Batch No. 8516918

Project Name or ID Hercules/RMT Inc No. of Coolers: 2 Temps: 0°C

A. Receipt Phase: Date cooler was opened: 3/10/05 **By:** AB

1: Were samples received on ice? (Must be ≤ 6 C)	(YES)	NO[2]	NA
2. Was there a Temperature Blank?	YES	(NO)	
3: Were custody seals present and intact on cooler? (Record on COC)	(YES)	NO	
4: Are COC documents present?	(YES)	NO[2]	
5: Does this Project require quick turn around analysis?	YES	(NO)	
6: Is there any sub-work?	YES	(NO)	
7: Are there any short *hold time tests*?	YES	(NO)	
8: Are any samples nearing expiration of hold-time? (Within 2 days)	YES[1]	(NO)	Contacted by/Who_____
9: Do any samples need to be Filtered or Preserved in the lab?	YES[1]	(NO)	Contacted by/Who_____

B. Check-in Phase: Date samples were Checked-in: 3/10/05 **By:** AB

1: Were all sample containers listed on the COC received and intact?	(YES)	NO[2]	NA
2: Sign the COC as received by En Chem. *Completed*	(YES)	NO	
3: Do sample labels match the COC?	(YES)	NO[2]	
4: Completed pH check on preserved samples. *(This statement does not apply to water: VOC, O&G, TOC, DRO, Total Rec. Phenolics)*	YES	NO	(NA)
5: Do samples have correct chemical preservation? *(This statement does not apply to water: VOC, O&G, TOC, DRO, Total Rec Phenolics)*	YES	NO[2]	(NA)
6: Are dissolved parameters field filtered?	YES	NO[2]	(NA)
7: Are sample volumes adequate for tests requested?	(YES)	NO[2]	
8: Are VOC samples free of bubbles >6mm	YES	NO[2]	(NA)
9: Enter samples into logbook. Completed	(YES)	NO	
10: Place laboratory sample number on all containers and COC. Completed	(YES)	NO	
11: Complete Laboratory Tracking Sheet (LTS). Completed	YES	NO	(NA)
12: *Start Nonconformance form*	YES	NO	(NA)
13: Initiate Subcontracting procedure Completed	YES	NO	(NA)
14: Check laboratory sample number on all containers and COC. 3/10/05	(YES)	NO	NA

Short Hold-time tests:

24 Hours or less	48 Hours	7 days	Footnotes
Coliform	BOD	Ash	1 Notify proper lab group immediately
Corrosivity = pH	Color	*Aqueous Extractable Organics- ALL*	2 Complete nonconformance memo.
Dissolved Oxygen	Nitrite or Nitrate	Flashpoint	
Hexavalent Chromium	Ortho Phosphorus	Free Liquids	
HPC	Surfactants	Sulfide	
Ferrous Iron	Turbidity	TDS	
Eh	En Core Preservation	TSS	
Odor	Power stop preservation	Total Solids	
Residual Chlorine		TVS	
Sulfite		TVSS	
		Unpreserved VOC's	

Rev. 2/05/04, Attachment to 1-REC-5.
Subject to QA Audit.

Reviewed by/date JK for JN 3/30/05

p:/everyone/forms/samplereceiving/crl doc

PROJECT WORK ORDER

Project: Hercules Landfill 009
Project Number: 70102.61
Sample Date: Week of 03/07/05
Project Manager: Steve Webb
Project Contact: Mark Miesfeldt/Charity Teamer

Enchem
1241 Bellevue St., Suite 9
Green Bay, WI 54302
Contact: Tom Trainor 800-736-2436
Fax: 920-469-8827

Annual Sample Collection Checklist for Hercules
March 2005 Sampling Event

LOCATION	Benzene Method 8260	Toxaphene Method SW-846	Total Suspended Solids	Field Ph Temp, Spec Cond	Comments/Instructions
	A	B	C		
N-01					Collect full round water levels.
N-02					Note visual observation of turbidity
N-03					
N-05					
N-06SR					
N-06DR					
N-07					
N-08					
N-09S					
N-09D					
N-10					
N-11					
N-12					
N-13					
N-14S					
N-14D					
N-15S					
N-15D					
RBLK-05101					
TBLK-05101					

A = 40mL Glass Container, Teflon Lined Septum\14 Day HT; HCL preserved.
B = 2 - 1 liter ambers for each location.
C = 1L nalgene w/no preservatives for each location.
Collect enough of one well for MS/MSD on Toxaphene (ie; 3 extra liters).

RMT Sampling Procedure
Please refer to the sampling procedure provided in the front pocket of the health and safety plan

RMT NOTES:
Conduct the landfill cap site inspection (include photographs)

8560946

✓1

75234

RMT®

CHAIN OF CUSTODY RECORD

30 Patewood Drive, Suite 100, Patewood Plaza One, Greenville, SC 29615-3535
Phone 864/281-0030 • Fax 864/281-0288

Project No.: 00-70102.61

Project/Client: Hercules / RMT Inc

Project Manager/Contact Person: Steve Webb / Charity Teamer

Filtered (Yes/No): No, No

Preserved (Code): A, A

Analyses Requested: Toxaphene/Method SW-8460; TSS

Lab No.	Yr. 05 Date	Time	Sample Station ID	Total Number of Containers	MATRIX	Toxaphene/Method SW-8460	TSS						Comments:
001	3/9	0915	N-14S	3	GW	X	X	1-1L poly[A]		2-1L amb[AA]			See Work Order
002	3/9	1110	N-14D	3	GW	X	X	↓		↓			
003	3/9	1400	N-10	3	GW	X	X	↓		↓			
004	3/9	1510	N-12	3	GW	X	X	↓		↓			
005	3/9	1520	N-08	3	GW	X	X	↓		↓			
006	3/9	1705	N-03	3	GW	X	X	↓		↓			
007	3/9	0915	MS/MSD	3	GW	X		3-1L amber[AAA]					
			#794572391010										

PRESERVED CODES
A — NONE
B — HNO_3
C — H_2SO_4
D — NaOH
E — HCl
F — METHANOL
G — ______

SPECIAL INSTRUCTIONS E-Mail Results to Steve Webb and Charity Teamer @ RMT Greenville SC.

SAMPLER Relinquished by (Signature)	Date/Time	Received by (Signature)	Date/Time
[signature]	3/9/05 1745	FED EX	3/9/05 1745
Relinquished by (Signature): FED EX	3/10/05 0930	Received by (Signature): Ashley Brusky	3/10/05 0930
Relinquished by (Signature)	Date/Time	Received by (Signature)	Date/Time

HAZARDS ASSOCIATED WITH SAMPLES
☐ Flammable
☐ Corrosive
☐ Highly Toxic
☐ Other (list) ______

Turn Around (circle one) (Normal) Rush

Report Due ______

(For Lab Use Only)

Receipt Temp: 0°C
Temp Blank Y (N)

Receipt pH (Wet/Metals) NA

Custody Seal: (Present)/Absent (Intact)/Not Intact Seal #s

F-268 (6/04)

WHITE—LABORATORY COPY YELLOW—REPORT APPENDIX PINK—SAMPLER/SUBMITTER

[illegible]

En Chem, Inc. Cooler Receipt Log

Batch No 857028

Project Name or ID Hercules No. of Coolers: 2 Temps: 2°C

A. Receipt Phase: Date cooler was opened: 3-11-05 By: SFaen

1: Were samples received on ice? (Must be ≤ 6 C)	(YES)	NO[2]	NA
2 Was there a Temperature Blank?	YES	(NO)	
3: Were custody seals present and intact on cooler? (Record on COC)	(YES)	NO	
4: Are COC documents present?	(YES)	NO[2]	
5: Does this Project require quick turn around analysis?	YES	(NO)	
6: Is there any sub-work?	YES	(NO)	
7: Are there any short hold time tests?	YES	(NO)	
8: Are any samples nearing expiration of hold-time? (Within 2 days)	YES[1]	(NO)	Contacted by/Who_____
9: Do any samples need to be Filtered or Preserved in the lab?	YES[1]	(NO)	Contacted by/Who_____

B. Check-in Phase: Date samples were Checked-in: 3-11-05 By: SFaen

1: Were all sample containers listed on the COC received and intact?	(YES)	NO[2]	NA
2: Sign the COC as received by En Chem Completed	(YES)	NO	
3: Do sample labels match the COC?	(YES)	NO[2]	
4: Completed pH check on preserved samples. *(This statement does not apply to water: VOC, O&G, TOC, DRO, Total Rec. Phenolics)*	YES	NO	(NA)
5: Do samples have correct chemical preservation? *(This statement does not apply to water: VOC, O&G, TOC, DRO, Total Rec. Phenolics)*	YES	NO[2]	(NA)
6: Are dissolved parameters field filtered?	YES	NO[2]	(NA)
7: Are sample volumes adequate for tests requested?	(YES)	NO[2]	
8: Are VOC samples free of bubbles >6mm	(YES)	NO[2]	NA
9: Enter samples into logbook Completed	(YES)	NO	
10: Place laboratory sample number on all containers and COC Completed	(YES)	NO	
11: Complete Laboratory Tracking Sheet (LTS) Completed	YES	NO	(NA)
12: Start Nonconformance form	YES	NO	(NA)
13: Initiate Subcontracting procedure Completed	YES	NO	(NA)
14: Check laboratory sample number on all containers and COC cf 3/11/05	(YES)	NO	NA

Short Hold-time tests:

24 Hours or less	*48 Hours*	*7 days*	*Footnotes*
Coliform	BOD	Ash	1 Notify proper lab group immediately.
Corrosivity = pH	Color	Aqueous Extractable Organics- ALL	2 Complete nonconformance memo.
Dissolved Oxygen	Nitrite or Nitrate	Flashpoint	
Hexavalent Chromium	Ortho Phosphorus	Free Liquids	
HPC	Surfactants	Sulfide	
Ferrous Iron	Turbidity	TDS	
Eh	En Core Preservation	TSS	
Odor	Power stop preservation	Total Solids	
Residual Chlorine		TVS	
Sulfite		TVSS	
		Unpreserved VOC's	

Rev 2/05/04, Attachment to 1-REC-5
Subject to QA Audit

Reviewed by/date [illegible] TH 3/30/05

PROJECT WORK ORDER

Project: Hercules Landfill 009
Project Number: 70102.61
Sample Date: Week of 03/07/05
Project Manager: Steve Webb
Project Contact: Mark Miesfeldt/Charity Teamer

Enchem
1241 Bellevue St., Suite 9
Green Bay, WI 54302
Contact: Tom Trainor 800-736-2436
Fax: 920-469-8827

Annual Sample Collection Checklist for Hercules
March 2005 Sampling Event

LOCATION	Benzene Method 8260	Toxaphene Method SW-846	Total Suspended Solids	Field Ph, Temp, Spec Cond	Comments/Instructions
	A	B	C		
N-01					Collect full round water levels.
N-02					Note visual observation of turbidity
N-03					
N-05					
N-06SR					
N-06DR					
N-07					
N-08					
N-09S					
N-09D					
N-10					
N-11					
N-12					
N-13					
N-14S					
N-14D					
N-15S					
N-15D					
RBLK-05101					
TBLK-05101					

A = 40mL Glass Container, Teflon Lined Septum\14 Day HT; HCL preserved.
B = 2 - 1 liter ambers for each location.
C = 1L nalgene w/no preservatives for each location.
Collect enough of one well for MS/MSD on Toxaphene (ie; 3 extra liters).

RMT Sampling Procedure
Please refer to the sampling procedure provided in the front pocket of the health and safety plan

RMT NOTES:
Conduct the landfill cap site inspection (include photographs)

85-1028

√4

75235

RMT®

CHAIN OF CUSTODY RECORD

30 Patewood Drive, Suite 100, Patewood Plaza One, Greenville, SC 29615-3535
Phone 864/281-0030 • Fax 864/281-0288

Project No. 00-70102.61
Project/Client: Hercules / RMT Inc.
Project Manager/Contact Person: Steve Webb / Charity Teamer

Filtered (Yes/No): No, No, No
Preserved (Code): A, A, E

Analyses Requested: Toxaphene / Method SW-846; TSS; Benzene / Method 8260

PRESERVED CODES
A — NONE
B — HNO_3
C — H_2SO_4
D — NaOH
E — HCl
F — METHANOL
G —

Lab No.	Yr. 05 Date	Time	Sample Station ID	Total Number of Containers	MATRIX	Toxaphene	TSS	Benzene	Comments
001	3/10	0940	N-05	6	GW	X	X	X	1-1L poly A, 2-1L amber, 3-40 ml VOA
002	3/10	1045	N-07	3	GW	X	X		
003	3/10	1110	N-13	3	GW	X	X		
004	3/10	1245	N-15S	3	GW	X	X		
005	3/10	1420	N-15D	3	GW	X	X		
006	3/10	0805	RBLK05101	5	GW	X		X	2, 3-40 ml
007	3/10		TBLK05101					X	2
			#847369845112			NON ENCHEM TRIP BLANK. 3-11-05 SF.			

SPECIAL INSTRUCTIONS E-Mail Results to Steve Webb and Charity Teamer @ RMT Inc Greenville SC.

SAMPLER Relinquished by (Signature)	Date/Time	Received by (Signature)	Date/Time
Joe DK	3/10/05 1530		
Relinquished by (Signature) FedEx	3-11-05 925	Received by (Signature) S Falk	3-11-05 925
Relinquished by (Signature)	Date/Time	Received by (Signature)	Date/Time

HAZARDS ASSOCIATED WITH SAMPLES
☐ Flammable
☐ Corrosive
☐ Highly Toxic
☐ Other (list)

Turn Around (circle one) (Normal) Rush
Report Due

(For Lab Use Only)
Receipt Temp: 2°C
Temp Blank Y (N)
Receipt pH (Wet/Metals) N/A

Custody Seal: (Present)/Absent (Intact)/Not Intact Seal #s

F-268 (6/04)   857078   WHITE—LABORATORY COPY   YELLOW—REPORT APPENDIX   PINK—SAMPLER/SUBMITTER

Attachment 2

Attachment 2
Summary of Groundwater Results

Well	Parameter	Extract ID	Analysis Method	EnChem	Flag	Result	EQL*	MDL	Units
N-01	Toxaphene	856921-001	SW846 8081A	EnChem	<	3.0	3.0		ug/L
N-01	Chlorinated Camphene	856921-001	SW846 8081A	EnChem	<	3.0	3.0		ug/L
N-01	Chlorinated Camphene	856921-001	ECNI-MS (RT= 30-62 min)	SKIO	<=	0.00584	2.22000	0.22200	ug/L
N-01	Chlorinated Camphene	856921-001	ECNI-MS (RT= 35-62 min)	SKIO	<=	0.00584	2.22000	0.22200	ug/L
N-01	Chlorinated Camphene	856921-001	ECD (RT= 24-49 min)	SKIO	<=	0.03260	2.22000	0.22200	ug/L
N-02	Toxaphene	856921-002	SW846 8081A	EnChem	<	3.0	3.0		ug/L
N-02	Chlorinated Camphene	856921-002	SW846 8081A	EnChem	<	3.0	3.0		ug/L
N-02	Chlorinated Camphene	856921-002	ECNI-MS (RT= 30-62 min)	SKIO	<=	0.02130	2.22000	0.22200	ug/L
N-02	Chlorinated Camphene	856921-002	ECNI-MS (RT= 35-62 min)	SKIO	<=	0.02480	2.22000	0.22200	ug/L
N-02	Chlorinated Camphene	856921-002	ECD (RT= 24-49 min)	SKIO	<=	0.03450	2.22000	0.22200	ug/L
N-9S	Toxaphene	856921-003	SW846 8081A	EnChem	<	3.0	3.0		ug/L
N-9S	Chlorinated Camphene	856921-003	SW846 8081A	EnChem	<	3.0	3.0		ug/L
N-9S	Chlorinated Camphene	856921-003	ECNI-MS (RT= 30-62 min)	SKIO	<=	0.04300	2.22000	0.22200	ug/L
N-9S	Chlorinated Camphene	856921-003	ECNI-MS (RT= 35-62 min)	SKIO	<=	0.04630	2.22000	0.22200	ug/L
N-9S	Chlorinated Camphene	856921-003	ECD (RT= 24-49 min)	SKIO	<=	0.02020	2.22000	0.22200	ug/L
N-9D	Toxaphene	856921-004	SW846 8081A	EnChem	<	3.0	3.0		ug/L
N-9D	Chlorinated Camphene	856921-004	SW846 8081A	EnChem	<	3.0	3.0		ug/L
N-9D	Chlorinated Camphene	856921-004	ECNI-MS (RT= 30-62 min)	SKIO	<=	0.01440	2.22000	0.22200	ug/L
N-9D	Chlorinated Camphene	856921-004	ECNI-MS (RT= 35-62 min)	SKIO	<=	0.01440	2.22000	0.22200	ug/L
N-9D	Chlorinated Camphene	856921-004	ECD (RT= 24-49 min)	SKIO	<=	0.03400	2.22000	0.22200	ug/L
N-6SR	Toxaphene	856921-005	SW846 8081A	EnChem	<	3.0	3.0		ug/L
N-6SR	Chlorinated Camphene	856921-005	SW846 8081A	EnChem	J	1.6	3.0		ug/L
N-6SR	Chlorinated Camphene	856921-005	ECNI-MS (RT= 30-62 min)	SKIO		1.38000	2.22000	0.22200	ug/L
N-6SR	Chlorinated Camphene	856921-005	ECNI-MS (RT= 35-62 min)	SKIO		1.55000	2.22000	0.22200	ug/L
N-6SR	Chlorinated Camphene	856921-005	ECD (RT= 24-49 min)	SKIO		1.02000	2.22000	0.22200	ug/L
N-6DR	Toxaphene	856921-006	SW846 8081A	EnChem	<	3.0	3.0		ug/L
N-6DR	Chlorinated Camphene	856921-006	SW846 8081A	EnChem	<	3.0	3.0		ug/L
N-6DR	Chlorinated Camphene	856921-006	ECNI-MS (RT= 30-62 min)	SKIO	<=	0.00258	2.22000	0.22200	ug/L
N-6DR	Chlorinated Camphene	856921-006	ECNI-MS (RT= 35-62 min)	SKIO	<=	0.00258	2.22000	0.22200	ug/L
N-6DR	Chlorinated Camphene	856921-006	ECD (RT= 24-49 min)	SKIO	<=	0.00513	2.22000	0.22200	ug/L
N-11	Toxaphene	856921-007	SW846 8081A	EnChem	<	3.0	3.0		ug/L
N-11	Chlorinated Camphene	856921-006	SW846 8081A	EnChem	<	3.0	3.0		ug/L
N-11	Chlorinated Camphene	856921-007	ECNI-MS (RT= 30-62 min)	SKIO		0.67100	2.22000	0.22200	ug/L
N-11	Chlorinated Camphene	856921-007	ECNI-MS (RT= 35-62 min)	SKIO		0.74000	2.22000	0.22200	ug/L
N-11	Chlorinated Camphene	856921-007	ECD (RT= 24-49 min)	SKIO		0.59900	2.22000	0.22200	ug/L
N-14S	Toxaphene	856996-001	SW846 8081A	EnChem	<	3.0	3.0		ug/L
N-14S	Chlorinated Camphene	856996-001	SW846 8081A	EnChem	<	3.0	3.0		ug/L

Attachment 2
Summary of Groundwater Results

Well	Parameter	Extract ID	Analysis Method	EnChem	Flag	Result	EQL*	MDL	Units
N-14S	Chlorinated Camphene	856996-001	ECNI-MS (RT= 30-62 min)	SKIO	<=	0.02630	2.22000	0.22200	ug/L
N-14S	Chlorinated Camphene	856996-001	ECNI-MS (RT= 35-62 min)	SKIO	<=	0.02630	2.22000	0.22200	ug/L
N-14S	Chlorinated Camphene	856996-001	ECD (RT= 24-49 min)	SKIO	<=	0.02900	2.22000	0.22200	ug/L
N-14D	Toxaphene	856996-002	SW846 8081A	EnChem	<	3.0	3.0		ug/L
N-14D	Chlorinated Camphene	856996-002	SW846 8081A	EnChem	<	3.0	3.0		ug/L
N-14D	Chlorinated Camphene	856996-002	ECNI-MS (RT= 30-62 min)	SKIO	<=	0.00091	2.22000	0.22200	ug/L
N-14D	Chlorinated Camphene	856996-002	ECNI-MS (RT= 35-62 min)	SKIO	<=	0.00091	2.22000	0.22200	ug/L
N-14D	Chlorinated Camphene	856996-002	ECD (RT= 24-49 min)	SKIO	<=	0.03700	2.22000	0.22200	ug/L
N-10	Toxaphene	856996-003	SW846 8081A	EnChem	<	3.0	3.0		ug/L
N-10	Chlorinated Camphene	856996-003	SW846 8081A	EnChem	<	3.0	3.0		ug/L
N-10	Chlorinated Camphene	856996-003	ECNI-MS (RT= 30-62 min)	SKIO	<=	0.00687	2.22000	0.22200	ug/L
N-10	Chlorinated Camphene	856996-003	ECNI-MS (RT= 35-62 min)	SKIO	<=	0.00687	2.22000	0.22200	ug/L
N-10	Chlorinated Camphene	856996-003	ECD (RT= 24-49 min)	SKIO	<=	0.07390	2.22000	0.22200	ug/L
N-12	Toxaphene	856996-004	SW846 8081A	EnChem	<	3.0	3.0		ug/L
N-12	Chlorinated Camphene	856996-004	SW846 8081A	EnChem	<	3.0	3.0		ug/L
N-12	Chlorinated Camphene	856996-004	ECNI-MS (RT= 30-62 min)	SKIO	<=	0.01690	2.22000	0.22200	ug/L
N-12	Chlorinated Camphene	856996-004	ECNI-MS (RT= 35-62 min)	SKIO	<=	0.02620	2.22000	0.22200	ug/L
N-12	Chlorinated Camphene	856996-004	ECD (RT= 24-49 min)	SKIO	<=	0.14100	2.22000	0.22200	ug/L
N-08	Toxaphene	856996-005	SW846 8081A	EnChem	<	3.0	3.0		ug/L
N-08	Chlorinated Camphene	856996-005	SW846 8081A	EnChem	<	3.0	3.0		ug/L
N-08	Chlorinated Camphene	856996-005	ECNI-MS (RT= 30-62 min)	SKIO	<=	0.00547	2.22000	0.22200	ug/L
N-08	Chlorinated Camphene	856996-005	ECNI-MS (RT= 35-62 min)	SKIO	<=	0.00547	2.22000	0.22200	ug/L
N-08	Chlorinated Camphene	856996-005	ECD (RT= 24-49 min)	SKIO	<=	0.03570	2.22000	0.22200	ug/L
N-03	Toxaphene	856996-006	SW846 8081A	EnChem	<	3.0	3.0		ug/L
N-03	Chlorinated Camphene	856996-006	SW846 8081A	EnChem	<	3.0	3.0		ug/L
N-03	Chlorinated Camphene	856996-006	ECNI-MS (RT= 30-62 min)	SKIO	<=	0.22200	2.22000	0.22200	ug/L
N-03	Chlorinated Camphene	856996-006	ECNI-MS (RT= 35-62 min)	SKIO	<=	0.22200	2.22000	0.22200	ug/L
N-03	Chlorinated Camphene	856996-006	ECD (RT= 24-49 min)	SKIO	<=	0.06080	2.22000	0.22200	ug/L
N-05	Chlorinated Camphene	857028-001	SW846 8081A	EnChem	<	3.0	3.0		ug/L
N-05	Toxaphene	857028-001	SW846 8081A	EnChem	<	3.0	3.0		ug/L
N-05	Chlorinated Camphene	857028-001	ECNI-MS (RT= 30-62 min)	SKIO	<=	0.01140	2.22000	0.22200	ug/L
N-05	Chlorinated Camphene	857028-001	ECNI-MS (RT= 35-62 min)	SKIO	<=	0.01140	2.22000	0.22200	ug/L
N-05	Chlorinated Camphene	857028-001	ECD (RT= 24-49 min)	SKIO	<=	0.04420	2.22000	0.22200	ug/L
N-07	Toxaphene	857028-002	SW846 8081A	EnChem	<	3.0	3.0		ug/L
N-07	Chlorinated Camphene	857028-002	SW846 8081A	EnChem	<	3.0	3.0		ug/L
N-07	Chlorinated Camphene	857028-002	ECNI-MS (RT= 30-62 min)	SKIO	<=	0.22200	2.22000	0.22200	ug/L
N-07	Chlorinated Camphene	857028-002	ECNI-MS (RT= 35-62 min)	SKIO	<=	0.22200	2.22000	0.22200	ug/L
N-07	Chlorinated Camphene	857028-002	ECD (RT= 24-49 min)	SKIO	<=	0.03280	2.22000	0.22200	ug/L

Attachment 2
Summary of Groundwater Results

Well	Parameter	Extract ID	Analysis Method	EnChem	Flag	Result	EQL*	MDL	Units
N-13	Toxaphene	857028-003	SW846 8081A	EnChem	<	3.0	3.0		ug/L
N-13	Chlorinated Camphenes	857028-003	SW846 8081A	EnChem	<	3.0	3.0		ug/L
N-13	Chlorinated Camphenes	857028-003	ECNI-MS (RT= 30-62 min)	SKIO	<=	0.22200	2.22000	0.22200	ug/L
N-13	Chlorinated Camphenes	857028-003	ECNI-MS (RT= 35-62 min)	SKIO	<=	0.22200	2.22000	0.22200	ug/L
N-13	Chlorinated Camphenes	857028-003	ECD (RT= 24-49 min)	SKIO	<=	0.02930	2.22000	0.22200	ug/L
N-15S	Toxaphene	857028-004	SW846 8081A	EnChem	<	3.0	3.0		ug/L
N-15S	Chlorinated Camphenes	857028-004	SW846 8081A	EnChem	<	3.0	3.0		ug/L
N-15S	Chlorinated Camphenes	857028-004	ECNI-MS (RT= 30-62 min)	SKIO	<=	0.00413	2.22000	0.22200	ug/L
N-15S	Chlorinated Camphenes	857028-004	ECNI-MS (RT= 35-62 min)	SKIO	<=	0.00413	2.22000	0.22200	ug/L
N-15S	Chlorinated Camphenes	857028-004	ECD (RT= 24-49 min)	SKIO	<=	0.03760	2.22000	0.22200	ug/L
N-15D	Toxaphene	857028-005	SW846 8081A	EnChem	<	3.0	3.0		ug/L
N-15D	Chlorinated Camphenes	857028-005	SW846 8081A	EnChem	<	3.0	3.0		ug/L
N-15D	Chlorinated Camphenes	857028-005	ECNI-MS (RT= 30-62 min)	SKIO	<=	0.22200	2.22000	0.22200	ug/L
N-15D	Chlorinated Camphenes	857028-005	ECNI-MS (RT= 35-62 min)	SKIO	<=	0.22200	2.22000	0.22200	ug/L
N-15D	Chlorinated Camphenes	857028-005	ECD (RT= 24-49 min)	SKIO	<=	0.01710	2.22000	0.22200	ug/L
RBLK-05101	Chlorinated Camphenes	857028-006	SW846 8081A	EnChem	<	3.0	3.0		ug/L
RBLK-05101	Toxaphene	857028-006	SW846 8081A	EnChem	<	3.0	3.0		ug/L
RBLK-05101	Chlorinated Camphenes	857028-006	ECNI-MS (RT= 30-62 min)	SKIO	<=	0.22200	2.22000	0.22200	ug/L
RBLK-05101	Chlorinated Camphenes	857028-006	ECNI-MS (RT= 35-62 min)	SKIO	<=	0.22200	2.22000	0.22200	ug/L
RBLK-05101	Chlorinated Camphenes	857028-006	ECD (RT= 24-49 min)	SKIO	<=	0.05460	2.22000	0.22200	ug/L

* EQL = Reported by EnChem and MDL X 10 for SKIO

A RE-ANALYSIS OF THE EUROPEAN MATT (2000) TOXICITY DATA AND DEVELOPMENT OF A REFERENCE DOSE FOR WEATHERED TOXAPHENE

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April 26, 2005

EXECUTIVE SUMMARY

Toxaphene is a mixture of chlorinated camphenes and bornanes that was produced and used in the United States until 1982. Toxaphene has the distinction of being the most used pesticide in history with 1.3 million tons having been released worldwide. So-called "Technical" Toxaphene (TT) consists of a mixture of up to 800 different chemicals, known as congeners. Once in the environment, technical toxaphene undergoes "weathering" by both biotic and abiotic processes and the number and identity of the chemicals in "weathered" toxaphene (WT) are different than TT.

One aspect of the weathering of TT is that the current human body burden consists of five congeners only, with three congeners predominant. These are considered the three persistent congeners (p-26, p-50 and p-62) (Σ3PC). A significant gap in our knowledge of the WT toxicity is the mode of action: is it the persistent congeners that make up the human body burden or the metabolism and/or metabolites of the non-persistent congeners that account for the adverse effects of WT?

The largest amount of information available on the toxicity of WT was developed by a group of European Union scientists tasked with addressing the risk of WT in fish of European waters and reported in the "Investigation Into the Monitoring, Analysis and Toxicity of Toxaphene" (MATT, 2000). The data presented in (MATT, 2000) was used here to derive a two-fold reference dose, specifically for assessing risk from fish ingestion at the Terry Creek Superfund site and possibly other hazardous waste sites with WT contamination. The critical effect considered is tumor promotion and this endpoint is considered protective for other endpoints as well. This use of the reference dose represents one of the first instances in which EPA has applied a threshold-type toxicity to a potentially carcinogenic chemical. This procedure is consistent with EPA's new cancer guidelines and with the goal of harmonization of cancer and non-cancer risk assessment.

The most appropriate reference dose for WT is based on consideration of the sum of the three most persistent congeners (Σ3PC) that comprise majority of the human body burden. The value of RfD is 2E-05 mg/kg-day for Σ3PC. Cleanup levels based on Σ3PC may be expressed in terms of the concentration of the entire WT mixture using the percentage of Σ3PC. This value derived here can be contrasted witht the Tolerable Daily Intake (TDI) from the MATT (2000) of 0.0069 mg/kg-day. This TDI value was based on the entire WT mixture. In gereral, cleanups based on Σ3PC RfD will be more stringent than those based on the MATT TDI.

Preliminary Remediation Goals (PRGs) were developed for WT based on the Σ3PC RfD and a representative percentage of Σ3PC in the WT mixture. These are shown in comparison to the PRGs from the Region 9 table that are based on TT. Please note that the proposed PRGs are almost 100 fold higher.

Comparison of PRGs developed with the WT RfD and the TT Cancer Slope Factor

	Region 9 PRGs (based on TT)	*Proposed PRGs (WT based on Σ3PC)*
Residential Soil (mg/kg)	0.44	12
Industrial Soil (mg/kg)	1.6	120
Ambient Air (μg/m^3)	0.006	0.7
Tap Water (μg/L)	0.061	0.8

The maximum contaminant level for toxaphene is 3 ug/L. Hence, considering TT, the cancer risk at the MCL is 5E-05 and considering WT, the hazard quotient at the MCL is 4. It may also be advisable to examine current fish advisories based on toxaphene to determine if they should be modified when the Σ3PC RfD is considered.

For Terry Creek fish, 95% upper confidence limits on the mean (95UCLs) were calculated for Σ3PC as 0.23 mg/kg respectively. Daily fish ingestion rates in terms of g/kg BW/day were obtained from Table 10-61 of EPA's Exposure Factors Handbook. A child's intake of Σ3PC was 9.8E-5 mg/kg-day respectively. This amount was threefold greater than an adult's intake from eating the same fish. Comparing this intake amount with the RfD gives an HQ value of 4 for Σ3PC.

The HQ value of 4 representing the hazard of Σ3PC in children was chosen as a basis for calculating a cleanup level or a "safe" level that would require no limits on fish consumption. Note this cleanup level would be protective of adults as well. The concentrations of Σ3PC and WT were highly correlated in Terry Creek fish and a linear regression was used to obtain the WT cleanup level based on an HQ of 4 for Σ3PC. These cleanup level for Σ3PC was 0.055 mg/kg and for WT was 1.3 mg/kg.

Any additional research on the toxicity of WT should concentrate upon determining the mode of action in order to address the data gap of whether the persistent congeners or the non-persistent congeners represent the toxic species. Specific experiments include repetition of the European in vivo and in vitro tumor promotion studies using different dose ranges in order to determine an effect threshold in both types of studies. Whole animal developmental studies for WT are also needed. The question of critical periods, i.e. increased susceptibility to the adverse effects of WT during a particular phase of life, should also be addressed. The question of the mode of action remains paramount. Seeking to answer this question will lead to the most relevant data needs and corresponding studies.

TABLE OF CONTENTS

1.0 INTRODUCTION

Toxaphene is a mixture of chlorinated camphenes and bornanes that was produced and used in the United States until 1982. Toxaphene has the distinction of being the most used pesticide in history with 1.3 million tons having been released worldwide. So-called "Technical" Toxaphene (TT) consists of a mixture of up to 800 different chemicals, known as congeners. Once in the environment, technical toxaphene undergoes "weathering" by both biotic and abiotic processes and the number and identity of the chemicals in "weathered" toxaphene (WT) are different than TT. Hence, toxaphene has presented a significant challenge to analytical chemists. In addition, the "weathered" toxaphene (WT) has dissimilar toxicity characteristics than technical toxaphene (TT).

The purpose of this assessment is fourfold: first, to summarize the available information about the toxicity of weathered toxaphene; second, to develop a toxicity criterion for WT(i.e. reference dose or cancer slope factor) to be used until additional scientific information about WT becomes available; third, to perform a preliminary risk assessment for fish in Terry Creek; and, fourth, to suggest potential avenues of research into the toxicity of WT.

Toxaphene was manufactured by the Hercules Chemical Company in Brunswick, Georgia and the toxicity of WT remaining at the Terry Creek and 009 Landfill sites is the primary subject of this assessment. Nonetheless, it is acknowledged that the results of this assessment may have bearing at other hazardous waste sites (USEPA, 2005).

One of the significant hurdles in risk assessment of WT is the issue of chemical analysis. The difficulties with EPA Method 8081 have been presented elsewhere (EPA, 2005). In short, Method 8081 fails to detect WT in environmental samples. A congener-specific method known as Gas Chromatography/Negative Ion Mass Spectroscopy(GC/NIMS)is considered the most appropriate method for detecting congeners of WT (EPA, 2005; Maruya, 2001).

In this paper, a comparison of the various regulatory levels of toxaphene will be presented. These regulatory levels are based on a cancer endpoint. It is clear from many studies (Young and Freeman, 2001; 2004)) that the mutagenicity, carcinogenicity and systemic toxicity of WT is not the same as TT. Hence, a consideration of the various toxic endpoints observed will also be presented.

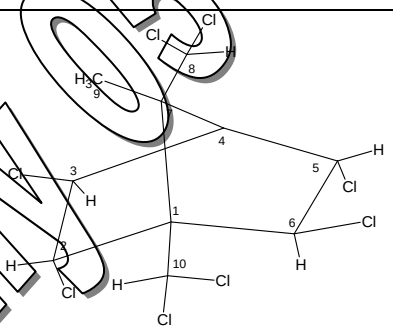
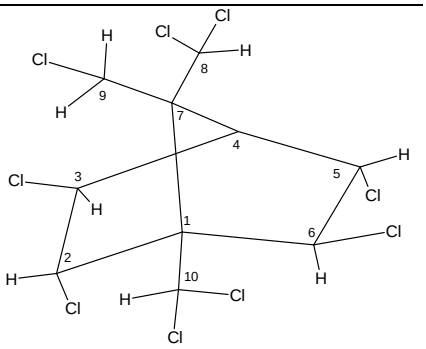
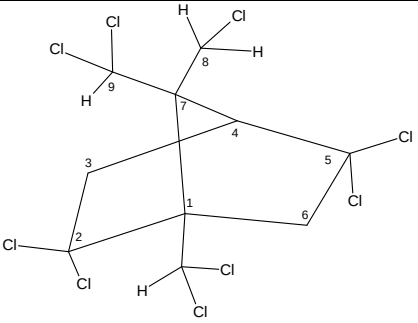
1.1 Nomenclature and Structure of Persistent Toxaphene Congeners

There is a large and growing literature on the analytical issues and toxicity of WT (e.g., MATT 2000; de Geus et al., 1999, 2000) Unfortunately, several nomenclature systems have evolved. Readers unfamiliar with toxaphene chemistry invariably find the various names for the same chemical a confusing issue. Regarding human exposure to WT, there are at most five and perhaps only three significant congeners.

Because of the analytical issues associated with toxaphene, individual congeners are available for purchase as analytical standards. These individual congeners are also known as "Parlars" and specific congeners have been given a Parlar number to easily

refer to them (e.g. p-26) Also, available as an analytical standard are a mixture of the 5 congeners predominant in biota and a mixture of 22 congeners as a standard for TT.

The major toxaphene congeners that persist in fish, marine mammals, human serum and human breast milk are p-26, p-50 and p-62. The congeners p-40, p-41, p-44 and p-62 are also observed. The table below gives the nomenclature and structure for the three predominant congeners of interest for human health risk assessment from fish consumption or other types of exposures.

Parlar Name	*Andrews-Vetter code*	*Wester code*	*IUPAC names of both chiral forms*	*Structure*
p-26	B8-1413	B[12012]-[202]r B[12012]-[202]s	2-endo, 3-exo, 5-endo, 6-exo, 8,8,10,10 octachlorobornane or 2-exo, 3-endo, 5-exo, 6-endo, 8,8,10,10 octachlorobornane Also known as T_2	
p-50	B9-1679	B[12012]-[212]r B[12012]-[212]s	2-endo, 3-exo, 5-endo, 6-exo, 8,8,9,10,10 nonachlorobornane or 2-exo, 3-endo, 5-exo, 6-endo, 8,8,9,10,10 nonachlorobornane Also known as T_{12}	
p-62	B9-1025		2,2,5,5,8,9,9,10,10 nonachlorobornane	

Although the Parlar code provides no information about the structure of the particular congener, it will be used here for convenience and consistency with other sources. Note that the IUPAC names and the Wester code provide structural information. Enantiomeric chiral forms exist for many of the congeners (p-26 and p-50 in the table); however, the structure of only one of the enantiomers is shown. For an additional discussion of nomenclature, the reader is referred to de Geus et al. (1999, 2000).

2.0 AVAILABLE TOXICITY CRITERIA FOR TOXAPHENE

2.1 Summary of Toxaphene Toxicity Criteria

In 1991, EPA's IRIS program developed a cancer slope factor for TT based on two rodent studies, the 1978 Litton Bionetics B6C3F1 mouse study and the 1979 NCI Osbourne-Mendel rat study. The slope factor was derived using the linearized multistage model with a value of 1.1 per mg/kg-day. The Ombudsman's Preliminary Technical Assessment (USEPA, 2005) indicates it is not appropriate to apply this value to WT or indivdual congeners.

In 2000, a peer review panel reevaluated the cancer slope factor and recommended that the value be reduced to 0.1 per mg/kg-day (Goodman et al., 2000). The basis of this recommendation was a reexamination of the original histological materials by an expert pathology working group and application of benchmark dose modeling to account for high background liver tumor rate.

In 2003, CAL-EPA (OEHHA, 2003) determined a cancer slope factor for TT and a public health goal for drinking water based on the 1978 Litton Bionetics rodent studies. The value of the slope factor was 1.2 per mg/kg-day, essentially the same as the EPA slope factor. The OEHHA public health goal document provides an excellent review of the toxicity. Several non-cancer endpoints are discussed and NOAELs/LOAELs are provided. The total safety factor of 1000 was used for NOAELs and a safety factor of 3000 was used for LOAELs. These safety factors account for interspecies extrapolation, subchronic to chronic extrapolation, human variability and LOAEL to NOAEL extrapolation.

In 2004, Buranatrevedh (2004) performed a risk assessment based on toxaphene concentrations reported in ATSDR's Toxicological Profile for Toxaphene (ATSDR, 1996). Buranatrevedh (2004) derived a slope factor of 0.86 per mg/kg-day from the original rat data. The methodology in this derivation is flawed – a t-distribution was used to determine confidence limits around the extrapolated low dose slope; this is incorrect: instead a chi-square distribution should have been used (Crump, 1984).

However, all of these derivations are based on the toxicity of TT to rodents and are not appropriate for evaluating the potential toxicity of WT (USEPA, 2005).

Germany has determined a maximum residue level in fish of 0.1 mg/kg as the sum of p-26, p-50 and p-62 concentrations (McHugh et al., 2004). The risk or health basis for this value is not known. Health Canada has proposed a TDI value of 200 ng/kg/day (Receveur et al., 1998) based on TT. The scientific basis for the Canadian value is not known; nor is it known whethr this TDI represents TT, WT or Σ3PC. Presently, Health Canada is performing toxicological studies with rodents and primates for the purpose of revising the toxaphene TDI (Tryphonas et al. 2000, 2001; Bryce et al., 2001; Arnold et al., 2001).

3.0 TOXAPHENE CONGENERS THAT COMPRISE THE HUMAN BODY BURDEN

Because of metabolism and excretion, toxaphene weathers in different ways in biotic and abiotic media (Angerhofer et al., 1999). In fact, different species of animals end up with different toxaphene congener profiles comprising their respective body burdens. Again, these differences in body burden are due to differences in the extent that the individual congeners are metabolized.

The congeners that comprise the human body burden are those for which we wish to know the toxicity. In 2004, Barr et al. reported concentrations of toxaphene congeners from pooled human serum from three US cities. Only p-26 and p-50 were positively identified in the serum samples. There were indications that p-40/41, p-44 and p-62 were also present. Gill et al. (1996) presented methods for toxaphene congener analysis in serum and observed p-26, p-40/41, p-44 and p-50 in human serum extracts. Polder et al. (2003) measured p-26, p-50 and p-62 in human milk samples from Kargopol, Severodvinsk, Arkhangelsk and Naryan-Mar, Russian towns all north of 60 degrees latitude. Skopp et al. (2002b) used GC-NIMS to measure p-26, p-41, p-44 and p-50 in human milk obtained from mothers in the north Rhine area of Germany.

One can determine the approximate ratio of the persistent congeners in the human body burden from concentrations in tables from Barr et al. (2004), Polder et al. (2003) and Skopp et al. (2002b) (Table 1). Average percentages of p-26, p50 and p-62 in the human body burden were calculated from these three sources and are shown in Table 1. These three congeners comprise almost 90% of the human body burden. These sum of these three persistent congeners will be termed Σ3PC.

In all these studies, the predominant congeners observed in humans were p-26 and p-50 but p-40/41, p-44 and p62 were also present. The conclusions that p-26, p-50 and p-62 are the persistent congeners and hence of interest for evaluating human health effects has been supported by the fact that toxaphene "weathers" very quickly in biological systems. Oral administration of toxaphene to mammals and birds results in fecal passage of about 40% of the administered chemical and metabolism of the remaining toxaphene on a a time scale of weeks (Andrews et al., 1996; Pollock and Hillstrand, 1982; Pollock and Kilgore, 1980; Biessman et al., 1983; Mohammed et al., 1983). Only five or six persistent congeners remain in the tissues of mammals and birds. Because of this rapid "weathering," the exposure of mammals to WT is equivalent to exposure to Σ3PC and the toxic effects are very likely due to Σ3PC. Consideration of the toxicity of TT in a risk assessment for Σ3PC is consistent with the practices in EPA's pesticide program. In that program, because many pesticides produce their effects through metabolites, the pesticides can be administered in animal studies to ascertain the toxic effect of the metabolites, so called "auto-toxicity" (James, Lamb, personal communication, 2005).

Because it is likely that the body burden of the persistent congeners produces the toxic effects, a toxicity assessment of WT should concentrate on these persistent congeners. The MATT (2000) based its toxicity assessment on the entire WT mixture and thus implicitly concluded that metabolism was likely needed for the tumor promotion

effects of WT. This implicit position of the MATT (2000) is not in agreement with the position here that the persistent congeners produce the toxic effects.

4.0 WEATHERING OF TOXAPHENE IN FISH AND BIOTA

It should be clear from the few (3-5) congeners that comprise the human body burden versus the many congeners present in technical toxaphene (>600) that changes in the composition of the material occurs once released in the environment. A multitude of studies indicate that toxaphene in the environment weathers and the resulting mixture consists of a much smaller number of congeners than in the original TT. As discussed, the persistent congeners that make up the human body burden are p-26, p-50 and p-62. However, a larger number of congeners occur in fish and other biota. The persistence of the various congeners is determined by their thermodynamic stabilities and molecular structural energies (Heimstad et al., 2001). Microbial degradation of toxaphene shows specificity for the removal of chlorines in particular positions on the molecule (Ruppe et al, 2003, 2004). In addition, biotic and abiotic transformation of toxaphene results in different mixtures of congeners (Angerhöfer et al., 1999).

The results of this report are expected to be used in future risk assessments of fish and other biota from Terry Creek, GA. Hence, it is necessary to show consistency between the WT obtained from Terry Creek samples, other biota samples from around the world and the biotic preparation of WT used to develop toxicity criteria (MATT, 2000).

Table 2 shows the percentage of the three congeners in biota from a variety of sources (Chan and Yeboah, 2000; Skopp et al, 2002a; Chan et al, 1998). The percentage of p-26 ranges from 0.33% to 30%, p-50 ranges from 0.45% to 25%, and p-62 ranges from 0.33% to 13%. Table 3 shows the percentages of the three congeners from fish samples obtained from Terry Creek collected in 1997 and analyzed by Dr. Keith Maruya of Skidaway Institute of Oceanography (Maruya, 2000). The percentage of p-26 ranges from 0.28% to 6%, p-50 ranges from 0.41% to 3.4%, and p-62 ranges from 0.21% to 1.21%.

The lower percentages of the persistent congeners in Terry Creek fish suggest that "weathering" of the original TT has been less extensive than in the the cold water fish reported in the literature (Chan and Yeboah, 2000; Skopp et al, 2002a; Chan et al, 1998).

5.0 THE MATT (INVESTINGATION INTO THE MOITORING, ANALYSIS AND TOXICITY OF TOXAPHENE) STUDY

So far, the MATT study produced by the European Union provides the only toxicity criterion based on weathered toxaphene. In the MATT report, the tumor promoting potency of TT, uv-irradiated TT and WT were examined in both in vivo and in vitro systems (MATT, 2000).

To prepare WT, cod fish were dosed with TT via feed pellets. Liver extracts from the cod were then used in both *in vitro* and *in vivo* experiments. The cod liver extract was analyzed and contained a mixture of toxaphene congeners, including p-26, p-50 and p-62. The cod fish were maintained in an outdoor facility in Bergen and were fed with

fish pellets containing 30 ppm toxaphene during June and July. The cod were fed a standard diet until August 14 when they were sacrificed and the liver extract of WT prepared (MATT, 2000).

There was a great deal of variation in the liver concentrations of toxaphene residues between individual fish (0 to 9 mg WT per liver). The MATT study attributed this variation to feeding competition between the fish. At the conclusion of the feeding period, a total of 1880 mg of toxaphene residue was obtained from the pooled cod livers (MATT, 2000).

A chromatogram provided in the MATT report shows that the WT in the cod liver extract was enriched in p-26, p-32, p-50 and p-62. However, the mixture of toxaphene residues in these cod livers is more complex and contains more individual congeners than those mentioned. The WT in the MATT cod liver extract was weathered for two months whereas WT from Terry Creek fish and biota from other sources has weathered for years. Hence, one would expect that the percentages of the persistent congeners would be lower in the samples that had been weathered for less time (Table 4).

The choice of dose in the MATT report was based on the total toxaphene amount in cod liver extract. There was only brief discussion of this choice in the MATT report, and implicit in this choice is that the non-persistent congeners are influential in producing any adverse effects of toxaphene whereas the human body burden consists of persistent congeners.

However, based on the rapid "weathering" of toxaphene in biological systems (Andrews et al., 1996; Pollock and Hillstrand, 1982; Pollock and Kilgore, 1980; Biessman et al., 1983; Mohammed et al., 1983), it seems much more likely that toxic effects are produced by the persistent congeners. Hence, in contrast to the choice of the MATT (2000), the risk assessment presented here is based on p-26, p-50, p-62 or their summed concentrations (Σ3PC).

5.1 In vivo Study of Tumor Promotion

Female Sprague-Dawley rats in groups of 3 or 4 animals were given partial hepatectomies at the age of six weeks. 30 mg/kg diethyl nitrosamine was given by intraperitoneal injection 24 hours after hepatectomy. Beginning 5 weeks later and lasting for 20 weeks, the rats were then dosed weekly via subcutaneous injection with either technical toxaphene (TT), uv-treated toxaphene (UVT), cod liver extract (CLE) or dioxin in a corn oil vehicle. The MATT (2000) did not discuss the decision to use UVT, but presumably, treatment with UV is another means of "weathering" toxaphene. A range of doses of TT, UVT and CLE were given. A single dose of dioxin was given as a positive control. One week after the last dose, the rats were sacrificed and their livers obtained (MATT, 2000; Besselink H et al., unpublished).

To obtain the dose of weathered toxaphene in CLE, the MATT study converted the concentrations in CLE to UVT equivalents. It is not entirely clear how this conversion was done. However, in table 2 of chapter 5 of the MATT report the dosing amounts of p-26, p-50 and p-62 in the CLE are provided.

The endpoint of the *in vivo* assay was the production of hepatic foci positive for the placental form of glutathione-S-transferase (GST-p). The GST-p assay has long been used as a measure of carcinogenic potency (Ito et al., 1989) Only TCDD produced hepatic foci positive for GST-p. The lower three doses of CLE were not significantly different than the corn oil control in terms of altered hepatic foci. The highest dose showed a decrease in hepatic foci, indicating some possible cytotoxic effect that may not be related to tumor promotion.

The liver concentration of the three persistent congeners in the rats' livers were measured at the end of the experiment. Only the highest dose (12.5 mg/kg-week) resulted in detectable liver concentrations with detections of 2 and 3 ug/kg wet weight for p-50 and p-62 respectively. Total toxaphene residues in the rats' liver were not presented.

5.2 In vitro Study of Gap Junctional Intercellular Communication

The *in vitro* study measured intracellular communication between Hep1c1c7 cells in culture by observing the spread of Lucifer yellow dye between adjacent cells. This dye has been used for a number of years to observe disruption of gap junctional communication and electrical junctions between nerve cells. (e.g. Spencer and Satterlie, 1980; McKarns and Doolittle, 1982). 2,3,7,8-TCDD was used as a positive control. A dose-dependent response was observed for CLE in this assay.

The lowest concentration for CLE was 1 mg/ml and an effect was observed – hence, this concentration represents a lowest effective concentration (LOEC). Because the persistent congeners are the likely toxicant(s), the LOEC for Σ3PC can be determined as a percentage based on the information from Table 2 of chapter 5 in the MATT (2000) and repeated here in Table 5. Multiplying the concentration of the CLE mixture by percentage of Σ3PC in the mixture gives the concentration of Σ3PC. The percentage of Σ3PC in the lowest dose CLE was 0.24%. This percentage of a concentration of 1mg/ml is 2.4 μg/ml.

5.3 The MATT Toxicity Criterion

The MATT study derived a toxicity criterion for total WT based on the *in vivo* rat study. A dose of 4.8 mg/kg-week based on TT equivalents was considered a NOAEL. This value is equal to 0.69 mg/kg-day. An uncertainty/safety factor of 100 was applied giving a tolerable daily intake (TDI) of 0.0069 mg/kg-day. There was no discussion of the origin of the safety factor (e.g. human variability, animal-to-human extrapolation, etc.) other than to indicate some disagreement between the Nordic Council of Ministers and the rest of the MATT participants. The Nordic Council suggested a safety factor of 1000, also without discussion of the origin of that value either.

5.4 Comparison of Risks based on the MATT TDI with Risks based on Σ3PC

Later in this report, a risk assessment for consumption of fish from Terry Creek is presented. The HQ calculated was 4 and was based on the RfD for Σ3PC. Using the MATT TDI, the HQ was 0.3. In all instances examined so far, the RfD developed here was more protective than the MATT TDI.

6.0 DERIVATION OF A REFERENCE DOSE FOR TUMOR PROMOTION BASED ON APPEARANCE OF GST-AHF IN RAT LIVER

The MATT value of 0.0069 mg/kg-day may be appropriate for assessing risk from fish in Terry Creek. However, the full details of the derivation were unfortunately not provided in the MATT (2000).

Here, in the interest of transparency and openness, a three-step process for deriving an RfD from the data in the MATT report was followed: (1) choosing a critical effect for WT; (2) deriving of NOAELs for both the persistent congeners and the WT mixture from the in vivo study in the MATT (2000); and (3) choosing and applying appropriate uncertainty factors to arrive at a reference dose.

Please note the RfD derived here is based on the sum of the three persistent congeners, p-26, p-50 and p-62, in the human body burden (Σ3PC). Cleanup level is based on Σ3PC should generally be expressed as WT concentrations based on the percentage of Σ3PC in the WT mixture. For fish in Terry Creek, application

6.1 Critical Effect of Weathered Toxaphene

Based on the early rodent studies, the critical effect for TT was liver cancer. EPA developed a cancer slope factor of 1.1 per mg/kg-day from these data using the linearized multistage model to perform the low-dose extrapolation. Later, Goodman et al. (1999), in a revisionist paper, suggested that the slope factor be lowered to 0.1 per mg/kg-day using benchmark dose modeling.

Here, three possibilities for the critical effect are considered– (1) WT acts as a genotoxic carcinogen; (2) WT acts as a developmental toxicant; and (3) WT acts as a carcinogen via tumor promotion.

6.1.1 Genotoxicity of TT or WT

In prokaryotic systems, TT has been shown to be mutagenic in the Ames test and others. Both TT and WT have been shown to be mutagenic in the Ames test (Young and Freeman, 2001, 2004), but the mutagenicity of WT appears less than that of TT. Mutagenicity of TT could not be shown in the mouse dominant lethal assay (NTP, 1979). Steinberg et al. (1998) could not demonstrate mutagenicity of WT. Bartos et al. (2005) showed both TT and WT to be genotoxic in bacterial systems. Activation of TT by human microsomal preparations produced a negative result in the Ames test with salmonella TA 98 and TA 100, which contain the pKm101 plasmid. Activation of TT by rat S9 liver fraction produced a positive Ames test in these strains (Hooper et al., 1979; Mortelsman et al., 1986).

In eukaryotic systems, the picture is less clear. In studies of sister chromatid exchange in Chinese hamster lung cells and human lymphocyte cultures, TT produced a weak response which was reduced by metabolic activation (Sobti et al, 1983; Steinel et al, 1990). Toxaphene was clearly not a mutagen by mouse dominant lethal assay (Epstein et al., 1972). Boon et al. (1998) observed genotoxicity for technical toxaphene

as well as the four toxaphene congeners in the Mutatox assay. Addition of rat S9 fraction or microsomes of harbour seal and albatross, decreased the genotoxic potential of the tested congeners and toxaphene. This suggests that metabolism of toxaphene produces potentially less toxic forms of the chemical and, regarding mutagenicity. that organisms with a low capacity to metabolize toxaphene might be more at risk than organisms with a high ability to metabolize toxaphene.

In humans, EPA did not observe leukocyte chromosomal aberrations in agricultural workers using toxaphene (USEPA, 1978). However, an accidental exposure of 8 women to airborne toxaphene resulted in an increase in chromosomal aberrations (Samosh, 1974).

There is certainly no clear and convincing evidence that TT or WT are genotoxic in humans and the critical effect is not cancer via a genotoxic mechanism.

6.1.2 Developmental Effects of Toxaphene

Calciu et al. (1997) investigated the effects of TT, p-26, p-50 and an equimolar mixture of p-26/p-50 on the development of cultured rat embryos. All treatments caused growth retardation of the embryos and the p-26/p50 mixture had the greatest effects, significantly greater than TT. Curiously, the mixture did not appear as toxic as the single congeners on the otic system. The results suggest that the congeners that are persistent in humans can have specific embryotoxic effects not associated with TT. These authors suggest that levels of toxaphene in umbilical cord blood estimated from measured levels in breast milk of Inuit women (Stern et al., 1992) are 1/1000 of the lowest concentrations used in the experiment and that it was unlikely that effects would be observed in humans. Calciu et al. (2002) discovered an interaction between TT, p-26 and p-50 on one hand and hyperglycemia on the other hand upon the development of cultured rat embryos. Again, the levels of toxaphene or individual congeners used appear to be 1000 times the levels of toxaphene predicted in cord blood based on plasma levels in Inuit women (Bjerregaard et al., 2001).

Because TT and the two congeners were added directly to the culture medium containing the rat embryos, it is difficult to determine an estimate of the human dose that would be associated with developmental effects. Nonetheless, a LOEC of 100 ng/ml or 100 µg/L can be established for TT, p-26 and p-50 from this study.

6.1.3 Toxaphene as a Tumor Promoter

Inhibition of gap junctional intercellular communication (GJIC) has been postulated to release initiated cells from suppressing effects of signals passing from surrounding cells (Yamasaki, 1990; Kao et al., 1995). Disruption of gap junctional communication can be measured by intercellular transfer of the dye Lucifer yellow following application of the tumor promoter 12-O-tetradecanoylphorbol-13-acetate (TPA) (McKarns and Doolittle, 1982). A study by Kang et al. (1996) showed that noncytotoxic concentrations of TT (0-10 mg/ml) inhibited GJIC in normal human breast epithelial cells in a dose-dependent way after 90 min of exposure.

The MATT study was based on WT acting as a tumor promotor and this assumption provided the conceptual basis for the in vivo and in vitro studies discussed

above. The cod liver extract used in the MATT study clearly disrupted gap junctions in the Hepa1c1c1 cells at all concentrations tested. This effect is similar to TPA, which is a known tumor promoter.

The tumor promotion endpoint was chosen as the critical effect for WT.

6.2 Determination of a NOAEL for Tumor Promotion from the MATT study

The MATT study was not completely clear about the dose calculations of weathered toxaphene obtained from the cod liver extract. A portion of Table 2 of chapter 5 of the MATT report is reproduced as Table 5 in this report. The doses of toxaphene in CLE as UVT equivalents and as Σ3PC were recalculated based on the rats' body weights and weight gains given in Table 4 of chapter 5 of the MATT report. These doses are also shown in Table 5 of this report.

A NOAEL was determined for Σ3PC. Σ3PC appears to be the most relevant indicator of exposure and hence toxicity for humans. As already indicated, Σ3PC is believed to be the ultimate toxicant(s) because of the rapid "weathering" of toxaphene in biological systems (Andrews et al., 1996; Pollock and Hillstrand, 1982; Pollock and Kilgore, 1980; Biessman et al., 1983; Mohammed et al., 1983).

6.2.1 Deriving a NOAEL based on the Sum of 3 persistent congeners

One can assuming that the concentration in the rats' livers of Σ3PC was 10 ug/kg wet weight or less at the highest dose in the *in vivo* study (Table 3 in chapter 5 of MATT (2000)). In the *in vitro* study, the lowest concentration of CLE used was 1 mg/ml (Besselink et al., unpublished). The percentage of Σ3PC in CLE at this lowest dose level was 0.24%. Hence, the liver concentration of Σ3PC would be 0.24% of the *in vitro* LOEC of 1 mg/ml of CLE. The liver concentration of Σ3Pc would be 2.4 μg/ml and would be considered a NOEC. Expressing this value in the same units as Σ3PC in the rats' livers, it becomes 2400 ug/L. Assuming that 1 kg of tissue occupies 1 L of volume, then this LOEC is 240 times larger than the NOEC of of 10 ug/kg in the rats' livers.

At the highest in vivo dose of 12.5 mg/kg-week CLE, there was a reduction in the number of altered hepatic foci in the rats' livers compared with the controls. This effect was thought to be due to cytotoxicity and the highest dose group result was not considered usable. No differences were observed in the number or size of altered hepatic foci at the three lower doses. Hence, the highest dose of these three (4.17 mg/kg-week CLE as UVT equivalents) can be considered a NOAEL.

The NOAEL for Σ3PC based on the percentages in the CLE mixture (Table 5) is 0.002 mg/kg-day. This value corresponds to 4.17 mg CLE per ml of corn oil

6.3 Application of Uncertainty Factors applied to obtain an RfD for Tumor Promotion

The usual uncertainty factors of 10 for animal-to-human extrapolation and 10 for human variability result in a combined UF of 100. To support this value, it is necessary to consider possible developmental effects and database insufficiencies.

Other endpoints were considered, although in less detail than those discussed above. The difficulty with these other endpoints is that the studies used TT and hence are not as relevant to WT. Tryphonas et al. (2000) observed immunologic effects in Cynomologous monkeys dosed with TT. A NOAEL for these effects is 0.1 mg/kg-day. Olson et al. (1980) observed neurodevelopmental effects in dogs for TT with a LOAEL of 0.5 mg/kg-day. Chu et al. (1986) observed liver effects in rats with a NOAEL of 0.35 mg/kg-day and thyroid effects with a NOAEL of 0.18 mg/kg-day. All of these point of departure values are similar to the NOAEL of 0.6 mg/kg-day for the CLE mixture. Although these other endpoints were studied using TT, because of the rapid "weathering" of TT in biological systemsas previously discussed (Andrews et al., 1996; Pollock and Hillstrand, 1982; Pollock and Kilgore, 1980; Biessman et al., 1983; Mohammed et al., 1983), these studies are appropriate for contributing to the breadth of endpoints considered for Σ3PC and an additional uncertainty factor for database insufficiency is not needed.

One can compare the concentration of TT, p-26 and p-50 used in the rat embryo study to estimates toxaphene intakes that would correspond to the LOAEL observed in that study and determine a margin of exposure (MOE). On the basis of this MOE value, the need an additional uncertainty factor for developmental effects can be determined.

The Inuit, a circumpolar aboriginal population, have high intakes of WT because of their dependence on fish and marine mammals as a food source. Estimates of daily WT intakes from fish and biota among the Inuit (Chan et al., 1997) can be compared to corresponding plasma levels (Bjerregard et al., 2001). These data can be used to estimate a relationship between daily intake and plasma levels. Assuming that maternal cord blood levels (i.e. fetal exposure concentrations) are the same as plasma levels, it can be shown the margin of exposure between the LOAEL for developmental effects in rat embryos and daily intakes in the Inuit of greater than 600 fold for WT. Assuming a combined uncertainty factor of 1000 and applying this to the NOAEL of 0.6 mg/kg-day for WT, the resulting RfD or TDI would be 0.6 ug/kg-day. This is very similar to the estimated intake of 0.26 ug/kg-day (Chan et al., 1997).

Table 6 shows these values and provides a calculation of the margin of exposure for frank effects based on the rat embryo data with p-26 and p-50 as the toxicants (Calciu et al, 1997). The MOEs for both p-26 and p-50 are both greater than 5000.

Using a UF of 1000, the RfD or TDI values for p-26 and p-50 are 5E-4 and 8E-4 ug/kg-day respectively, which are less than the estimated Inuit intake of 0.005 ug/kg-day for each congener (Table 6).

There is no evidence that the Inuit experience greater developmental health effects due to toxaphene exposure than do developed societies (Landrigan et al. 2002; Faustman et al., 2000). Birth defects among the Inuit have been attributed to other causes such as nutrition and alcoholism and increasing "Westernization" of their culture (Arbour et al., 2004; Macaulay et al., 2003).

With regard to cancer, there is also currently no definitive evidence that the Circumpolar Inuit experience greater cancer risk due to toxaphene exposure than developed societies. The Inuit do present a different picture regarding the relative

frequencies of various types of cancers within the population but the etiology remains unclear (Neilsen et al. 1996: Gaudette et al., 1993).

With regard to breast cancer specifically, TT, p-26 and p-50 all enhance proliferation of MCF7-E3 human breast cancer estrogen-sensitive cells (Stelzer and Chan, 1999). In this same cell line, TT, p-26 and p-60 have weak anti-estrogenic properties and can potentially disturb the intracellular signaling mediated by the estrogen receptor (Bonefield et al., 1997; Arcaro et al., 2000) and a link has been suggested between toxaphene and breast cancer. However, the Inuit have been shown to have very low breast cancer rates, about 1/10 of the Canadian average rate (Miller and Gaudette, 1996; Gaudette et al, 1996).

For the reasons of the high MOE and the lack of concordance of laboratory and population effects of toxaphene, an additional UF for developmental effects will not be included and the combined UF for Σ3PC is 100.

Reference Dose (mg/kg-day) for Σ3PC
2E-05

One can determine the corresponding RfD for the WT mixture assuming that the CLE contained 0.3 percent ΣPC. This value is 0.0067 mg/kg-day which is very close to the value of 0.0069 mg/kg-day derived in the MATT (2000).

6.4 Application and development of PRGs for WT using the RfD for ΣPC

In applying the RfD for Σ3PC to cleanup of WT, it is necessary to determine the percentage of Σ3PC in the WT mixture. This practice will ensure that cleanup levels will be expressed as WT and will be consistent with past practices. If, in the future, analytical methods for WT become standardized, there will need to be consideration that p-25, p-50 and p-62 be part of the analysis.

The Region 9 Preliminary Remediation Goal methodology and exposure assumptions were used to develop PRGs for WT based on the toxicity of Σ3PC. For air, the percentage of Σ3PC in WT was estimated from Bidleman et al. (2004) at 8.8%. For soil, the percentage of Σ3PC in WT was assumed to be 10%; for water, 5%.

Preliminary Remediation Goals for Weathered Toxaphene	
Residential Soil	12 mg/kg
Industrial Soil	120 mg/kg
Tap Water	0.7 µg/L
Ambient Air	0.8 µg/m^3

Please note that these PRG values represent total WT but are based on Σ3PC at specific percentages. Presently, it is not appropriate to use these WT PRGs as cleanup levels without site-specific determination of the percentage of Σ3PC in WT.

6.5 Fish Advisories

Toxicity values are also used to determine fish advisories and it might be appropriate to revise fish advisory levels for toxaphene based on these toxicity criteria for WT.

7.0 COMPARISON OF TOXAPHENE TOXICITY CRITERIA

A number of regulatory agencies around the world have developed toxicity criteria for toxaphene. Table 7 shows the various toxicity criteria. These values are shown in their original units, either tolerable daily intakes (TDIs) in units of mg/kg-day or slope factor in units of $(mg/kg\text{-}day)^{-1}$. Converting between these two units is not as simple as merely taking the reciprocal. In fact, MATT (2000) incorrectly applied the EPA slope factor as a TDI for determining daily intake. EPA considers cancer risks as unitless probabilities and it is not known what cancer probability value provided the basis for the TDI values.

7.1 Representative Fish Concentrations for Comparing Toxicity Criteria

Both slope factors and TDIs were expressed in terms of representative fish concentrations. The characteristic lifetime receptor for carcinogenic effects was defined as an individual exposed from age 0 to 30. Age-specific body weights and fish consumption rates were obtained from table 11-6 in EPA's Child-Specific Exposure Factors Handbook and table 10-61 in EPA's Exposure Factors Handbook respectively. For each year from 1 to 30, the fish consumption rate in g/day was divided by the body weight. These resulting values were averaged over all 30 years to obtain the Lifetime Receptor fish consumption rate (FCR) of 0.194 g/kg-day. This FCR was used in a risk equation as follows:

$$Risk = CSF \frac{C\ FCR\ CF\ ED\ EF}{AT} \qquad (7\text{-}1)$$

where C = Concentration in mg/kg
CSF = Cancer Slope Factor in $(mg/kg\text{-}day)^{-1}$
FCR = Fish Consumption Rate in g/kg-day = 0.194 g/kg-day
CF = Conversion Factor in kg/g = 0.001 kg/g
ED = Exposure Duration in years = 30 yr
EF = Exposure Frequency in days/year = 365 day/yr
AT = Averaging Time in days = 25550 days

Equation 1 was solved interatively by changing concentration until the risk was equal to 10^{-5}. This value is the middle of the risk range given in the NCP and was chosen in lieu of the point of departure because the TDI values were based on an unknown risk value.

To obtain intake estimates for comparison with TDI or RfD values, equation 2 (below) was also solved iteratively for concentration until the daily intake equaled the TDI or RfD. However, in eq. 2, the fish consumption rate used was that for children – 0.369 g/kg-day and the ED was 6 years.

$$Daily\ Intake = \frac{C\ FCR\ CF\ ED\ EF}{AT} \qquad (7\text{-}2)$$

The values and units for eq. 2 are the same as those for eq. 1. Table 7 shows the various fish concentrations calculated using various regulatory values.

The results of this analysis are somewhat surprising. The MATT study has the reputation of being highly protective. The uncertainty with this analysis is the lack of knowledge regarding the calculation of the MATT value of 0.41 mg/day/ 60 kg. Assuming both a 70 year and a 30 year exposure duration yielded fish concentrations considerably above those determined by any of the array of cancer slope factors.

Another key point of the table above is that the only regulatory value based on weathered toxaphene is that based on the European MATT study. Although the MATT document did not report concentrations of p-26 or others known to be the major players in humans (see above) that were present in the cod liver extract, these can be deduced from a large number of studies of toxaphene occurrence in fish.

For this reason, the MATT criterion of 0.41 mg/day/60 kg BW is the most appropriate to use as an interim value for assessing risk of WT.

If one considers the percentage of Σ3PC in WT as between 5% and 18% (Tables 2 and 4), one can determine a concentration of WT in fish corresponding to the German limit of 0.1 mg/kg based on Σ3PC. This corresponding concentration of WT ranges from 0.6 mg/kg to 2 mg/kg.

8.0 Comparison of Terry Creek Fish Concentrations with Risk-Based Levels for WT

From the MATT report, the percentages of the three congeners in the corn oil used as a dosing vehicle in the in vitro or in vivo studies are compared with the median values from the Terry Creek biota or biota from other sources in Table 4. Note that the percentages of the three congeners are lower in the MATT dosing vehicle than in fish or biota (Table 5). Because the congeners other than those that comprise the human body burden, it will be important to account for this concentration difference in the risk analysis.

Data from Terry Creek fish and shellfish were obtained from Maruya (2000). 95% upper confidence limits on the WT and 3PC concentrations in the fish were calculated with PROUCL software (USEPA, 2004). Finfish and shellfish concentrations in ng/g wet weight were both included in the calculation. WT concentrations were consistent with a lognormal distribution. Because several nondetects were present, the bootstrap-t method was used for the UCL. The UCL value for WT was 5348 ug/kg. 3PC concentrations were consistent with a gamma distribution and the adjusted gamma method was used for the UCL. The UCL value for ΣPC was 228 ug/kg.

Fish ingestion rates for both children and adults were obtained from the Exposure Factors Handbook (USEPA, 2000). Table 10-61 provides mean fish ingestion rates for a

number of age groups in g/kg-day. The highest value of 0.369 g/kg-day for consumption of recreationally caught fish is representative of children ages 1-5 and was used here. Please note that this value will also be protective of adults. In general, site-specific data on fish consumption is preferable to default values such as these. Hence, it may be advisable to obtain site-specific fish consumption data for Terry Creek in the future. In this report, the risk estimates for consumption of fish in Terry Creek are based on these default consumption rates.

$$HQ = \frac{C_{fish} \cdot IR_{fish}}{RfD} \qquad (8\text{-}1)$$

An hazard quotient value of 4 for Σ3PC were calculated with eq. 8.1.

To obtain a cleanup level based on Σ3PC, the UCL for Σ3PC is divided by the HQ resulting in a value of 55 ug/kg. The corresponding cleanup level for WT can be obtained by dividing the cleanup level for Σ3PC by the percentage of 3 Σ3PC in Terry Creek fish (Table 4), resulting in a cleanup level of 1300 ug/kg for WT.

9.0 SUGGESTIONS FOR ADDITIONAL RESEARCH

There are three areas of possibly productive research regarding the toxicity of WT.

9.1 In Vitro Testing of Tumor Promotion

Repetition of a gap junction intercellular communication assay may shed some light on this. The lowest dose of CLE used in MATT (2000) represented a LOEC in this *in vitro* assay. Even lower doses should be used in any future assay in order to determine a NOEC and thus provides bounds on the threshold dose. Additionally, incubation of WT with microsomal fractions prior to use in the assay would remove by metabolism more of the non-persistent congeners. It would be intersting to know whether this would enhance or reduce toxicity.

9.2 In Vivo Testing of Tumor Promotion

The *in vivo* rat assay for altered hepatic foci should be repeated. The issue with the test as performed by MATT (2000) is that the doses of CLE did not produce liver concentrations that caused an increase in AHP over control. One of the problems is that CLE was administered by subcutaneous injection in corn oil. Gavage would have been a better method since material in the gut travels first to the liver via portal flow. A physiologically-based pharmacokinetic model for toxaphene in the rat has been developed and this model could be used to predict and optimize the internal liver dose to attempt to obtain a threshold dose and a dose-response curve (Wen and Chan, 2000).

9.3 Whole Animal Developmental Studies

Although effects were observed in cultured rat embryos, developmental tests with whole animals are needed to determine a dose response for any possible developmental effects. Administration of WT to pregnant females would provide this information.

9.4 Critical Periods/Early or Late Life Exposure

Some chemicals such as vinyl chloride act during early life to produce cancer or other harmful effects later in life. It may be possible to develop studies that test animals during specific phases of their lives and look for adverse effects showing up later.

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TABLES

Table 1. Toxaphene Congeners and Their Average Percentages in the Human Body Burden (from Gill et al., 1996; Polder et al., 2003; Skopp et al., 2002b)

Congener	*p-26*	*p-40/41*	*p-44*	*p-50*	*p-62*
Average Percent in human body burden	32.6%	3.1%	4.1%	55.2%	5%

Table 2. Percentage of Three Congeners in Various Biotic Samples (based on ng/g wet weight)

Source	*% -p26*	*%p-50*	*%p-62*	*%3PC*	*Reference*
King Salmon	4.12%	5.76%	9.41%	19.29%	Chan & Yeboah, 2000
Dogfish	4.09%	5.16%	9.14%	18.39%	Chan & Yeboah, 2000
King Salmon	4.11%	6.56%	7.67%	18.33%	Chan & Yeboah, 2000
King Salmon	3.70%	6.30%	7.26%	17.26%	Chan & Yeboah, 2000
King Salmon	4.59%	6.89%	10.00%	21.49%	Chan & Yeboah, 2000
Whitefish	3.50%	6.50%	9.75%	19.75%	Chan & Yeboah, 2000
King Salmon	4.00%	5.00%	7.67%	16.67%	Chan & Yeboah, 2000
King Salmon	3.66%	5.00%	7.93%	16.59%	Chan & Yeboah, 2000
Trout	3.96%	3.96%	6.04%	13.96%	Chan & Yeboah, 2000
King Salmon	3.08%	5.94%	6.78%	15.80%	Chan & Yeboah, 2000
King Salmon	5.00%	7.42%	7.26%	19.68%	Chan & Yeboah, 2000
Halibut	7.86%	9.29%	6.19%	23.33%	Chan & Yeboah, 2000
Ooligan	2.26%	2.71%	4.71%	9.68%	Chan & Yeboah, 2000
Chum Salmon	2.84%	4.33%	6.72%	13.88%	Chan & Yeboah, 2000
Trout	5.02%	6.39%	13.24%	24.66%	Chan & Yeboah, 2000
Trout	4.12%	7.41%	10.70%	22.22%	Chan & Yeboah, 2000
Trout	4.42%	7.28%	13.11%	24.81%	Chan & Yeboah, 2000
Sockeye	2.29%	1.98%	3.75%	8.02%	Chan & Yeboah, 2000
Lake Trout	6.36%	5.45%	11.82%	23.64%	Chan & Yeboah, 2000
SRM 1588 cod liver oil	6.38%	9.83%	4.84%	21.06%	Chan & Yeboah, 2000
burbot	2.79%	4.12%	1.76%	8.68%	Skopp et al, 2002
burbot	6.88%	5.74%	2.10%	14.72%	Skopp et al, 2002
muskox	0.33%	0.45%			Skopp et al, 2002
beluga	14.01%	25.07%	0.80%	39.88%	Skopp et al, 2002
narwhal	16.41%	20.00%	2.24%	38.65%	Skopp et al, 2002
walrus	12.03%	6.86%	0.33%	19.22%	Skopp et al, 2002
Whitefish	13.48%	23.60%		40.45%	Chan et al, 1998
Lake Trout	10.37%	23.70%		40.37%	Chan et al, 1998
Narwhal	29.99%	25.40%		58.05%	Chan et al, 1998
Minimum	0.33%	0.45%	0.33%	8.02%	
Maximum	29.99%	25.40%	13.24%	58.05%	
Median	4.12%	6.39%	7.26%	19.49%	
Average	6.61%	8.76%	6.85%	22.45%	

Table 3. Percentages of the Three Congeners in Fish Samples from Terry Creek, GA

Source	*% P-26*	*% P-50*	*% P-62*	*% 3PC*	*Source*	*% P-26*	*% P-50*	*% P-62*	*% 3PC*
Black Drum	2.71%			2.71%	Spot	3.63%	1.24%	0.39%	5.26%
Blue Crab	6.09%	1.50%	0.33%	7.92%	Spot	4.08%	1.39%	0.29%	5.76%
Blue Crab	4.72%	2.37%	1.09%	8.19%	Spot	3.72%	1.15%	0.35%	5.22%
Blue Crab	5.39%	2.86%		8.24%	Spot	3.23%	1.02%		4.25%
Blue Crab	4.39%			4.39%	Spot	3.51%	0.76%	0.36%	4.63%
Blue Crab	0.89%	0.66%		1.56%	Spot	2.64%	0.52%		3.15%
Blue Crab	2.10%	2.15%		4.25%	Spot	2.79%	0.45%	0.21%	3.46%
Blue Crab	4.45%	2.29%	0.65%	7.39%	Spot	3.20%	0.65%		3.85%
Blue Crab	4.52%	1.20%		5.72%	Spot	2.05%			2.05%
Croaker	2.39%	0.59%		2.98%	Yellowtail	3.20%	1.01%		4.21%
Croaker	2.32%	0.63%		2.95%	Yellowtail	3.12%	0.91%	0.35%	4.38%
Croaker	2.24%	0.41%		2.64%	Yellowtail	2.98%	1.12%	0.44%	4.53%
Croaker	2.47%	0.62%		3.10%	Flounder	3.54%		0.71%	4.26%
Mullet	4.19%	1.37%	0.73%	6.29%	Whiting	3.45%			3.45%
Mullet	1.94%	1.91%	0.74%	4.59%					
Mullet	3.62%	1.47%	0.47%	5.55%					
Mullet	2.59%	0.92%		3.51%					
Mullet	3.92%	1.23%	0.67%	5.82%					
Mullet	0.28%	1.43%	0.49%	2.21%					
Mullet	3.13%	1.50%	0.61%	5.24%					
Mullet	4.42%	0.69%		5.11%					
Mullet	4.03%	1.57%		5.60%					
Red Drum	3.37%	1.29%	0.64%	5.29%					
Red Drum	2.78%	0.88%	0.46%	4.12%					
Red Drum	2.85%			2.85%					
Sea Trout	3.92%	1.40%	0.54%	5.86%					
Sea Trout	0.92%	1.05%	0.70%	2.67%					
Sea Trout	3.19%	0.92%	0.53%	4.63%					
Sea Trout	2.54%	0.50%	0.24%	3.28%					
Sea Trout	2.09%	0.57%	0.25%	2.91%					
Sea Trout	3.19%	0.70%	0.42%	4.31%					
Sea Trout	3.65%	1.04%		4.68%					
Shrimp	4.61%	2.84%	0.66%	8.11%					
Shrimp	4.42%	2.70%	0.67%	7.79%					
Shrimp	2.73%	1.57%	0.55%	4.85%					
Shrimp	4.73%	2.74%	0.51%	7.98%					
Shrimp	4.06%	3.43%	1.21%	8.69%					
Shrimp	5.82%	1.82%		7.63%					

Table 4. Median Values of the Percentages of Three Congeners in Fish Samples and in the MATT dosing vehicle.

	p-26	p-50	p-62	Σ3PC
Other Sources (Table 2)	4.12%	6.39%	7.26%	17.77%
Terry Creek (Table 3)	3.22%	1.18%	0.52%	4.92%
MATT dosing vehicle	0.08%	0.12%	0.13%	0.33%

Table 5. Percentage of p-26, p-50 and p-62 in the Dosing Vehicle of the MATT Study

Concentration UV-T equivalents (mg/ml oil)	*0.46*	*1.39*	*4.17*	*12.5*
Concentration (µg/ml oil)				
P26	0.3	1.2	3.4	9.9
P50	0.4	2	5.5	16.8
P62	0.4	2.1	6	17.5
Σ3PC	1.1	5.3	14.9	44.1
Percentages of Three Congeners in CLE				
% p-26	0.065%	0.086%	0.082%	0.079%
% p-50	0.087%	0.144%	0.132%	0.134%
% p-62	0.087%	0.151%	0.144%	0.140%
Σ3PC	0.239%	0.381%	0.357%	0.354%
Doses (mg/kg-day) used in the MATT Study				
CLE as UVT	0.066	0.198	0.60	1.79
p-26	4.28E-05	0.00017	0.00049	0.0014
p-50	5.71E-05	0.00029	0.00079	0.0024
p-62	5.71E-05	0.00030	0.00086	0.0025
Σ3PC	1.57E-04	0.00076	**0.0021**	.0.0063

Please note the value of 0.0021 used as the NOAEL for deriving the Σ3PC RfD is shaded and shown in bold type

Table 6. Margin of Exposure for Developmental Effects among the Inuit based on Developmental Effects in Cultured Rat Embryos

Chemical	*Intake ug/kg-day*	*Plasma Conc. ug/L*	*Ratio Intake/Plasma Conc.*	*Effect Level ug/L*	*Margin of Exposure*
WT	0.26	1.55	6	100	600
p-26	*0.0052	0.66	53	100	6350
p-50	*0.0052	0.81	61	100	5200

*Intakes for p-26 and p-50 were estimated based on 4% p-26 and 6% p-50 (Table 4).

Table 7. Comparison of Toxaphene Toxicity Criteria in terms of Representative Fish Concentrations

Source	*Value*	*Risk or HQ Level*	*Representative Fish Concentration*
Slope Factor Values			
EPA (1991)	1.1 per mg/kg-day	1E-5	0.11 mg/kg
CAL-EPA (2003)	1.2 per mg/kg-day	1E-5	0.10 mg/kg
Buranatrevedh (2004)	0.86 per mg/kg-day	1E-5	0.14 mg/kg
Goodman et al. (2000)	0.1 per mg/kg-day	1E-5	1.3 mg/kg
Tolerable Daily Intake Values			
Health Canada	12 µg/day/ 60 kg BW or 0.0002 mg/kg-day	1	0.5 mg/kg (child)
MATT	0.41 mg/day/ 60 kg BW or 0.007 mg/kg-day	NA	19 mg/kg (child)
RfD (this report) Based on the MATT (2000) data	2E-05 mg/kg-day based on Σ_{3PC} (assuming 5% Σ_{3PC} in the WT mixture)		1.2 mg/kg (child)
Tissue Concentration Values			
Germany - Maximum Residue Level	Based on Σ_{PC}		0.1 mg/kg

DIFFERENCES BETWEEN CANCER AND NON-CANCER RISK ASSESSMENT USING TOXAPHENE AS AN EXAMPLE

The purpose of this informal paper is to explain the differences in cancer and non-cancer risk assessment and to point out a basic error that was made in the interpretation of EPA's cancer risk assessment methodology regarding toxaphene.

How Cancer Risk Assessment Works

The basic concept of a dose threshold separates the cancer and non-cancer endpoints in EPA risk assessments. The presumption of a threshold for the cancer endpoint is considered to be inappropriate and any dose, no matter how low, produces an increased probability of cancer. In fact, the cancer toxicity criteria or cancer slope factor for many chemicals regulated by EPA and presented in the IRIS database is a numerical value that relates probability of cancer to dose.

If one assumes that a dose-response relationship for cancer passes through the origin (i.e. zero dose and zero cancer probability), then, in deriving a cancer slope factor, an extrapolation must be made from doses at which cancer can be observed down to extremely low probability (1E-06) and a correspondingly low dose. Note that the relationship of probability and dose is the slope of the dose-reponse curve and this relationship provides the reason for the term "slope factor."

The exposure assessment for cancer determines a lifetime average daily intake in units of mg of chemical per kg of body weight per day and 25550, the number of days in a 70 year lifespan occurs in the denominator. The cancer slope factor is in units of unitless cancer probability per dose expressed as per (mg/kg-day) or as $(mg/kg\text{-}day)^{-1}$.

Let us now consider an numerical example with fish consumption of toxaphene contaminated fish. The EPA slope factor for technical toxaphene will be the toxicity criterion. The risk equation is:

$$Risk = CSF \frac{C \;\; IR \;\; EF \;\; ED}{BW \;\; AT}$$

$$= 1.1 \frac{1}{mg/kg-day} \frac{1 \frac{mg}{kg\ fish} \; 0.14 \frac{kg\ fish}{meal} \; 36 \frac{meals}{yr} \; 30\,yr}{70\,kg\ BW \;\; 25550\,days}$$

$$= 9E-05$$

CSF	= Cancer Slope Factor	= 1.1 per mg/kg-day
C	= Fish Concentration	= 1 mg/kg fish
IR	= Fish Ingestion Rate	= 0.14 kg fish/meal
EF	= Exposure Frequency	= 36 meals/yr
ED	= Exposure Duration	= 30 years
BW	= Body Weight	= 70 kg BW
AT	= Averaging Time	= 25550 days (70 yr)

Hence, in this example with technical toxaphene, the risk is 9E-05. A fish concentration corresponding to a risk of 1E-06 can be determined as 0.011 mg/kg

How Reference Doses (RfD) or Tolerable Daily Intakes (TDI) are Threshold Doses

The concept of an RfD or TDI for non-cancer effects is that of a threshold dose below which adverse effects will not occur. RAGS, Part A defines the chronic RfD as an estimate with uncertainty spanning perhaps an order of magnitude of a daily exposure level for the human population, including sensitive subpopulations, that is likely to be without an appreciable risk of deleterious effects during a lifetime. Hence, the RfD is a highly protective estimate of the human threshold dose for adverse effects. The units of the RfD are in mg/kg-day.

Let's consider a numerical example, with weathered toxaphene this time, using the European TDI value from the MATT report. This value is 0.0069 mg/kg-day. For an RfD or TDI, the toxicity criterion appears in the denominator as follows:

$$Hazard\ Quotient = \frac{1}{TDI}\frac{C\ IR\ EF\ ED}{BW\ AT}$$

$$= \frac{1}{0.0069\frac{mg}{kg\ BW\ day}}\frac{1\frac{mg}{kg\ fish}\ 0.14\frac{kg\ fish}{meal}\ 36\frac{meals}{yr}\ 30\,yr}{70\,kg\ BW\ 10950\,days}$$

$$= 0.03$$

TDI	= Tolerable Daily Intake	= 0.0069 mg/kg-day
C	= Fish Concentration	= 1 mg/kg
IR	= Fish Ingestion Rate	= 0.14 kg/meal
EF	= Exposure Frequency	= 36 meals/yr
ED	= Exposure Duration	= 30 years
BW	= Body Weight	= 70 kg
AT	= Averaging Time	= 10950 days (30 yr)

Note that the averaging time used for non-cancer effects is the exposure duration expressed in days. This is consistent with long-term EPA practice and RAGS, Part A. Hence, the resulting HQ is 0.03 and a fish concentration corresponding to an HQ of 1 is 35 mg/kg.

How the MATT misinterpreted the Technical Toxaphene CSF

The writers of the MATT assumed the units of the CSF were mg/kg-day rather than per (mg/kg-day). They wrote:

> *For the tumour promotion potency a TDI of 0.41 mg for total toxaphene per day for a person of 60 kg was calculated. This value is much lower than the TDI value, 66 mg, calculated from the EPA reference dose for carcinogenicity.*

The use of the term "reference dose for carcinogenicity" indicates their lack of understanding of how EPA determines carcinogenic risk. Let's see how using the value of 1.1 as a reference dose works out in calculation

$$Hazard\ Quotient = \frac{1}{RfD}\frac{C\ IR\ EF\ ED}{BW\ AT}$$

$$= \frac{1}{1.1\frac{mg}{kg\ BW\ day}}\frac{1\frac{mg}{kg\ fish}\ 0.14\frac{kg\ fish}{meal}\ 36\frac{meals}{yr}\ 30\,yr}{70\,kg\ BW\ 10950\,days}$$

$$= 0.0002$$

RfD	= Reference Dose	= 1.1 mg/kg-day
C	= Fish Concentration	= 1 mg/kg
IR	= Fish Ingestion Rate	= 0.14 kg/meal
EF	= Exposure Frequency	= 36 meals/yr
ED	= Exposure Duration	= 30 years
BW	= Body Weight	= 70 kg
AT	= Averaging Time	= 10950 days (30 yr)

Please note that the misinterpretation of the cancer slope factor for toxaphene results in an estimated HQ that suggests that the MATT TDI value is more protective (conservative) than the EPA slope factor.

The MATT expresses toxicity criterion on the basis of a daily intake of a 60 kg individual. The simple calculation they perform is to multiply the toxicity criterion by 60 kg.

$$Daily\ Intake\ for\ a\ 60\,kg\ person = 0.0069\frac{mg}{kg\ BW\ day}\cdot 60\,kg\ BW = 0.41\frac{mg}{day}$$

$$= 1.1\frac{mg}{kg\ BW\ day}\cdot 60\,kg\ BW \qquad = 66\frac{mg}{day}$$

Hence, the writers of the MATT got it wrong! The MATT report wrongly claimed that their TDI value was 66/0.41 or 160 times more protective than EPA toxicity criterion. In truth, the EPA toxicity criterion is much more stringent (protective or conservative) than the MATT TDI, perhaps up to 3000 times more conservative depending on the choice of risk level within the risk range of 1E-6 to 1E-4.

United States Environmental Protection Agency
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December 17, 2003

4WD-TSS

MEMORANDUM

SUBJECT: Review of Remaining data for the 2002 Annual Groundwater Sampling Results, Inspection Report and Five Year Review

FROM: Kay Wischkaemper, Hydrologist, P. G.
Technical Services Section
Superfund Remedial and Technical Services Branch

TO: Leo Francendese
Remedial Project Manager
Superfund Remedial and Technical Services Branch

I have reviewed the requested historical data for the Five Year Review and conclude that the remedy is protective. In evaluating the hydraulic data the vertical head distribution between shallow, intermediate and deep zones, the wells are sufficient to evaluate migration of any suspected plume. Toxaphene has not been detected in ground water at the site and remains a non-detect following the implementation of the remedy. I remain in my position of continuing the collection of water level data with each sampling exercise. Remaining outstanding specific comments are supplied below:

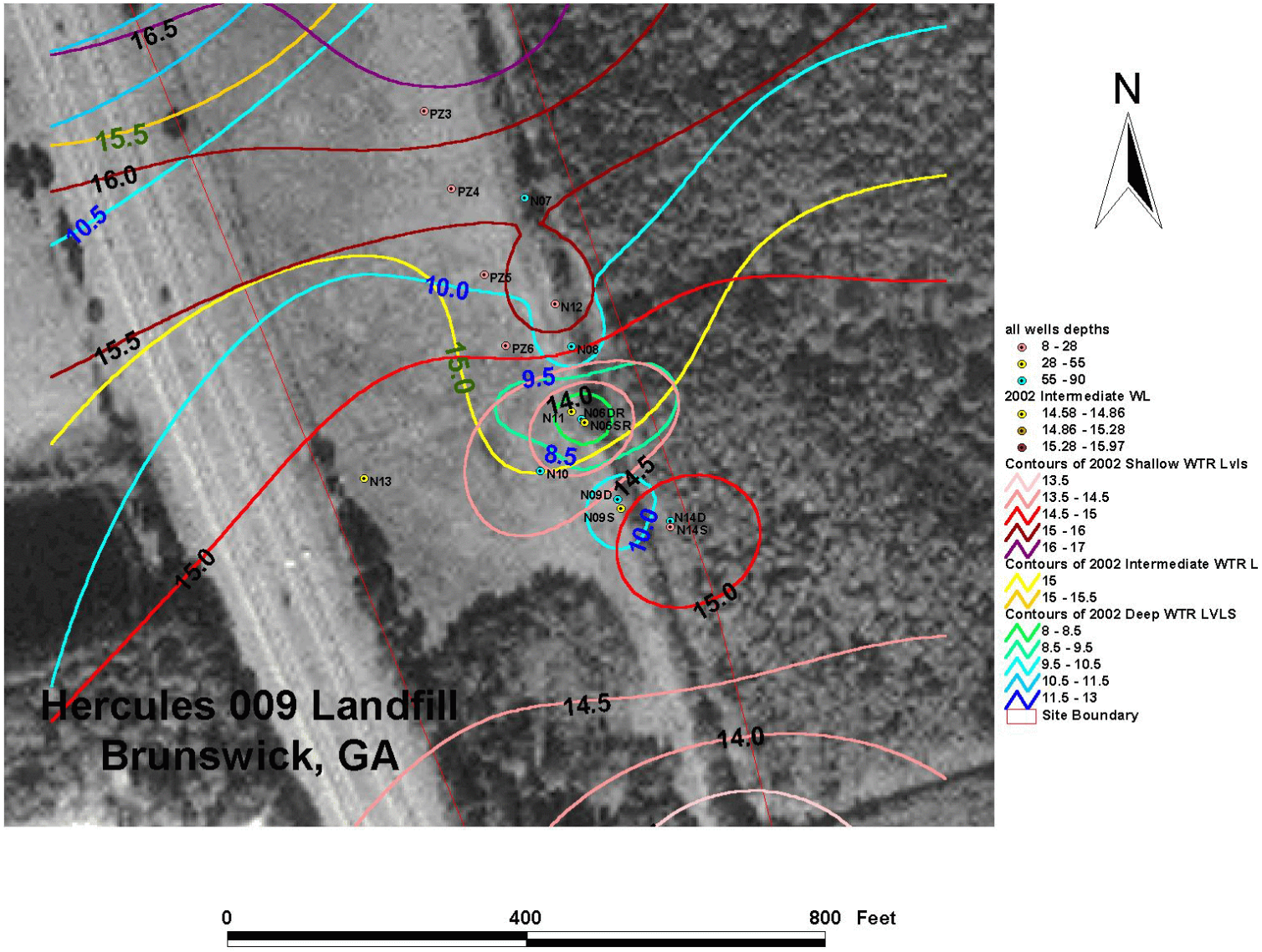
1. Explain how the water level contours were constructed. I drew contours of my own both by hand and using Surfer and could not duplicate RMT's map. A discussion of the head perturbations(see Figure in the next page) in the intermediate and deep zones needs to be discussed in support of the ability of the monitoring systeim to detect contamination if it existed.

2. RMT concludes that the monitoring system is sufficient to detect contamination. In support of that statement the cross-sections on Sheets 1 and 2 should include the monitoring wells in order to depict that clearly.

3. A discussion of the vertical ground water velocity should be included in the groundwater monitoring results section. This information is valuable in substantiating that migration of contamination would be reflected in the monitoring system if it was indeed occurring.

In conclusion, my observation is that the remedy is protective. If you have any questions feel free to contact me at X28641.

cc: Jim McGuire


16.5
15.5
16.0
10.5
10.0
15.5
15.0
9.5
14.0
8.5
14.5
10.0
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PZ3
PZ4
N07
PZ5
N12
PZ6
N08
N11
N06DR
N05SR
N10
N13
N09D
N09S
N14D
N14S
Hercules 009 Landfill
Brunswick, GA
N
all wells depths
8 - 28
28 - 55
55 - 90
2002 Intermediate WL
14.58 - 14.86
14.86 - 15.28
15.28 - 15.97
Contours of 2002 Shallow WTR Lvls
13.5
13.5 - 14.5
14.5 - 15
15 - 16
16 - 17
Contours of 2002 Intermediate WTR L
15
15 - 15.5
Contours of 2002 Deep WTR LVLS
8 - 8.5
8.5 - 9.5
9.5 - 10.5
10.5 - 11.5
11.5 - 13
Site Boundary
0
400
800 Feet

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 4

Science and Ecosystem Support Division
980 College Station Road
Athens, Georgia 30605-2720

June 21, 2005

MEMORANDUM

SUBJECT: SESD Comments on the May 16, 2005, Draft OIG Report on Hercules 009 Landfill Superfund Site, Brunswick, Georgia

FROM: Gary Bennett, Acting Chief
Analytical Support Branch

TO: Leo Francendese, Remedial Project Manager
Waste Management Division

Attached you will find comments on the subject report which were prepared by staff in the Analytical Support Branch. These comments are focused on Chapter 2 of the report and discuss the analytical methodology which has been previously used at the subject site, as well as the OIG's proposed negative ion mass spectroscopy method which was recommended in the report.

Please contact me if you have any questions.

Attachment

cc: Mike Peyton, SESD
Charles Hooper, SESD

OIG Report, page 5*: In the case of Hercules 009 Landfill, the surrounding groundwater is being periodically tested for the original toxaphene mixture put in the site between 1975 and 1980. However, this routine testing does not specifically look for, or definitively identify, individual breakdown products of toxaphene, i.e., the toxaphene breakdown products.*

SESD Comment #1: Over time, the focus of environmental monitoring in and around the Hercules facilities has shifted from determining regulatory compliance by monitoring technical toxaphene to assessing the health effects of toxaphene degradation products. This is best illustrated in the details of two meetings which took place, one in 1991 and another in 1997.

(1) On September 30, 1991, a meeting to discuss monitoring of technical toxaphene in water and soil samples was held between representatives of Hercules, EPA Region 4, Georgia Environmental Protection Division (EPD), and Law Environmental, Inc. The primary objective of the meeting was to agree on a consensus toxaphene method in order to assure data comparability between various laboratories involved in the measurement of the technical toxaphene mixture in environmental samples. According to the minutes of that meeting, one reason for focusing on technical toxaphene was the fact that both the Georgia water quality standards and Hercules' NPDES wastewater discharge permit were based on the technical toxaphene mixture. Therefore, working under the premise that exceeding these allowable limits would pose an unacceptable risk to human health and/or the environment, the regulators were focused on the measurement of technical toxaphene to determine if water quality standards and/or permit limits were being met.

(2) On May 15, 1997, a meeting was held between representatives of the Glynn Environmental Coalition (GEC), EPA Region 4, and Georgia EPD to discuss issues related to monitoring around the Hercules facilities. At this meeting, concerns were voiced by GEC representatives about the potential health effects of toxaphene breakdown products. EPA representatives acknowledged that scientific literature describes the degradation of the individual chemical compounds in the technical toxaphene mixture. However, EPA representatives also pointed out two difficulties in determining the health effects of toxaphene degradation products: (1) analytical calibration standards, which are required to identify and quantify chemical compounds, were not available for most of the individual compounds (congeners) comprising the technical toxaphene mixture, and (2) there was a notable lack of toxicity data for the individual compounds in the toxaphene mixture.

Subsequent to the 1997 meeting, at the request of the EPA remedial project manager, the Region 4 laboratory investigated several different analytical options. The first involved the acquisition and analysis of analytical standards containing the individual congeners of technical toxaphene which had been isolated and purified. At that time, standards were analyzed by Gas Chromatography/Electron Capture Detector (GC/ECD) for 22 individual congeners. The second approach involved the GC/ECD analysis of environmental samples from Hercules and surrounding area using the "total area under the curve" approach for quantifying technical toxaphene. This approach was an attempt to obtain

concentrations for toxaphene which were the worst case scenario, i.e., assuming that all compounds present in the chromatogram from the first to last peak in the technical toxaphene chromatogram were toxaphene congeners and/or breakdown products, and assigning the toxicity of the technical toxaphene mixture to this estimated concentration. This approach was used because of the lack of human toxicity data for the individual toxaphene congeners. These efforts demonstrate that between 1997 and 2004, regional personnel, using instrumentation and resources which were available at the time, continued to investigate different analytical options for measuring toxaphene and its breakdown products.

OIG Report, page 6*: EPA's method (Method 8081) is a test procedure designed to look for the original, unaltered toxaphene mixture. However, microbes in the soil are known to decompose the original toxaphene mixture into just two major breakdown products (i.e., Hx-Sed and Hp-Sed) and several minor breakdown products. The original toxaphene mixture and the two principle toxaphene breakdown products look completely different to the analytical instruments.*

SESD Comment #2: This section discusses EPA Method 8081, the analytical method which was used to measure the technical toxaphene mixture. Method 8081 is a GC/ECD method which can be used to analyze for the technical toxaphene mixture. However we disagree with the implication that this method is only useful for the analysis of technical toxaphene. Method 8081 could be used for measuring any of a number of different chlorinated organic compounds, even the toxaphene congeners, and in many cases demonstrates better sensitivity than other analytical methods. The fact that Method 8081 could be used to measure toxaphene congeners is evident in Appendix A of the OIG report in the section entitled "Estimated Retention Time of Toxaphene Degradation Products" on page 24. Method 8081 might be used to analyze the toxaphene congeners if analytical standards were available for all congeners being measured. While the lack of a mass spectral "fingerprint" for positive identification is one of the weaknesses of Method 8081, another problem is the lack of commercially available analytical standards for the toxaphene congeners. The existence of fewer than 30 analytical standards for toxaphene congeners is an analytical problem whether Method 8081 or a negative ion mass spectrometry method is used for analysis.

One might also infer from this section of the report that Hx-Sed and Hp-Sed are the breakdown products which are of primary concern to human health. However, Appendix A, page 17, notes Hx-Sed and Hp-Sed are readily metabolized by the body and the minor breakdown products, including p26, p50, p40, p41, and p44, pose the majority of risk to human health because they are not effectively metabolized. Therefore based on the technical discussion provided in Appendix A, any analysis of toxaphene congeners to assess human health effects should be focused on the measurement of p26, p50, p40, p41, and p44, not on Hx-Sed and Hp-Sed.

OIG Report, page 7: *A new analytical method using Negative Ion Mass Spectroscopy (NIMS, or called new method hereafter) should be used to test for toxaphene breakdown products in the groundwater. Academia and the European Union have successfully used*

the new analytical method for at least 5 years to test for toxaphene breakdown products in the environment.

The new technique provides definitive test results because the technique generates a mass spectrum for each compound in an environmental sample. A mass spectrum is analogous to a chemical "fingerprint." By comparing the "fingerprint" of an unknown compound in the Hercules 009 groundwater sample with the known "fingerprint" of the toxaphene breakdown products, a resulting match of the "fingerprints" would definitively identify the presence of toxaphene breakdown products.

SESD Comment #3: We agree that the NIMS method is a possible option for the analysis of toxaphene congeners. The method being advocated by the OIG, mass spectrometry, is a powerful qualitative tool for identifying compounds based on the molecular structure. However, for a positive, confirmed molecular identification, a pure analytical standard of the congener is required. In order to accurately quantify a particular toxaphene congener, an analytical standard of the same congener is absolutely essential to characterize the response of an individual GC/MS and to provide an unequivocal identification of the congener. Some estimates put the number of chlorinated compounds in the technical toxaphene mixture at more than 200 compounds, but for the chemical family of chlorinated bornanes, which is one of the primary constituents of technical toxaphene, there are over 32,000 congeners. Since there are numerous breakdown products of toxaphene, many with molecular structures not completely identified and isolated, it is not feasible to analyze for all the possible congeners.

In order to shorten the list of potential congener target analytes, information on which congeners are a threat to human health must be considered. Appendix A, page 17, of the OIG report indicates detailed information is lacking on the potential human exposure to toxaphene degradation products and their toxicity limiting the ability to conduct a thorough risk assessment. However, page 29 of Appendix A indicates that five congeners, p26, p50, p40, p41, and p44, are most likely to accumulate in the human body. Therefore any attempt to produce analytical data which is meaningful for human risk assessment must be focused on those congeners which are most toxic with a concurrent consideration of which congeners have analytical standards available to perform the analysis. If the list of toxic congeners can indeed be narrowed to the p26, p50, p40, p41, and p44 congeners, this makes the analytical task of measuring these congeners much more manageable whether Method 8081, a NIMS method, or some other type of method is employed.

OIG Report, page 7: *Region 4 officials are concerned about using the new (or NIMS) method to test groundwater at the Hercules 009 Landfill because the method is not approved by EPA. When using environmental data for public health decisions, Region 4 prefers to use approved methods that have been validated by several laboratories. The EPA conventional method (Method 8081) was validated. Consequently, EPA knows the results will be accurate when Method 8081 is used to test for the original toxaphene mixture.*

The OIG agrees that using an EPA-approved method is better than a method not approved by EPA. However, EPA has no approved method to identify toxaphene breakdown products. Thus, to decide if the cleanup is effective, Region 4 must use an unapproved method to obtain the necessary information on the presence or absence of toxaphene breakdown products.

SESD Comment # 4: We agree that the EPA hazardous waste program does not require the use of an approved method. Region 4's concern about the use of the NIMS method does not relate to method approval by the Agency, but to method validation. Multi-laboratory validation of an analytical method assures the data produced with the method can meet certain criteria for acceptable precision and bias as well as withstand challenge from those who may disagree with the results. Collaborative testing among multiple laboratories helps to assure that different labs can obtain comparable data using the method, that the method is free of bias due to effects of a particular sample matrix, and that the data produced with the method is defensible in regulatory and legal proceedings. Therefore prior to using the NIMS method, or any other method, to generate data which must ultimately be defended by the laboratory which produced it, we believe the Agency as whole must develop a standard, validated analytical method which has been shown to produce data which is reliable, reproducible, and accurate. Ultimately any data produced by NIMS or another new method must be suitable for its intended purpose, which in this case is to assess human risk from toxaphene congeners and breakdown products.

OIG Report, page 8: *The EPA Region 4 laboratory has the capability to run the new (NIMS) method. However, the Region 4 laboratory personnel will need to learn the procedures, show they work, and practice using them before actually testing groundwater samples from the Hercules 009 Landfill. Thus, implementing the new method will take more laboratory resources, but is needed to obtain the necessary information to decide if the cleanup is effective.*

We recommend that the Regional Administrator, Region 4:

2.1 Use negative ion mass spectroscopy to definitively determine if toxaphene breakdown products are present in the surrounding groundwater at the Hercules 009 Landfill site, and (if so) in what amounts.

SESD Comment # 5: The Region 4 laboratory acquired the instrumentation needed to perform the NIMS procedure on January 30, 2004, after the monitoring had been performed for the five year review. We agree the NIMS procedure has the potential to identify and quantify the toxaphene congeners and also agree that additional resources would be needed to implement the method in the Region 4 laboratory. In the absence of new resources, existing staff must be shifted from performing other project analyses to implementation of the NIMS method for toxaphene congeners. As noted elsewhere in the Region 4 comments, the analysis of toxaphene breakdown products needs to be focused on those congeners which demonstrate toxicity to humans. Attempting to identify all possible toxaphene breakdown products is not feasible because of the large number of

potential targets, and the fact that analytical standards are available for a limited number of toxaphene congeners.

The lack of a validated method for the analysis of toxaphene congeners, as well as the apparent dearth of congener toxicity data is not isolated to Region 4. The issues cut across the entire Agency and the resources of the Agency must be focused on the problem to reach a satisfactory resolution. Region 4's laboratory is willing to participate in a multi-laboratory method validation study for toxaphene congeners in environmental samples. However, since the Agency as a whole would obviously benefit from a validated NIMS method for toxaphene congeners, we believe that a multi-laboratory method validation study should be initiated at the program level by the Office of Solid Waste and Emergency Response. A validated method will serve both the regulated community and the Agency by assuring that analytical data produced by the method is defensible, of known quality, and suitable for risk assessment decision making.

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 4
ATLANTA FEDERAL CENTER
61 FORSYTH STREET
ATLANTA, GEORGIA 30303-8960

June 22, 2005

MEMORANDUM

SUBJECT: Comments on the May 16, 2005, EPA Ombudsman Draft Report Concerning the Hercules 009 Landfill Superfund Site, Brunswick, Georgia

FROM: Gregory D. Luetscher
Associate Regional Counsel
Office of Environmental Accountability

TO: Leo Francendese
Remedial Project Manager
Waste Management Division

Attached hereto are my comments pertaining to the legal issues identified in the subject Draft Report as transmitted to Mr. Palmer via OIG memorandum dated May 16, 2005. As you know, the attachment itself is typically classified under the rubric of attorney-work-product and thus exempt from release to the public under FOIA. However, the issues identified and opinions reflected by the comments may, of course, nevertheless become a part of the Region's reply to the extent you find them suitable for that purpose. Finally, since I am clearly unqualified to comment on the technical matters, please note that my review focused upon only the first fifteen pages or so of the draft.

If you would like clarification on any matter I have addressed or if you simply have any questions, please do not hesitate to get in touch. Thanks.

Attachment

Review of draft IG Audit Report, Hercules 009 Landfill:

The draft IG audit contains terms, such as "required" and "must," that reflect non-discretionary obligations arising from legal authority, but which have mistakenly been ascribed by the authors to EPA's discretionary, policy-based, duties. In Chapters 2 and 3, mistakes of this variety are notable in both the text and the findings which highlight the text. While EPA clearly has various non-discretionary duties that it must perform under particular circumstances (e.g., a five year review), EPA's directives, guidance documents, and other expressions of policy produce duties which are, by their very nature, discretionary. In various places throughout the draft errors of this type are all-the-more obvious because italicized language drawn verbatim from EPA guidance documents clearly contains terms descriptive of a discretionary act (e.g., "should"), but which, when included as part of a finding, have been transformed into language containing terms, such as "required" or "requires," that describe non-discretionary acts. Unfortunately, the repeated failure to adequately distinguish between those sources of statutory authority from which EPA is legally obligated to act, and those instances arising from EPA policy in which an action may be discretionary, tends to cast doubt upon the credibility of other conclusions contained in the document.

Curiously, at the end of Chapter 2 the drafters expressly acknowledge the proposition that the draft otherwise appears to disclaim, i.e., that EPA's policies produce discretionary, rather than mandatory, duties. To support the drafters' apt conclusion that "EPA must use an unapproved method" to properly test for toxaphene's breakdown products, the authors opine that EPA has the authority to deviate from the conventional method (i.e., Method 8081) specifically because the document in which the official methods are published, (i.e., EPA publication SW-846) "functions primary as a guidance document setting forth acceptable, although not required, methods ... ." The drafters then buttress this conclusion, while at the same time undermining several of their earlier findings, by observing that "the methods are guidance and not mandatory."

Specific examples of language that should be corrected include the following:

- The third word of Chapter 2 (on page 5) mis-characterizes the scope and nature of EPA's authority by asserting, "EPA policy *requires* ... ." It should state something akin to "EPA policy *encourages* ... ." (Emphasis added.)

- Further along on page 5, EPA guidance is mistakenly alleged to "*require*(s) EPA to look for the potential presence of toxic transformation products." This misplaced assertion concerning EPA's duty is then transformed into a further "requirement" via the finding entitled, "Testing for Toxaphene Breakdown Products is Required." These errors are twice emphasized in the text by including quotes from the pertinent guidance. ("... and *should* be evaluated to determine..." and "...programs *should* be designed"... .) (Emphasis added.)

- On page 6 the authors mistakenly ascribe a non-discretionary duty to expressions of policy by asserting, "EPA's guidance *requires* groundwater monitoring..." and "EPA *must* evaluate the groundwater... ." As both duties arise from EPA policy, each is a discretionary duty and hence not required. (Emphasis added.)

- The third word of Chapter 3 is "report" but should instead be "review"; thereby explaining (correctly) that, "EPA must *review* every 5 years ..." rather than "EPA must *report* every 5 years..." as currently drafted. The obligation to "review" is a non-discretionary duty assigned to EPA under CERCLA, but the duty to "report" the findings derives from EPA's policy and is therefore merely a discretionary duty. (Emphasis added.)

- The first finding in Chapter 3 should be changed to reflect what actually is required under law, i.e., "Review," not "Reporting," Must Occur Every 5 Years.

- On page 11, a finding states, "EPA Policy Requires the Reviewer to Make a Decision." In the associated text, the authors assert that EPA's June 2001 Five-year Review Guidance directs that "the reviewer must make a decision ... ." However, no such requirement can be located within the referenced EPA document which, by its very title, seems to be an expression of EPA policy rather than a statutory or regulatory requirement.

AFFIDAVIT

STATE OF GEORGIA)
) ss:
COUNTY OF GLYNN)

000057

JAMES B. GILBERT, JR., affiant herein, being duly sworn, deposes and says this 14th day of December, 1993:

1. That affiant is the attorney of Hercules Incorporated.

2. That attached hereto as Exhibit A is a copy of a Consent Decree dated October 1, 1993, entered in the United States District Court for the Southern District of Georgia at Civil Action No. CV293-132, certified from the record on December 10, 1993 by the Deputy Clerk of said court.

3. That Hercules Incorporated is required, pursuant to Article V of said Consent Degree, to record a certified copy thereof in the Recorder of Deeds Office in Glynn County.

(L.S.)
James B. Gilbert, Jr.
Attorney for Hercules Incorporated

Sworn to and subscribed before me
this 14th day of December 1993:

Lisa Sullivan
Notary Public
Glynn County, Georgia

My Commission Expires: Notary Public, Glynn County, Georgia My Commission Expires May 9, 1997.

LISA SULLIVAN NOTARY PUBLIC GLYNN COUNTY, GA.

000058

ATTEST: A TRUE COPY

Certified to Dec 13 19 83

Deputy Clerk

4 August 2005

Mr. Leo Francendese
U.S. Environmental Protection Agency Region 4
Hazardous Waste Management Division
Atlanta Federal Center
61 Forsyth Street
Atlanta, GA 30303

Dear Mr. Francendese:

In response to your request for clarification on the effects of specific sample processing procedures – namely acid-activated copper and strong acid treatments -- for environmental toxaphene residues, please consider the following:

1. ***Acid-activated copper treatment (ACT).*** Copper granules that have been "activated" (surface oxide layer removed) by strong acid treatment (e.g. HCl) are routinely used to remove elemental sulfur from organic extracts containing chlorinated hydrocarbon pollutant target analytes (e.g. the chlorinated monoterpenes that comprise toxaphene). High quality ACT protocols also minimize acid residual associated with this procedure via exhaustive water washing followed by polar and non-polar solvent rinses of the activated copper granules. As a result, the chances of acid contamination in the treated sample extract resulting from ACT are extremely low. Application of ACT for extracts of sulfur-containing media -- e.g. sediments and groundwater -- has been shown repeatedly not to affect the quantitative recovery of target (toxaphene) analytes (Maruya et al. 2000; Vetter and Maruya 2000) and, as such, continues to be used by the top research labs in the world (Raff and Hites 2004). For example, the recoveries of four individual toxaphene components and of technical toxaphene fortified into a reference estuarine sediment sample averaged between 90-100% and between 70-80% when analyzed by GC-ECD, respectively (Maruya et al. 2000). Although some target analyte loss due to ACT is possible (e.g. via sorption, residual acid hydrolysis), the aforementioned QA/QC results strongly indicate that any losses are minimal.

2. ***Strong acid treatment (SAT)***. Strong acids such as hydrochloric (HCl) or sulfuric (H_2SO_4) hydrolyze various organic moieties (i.e. functional groups) leading to a reduction in the number and types of chemical compounds present in an organic extract of an environmental sample. Polar compounds (e.g. those with O, S, -OH, -COOH) and in particular lipids are susceptible to acid hydrolysis. However, most legacy organic

pollutants are acid-resistant (i.e. not chemically modified or transformed) and are thus preserved in organic extracts subjected to SAT. These pollutants include HCHs, α- and γ-chlordane, DDTs, and the chlorinated monoterpenes that comprise technical toxaphene. A round-robin exercise using EPA SW-846 approved analytical protocols for technical toxaphene modified by the addition of a sulfuric acid cleanup step for various environmental matrices (including sludge, soil and water) concluded no substantial differences in recovered mass or chromatographic profile (Carlin et al. 1998). SAT has also been used successfully as a cleanup step to quantify toxaphene residues in fish and marine mammal blubber samples (Føreid et al. 2000).

3. ***Method validation for Hercules 009 Landfill groundwater samples***. As part of the QA/QC effort for this project, technical toxaphene was fortified into three different groundwater samples (856996-007MS, 856996-008MSD and svk1082-075mbLCS) and analyzed in conjunction with the remainder of field samples. Quantitative/near quantitative recovery was reported in each of the three matrix spike samples, before/after ACT was employed to remove sulfur.

In conclusion, ACT and SAT have minimal, if any, quantitative or qualitative effect on toxaphene residues in environmental samples. As a final (combined) example, both ACT and SAT are approved for the analysis of organochlorines (including toxaphene) in sediment for the Greenland Artic Monitoring and Assessment Programme (AMAP) (Asmund and Cleeman 2000). As a scientist who specialized in the analysis of toxaphene residues in sediments, water and tissue samples since 1997, I am confident that ACT and SAT (if applied) had little/no effect on the identification and quantitation of toxaphene residues in Hercules 009 Landfill groundwater samples collected in 2004.

Sincerely yours,

/s/

Keith A. Maruya, Ph.D.
Principal Scientist
Southern California Coastal Water Research Project (SCCWRP)

Literature cited

Asmund G, Cleemann M. 2000. Analytical methods, quality assurance and quality control used in the Greenland AMAP programme. *Sci Tot Environ* 245: 203-219.

Carlin FJ, Revells HL, Reed DL. 1998. The application of standard methods for the determination of toxaphene in environmental media. *Chemosphere* 41:481-486.

Føreid S, Rundberget T, Severinsen T, Wiig Ø, Skaare JU. 2000. Determination of toxaphenes in fish and marine mammals. *Chemosphere* 41:521-528.

Maruya KA, Wakeham SG, Vetter W, Francendese L. 2000. Prominent chlorobornane residues in estuarine sediments contaminated with toxaphene. *Environ Toxicol Chem* 19:2198-2203.

Raff JD, Hites RA. 2004. Transport of Suspended-Sediment-Bound Toxaphene in the Mississippi River. *Environ Sci Technol* 38:2785-2791.

Vetter W, Maruya KA. 2000. Congener and enantioselective analysis of toxaphene residues in sediment and biota from a contaminated estuarine wetland. *Environ Sci Technol* 34:1627-1635.

BR 11-10-04

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

Region 4

Science and Ecosystem Support Division
980 College Station Road
Athens, Georgia 30605-2720

MEMORANDUM

Date: 11/08/2004

Subject: Results of PESTICIDES/PCB Sample Analysis
04-0943 *Hercules 009 Landfill*
Brunswick, GA

From: Revells, Lavon

To: Francendese, Leo

CC: Brittany Barnes
RMT

Thru: Bennett, Gary
Chief,Organic Chemistry Section
Analytical Support Branch

Attached are the results of analysis of samples collected as part of the subject project. If you have any questions, please contact me.

Sample Disposal Policy:

According to our records this project is not part of a criminal investigation.
Because of our limited space for long term sample storage, we must perform disposals on a routine basis.

Therefore, please take note that within 90 days of the date of this memo, the original samples and all extracts associated with the samples will be disposed of as required by all applicable and appropriate statutes.

These samples may be held in custody for longer than 90 days only by contacting our sample coordinator, Debbie Colquitt, by e-mail at Colquitt.Debbie@epa.gov.

ATTACHMENT

Sample **10623** FY **2004** Project: **04-0943**

SPECIFIED TESTS

Facility: Hercules 009 Landfill Brunswick, GA
Program: SF
Id/Station: N-07 /
Media: GROUNDWATER

Produced by: Revells, Lavon
Requestor:
Project Leader: LFRANCEN
Beginning: 09/28/2004 18:20
Ending:

RESULTS	UNITS	ANALYTE
2.0 U	UG/L	Toxaphene
2.5 U	UG/L	Chlorinated Camphenes

U-Analyte not detected at or above reporting limit. | J-Identification of analyte is acceptable; reported value is an estimate. | UJ-Analyte not detected at or above reporting limit. Reporting limit is an estimate.
N-Presumptive evidence analyte is present; analyte reported as tentative identification. | NJ-Presumptive evidence analyte is present; analyte reported as tentative identification. Reported value is an estimate.
K-Identification of analyte is acceptable; reported value may be biased high. Actual value expected to be less than the reported value.
L-Identification of analyte is acceptable; reported value may be biased low. Actual value expected to be greater than reported value.
NA-Not Analyzed. | NAI-Not Analyzed due to Interferences. | A-Analyte analyzed in replicate. Reported value is "average" of replicates.
R-Presence or absence of analyte can not be determined from data due to severe quality control problems. Data are rejected and considered unusable.
C-confirmed by GCMS | /1-when no value is reported, see chlordane constituents | /2-constituents or metabolites of technical chlordane

Sample **10624** FY **2004** Project: **04-0943**

SPECIFIED TESTS

Facility: Hercules 009 Landfill Brunswick, GA
Program: SF
Id/Station: N-15D /
Media: GROUNDWATER

Produced by: Revells, Lavon
Requestor:
Project Leader: LFRANCEN
Beginning: 09/29/2004 16:55
Ending:

RESULTS	UNITS	ANALYTE
2.0 U	UG/L	Toxaphene
2.5 U	UG/L	Chlorinated Camphenes

U-Analyte not detected at or above reporting limit. | J-Identification of analyte is acceptable; reported value is an estimate. | UJ-Analyte not detected at or above reporting limit. Reporting limit is an estimate.
N-Presumptive evidence analyte is present; analyte reported as tentative identification. | NJ-Presumptive evidence analyte is present; analyte reported as tentative identification. Reported value is an estimate.
K-Identification of analyte is acceptable; reported value may be biased high. Actual value expected to be less than the reported value.
L-Identification of analyte is acceptable; reported value may be biased low. Actual value expected to be greater than reported value.
NA-Not Analyzed. | NAI-Not Analyzed due to Interferences. | A-Analyte analyzed in replicate. Reported value is "average" of replicates.
R-Presence or absence of analyte can not be determined from data due to severe quality control problems. Data are rejected and considered unusable.
C-confirmed by GCMS | /1-when no value is reported, see chlordane constituents | /2-constituents or metabolites of technical chlordane

Sample **10625** FY **2004** Project: **04-0943**

SPECIFIED TESTS

Facility: Hercules 009 Landfill Brunswick, GA
Program: SF
Id/Station: N-09S /
Media: GROUNDWATER

Produced by: Bennett, Gary
Requestor:
Project Leader: LFRANCEN
Beginning: 09/28/2004 09:30
Ending:

RESULTS	UNITS	ANALYTE
2.0 U	UG/L	Toxaphene
0.76 JN	UG/L	*Chlorinated Camphenes*

Value for camphenes is estimated maximum possible concentration based on peaks within the toxaphene retention time window.

Chlorinated camphenes not positively identified.

U-Analyte not detected at or above reporting limit. | J-Identification of analyte is acceptable; reported value is an estimate. | UJ-Analyte not detected at or above reporting limit. Reporting limit is an estimate.
N-Presumptive evidence analyte is present; analyte reported as tentative identification. | NJ-Presumptive evidence analyte is present; analyte reported as tentative identification. Reported value is an estimate.
K-Identification of analyte is acceptable; reported value may be biased high. Actual value expected to be less than the reported value.
L-Identification of analyte is acceptable; reported value may be biased low. Actual value expected to be greater than reported value.
NA-Not Analyzed. | NAI-Not Analyzed due to Interferences. | A-Analyte analyzed in replicate. Reported value is "average" of replicates.
R-Presence or absence of analyte can not be determined from data due to severe quality control problems. Data are rejected and considered *unusable*.
C-confirmed by GCMS | /1-when no value is reported, see chlordane constituents | /2-constituents or metabolites of technical chlordane

Sample **10626** FY **2004** Project: **04-0943**

SPECIFIED TESTS

Facility: Hercules 009 Landfill Brunswick, GA
Program: SF
Id/Station: N-09D /
Media: GROUNDWATER

Produced by: Revells, Lavon
Requestor:
Project Leader: LFRANCEN
Beginning: 09/28/2004 10:35
Ending:

RESULTS	UNITS	ANALYTE
2.0 U	UG/L	Toxaphene
2.5 U	UG/L	Chlorinated Camphenes

U-Analyte not detected at or above reporting limit. | J-Identification of analyte is acceptable; reported value is an estimate. | UJ-Analyte not detected at or above reporting limit. Reporting limit is an estimate.
N-Presumptive evidence analyte is present; analyte reported as tentative identification. | NJ-Presumptive evidence analyte is present; analyte reported as tentative identification. Reported value is an estimate.
K-Identification of analyte is acceptable; reported value may be biased high. Actual value expected to be less than the reported value.
L-Identification of analyte is acceptable; reported value may be biased low. Actual value expected to be greater than reported value.
NA-Not Analyzed. | NAI-Not Analyzed due to Interferences. | A-Analyte analyzed in replicate. Reported value is "average" of replicates.
R-Presence or absence of analyte can not be determined from data due to severe quality control problems. Data are rejected and considered unusable.
C-confirmed by GCMS | /1-when no value is reported, see chlordane constituents | /2-constituents or metabolites of technical chlordane

Sample **10627** FY **2004** Project: **04-0943**

SPECIFIED TESTS

Facility: Hercules 009 Landfill Brunswick, GA
Program: SF
Id/Station: N-06SR /
Media: GROUNDWATER

Produced by: Bennett, Gary
Requestor:
Project Leader: LFRANCEN
Beginning: 09/28/2004 12:10
Ending:

RESULTS	UNITS	ANALYTE
2.0 U	UG/L	Toxaphene
2.0 JN	UG/L	Chlorinated Camphenes

Value for camphenes is estimated maximum possible concentration based on peaks within the toxaphene retention time window.

Chlorinated camphenes not positively identified.

U-Analyte not detected at or above reporting limit. | J-Identification of analyte is acceptable; reported value is an estimate. | UJ-Analyte not detected at or above reporting limit. Reporting limit is an estimate.
N-Presumptive evidence analyte is present; analyte reported as tentative identification. | NJ-Presumptive evidence analyte is present; analyte reported as tentative identification. Reported value is an estimate.
K-Identification of analyte is acceptable; reported value may be biased high. Actual value expected to be less than the reported value.
L-Identification of analyte is acceptable; reported value may be biased low. Actual value expected to be greater than reported value.
NA-Not Analyzed. | NAI-Not Analyzed due to Interferences. | A-Analyte analyzed in replicate. Reported value is "average" of replicates.
R-Presence or absence of analyte can not be determined from data due to severe quality control problems. Data are rejected and considered unusable.
C-confirmed by GCMS | /1-when no value is reported, see chlordane constituents | /2-constituents or metabolites of technical chlordane

Sample **10628** FY **2004** Project: **04-0943**

SPECIFIED TESTS

Facility: Hercules 009 Landfill *Brunswick, GA*
Program: SF
Id/Station: N-11 /
Media: GROUNDWATER

Produced by: Bennett, Gary
Requestor:
Project Leader: LFRANCEN
Beginning: 09/28/2004 11:45
Ending:

RESULTS	UNITS	ANALYTE
4.0 U	UG/L	Toxaphene
4.0 JN	UG/L	Chlorinated Camphenes

Value for camphenes is estimated maximum possible concnetration based on peaks within toxaphene retention time window

Chlorinated camphenes not positively identified

U-Analyte not detected at or above reporting limit. | J-Identification of analyte is acceptable; reported value is an estimate. | UJ-Analyte not detected at or above reporting limit. Reporting limit is an estimate.
N-Presumptive evidence analyte is present; analyte reported as tentative identification. | NJ-Presumptive evidence analyte is present; analyte reported as tentative identification. Reported value is an estimate.
K-Identification of analyte is acceptable; reported value may be biased high. Actual value expected to be less than the reported value.
L-Identification of analyte is acceptable; reported value may be biased low. Actual value expected to be greater than reported value.
NA-Not Analyzed. | NAI-Not Analyzed due to interferences. | A-Analyte analyzed in replicate. Reported value is "average" of replicates.
R-Presence or absence of analyte can not be determined from data due to severe quality control problems. Data are rejected and considered unusable.
C-confirmed by GCMS | /1-when no value is reported, see chlordane constituents | /2-constituents or metabolites of technical chlordane

Sample **10629** *FY* **2004** *Project:* **04-0943**

SPECIFIED TESTS

Facility: Hercules 009 Landfill Brunswick, GA
Program: SF
Id/Station: N-08 /
Media: GROUNDWATER

Produced by: Revells, Lavon
Requestor:
Project Leader: LFRANCEN
Beginning: 09/28/2004 16:20
Ending:

RESULTS	UNITS	ANALYTE
2.0 U	UG/L	Toxaphene
2.5 U	UG/L	Chlorinated Camphenes

U-Analyte not detected at or above reporting limit. | J-Identification of analyte is acceptable; reported value is an estimate. | UJ-Analyte not detected at or above reporting limit. Reporting limit is an estimate.
N-Presumptive evidence analyte is present; analyte reported as tentative identification. | *NJ-Presumptive evidence analyte is present; analyte reported as tentative identification. Reported value is an estimate.*
K-Identification of analyte is acceptable; reported value may be biased high. Actual value expected to be less than the reported value.
L-Identification of analyte is acceptable; reported value may be biased low. Actual value expected to be greater than reported value.
NA-Not Analyzed. | NAI-Not Analyzed due to Interferences. | A-Analyte analyzed in replicate. Reported value is "average" of replicates.
R-Presence or absence of analyte can not be determined from data due to severe quality control problems. Data are rejected and considered unusable.
C-confirmed by GCMS | /1-when no value is reported, see chlordane constituents | /2-constituents or metabolites of technical chlordane

Sample **10630** FY **2004** Project: **04-0943**

SPECIFIED TESTS

Facility: Hercules 009 Landfill Brunswick, GA
Program: SF
Id/Station: N-05 /
Media: GROUNDWATER

Produced by: Bennett, Gary
Requestor:
Project Leader: LFRANCEN
Beginning: 09/28/2004 18:00
Ending:

RESULTS	UNITS	ANALYTE
2.0 U	UG/L	Toxaphene
1.6 JN	UG/L	Chlorinated Camphenes

Value for camphenes is estimated maximum possible concentration based on peaks within toxaphene retention time window.

Chlorinated camphenes not positively identified.

U-Analyte not detected at or above reporting limit. | J-Identification of analyte is acceptable; reported value is an estimate. | UJ-Analyte not detected at or above reporting limit. Reporting limit is an estimate.
N-Presumptive evidence analyte is present; analyte reported as tentative identification. | NJ-Presumptive evidence analyte is present; analyte reported as tentative identification. Reported value is an estimate.
K-Identification of analyte is acceptable; reported value may be biased high. Actual value expected to be less than the reported value.
L-Identification of analyte is acceptable; reported value may be biased low. Actual value expected to be greater than reported value.
NA-Not Analyzed. | NAI-Not Analyzed due to Interferences. | A-Analyte analyzed in replicate. Reported value is "average" of replicates.
R-Presence or absence of analyte can not be determined from data due to severe quality control problems. Data are rejected and considered unusable.
C-confirmed by GCMS | /1-when no value is reported, see chlordane constituents | /2-constituents or metabolites of technical chlordane

Sample **10631** FY **2004** Project: **04-0943**

SPECIFIED TESTS

Facility: Hercules 009 Landfill Brunswick, GA
Program: SF
Id/Station: N-10 /
Media: GROUNDWATER

Produced by: Revells, Lavon
Requestor:
Project Leader: LFRANCEN
Beginning: 09/28/2004 19:30
Ending:

RESULTS	UNITS	ANALYTE
2.0 U	UG/L	Toxaphene
2.5 U	UG/L	Chlorinated Camphenes

U-Analyte not detected at or above reporting limit. | J-Identification of analyte is acceptable; reported value is an estimate. | UJ-Analyte not detected at or above reporting limit. Reporting limit is an estimate.
N-Presumptive evidence analyte is present; analyte reported as tentative identification. | NJ-Presumptive evidence analyte is present; analyte reported as tentative identification. Reported value is an estimate.
K-Identification of analyte is acceptable; reported value may be biased high. Actual value expected to be less than the reported value.
L-Identification of analyte is acceptable; reported value may be biased low. Actual value expected to be greater than reported value.
NA-Not Analyzed. | NAI-Not Analyzed due to Interferences. | A-Analyte analyzed in replicate. Reported value is "average" of replicates.
R-Presence or absence of analyte can not be determined from data due to severe quality control problems. Data are rejected and considered unusable.
C-confirmed by GCMS | /1-when no value is reported, see chlordane constituents | /2-constituents or metabolites of technical chlordane

PESTICIDES/PCB SAMPLE ANALYSIS | **EPA - REGION IV SESD, ATHENS, GA** | **Production Date: 11/10/2004 09:00**

Sample **10632** FY **2004** Project: **04-0943**

SPECIFIED TESTS

Facility: Hercules 009 Landfill Brunswick, GA
Program: SF
Id/Station: N-12 /
Media: GROUNDWATER

Produced by: Bennett, Gary
Requestor:
Project Leader: LFRANCEN
Beginning: 09/28/2004 16:00
Ending:

RESULTS	UNITS	ANALYTE
2.0 U	UG/L	Toxaphene
1.4 JN	UG/L	Chlorinated Camphenes

Value for camphenes is estimated maximum possible concentration based on peaks within toxaphene retention time window.

Chlorinated camphenes not positively identified.

U-Analyte not detected at or above reporting limit. | J-Identification of analyte is acceptable; reported value is an estimate. | UJ-Analyte not detected at or above reporting limit. Reporting limit is an estimate.
N-Presumptive evidence analyte is present; analyte reported as tentative identification. | NJ-Presumptive evidence analyte is present; analyte reported as tentative identification. Reported value is an estimate.
K-Identification of analyte is acceptable; reported value may be biased high. Actual value expected to be less than the reported value.
L-Identification of analyte is acceptable; reported value may be biased low. Actual value expected to be greater than reported value.
NA-Not Analyzed. | NAI-Not Analyzed due to Interferences. | A-Analyte analyzed in replicate. Reported value is "average" of replicates.
R-Presence or absence of analyte can not be determined from data due to severe quality control problems. Data are rejected and considered unusable.
C-confirmed by GCMS | /1-when no value is reported, see chlordane constituents | /2-constituents or metabolites of technical chlordane

Sample **10633** FY **2004** Project: **04-0943**

SPECIFIED TESTS

Facility: Hercules 009 Landfill Brunswick, GA
Program: SF
Id/Station: N-06DR /
Media: GROUNDWATER

Produced by: Bennett, Gary
Requestor:
Project Leader: LFRANCEN
Beginning: 09/28/2004 13:30
Ending:

RESULTS	UNITS	ANALYTE
2.0 U	UG/L	Toxaphene
0.75 JN	UG/L	Chlorinated Camphenes

Value for camphenes is estimated maximum possible concentration based on peaks within toxaphene retention time window.

Chlorinated camphenes not positively identified.

U-Analyte not detected at or above reporting limit. | J-Identification of analyte is acceptable; reported value is an estimate. | UJ-Analyte not detected at or above reporting limit. Reporting limit is an estimate.
N-Presumptive evidence analyte is present; analyte reported as tentative identification. | NJ-Presumptive evidence analyte is present; analyte reported as tentative identification. Reported value is an estimate.
K-Identification of analyte is acceptable; reported value may be biased high. Actual value expected to be less than the reported value.
L-Identification of analyte is acceptable; reported value may be biased low. Actual value expected to be greater than reported value.
NA-Not Analyzed. | NAI-Not Analyzed due to Interferences. | A-Analyte analyzed in replicate. Reported value is "average" of replicates.
R-Presence or absence of analyte can not be determined from data due to severe quality control problems. Data are rejected and considered unusable.
C-confirmed by GCMS | /1-when no value is reported, see chlordane constituents | /2-constituents or metabolites of technical chlordane

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

Region 4

Science and Ecosystem Support Division
980 College Station Road
Athens, Georgia 30605-2720

MEMORANDUM

Date: 03/02/2005

Subject: Results of PESTICIDES/PCB Sample Analysis
05-0204 Hercules 009 Landfill
Brunswick, GA

From: Revells, Lavon HLR

To: Francendese, Leo

CC: Brittany Barnes
RMT

Thru: Bennett, Gary
Chief,Organic Chemistry Section
Analytical Support Branch

Attached are the results of analysis of samples collected as part of the subject project. EPA Region 4 Lab received four 1-liter bottles for a water sample from the Hercules 009 site. The four 1-liter bottles were labled A, B, C, and D. Samples from bottles A and B were used for a Matrix Spike and Matrix Spike Duplicate. Samples from bottles C and D were treated as laboratory duplicates. The Toxaphene results for both duplicates were 2.1 ug/L. The Chlorinated Camphene result from bottle C was 6.1 NJ ug/L and from bottle D was 6.7 NJ ug/L. The average result of 6.4 ANJ ug/L for Chlorinated Camphene was reported. If you have any questions, please contact me.

Sample Disposal Policy:

According to our records this project is not part of a criminal investigation.
Because of our limited space for long term sample storage, we must perform disposals on a routine basis.

Therefore, please take note that within 90 days of the date of this memo, the original samples and all extracts associated with the samples will be disposed of as required by all applicable and appropriate statutes.

These samples may be held in custody for longer than 90 days only by contacting our sample coordinator, Debbie Colquitt, by e-mail at Colquitt.Debbie@epa.gov.

ATTACHMENT

Sample 1711 FY 2005 Project: 05-0204

SPECIFIED TESTS

Facility: Hercules 009 Landfill Brunswick, GA
Program: SF
Id/Station: N-11 / 111305 N-11
Media: WATER

Produced by: Revells, Lavon
Requestor:
Project Leader: LFRANCEN
Beginning: 01/13/2005 12:50
Ending:

RESULTS	UNITS	ANALYTE
2.1 U	UG/L	Toxaphene
6.4 ANJ	UG/L	Chlorinated Camphenes

U-Analyte not detected at or above reporting limit. | J-Identification of analyte is acceptable; reported value is an estimate. | UJ-Analyte not detected at or above reporting limit. Reporting limit is an estimate.
N-Presumptive evidence analyte is present; analyte reported as tentative identification. | NJ-Presumptive evidence analyte is present; analyte reported as tentative identification. Reported value is an estimate.
K-Identification of analyte is acceptable; reported value may be biased high. Actual value expected to be less than the reported value.
L-Identification of analyte is acceptable; reported value may be biased low. Actual value expected to be greater than reported value.
NA-Not Analyzed. | NAI-Not Analyzed due to Interferences. | A-Analyte analyzed in replicate. Reported value is "average" of replicates.
R-Presence or absence of analyte can not be determined from data due to severe quality control problems. Data are rejected and considered unusable.
C-confirmed by GCMS | /1-when no value is reported, see chlordane constituents | /2-constituents or metabolites of technical chlordane