## APPENDIX B: OPEN SCAN VOLATILES IN SLUDGE

### OVERVIEW:

Upon receipt at Enviro-Test Labs, the sample was stored in a walk-in cooler maintained at approximately 4°C. On March 30, 1998, approximately 2 g of the sample was weighed into a 20 mL headspace vial, brought to a 15 mL final volume with pre-purged water, and sealed with a teflon-lined septum. A method blank vial containing 15 mL of the same pre-purged water was prepared along with the sample to account for any background contamination. The vials were spiked with three surrogates and three internal standard compounds to monitor the headspace extraction efficiency and system performance. Analysis was by automated headspace injection into a capillary gas chromatograph equipped with a mass selective detector (GC/MSD) in normal scanning aquisition (SCAN) mode.

The mass spectra for all the major components were then generated along with an area report. The spectra were manually interpreted using first principles and matching to hardcopy library spectra, when available. Each compound was assigned a confidence of identification value based on the quality of the mass spectra and the analyst's opinion of the match to hardcopy spectra. These values are fully explained in the attached table.

Semi-quantitation was performed by external standard method using the average response factor of the surrogates and internal standards.

### METHOD REFERENCE:

ETL MSOP# 52.04 (modified method EPA8240 with automated headspace and GC/MSD/SCAN analysis).

## APPENDIX C

## OPEN SCAN OF EXTRACTABLE ORGANICS

### OVERVIEW:

A pulpmill sludge sample was submitted for open scan analysis. A moisture determination found the sample contained 73% moisture. A portion of the sample was soxhlet extracted with dichloromethane (DCM), under acidic pH conditions, for 16 hours. The soxhlet extract then went through a base/neutral and acid partition. The soxhlet extract was added to 1L of lab water, the pH adjusted to pH > 12 and serially extracted with DCM. The pH was then re-adjusted to pH < 2 and again serially extracted with DCM. Each DCM extract was concentrated to a low known final volume, resulting in 2 extracts, a base/neutral (B/N) extract and acid extract. A method blank was extracted and concentrated in the same manner as the sample. An aliquot of each extract was injected onto a gas chromatograph/mass spectrometer (GC/MS) operating in the scanning acquisition mode. The spectra for all major resolved components were generated along with an area % report. The spectra were then manually interpreted using first principles and matching to hard copy spectra, when available. Each compound identified is assigned a confidence of identification value based on the quality of the spectra and the analyst's opinion of the match. These values are fully explained in Table 1 below. The components identified are semi-quantitated by comparing peak areas to that of the peak area of internal standard, Phenanthrene $d_{10}$, which was added to the extract before being injected. Tables 2 and 3 list the compounds identified and their approximate concentrations. The total ion chromatograms from the GC/MS analysis are also included.

**QA/QC:**

To ensure extraction/method efficiency, the samples were fortified with a surrogate compound mix prior to extraction. The surrogate recoveries reflect the overall method efficiency. Below are the surrogate recoveries for this sample. Glassproofs were collected from both the soxhlet glassware and the glassware from the base/neutral & acid partition. The method blank and glassproofs were analyzed, and used to account for any lab reagent and glassware contribution.

| SURROGATE | RECOVERY (%) |
|---|---|
| 2-Fluorophenol | 49 |
| Phenol $d_5$ | 66 |
| 2,4,6-Tribromophenol | 107 |
| 5-Nitrobenzene $d_5$ | 92 |
| 2-Fluorobiphenyl | 102 |
| p-Terphenyl $d_{14}$ | 95 |

**TABLE 1: TABLE OF CONFIDENCE OF IDENTIFICATION**

**NUMBER 1:** Very confident, clean spectra, excellent match with library spectra.

**NUMBER 2:** Some small spectra differences with library match. A good comparison. Reference standard needed for positive identification.

**NUMBER 3:** Possible compound or very similar to. Spectra is definitely contaminated. Reference standard needed for positive identification.

**NUMBER 4:** Evidence of possible structure and molecular weight. Compound not identified.

**NUMBER 5:** Unable to identify.

**RESULTS:**

## SUMMARY OF IDENTIFICATION BY GC/MS

**SAMPLE I.D.:** PULPMILL SLUDGE
**LAB SAMPLE #:** E8-03-641-01

## SPECIFIC COMPOUNDS IDENTIFIED

| COMPOUND IDENTIFIED: | CONFIDENCE OF IDENTIFICATION: | CONCENTRATION: (µg/g or ppm) |
|---|---|---|
| α-pinene | 1 | 2.0 |
| camphene | 1 | 0.11 |
| $C_{10}$-di-olefinic/cyclic | 2 | 2.3 |
| β-pinene | 2 | 1.3 |
| 3-carene | 1 | 1.8 |
| limonene | 2 | 0.64 |
| $C_4$-alkyl sub't benzene | 1 | 0.71 |

Detection Limit: 0.1 ppm for hydrophobic volatiles, 1 ppm for light gases, 10 ppm for water-solubles.

## TABLE OF CONFIDENCE OF IDENTIFICATION

**NUMBER 1:** Very confident, clean spectra, excellent match with library spectra.

**NUMBER 2:** Some small spectra differences with library match. A good comparison. Reference standard needed for positive identification.

**NUMBER 3:** Possible compound or very similar to. Spectra is definitely contaminated. Reference standard needed for positive identification.

**NUMBER 4:** Evidence of possible structure and molecular weight. Compound not identified.

**NUMBER 5:** Unable to identify.

**RESULTS:**

## TABLE 2: SUMMARY OF IDENTIFICATION BY GC/MS

**SAMPLE:** Pulpmill Sludge, 03/06/98 - B/N extract **LAB SAMPLE #:** E8-03-641-01 base/neutral extract

**CONCENTRATION:** Semi-quantitation versus Phenanthrene $d_{10}$

### SPECIFIC COMPOUNDS IDENTIFIED

| COMPOUND | CONFIDENCE OF IDENTIFICATION | APPROXIMATE CONCENTRATION µg/g (ppm) |
|---|---|---|
| Alpha pinene | 1 | 3.3 |
| Beta pinene | 1 | 2.4 |
| 3-Methyl-6-isopropyl-cyclohexene | 2 | 6.7 |
| Alpha-phellandrene | 1 | 1.7 |
| Delta-3-carene | 1 | 4.7 |
| Alpha terpinene | 2 | 1.8 |
| 1-Methyl-4-isopropyl-cyclohexene | 2 | 2.2 |
| Methyl-isopropyl-benzene | 2 | 3.1 |
| Beta-phellandrene | 1 | 4.8 |
| Dihydro-terpineol | 2 | 1.6 |
| Isopropyl-cyclohexanone | 3 | 0.6 |
| Isopropyl-cyclohexen-one | 3 | 1.0 |
| Alpha terpineol | 2 | 1.8 |
| $C_{13}$ Alkane | 1 | 0.7 |
| Alpha copaene | 2 | 1.0 |
| Tetramethyl-tricyclo-undecene, mol. wt. = 204 | 3 | 0.6 |
| Junipene | 1 | 2.0 |
| Alpha muurolene | 1 | 1.5 |
| Dimethyl-isopropyl-hexahydro-naphthalene | 3 | 1.0 |
| Delta cadinene | 3 | 1.7 |
| Dimethyl-isopropyl-tetrahydro-naphthalene | 3 | 0.6 |

**RESULTS cont'd:**

**TABLE 2 cont'd: SUMMARY OF IDENTIFICATION BY GC/MS**

**SAMPLE:** Pulpmill Sludge, 03/06/98 - B/N extract cont'd **LAB SAMPLE #:** E8-03-641-01 base/neutral extract

**CONCENTRATION:** Semi-quantitation versus Phenanthrene $d_{10}$

**SPECIFIC COMPOUNDS IDENTIFIED**

| COMPOUND | CONFIDENCE OF IDENTIFICATION | APPROXIMATE CONCENTRATION µg/g (ppm) |
|---|---|---|
| 1,2,3,5,6-Pentathiepane | 3 | 0.6 |
| Longifolene chloride, mol. wt. = 238 | 2 | 0.9 |
| Oxygen & chlorine containing, Dimethyl-isopropyl-octahydro-naphthalene mol. wt. = 256 | 3 | 1.0 |
| Dimethyl-isopropyl-naphthalene | 3 | 0.6 |
| Ethenyl-dimethyl-methylene-dodecahydro-phenanthrene, mol. wt. = 256 | 3 | 2.2 |
| Alkyl subst'd, unsaturated, multicyclic hydrocarbon, mol. wt. = 272 | 3 | 3.6 |
| Ethenyl-tetramethyl-dodecahydro-phenanthrene mol. wt. = 272 (2 isomers) | 3 | 19 |
| Alkyl subst'd, multicyclic hydrocarbon, mol. wt. = 274 | 3 | 2.9 |
| Ethenyl-pentamethyl-naphthopyran (2 isomers) mol. wt. = 290 | 3 | 8.5 |
| Alkyl subst'd, multicyclic hydrocarbon, mol. wt. = 274 | 3 | 2.4 |
| Trimethyl-methylene-octahydro-phenanthrene | 2 | 11 |
| Alkyl subst'd, multicyclic hydrocarbon, mol. wt. = 274 | 3 | 3.7 |
| Trimethyl-isopropyl-cyclotetradecatetraene, mol. wt. = 272 | 3 | 3.7 |
| Pentamethyl-ethenyl-dodecahydro-naphthofuran-methanol, mol. wt. = 274 | 3 | 2.0 |

**RESULTS cont'd:**

**TABLE 2 cont'd: SUMMARY OF IDENTIFICATION BY GC/MS**

**SAMPLE:** Pulpmill Sludge, 03/06/98 - B/N extract cont'd **LAB SAMPLE #:** E8-03-641-01

**CONCENTRATION:** Semi-quantitation versus Phenanthrene $d_{10}$

**SPECIFIC COMPOUNDS IDENTIFIED**

| COMPOUND | CONFIDENCE OF IDENTIFICATION | APPROXIMATE CONCENTRATION µg/g (ppm) |
|---|---|---|
| Ethyl-trimethyl-methylene-dodecahydro-phenanthrene, mol. wt. = 274 | 3 | 11 |
| Labdadien-ol, mol. wt. = 290 | 4 | 9.0 |
| Alkyl subst'd, multicyclic hydrocarbon, mol. wt. = 286 | 4 | 8.2 |
| Dehydroabietic aldehyde, mol. wt. = 284 | 2 | 3.4 |
| Alkyl subst'd, multicyclic hydrocarbon, mol. wt. = 290 | 4 | 6.1 |
| Trimethyl-isopropyl-hexahydro-phenanthrone mol. wt. = 264 | 3 | 2.7 |
| Di-chlorine subst'd, alkyl subst'd multicyclic mol. wt. = 308 (2 isomers) | 4 | 5.6 |

The total ion chromatogram also contains an unresolved "hump" of material. The hump contains approximately 1000 ug/g of material.

E8-03-641 cont'd

**RESULTS:**

## TABLE 3: SUMMARY OF IDENTIFICATION BY GC/MS

**SAMPLE:** Pulpmill Sludge, 03/06/98 - Acid extract **LAB SAMPLE #:** E8-03-641-01 acid extract

**CONCENTRATION:** Semi-quantitation versus Phenanthrene $d_{10}$

### SPECIFIC COMPOUNDS IDENTIFIED

| COMPOUND | CONFIDENCE OF IDENTIFICATION | APPROXIMATE CONCENTRATION µg/g (ppm) |
|---|---|---|
| $C_4$ carboxylic acid | 1 | 4.7 |
| $C_5$ carboxylic acid (3 isomers) | 1-2 | 3.4 |
| $C_8$ carboxylic acid | 2 | 0.5 |
| Methoxy subst'd phenol | 2 | 0.5 |
| Ethyl subst'd phenol | 2 | 0.6 |
| Propyl subst'd phenol | 2 | 0.6 |
| $C_{10}$ carboxylic acid | 3 | 0.3 |
| Benzene propanoic acid | 1 | 0.6 |
| 4-Hydroxy-3-methoxy-benzaldehyde | 1 | 1.5 |
| $C_{15}$ carboxylic acid | 2 | 0.7 |
| $C_{16}$ carboxylic acid | 1 | 12 |
| $C_{17}$ carboxylic acid | 2 | 1.2 |
| Mono unsaturated $C_{18}$ carboxylic acid (2 isomers) | 1 | 3.4 |
| Subst'd alkyl phenol, mol. wt. = 214 | 4 | 10 |
| Trimethyl-ethenyl-dodecahydro-phenanthrene carboxylic acid, similar to pimaric acid, mol. wt. = 302 | 3 | 3.6 |
| Trimethyl-ethenyl-dodecahydro-phenanthrene carboxylic acid, mol. wt. = 302 | 3 | 4.4 |
| Alkyl subst'd, oxygen containing, unsaturated, multicyclic hydrocarbon, mol. wt. = 304 | 4 | 2.2 |
| Dimethyl-isopropyl-octahydro-phenanthrene carboxylic acid, mol. wt. = 300 (2 isomers) | 2 | 1.5 |