

# **ENVIRONMENTAL RESOURCE MANAGEMENT**

## **SOLID SAMPLES**

### **DIOXIN/FURAN ANALYSIS**

**AXYS METHOD: DX-S-8290/VER.2**

**Data Package:  
4184  
L4974 -1 to -5, -7**

**Prepared for:  
Rachel Leary  
Environmental Resource Management  
399 Boylston Street. 6<sup>th</sup> Floor  
Boston, MA 02116  
USA**

**Prepared by:  
AXYS Analytical Services Ltd.  
P.O. Box 2219, 2045 Mills Road  
Sidney, British Columbia V8L 3S8  
CANADA**

**Contact: Dr. M.C. Hamilton**

**DECEMBER 2002**

18 December 2002

## NARRATIVE

This narrative describes the analysis of six solid samples for the determination of polychlorinated dibenzodioxins and polychlorinated dibenzofurans.

### 1. SAMPLE RECEIPT AND STORAGE

Samples were received on the 15<sup>th</sup> August 2002. Details of sample conditions upon receipt are provided on the Sample Receiving Record form included in this data package. The samples were stored at -20°C prior to homogenization, extraction and analysis.

### 2. SAMPLE PREPARATION

Samples were homogenized as described on the Sample Preparation Record forms included in this data package. A portion of sample SS-21 (AXYS ID L4974-1) was taken and Matrix Spiked (MS) / Matrix Spike Duplicate (MSD) pair was prepared. Approximately 10g (dry weight) of each of the sample homogenates along with the MS/MSD were spiked with the labeled quantification standards and soxhlet extracted. The extracts were cleaned up using base acid wash followed by chromatographic cleanup using silver nitrate layered acid base silica, Florisil, carbon /Celite and alumina columns. The resulting extracts were spiked with labeled recovery (internal) standard prior to instrumental analysis.

### 3. ANALYSIS

- i. Sample and QC samples were analyzed in a single batch named, DXWG6796. The composition of the batch is shown on the Sample Batch Summary forms included with this data package.
- ii. Analysis procedures were in general accordance with 'USEPA Method 8290, as documented in AXYS Method DX-S-8290/Ver. 2
- iii. Instrumental analysis was conducted by high-resolution gas chromatography/high resolution mass spectrometry (HRGC/HRMS) using Autospec Ultima high resolution MS equipped with an HP 6890 gas chromatograph, a CTC auto-sampler, and an Alpha data system running Micromass software. A DB-5 (60m, 0.25mm i.d. 0.1 µm film thickness) chromatography column was coupled directly to the MS source. The MS was operated at a mass resolution of 10000 (static) in the electron impact ionization mode using multiple ion detection, acquiring the ions listed in Table 6 of USEPA Method 8290, for each target analyte and surrogate standard with the following exception.
  - AXYS routinely uses M/Z channels 354/356 and 366/368 to confirm and quantify the native and surrogate penta-substituted dioxins, respectively; this change from the method's specification has been made in the instrument method in order to avoid a persistent interference due to coplanar PCB's in the 356/358 and 368/370 M/Z channels. The amendment has been noted on each report page in the data package. The theoretical ratio for the P5CDD M/M+2 ions is 0.61; therefore, the acceptance range is 0.52 - 0.70.
- i. The qualitative identification procedures followed the criteria as set out in Method 8290, Section 7.8.4; the specifications refer to absolute and relative retention time, signal to noise ratio and relative abundance ratio. Chromatograms were visually examined and evaluated against the areas and ratios of PCDD/PCDF peaks as determined by the OPUSquan software. If the ratio determined by the software was just marginally out of specification, the chromatogram was

visually inspected to ensure that peaks were correctly integrated and peak areas were adjusted where correction was necessary.

- ii. Target concentrations were determined by isotope dilution using Micromass OPUSQUAN software. Congener peaks that did not meet the ion abundance ratio requirements were not included in the homologue total calculations.
- iii. The concentration for 2,3,7,8-TCDF detected in samples or lab blanks was confirmed by analysis using a DB-225 (30m, 0.25mm i.d., 0.15µm film thickness) chromatography column and this result is reported separately.
- iv. Sample specific detection limits (SDLs) were determined from the analysis data by converting the average noise signal to a concentration following the same procedures used to convert target peak responses to concentrations. In cases when the software selected unrepresentative area for the detection limit calculation, the data interpretation chemist and/or the QA chemist made corrections. These corrections are noted on the quantification report pages.

#### 4. REPORTING CONVENTIONS

- i. The AXYS contract number assigned for internal tracking was 4184. Samples were assigned a unique laboratory identifier of the form L4974-X, where 'X' are numerals; all data reports reference this unique AXYS ID plus the client sample identifier.
- ii. Any extra work required and performed after the initial instrumental analysis of the sample's extract is given an extra test suffix code. The single letter code per extra work performed is added to the AXYS sample ID as a suffix, and is combined with any other applicable test suffix codes:
  - i = instrumental reanalysis of sample extract
  - L = to remove interference extract was given further chromatographic cleanup
  - K = re-spike with the labeled standards
  - W = small dilution within the microvial followed by instrumental reanalysis
  - N = large dilution into a fresh microvial
- iii. Laboratory qualifier flags:
  - J = indicates an estimated value where the concentration of the analyte is less than the LMCL but greater than EDL.
  - EMPC = peak detected but did not meet confirmation criteria.
  - U = identifies a compound that was not detected
- iv. Results are reported in concentration units of pico grams per gram (pg/g, dry weight basis).

#### 5. QA/QC NOTES

Samples were analyzed in a single batch carried intact through the entire analytical process. The sample data were reviewed and evaluated in relation to the batch QC samples. Note that sample data are not blank corrected. All results fell within the quality acceptance specifications of the method and the contract.

#### 6. ANALYTICAL DISCUSSION

##### Analysis using DB-5 column

- Due to erratic separation observed using the carbon / Celite column all sample and QC sample extract fractions from the carbon / Celite column were re-combined and cleaned up using carbon/Celite, Florisil and alumina chromatographic columns. All the QC indicators indicated that data are not affected by this variance.

- Sample extract SS-21, SS-24 and the MS/MSD pairs (AXYS ID L4974-1 , -4, WG6796-103, and – 104 respectively) could not be reduced to dryness following chromatographic cleanup using the silver nitrate layered acid base silica columns. These sample extracts were given additional cleanup using gel permeation chromatographic columns.
- Due to the high levels of 1,2,3,4,6,7,8-HpCDD, OCDD, 1,2,3,4,6,7,8-HpCDF and 1,2,3,4,7,8,9-HpCDF present in sample SS-21 (AXYS ID L4974-1) the sample extract was diluted and re-spiked with another aliquot of the quantification standards. However, this dilution / re-spike analysis for the above noted analytes was not successful. As a result a portion of the original extract was given 20X dilution and the dilution data are reported. The MS/MSD pair prepared using this sample was similarly analyzed. This was communicated to the client and the relevant e-mail correspondences are included in this data package.
- To bring the response for some analytes within the linear calibration range of the instrument sample extracts SS-21, SS-22, SS-23, SS-24, SS-25 and the MS/MSD pair (AXYS ID L4974-1, to –5 and WG6797-103 / –104 pair respectively) were diluted and re-analyzed. The dilution factors are provided on the report forms and the affected analytes are separately reported from the dilution data.
- The Lab blank extract (AXYS ID WG6796-101) went to dryness during Rotary evaporation following the carbon/ Celite chromatographic cleanup. Given that the percent recovery values for all the labelled standards were within the method criteria data are not affected.

#### Analysis using DB-225 column

- Closing calibration verification associated with the initial injection analysis using DB-225 chromatographic column did not meet the method criteria. Consequently, the extracts were re-injected, the re-injection was not successful and all the extracts were re-injected and the second re-injection data met the method criteria. 2,3,7,8-TCDF concentrations are reported from the re-injection data.
- Due to concerns of instrumental carryover from preceding sample injections using the DB-225 column, sample extract SS-22 and SS-23 (AXYS ID L4974,-2 and -3) were re-injected and the re-injection data confirmed that there was no carryover. The original data are reported.

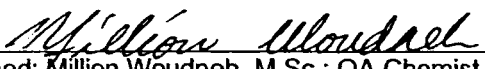
#### 7. SAMPLE NOTES

The analysis results for sample SS-26 (AXYS ID L4974-6) did not meet the method requirements. As a result repeat analysis is being conducted for this sample and the repeat analysis data will be submitted as soon as available.

#### 8. DATA PACKAGE

Included in the data package are narratives and supporting documentations, sample data, laboratory blanks, LCS, MS/MSD, and instrument QC reports.

**I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, for other than the conditions detailed above. In addition, I certify, that to the best of my knowledge and belief, the data as reported are true and accurate. Release of the data contained in this data package has been authorized by the Laboratory Manager or her designee, as verified by the following signature.**

  
Signed: Million Woudneh, M.Sc.; QA Chemist

  
Date Signed

# DX-DB5-1\_01

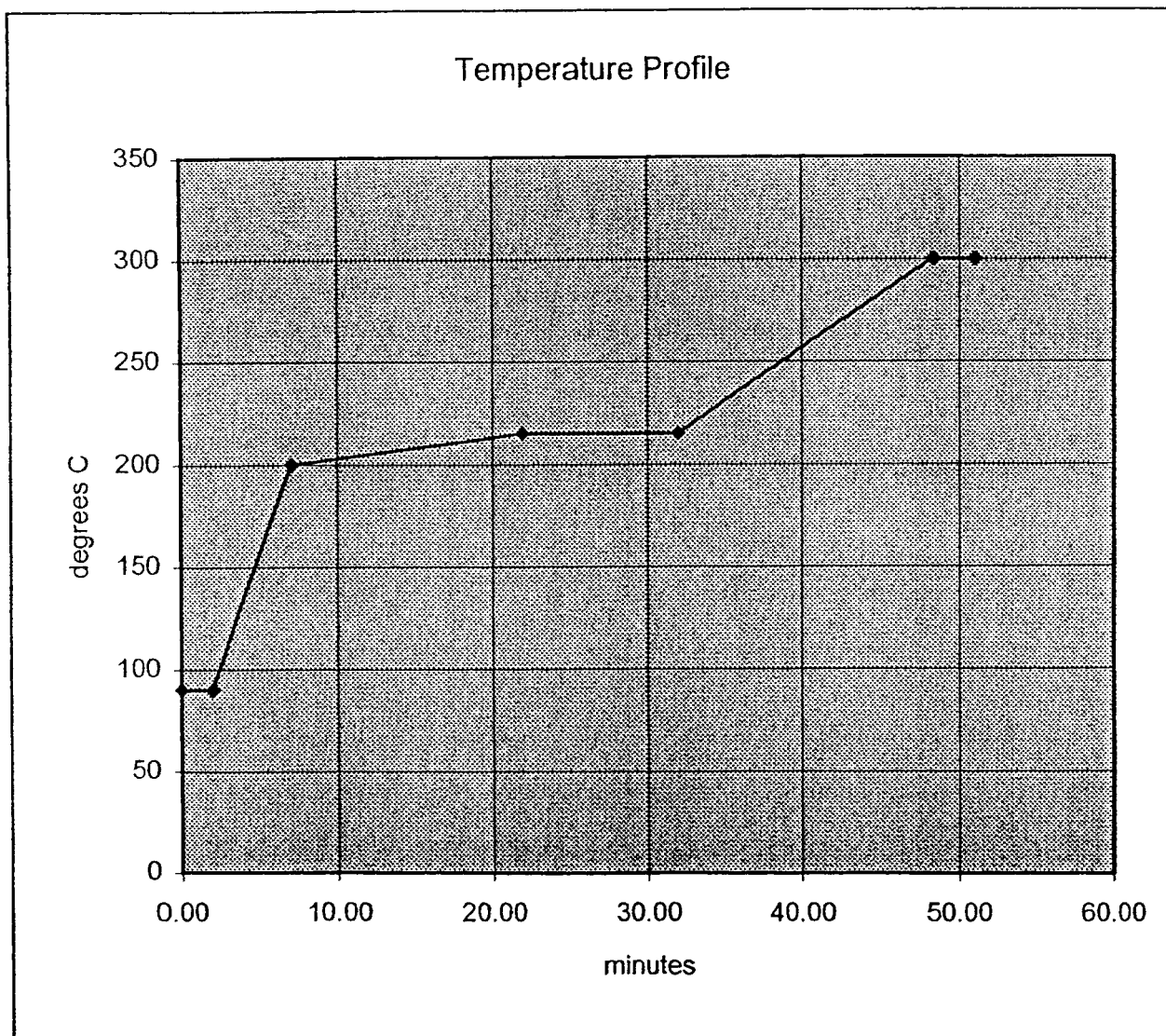
## COLUMN CONDITIONS:

60m DB-5 0.25mm id, 0.1um film, 5m guard column

	Temp	TIME/min.	TEMP/C
Temp	90		
Hold time	2		
Rate	22	0.00	90
Temp	200	2.00	90
Hold time	0	7.00	200
Rate	1	7.00	200
Temp	215	22.00	215
Hold time	10	32.00	215
Rate	5.2	48.35	300
Temp	300	51.05	300
Hold time	2.7		

## GC CONDITIONS:

Injector Temp/C	290
Splitless time/min.	2
He/kPa (new column)	-200
Maximum Temp/C	325



# DX-DB225-1\_01

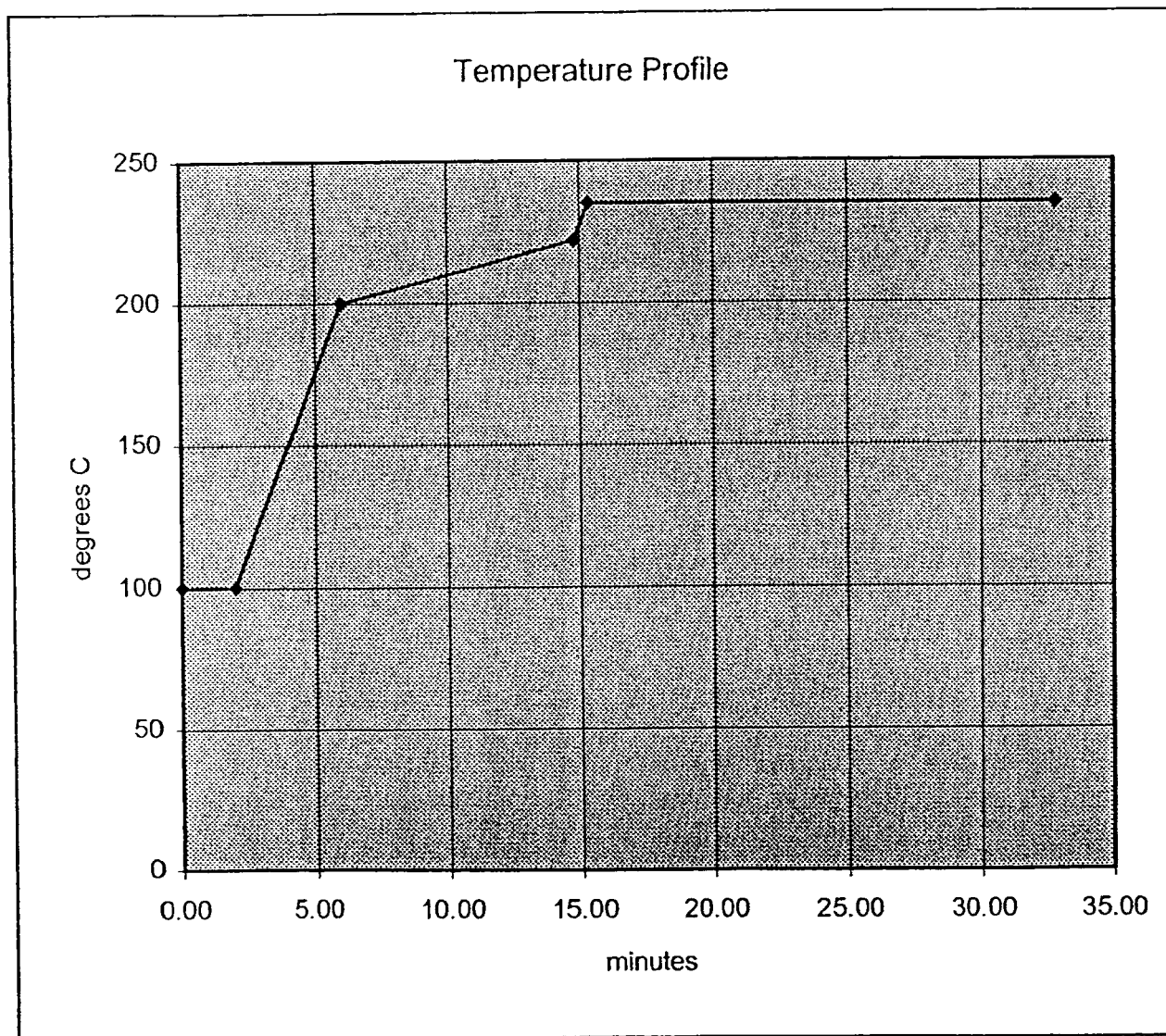
## COLUMN CONDITIONS:

30m DB-225 0.25mm id, 0.15um film, no guard column

## GC CONDITIONS:

	Temp	TIME/min.	TEMP/C
Temp	100		
Hold time	2		
Rate	25	0.00	100
Temp	200	2.00	100
Hold time	0	6.00	200
Rate	2.5	6.00	200
Temp	222	14.80	222
Hold time	0	14.80	222
Rate	25	15.32	235
Temp	235	32.82	235
Hold time	17.5		

Injector Temp/C	240
Splitless time/min.	2
He/kPa (new column)	-150
Maximum Temp/C	240



- 0005

Rachel Leary  
 Environmental Resource Management  
 399 Boylston Street, 6th Floor  
 Boston MA USA 02116

COVER PAGE - DIOXIN/FURAN ANALYSIS

Lab Name: AXYS Analytical Services Ltd.	Contract Number: 4184
Project No: NA	AXYS Methods: DX-S-8290/VER.2
Industrial Category: NA	Program: Solid Samples
Client Sample Number	Lab Sample Identification
LAB BLANK	WG6796-101
OPR	WG6796-102
SS-21	L4974-1 MS/MSD
SS-22	L4974-2
SS-23	L4974-3
SS-24	L4974-4
SS-25	L4974-5
DUP	L4974-7
Comments: Narrative Report is attached. (yes)	
<p>I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, for other than the conditions detailed in the Narrative Report. Release of the data contained in this hardcopy data package (and in the data submitted on magnetic media, if data are submitted on magnetic media), has been authorized by the Laboratory Manager or the Manager's designee, as verified by the following signature.</p>	
Signature: <i>Million Woudneh</i>	Name: Million Woudneh M.Sc.
Date: December 2002	Title: QA/QC Chemist





# CHAIN OF CUSTODY



Post Office Box 2219, 2045 Mills Road West  
Sidney, British Columbia, Canada V8L 3S8

AXYS CLIENT #:

ANALYSIS REQUESTED

Company: ERM  
Address: 399 Balmain St, Gm F1  
Boston, MA 02116  
USA  
Contact: Rachel Leary  
Phone: (617) 646-7841  
Fax: (617) 267-8377

Company: Same  
Address: \_\_\_\_\_  
Contact: \_\_\_\_\_  
Phone: \_\_\_\_\_  
Fax: \_\_\_\_\_

Project Name/Number: 143.72

Sampler's Name: Rachel Leary  
Signature: \_\_\_\_\_

Client Sample Identification	Matrix	Sampling Date	Sampling Time	Container Type/No	Axys Lab Sample ID (Lab use only)
SS-21	S	8/12/02	15:45	500ml jar	L4974-1
SS-22	S	8/12/02	15:20		-2
SS-23	S	8/12/02	15:20		-3
SS-24	S	8/12/02	16:00		-4
SS-25	S	8/12/02	14:30		-5
SS-20	S	8/12/02	14:50		-6
DUP	S	8/12/02	9:00	A	L4974-7

Received by (Signature)	Date	Time
<u>Rachel Leary</u>	8/12/02	3:45pm
<u>Rachel Leary</u>	15 Aug 02	10:00

Temp °C	Custody Seal #	Seal Intact	Sample Tags
		Y/N	Y/N

Received by (Signature)	Date	Time

Remarks:

SAMPLE RECEIVING RECORD

Waybill: Present <input checked="" type="checkbox"/> Absent		Waybill #: 828850130040	
Date Shipped: 13/Aug/02	Date Received: 15/Aug/02	Time Received: 10:00	
Received By (print): R Pingle		Signature: <i>[Signature]</i>	
Axy's Client and Contract #: 4184 ERM		Client Reference #:	
Condition of Shipping Container: Intact Cooler			
Temperature of Shipping Container on Receipt: 22°C on ice packs			
Custody Seals: Absent: <input checked="" type="checkbox"/> Present: <input type="checkbox"/> Intact <input type="checkbox"/> Broken <input type="checkbox"/>		Custody Seal Numbers: NA	
On: Shipping Container: <input type="checkbox"/> Sample Container: <input type="checkbox"/>			
Axy's Sample IDs: L4974-157			
Log-in by (print): R Pingle		Signature: <i>[Signature]</i>	
Chain of Custody or Documents: Present: <input checked="" type="checkbox"/> Absent: <input type="checkbox"/>		Traffic Report/Packing List: Y <input checked="" type="checkbox"/> N	
Sample IDs	<input checked="" type="checkbox"/> Y <input type="checkbox"/> N	Sample Tag Numbers	<input checked="" type="checkbox"/> Y <input type="checkbox"/> N
Location	<input checked="" type="checkbox"/> Y <input type="checkbox"/> N	Sample Type	<input checked="" type="checkbox"/> Y <input type="checkbox"/> N
Date & Time of Collection	<input checked="" type="checkbox"/> Y <input type="checkbox"/> N	Preservative Added	<input checked="" type="checkbox"/> Y <input type="checkbox"/> N
Collector's Name	<input checked="" type="checkbox"/> Y <input type="checkbox"/> N	Preservation Requested	<input checked="" type="checkbox"/> Y <input type="checkbox"/> N
			(details)
			(details)
Sample Acceptance Criteria:		Matrix Type: Solid	
Appropriate Container	<input checked="" type="checkbox"/> Y <input type="checkbox"/> N	Correct Labelling	<input checked="" type="checkbox"/> Y <input type="checkbox"/> N
Damaged Container	<input checked="" type="checkbox"/> Y <input type="checkbox"/> N	Holding Time Exceeded	<input checked="" type="checkbox"/> Y <input type="checkbox"/> N
Adequate Sample Size	<input checked="" type="checkbox"/> Y <input type="checkbox"/> N	Appropriate Temperature	<input checked="" type="checkbox"/> Y <input type="checkbox"/> N
Aqueous Samples: pH adjustment required Y/N		Residual Cl required Y/N	
Sample Tags: Present: <input type="checkbox"/> Absent: <input checked="" type="checkbox"/>		Sample Labels: Present: <input checked="" type="checkbox"/> Absent: <input type="checkbox"/>	
Sample Labels Cross Referenced to Chain of Custody <input checked="" type="checkbox"/> Y <input type="checkbox"/> N		Information Agrees Y <input checked="" type="checkbox"/> N	
Sample Tags Cross Referenced to Sample Labels Y/N		Information Agrees Y/N	
Sample Tags Cross-Referenced to Chain of Custody Y/N		Information Agrees Y/N	
Problems or Discrepancies: Solid samples should arrive @ ≤ 4°C Sample SS-22 does not have the same sample times on label + COC - COC states 15:20 label states 15:05			
Action Taken: Notified Laurie + PCs, client replied back to use label ID			



Axys Analytical Services Ltd.  
 Login Chain of Custody Report (In01)  
 Aug. 27, 2002  
 10:20 AM

Login Number: L4974  
 Account: 4184 ERM  
 Project:

AXYS ID	Client Sample Identification	Collect Date	Receive Date	Due PR Date
L4974-1	SS-21	12-AUG-02 15:45	15-AUG-02	14-SEP-02
	WIF-4,3C			
Solid	2:SOLIDS		:	USD
Solid	7:MOISTURE		:	USD
Solid	DX8290 (DB 5)		:	USD
Solid	DX8290 (DB225)		:	USD
Solid	HOMOGENIZATION		2 :500 ml Jar	USD
D.Package	DX DATA PKG		:	USD
D.Package	DX DATA PKG-CD		:	USD
ANY	DX MS		:	USD
ANY	DX MSD		:	USD
L4974-2	SS-22	12-AUG-02 15:05	15-AUG-02	14-SEP-02
	WIF-4,3C, client wants collection time from label used			
Solid	2:SOLIDS		:	USD
Solid	7:MOISTURE		:	USD
Solid	DX8290 (DB 5)		:	USD
Solid	DX8290 (DB225)		:	USD
Solid	HOMOGENIZATION		2 :500 ml Jar	USD
D.Package	DX DATA PKG		:	USD
D.Package	DX DATA PKG-CD		:	USD
L4974-3	SS-23	12-AUG-02 15:20	15-AUG-02	14-SEP-02
	WIF-4,3C			
Solid	2:SOLIDS		:	USD
Solid	7:MOISTURE		:	USD
Solid	DX8290 (DB 5)		:	USD
Solid	DX8290 (DB225)		:	USD
Solid	HOMOGENIZATION		2 :500 ml Jar	USD
D.Package	DX DATA PKG		:	USD
D.Package	DX DATA PKG-CD		:	USD

\*As part of S290 must be extracted by Sept 12 so as not exceed hold times:

Signature: Katrina Biss 0010  
 Date: Aug 27-02



Axys Analytical Services Ltd.  
 Login Chain of Custody Report (In01)  
 Aug. 27, 2002  
 10:20 AM

Login Number: L4974  
 Account: 4184 ERM  
 Project:

AXYS ID	Client Sample Identification	Collect Date	Receive Date	Due PR Date
L4974-4	SS-24	12-AUG-02 16:00	15-AUG-02	14-SEP-02
	WIF-4,3C			
	Solid 2:SOLIDS		:	USD
	Solid 7:MOISTURE		:	USD
	Solid DX8290 (DB 5)		:	USD
	Solid DX8290 (DB225)		:	USD
	Solid HOMOGENIZATION		2 : 500 ml Jar	USD
	D.Package DX DATA PKG		:	USD
	D.Package DX DATA PKG-CD		:	USD
L4974-5	SS-25	12-AUG-02 14:30	15-AUG-02	14-SEP-02
	WIF-4,3C			
	Solid 2:SOLIDS		:	USD
	Solid 7:MOISTURE		:	USD
	Solid DX8290 (DB 5)		:	USD
	Solid DX8290 (DB225)		:	USD
	Solid HOMOGENIZATION		2 : 500 ml Jar	USD
	D.Package DX DATA PKG		:	USD
	D.Package DX DATA PKG-CD		:	USD
L4974-6	SS-26	12-AUG-02 14:50	15-AUG-02	14-SEP-02
	WIF-4,3C			
	Solid 2:SOLIDS		:	USD
	Solid 7:MOISTURE		:	USD
	Solid DX8290 (DB 5)		:	USD
	Solid DX8290 (DB225)		:	USD
	Solid HOMOGENIZATION		2 : 500 ml Jar	USD
	D.Package DX DATA PKG		:	USD
	D.Package DX DATA PKG-CD		:	USD
L4974-7	DUP	12-AUG-02 09:00	15-AUG-02	14-SEP-02
	WIF-4,3C			
	Solid 2:SOLIDS		:	USD
	Solid 7:MOISTURE		:	USD
	Solid DX8290 (DB 5)		:	USD
	Solid DX8290 (DB225)		:	USD
	Solid HOMOGENIZATION		2 : 500 ml Jar	USD
	D.Package DX DATA PKG		:	USD
	D.Package DX DATA PKG-CD		:	USD

Signature: Katherine Balle  
 Date: Aug 27-02 - 0011



**TO:** Rachel Leary  
**FAX:** (617) 267-6447  
Environmental Resource Management

**DATE:** August 28, 2002  
**TOTAL PAGES:** 2

Please review this information and contact Laurie Phillips immediately if any problems exist. Unless instructed otherwise we will use this information:

Billing Address:  
Rachel Leary  
Environmental Resource Management  
399 Boylston Street, 6th floor  
Boston, MA, USA 02116

Reporting Address:  
Rachel Leary  
Environmental Resource Management  
399 Boylston Street, 6th floor  
Boston, MA, USA 02116  
rachel.leary@erm.com

**Sample(s) Received :** 15-AUG-2002

**ERM**

**Your Sample ID**

**AXYS Client Account No.:**

**4184**

**Product- Description**

**AXYS ID**

**SS-21 12-AUG-02 ( )**

**L4974-1**

Percent Solids Content	{2.SOLIDS}
Percent Moisture Content	{7.MOISTURE}
Dioxins/Furans EPA 8290 (DB5)	{DX8290 (DB 5)}
Dioxins/Furans EPA8290 (DB225)	{DX8290 (DB225)}
Homogenization of Sample	{HOMOGENIZATION}
Dioxin/Furan Data Package	{DX DATA PKG}
Dioxin/Furan Data Package on CD Rom	{DX DATA PKG-CD}
Dioxins/Furans MS	{DX MS}
Dioxins/Furans MSD	{DX MSD}

**SS-22 12-AUG-02 ( )**

**L4974-2**

Percent Solids Content	{2.SOLIDS}
Percent Moisture Content	{7.MOISTURE}
Dioxins/Furans EPA 8290 (DB5)	{DX8290 (DB 5)}
Dioxins/Furans EPA8290 (DB225)	{DX8290 (DB225)}
Homogenization of Sample	{HOMOGENIZATION}
Dioxin/Furan Data Package	{DX DATA PKG}
Dioxin/Furan Data Package on CD Rom	{DX DATA PKG-CD}

**SS-23 12-AUG-02 ( )**

**L4974-3**

Percent Solids Content	{2.SOLIDS}
Percent Moisture Content	{7.MOISTURE}
Dioxins/Furans EPA 8290 (DB5)	{DX8290 (DB 5)}
Dioxins/Furans EPA8290 (DB225)	{DX8290 (DB225)}
Homogenization of Sample	{HOMOGENIZATION}
Dioxin/Furan Data Package	{DX DATA PKG}
Dioxin/Furan Data Package on CD Rom	{DX DATA PKG-CD}

**SS-24 12-AUG-02 ( )**

**L4974-4**

Percent Solids Content	{2.SOLIDS}
Percent Moisture Content	{7.MOISTURE}
Dioxins/Furans EPA 8290 (DB5)	{DX8290 (DB 5)}
Dioxins/Furans EPA8290 (DB225)	{DX8290 (DB225)}
Homogenization of Sample	{HOMOGENIZATION}
Dioxin/Furan Data Package	{DX DATA PKG}



Sample(s) Received : 15-AUG-2002

ERM

Your Sample ID

AXYS Client Account No.:

4184

Product- Description	AXYS ID
SS-24 12-AUG-02 ()	L4974-4
Dioxin/Furan Data Package on CD Rom	(DX DATA PKG-CD)
SS-25 12-AUG-02 ()	L4974-5
Percent Solids Content	{2:SOLIDS}
Percent Moisture Content	{7:MOISTURE}
Dioxins/Furans EPA 8290 (DB5)	{DX8290 (DB 5)}
Dioxins/Furans EPA8290 (DB225)	{DX8290 (DB225)}
Homogenization of Sample	{HOMOGENIZATION}
Dioxin/Furan Data Package	{DX DATA PKG}
Dioxin/Furan Data Package on CD Rom	{DX DATA PKG-CD}
SS-26 12-AUG-02 ()	L4974-6
Percent Solids Content	{2:SOLIDS}
Percent Moisture Content	{7:MOISTURE}
Dioxins/Furans EPA 8290 (DB5)	{DX8290 (DB 5)}
Dioxins/Furans EPA8290 (DB225)	{DX8290 (DB225)}
Homogenization of Sample	{HOMOGENIZATION}
Dioxin/Furan Data Package	{DX DATA PKG}
Dioxin/Furan Data Package on CD Rom	{DX DATA PKG-CD}
DUP 12-AUG-02 ()	L4974-7
Percent Solids Content	{2:SOLIDS}
Percent Moisture Content	{7:MOISTURE}
Dioxins/Furans EPA 8290 (DB5)	{DX8290 (DB 5)}
Dioxins/Furans EPA8290 (DB225)	{DX8290 (DB225)}
Homogenization of Sample	{HOMOGENIZATION}
Dioxin/Furan Data Package	{DX DATA PKG}
Dioxin/Furan Data Package on CD Rom	{DX DATA PKG-CD}

Number:	Matrix:	Product Description:
7	Solid	Percent Solids Content
7	Solid	Percent Moisture Content
7	Solid	Dioxins/Furans EPA 8290 (DB5)
7	Solid	Dioxins/Furans EPA8290 (DB225)
7	Solid	Homogenization of Sample
7	D.Package	Dioxin/Furan Data Package
7	D.Package	Dioxin/Furan Data Package on CD Rom
1	Any	Dioxins/Furans MS
1	Any	Dioxins/Furans MSD

0013



4184

COMMERCIAL INVOICE

DATE OF EXPORTATION: 8/13/02	EXPORTER REFERENCE (i.e., order no., invoice no., etc.):
SHIPPER/EXPORTER (complete name and address):  ERM 399 Boylston St., 6 <sup>th</sup> Floor Boston, MA 02116 USA Attn: Rachel Leary	CONSIGNEE (complete name and address):  AXYS Analytical Services 2045 Mills Road West Sidney, BC CANADA V8L 3S8  Phone: 250-655-5800
Country of Export: USA	REASON FOR SHIPMENT:  <i>Samples for Analysis Only</i>  <i>No Commercial Value</i>
Country of Manufacture: USA	
Country of Ultimate Destination: CANADA	

International Air Waybill No.:

MARKS / Nos.	No. of PKGS	TYPE OF PACKAGING	FULL DESCRIPTION OF GOODS	Qty.	UNIT OF MEASURE	WEIGHT	UNIT VALUE	TOTAL VALUE
N/A	1	Cooler	Jars of Sediment	14	Lbs	30	\$1.00	\$14.00
						TOTAL WEIGHT		TOTAL INVOICE VALUE
						30 lbs		\$14.00

THESE COMMODITIES ARE LICENSED FOR THE ULTIMATE DESTINATION SHOWN. DIVERSION CONTRARY TO UNITED STATES LAW IS PROHIBITED.

I DECLARE ALL THE INFORMATION CONTAINED IN THIS INVOICE TO BE TRUE AND CORRECT.

SIGNATURE OF SHIPPER/EXPORTER (Type name and title, and sign).

DATE

*Rachel Leary*  
 \_\_\_\_\_  
 RACHEL LEARY  
 PROJECT ENGINEER

*8/13/02*  
 \_\_\_\_\_

0015



4184

COMMERCIAL INVOICE

DATE OF EXPORTATION: 8/13/02	EXPORTER REFERENCE (i.e., order no., invoice no., etc.):
SHIPPER/EXPORTER (complete name and address):  ERM 399 Boylston St., 6 <sup>th</sup> Floor Boston, MA 02116 USA Attn: Rachel Leary	CONSIGNEE (complete name and address):  AXYS Analytical Services 2045 Mills Road West Sidney, BC CANADA V8L 3S8  Phone: 250-655-5800
Country of Export: USA	REASON FOR SHIPMENT:  <i>Samples for Analysis Only</i>  <i>No Commercial Value</i>
Country of Manufacture: USA	
Country of Ultimate Destination: CANADA	

International Air Waybill No.:

MARKS / Nos.	No. of PKGS	TYPE OF PACKAGING	FULL DESCRIPTION OF GOODS	Qty.	UNIT OF MEASURE	WEIGHT	UNIT VALUE	TOTAL VALUE
N/A	1	Cooler	Jars of Sediment	14	Lbs	30	\$1.00	\$14.00
						TOTAL WEIGHT		TOTAL INVOICE VALUE
						30 lbs		\$14.00

THESE COMMODITIES ARE LICENSED FOR THE ULTIMATE DESTINATION SHOWN. DIVERSION CONTRARY TO UNITED STATES LAW IS PROHIBITED.

I DECLARE ALL THE INFORMATION CONTAINED IN THIS INVOICE TO BE TRUE AND CORRECT.

SIGNATURE OF SHIPPER/EXPORTER (Type name and title, and sign).

DATE

*Rachel Leary*

*8/13/02*

RACHEL LEARY  
PROJECT ENGINEER

### SAMPLE PREPARATION RECORD

Axys Sample ID: L4974-1 1 of 2 Axys Contract No.: 4184

Date: 30-AUG-02 Record Keeper: Quyen Do

Filing/Dissection: \_\_\_\_\_ Homogenization: QD 11:24  
Initials Time Initials Time

Client Label: SS-21

Sample Type: solid; sediment

Sample Description: - very wet, fine clay-like sediment; standing water present  
- original jar was broken  
- very fibrous w/ roots, leaves mixed in  
\* standing water was not weighed due to broken jar

Equipment Used: 4 mm sieve  Stainless bowl  Spatula  Scissors  Forceps  Shears  Knife   
 Blender: BO6  BO7  Grinder: GO1  GO2  GO3  GO4   
 Virtis: Large blade  Stator

Other \_\_\_\_\_

Procedure: - sample was transferred from broken jar into sieve and  
homogenized  
- all vegetations were crush with a spatula & forced through sieve  
- sample was thoroughly mixed with a spatula & returned  
to a new 500 ml amber jar

Single Sample:  Composite sample: \_\_\_\_\_

Sample weight before preparation: 499 g

Sample weight before preparation: \_\_\_\_\_

Sample weight before preparation: \_\_\_\_\_

Sample weight before preparation: \_\_\_\_\_

Sample weight before preparation: \_\_\_\_\_

Sample weight before preparation: \_\_\_\_\_

Sample weight before preparation: \_\_\_\_\_

Total sample weight after preparation: 368 g

Jars or Containers Filled: 1 x 500ml baked ICHM amber jar

<b>Verification Checklist:</b>			
Client/AXYS ID correlation <input checked="" type="checkbox"/>	Basic Information <input checked="" type="checkbox"/>	LIMS Updated <input checked="" type="checkbox"/>	Requested Work Completed <input checked="" type="checkbox"/>
Initial of Verification: <u>QD</u>		Date: <u>17-Sep-02</u>	

### SAMPLE PREPARATION RECORD

Axys Sample ID: L4974-2 1 of 2 Axys Contract No.: 4184  
 Date: 30- Aug-02 Record Keeper: Quyen Do  
 Filing/Dissection: \_\_\_\_\_ Homogenization: QD 10:30  
Initials Time Initials Time  
 Client Label: SS-22

Sample Type: solid; sediment

Sample Description: - original jar was broken  
- very wet; soft clay-like sediment  
- small pieces of well decomposed vegetations

Equipment Used: 4 mm sieve  Stainless bowl  Spatula  Scissors  Forceps  Shears  Knife   
 Blender: BO6  BO7  Grinder: GO1  GO2  GO3  GO4   
 Virtis: Large blade  Stator

Other \_\_\_\_\_

Procedure: - sample was carefully <sup>GD</sup> transferred from broken jar onto  
sieve; homogenized and collected in stainless bowl  
- all vegetations were pushed through sieve w/ a spatula  
- sample thoroughly mixed with a spatula & returned  
to 500ml amber jar

Single Sample:  Composite sample: \_\_\_\_\_

Sample weight before preparation: 658g  
 Sample weight before preparation: \_\_\_\_\_  
 Sample weight before preparation: \_\_\_\_\_  
 Sample weight before preparation: \_\_\_\_\_  
 Sample weight before preparation: \_\_\_\_\_  
 Sample weight before preparation: \_\_\_\_\_  
 Sample weight before preparation: \_\_\_\_\_  
 Total sample weight after preparation: 587g

Note: 119 g of sample was discarded to facilitate storage in WIF4 (less chance of jar breaking).  
 468 g of sample actually present.

Jars or Containers Filled: 1 x 500 ml ICHM baked amber jar

Verification Checklist:  
 Client/AXYS ID correlation  Basic Information  LIMS Updated  Requested Work Completed   
 Initial of Verification: EG Date: 17-Sep-02

0021

### SAMPLE PREPARATION RECORD

Axys Sample ID: L 4974-3, jar 1 of 2 Axys Contract No.: 4184  
 Date: 4-Sep-02 Record Keeper: Elza Perkins  
 Filing/Dissection: \_\_\_\_\_ Homogenization: EP 9.20  
Initials Time Initials Time  
 Client Label: SS-23

Sample Type: solid, sediment

Sample Description: - sample was black in colour, appeared to be homogeneous  
- Some vegetation was present in sample.

Equipment Used: 4 mm sieve  Stainless bowl  Spatula  Scissors  Forceps  Shears  Knife   
 Blender: BO6  BO7  Grinder: GO1  GO2  GO3  GO4   
 Virtis: Large blade  Stator

Other \_\_\_\_\_

Procedure: Sample was put through a 4mm sieve into a stainless bowl using a spatula. Vegetation present was cut into smaller pieces using scissors & forceps. One in bowl, sample was thoroughly mixed with a spatula and then transferred back into its jar.

Single Sample:  Composite sample: \_\_\_\_\_

Sample weight before preparation: 611g  
 Sample weight before preparation: \_\_\_\_\_  
 Sample weight before preparation: \_\_\_\_\_  
 Sample weight before preparation: \_\_\_\_\_  
 Sample weight before preparation: \_\_\_\_\_  
 Sample weight before preparation: \_\_\_\_\_

Balance used was # 105  
 Mettler BB 2440  
Jar # 1 of 2

Total sample weight after preparation: 585g

Note: 118g of sample was discarded to facilitate storage in WIF 4 (less chance of jar breaking). 467g of sample actually present.

Jars or Containers Filled: 1 x 500ml amber jar.

Verification Checklist:	
Client/AXYS ID correlation <input checked="" type="checkbox"/>	Basic Information <input checked="" type="checkbox"/> LIMS Updated <input checked="" type="checkbox"/> Requested Work Completed <input checked="" type="checkbox"/>
Initial of Verification: <u>EP</u>	Date: <u>17-Sep-02</u>

0022

**SAMPLE PREPARATION RECORD**

Axys Sample ID: L9974-4 1 of 2 Axys Contract No.: 4184  
 Date: 30-AUG-02 Record Keeper: Quyen Do  
 Filing/Dissection: \_\_\_\_\_ Homogenization: QD 9:04  
Initials Time Initials Time  
 Client Label: SS-24

Sample Type: solid ; sediment

Sample Description: - very wet, fine dark brown sediment  
- large pieces of roots & other vegetations well decomposed  
- original jar was broken

Equipment Used: 4 mm sieve  Stainless bowl  Spatula  Scissors  Forceps  Shears  Knife   
 Blender: B06  B07  Grinder: G01  G02  G03  G04   
 Virtis: Large blade  Stator

Other \_\_\_\_\_

Procedure: - sample carefully transferred from broken jar onto a  
sieve; homogenized & collected in a stainless bowl  
- sample was thoroughly mixed with a spatula &  
returned to original <sup>500 mL</sup> jar  
QD

Single Sample:  Composite sample: \_\_\_\_\_

Sample weight before preparation: 495 g  
 Sample weight before preparation: \_\_\_\_\_  
 Sample weight before preparation: \_\_\_\_\_  
 Sample weight before preparation: \_\_\_\_\_  
 Sample weight before preparation: \_\_\_\_\_  
 Sample weight before preparation: \_\_\_\_\_  
 Sample weight before preparation: \_\_\_\_\_

Total sample weight after preparation: 445 g

Jars or Containers Filled: 1 x 500 mL baked ICHM amber jar

**Verification Checklist:**  
 Client/AXYS ID correlation  Basic Information  LIMS Updated  Requested Work Completed   
 Initial of Verification: EQ Date: 17-Sep-02

- 0023

**SAMPLE PREPARATION RECORD**

Axys Sample ID: L4974-5 1 of 2 Axys Contract No.: 4184

Date: 30-AUG-02 Record Keeper: Queen D

Filing/Dissection: \_\_\_\_\_ Homogenization: QD 12:46  
Initials Time Initials Time

Client Label: SS-25

Sample Type: solid; sediment

Sample Description: - very wet, fine clay-like sediment  
- lots of fibre, leaves & decomposing stems  
- original jar broken

Equipment Used: 4 mm sieve  Stainless bowl  Spatula  Scissors  Forceps  Shears  Knife   
 Blender: BO6  BO7  Grinder: GO1  GO2  GO3  GO4   
 Virtis: Large blade  Stator

Other \_\_\_\_\_

Procedure: - sample was transferred from broken jar to sieve & homogenized  
- all vegetations were crushed with a spatula and forced through sieve  
- sample thoroughly mixed with a spatula before returning to a new 500ml amber jar

Single Sample:  Composite sample: \_\_\_\_\_

Sample weight before preparation: 527 g

Sample weight before preparation: \_\_\_\_\_

Sample weight before preparation: \_\_\_\_\_

Sample weight before preparation: \_\_\_\_\_

Sample weight before preparation: \_\_\_\_\_

Sample weight before preparation: \_\_\_\_\_

Sample weight before preparation: \_\_\_\_\_

Total sample weight after preparation: 451 g

Jars or Containers Filled: 1 x 500ml ICHER baked amber jar

<b>Verification Checklist:</b>			
Client/AXYS ID correlation <input checked="" type="checkbox"/>	Basic Information <input checked="" type="checkbox"/>	LIMS Updated <input checked="" type="checkbox"/>	Requested Work Completed <input checked="" type="checkbox"/>
Initial of Verification: <u>QD</u>	Date: <u>17-Sep-02</u>		

0024

### SAMPLE PREPARATION RECORD

Axys Sample ID: L4974-6 (1 of 2) Axys Contract No.: 4184

Date: 30-Aug-02 Record Keeper: Marta Marbella

Filing/Dissection: \_\_\_\_\_ Homogenization: MN 12:10  
Initials Time Initials Time

Client Label: SS-26  
8/12/02

Sample Type: Solid; sediment

Sample Description: - wet, soft, black, clumpy sediment containing small bits of vegetation. Original jar was broken. parts of sample in contact with the broken glass was carefully separated from the rest of the sample.

Equipment Used: 4 mm sieve  Stainless bowl  Spatula  Scissors  Forceps  Shears  Knife   
Blender: B06  B07  Grinder: G01  G02  G03  G04   
Virtis: Large blade  Stator

Other \_\_\_\_\_

Procedure: - initial sample weight obtained. Sample was put through a sieve and collected in a stainless steel bowl. Sample was mixed thoroughly in the bowl, then returned back to the original <sup>MN</sup> jar. Final sample weight was obtained.  
transferred to a new, pre-weighed baked jar.

Single Sample:  Composite sample: \_\_\_\_\_

Sample weight before preparation: 551 g  
Sample weight before preparation: \_\_\_\_\_  
Sample weight before preparation: \_\_\_\_\_  
Sample weight before preparation: \_\_\_\_\_  
Sample weight before preparation: \_\_\_\_\_  
Sample weight before preparation: \_\_\_\_\_  
Sample weight before preparation: \_\_\_\_\_

Total sample weight after preparation: 4.32 g

Jars or Containers Filled: 1x new baked 500 ml amber jar

Verification Checklist:  
Client/AXYS ID correlation  Basic Information  LIMS Updated  Requested Work Completed   
Initial of Verification: EG Date: 17-Sep-02

0025

### SAMPLE PREPARATION RECORD

Axys Sample ID: L4974-6 (2 of 2) Axys Contract No.: 4184

Date: 30-Aug-02 Record Keeper: Marta Mariella

Filing/Dissection: \_\_\_\_\_ Homogenization: UM 13:45

Client Label: SS-26 <sup>Initials</sup> 8/12/02 <sup>Time</sup>

Sample Type: solid, sediment

Sample Description: - wet, soft, black, clumpy sediment containing a few small bits of vegetation and garbage. Original jar was broken. Faint hydrocarbon odour present. Parts of sample in contact with the broken glass was carefully separated from the rest of the sample.

Equipment Used: 4 mm sieve  Stainless bowl  Spatula  Scissors  Forceps  Shears  Knife   
Blender: BO6  BO7  Grinder: GO1  GO2  GO3  GO4   
Virtis: Large blade  Stator

Other \_\_\_\_\_

Procedure: - initial sample weight obtained. Sample was put through a sieve and collected in a stainless steel bowl. Sample was mixed thoroughly in the bowl, then transferred to a new, pre-weighed baked jar. Final sample weight was obtained.

Single Sample:  Composite sample: \_\_\_\_\_

Sample weight before preparation: 548g  
Sample weight before preparation: \_\_\_\_\_  
Sample weight before preparation: \_\_\_\_\_  
Sample weight before preparation: \_\_\_\_\_  
Sample weight before preparation: \_\_\_\_\_  
Sample weight before preparation: \_\_\_\_\_  
Sample weight before preparation: \_\_\_\_\_  
Total sample weight after preparation: 475g

Jars or Containers Filled: 1x new baked 500 ml amber jar

**Verification Checklist:**  
Client/AXYS ID correlation  Basic Information  LIMS Updated  Requested Work Completed   
initial of Verification: OG Date: 17-Sep-02

0026



### SAMPLE PREPARATION RECORD

Axys Sample ID: L4974-7 1 of 2 Axys Contract No.: 4184

Date: 30-AUG-02 Record Keeper: Quyen Do

Filing/Dissection: \_\_\_\_\_ Homogenization: QD 14:00  
Initials Time Initials Time

Client Label: DUP

Sample Type: solid; sediment

Sample Description: - very wet; soft clay-like sediment  
- fibrous, dense with lots of roots and pieces of leaves  
- original jar was broken (lid)

Equipment Used: 4 mm sieve  Stainless bowl  Spatula  Scissors  Forceps  Shears  Knife   
Blender: BO6  BO7  Grinder: GO1  GO2  GO3  GO4   
Virtis: Large blade  Stator

Other \_\_\_\_\_

Procedure: - sample was homogenized through sieve and collected  
in a stainless bowl  
- all vegetations were crushed with a spatula & pushed  
through sieve  
- sample was thoroughly mixed w/ a spatula + returned to original jar

Single Sample:  Composite sample: \_\_\_\_\_

Sample weight before preparation: 694g

Sample weight before preparation: \_\_\_\_\_

Sample weight before preparation: \_\_\_\_\_

Sample weight before preparation: \_\_\_\_\_

Sample weight before preparation: \_\_\_\_\_

Sample weight before preparation: \_\_\_\_\_

Sample weight before preparation: \_\_\_\_\_

Total sample weight after preparation: 603g

Note: 119 g of sample was discarded to facilitate storage in WIF4 (less than of jar breaking). 484 g of sample actually present. MM

Jars or Containers Filled: 1 x original 500ml amber jar

**Verification Checklist:**  
Client/AXYS ID correlation  Basic Information  LIMS Updated  Requested Work Completed   
Initial of Verification: QD Date: 17-Sep-02

0027



25  
T1047

Fraction: Tray # 96

# BATCH LIST

Analysis Type: DIOXINS (8290)		Batch ID: DXWG6796	
		Blank: WG6796-101 BLK BAKED NA2S04	
		Reference or Spike: WG6796-102 SPM	
		10G 8SREF SPIKED WITH 20UL DX012B-AUT/02 #35/10	
		Matrix Spike: WG6796-103 MS L	
Matrix: Solids		46.5g L4974-1 spiked with 20uL DX012B-AUT/02 #35/10	
Method ID: DX-S-8290/Ver.2		Matrix Spike Duplicate: WG6796-104 MSD L	
Analyst: Jas Hundal		46.5g L4974-1 spiked with 20uL DX012B-AUT/02 #35/10	
Date Due: 15-Sep-02		Samples: (4184)	
Date Started: 10-Sep-02		L4974-1L 46.5g Re-combine	
Date Finished:		L4974-2L 25.0g	
		L4974-3L 24.5g	
Surrogate:		L4974-4L 36.0g	
DX013A-SUR/06 #35/10, 20uL		L4974-5L 35.5g	
		L4974-6L 34.0g	
		L4974-7L 23.5g FIELD DUPLICATE	
Recovery:			
DX005A-REC/18 #35/10, 20uL		20uL	
Calibration:		SAR: WG6796-105 SAR #35/10	
DX018C-CAL/		20uL DX013A-SUR/06 + 20UL DX012B-AUT/02 +	
		20UL DX005A-REC/18 FV=20UL	
<p>Comments:</p> <p>10g dry weight -- use weights as above</p> <p>% moisture required</p> <p>USE LARGE SOXHLET GLASSWARE AND USE EXTRA NA2S04 TO DRY THE LARGE SAMPLE SIZE</p> <p>-SOXHLET IN 600ML 80:20 TOLUENE/ACETONE NK (100x, surrogate respite) L4974-1, wg6796-103, wg6796-104</p> <p>ALSO - DON'T FORGET TO PRE-SOXHLET YOUR GLASSWARE</p> <p>Clean up c/c EI : combine with existing extract. EA 25-Sept-02</p> <p>(c/c #106 was used)</p> <p>Repeat L4974-6L low sur rec.</p> <p>NK (100x dil and respite sur) L4974-1, wg6796-103, -104 to 28 Nov 02 to 9 Dec 02</p>			
QA/QC SPECIFICATIONS (State if different from AXYS Criteria)	PASS Y N	Checked By	QA/QC SPECIFICATIONS (State if different from AXYS Criteria)
BLANK:			PASS Y N
REFERENCES			Checked By
DUPLICATES			SURROGATE RECs
			DETECTION LIMITS

Dil L4974-2L 2.5X oCDD OLR to 9 Dec 02

Redo NK from original L4974-1, wg6796-103, -104 to 13 Dec 02

Dil original L4974-1, wg6796-103, -104 (Lnd) to 13 Dec 02

0029

DIOXIN/FURAN SAMPLE ANALYSIS RECORD

Method #s: Pulp: DX-P-01/Ver.4 Sludge: DX-SL-01/Ver.4, DX-SL-8290/Ver.2 Filter/Pufs: DX-F-01/Ver.2  
Sediment: DX-S-01/Ver.4, DX-S-8290/Ver.2 Wood Chips: DX-WC-01/Ver.4 Black Liquor: DX-L-01/Ver.2

Level: DC \_\_\_\_\_ MDT \_\_\_\_\_ HR \_\_\_\_\_ LR \_\_\_\_\_ Due Date: 15 SEPT 02  
 Sample ID: WG 6796 - 101 BLK # 10 SEPT 02 Date: 10 SEPT 02 Analyst: JAS HUNDAK BATCH #: DXWG 6796  
 Blank: WG 6796 - 101 BLK Rec'd: \_\_\_\_\_ Back-up: \_\_\_\_\_ g Stored In: \_\_\_\_\_  
 Original Labelling: POWDER Na<sub>2</sub>SO<sub>4</sub> LAB 2

Sample Type and Description: PROCEDURAL BLANK

Sample Pre-Treatment at Axys: \_\_\_\_\_ Date: \_\_\_\_\_ Homogenizer: \_\_\_\_\_  
 at other: \_\_\_\_\_ Specifics: \_\_\_\_\_

AUTHENTIC STANDARD

	name	volume	date	<u>14:34</u> time	<u>JH</u> analyst
Sample Weight:	Wet <u>78.67</u> g	Moisture: <u>0%</u>		Tare: <u>0.95</u> g	
		Dry <u>78.67</u> g		Dry + Tare: <u>0.95</u> g	
				Wet + Tare: <u>0.95</u> g	
				Dry/Wet Ratio: <u>0</u>	

SURROGATE STANDARD ID	<u>DX013A-SUR106</u>	<u>20uL</u>	<u>11 SEPT 02</u>	<u>14:30</u>	<u>JH</u>
	name	quantity	date	time	analyst

Extraction:

Start:	<u>11 SEPT 02</u>	<u>17:45</u>	<u>JH</u>	End:	<u>12 SEPT 02</u>	<u>9:00</u>	<u>d.a.</u>
	date	time	analyst		date	time	analyst

Cleanup:

Base/Acid Wash: 1 B 1 W 2 A 1 W 12 SEPT 02 d.a.

Chromatography: Cu rxn: 1 meq. 2

Procedure No.

( ) Ag/AB Silica Gel	(Batch # <u>170</u> )	<u>12 SEPT 02</u>	<u>d.a.</u>
( ) Florisil	(Batch # <u>47B</u> )	<u>13 SEPT 02</u>	<u>JH</u>
( ) Carbon/celite	(Batch # <u>106</u> )	<u>13 SEPT 02</u>	<u>JH</u>
( ) Alumina	(Batch # <u>226</u> )	<u>14 SEPT 02</u>	<u>d.a.</u>
		date	analyst

RECOVERY STANDARD ID	<u>DX005A-REC18</u>	<u>20uL</u>	<u>14 SEPT 02</u>	<u>3:00</u>	<u>d.a.</u>
	name	quantity	date	time	analyst

Final microvial volume: 20uL

Additional Work

Action:	Reason:	GC Data File:	Requested by/date:	Completed in lab by/date:	Run on Instr. by/date:
<u>Clean up c/c EI; combine with existing extract</u>	<u>Low TCDP rec</u>	<u>3_243</u>	<u>EA 25-sept-02</u>	<u>WN 16 Oct 02</u>	<u>#261 16 Nov. 02</u>

Comments

-went dry on the rotavap after carbon celite when rotavap spiked to ~48°C.  
Carbon-Celite #107 11 Oct 02; Florisil #48A 15 Oct 02; Alumina #228 16 Oct 02 WJH  
Recombined GC EI into existing μ-vial. 16 Oct 02 WJH

DIOXIN/FURAN SAMPLE ANALYSIS RECORD

Method #s: Pulp: DX-P-01/Ver.4 Sludge: DX-SL-01/Ver.4, DX-SL-8290 / Ver. 2 Filter/Pufs: DX-F-01/Ver.2  
 Sediment: DX-S-01/Ver.4, DX-S-8290/Ver.2 Wood Chips: DX-WC-01/Ver.4 Black Liquor: DX-L-01/Ver.2

Level: DC \_\_\_\_\_ MDT \_\_\_\_\_ HR \_\_\_\_\_ LR \_\_\_\_\_ Due Date: 15 SEPT. 02

Sample ID: WG6796-10A SPM Date: 10 SEPT. 02 Analyst: JAS HUNDAL BATCH #: DXWG 6796

Blank: WG6796-10 BLK Rec'd: \_\_\_\_\_ Back-up: \_\_\_\_\_ g Stored In: \_\_\_\_\_

Original Labelling: 95 REF 24-MARCH 1999 NOT SEXHALETED  
SEDIMENT ~~NOT~~ REFERENCE MATERIAL

Sample Type and Description: SPIKED MATRIX

Sample Pre-Treatment at Axys: \_\_\_\_\_ Date: \_\_\_\_\_ Homogenizer: \_\_\_\_\_  
 at other: \_\_\_\_\_ Specifics: \_\_\_\_\_

AUTHENTIC STANDARD DX02B-AUT/02 20ul 11 SEPT. 02 14:34 JH  
 name volume date time analyst

Sample Weight: Wet 10.73 g Moisture: 4.8% Tare: 0.95 g  
 Dry 10.23 g Dry + Tare: 2.35 g  
 Wet + Tare: 2.42 g  
 Dry/Wet Ratio: 1.40/1.47 = 0.95

SURROGATE STANDARD ID DX02A-SUR/06 20ul 11 SEPT. 02 14:31 JH  
 name quantity date time analyst

Extraction: Start: 11 SEPT. 02 17:45 JH End: 12 SEPT. 02 9:00 J.A.  
 date time analyst date time analyst

Cleanup: Base/Acid Wash: 1 B 1 W 2 A 1 W 12 SEPT. 02 J.A.  
 Chromatography: Cu rxn: 1 mg. 2  
 Procedure No.

- ( ) Ag/AB Silica Gel (Batch # 170) 12 SEPT. 02 J.A.
- ( ) Florisil (Batch # 47B) 13 SEPT. 02 JH
- ( ) Carbon/celite (Batch # 106) 13 SEPT. 02 JH
- ( ) Alumina (Batch # 226) 14 SEPT. 02 J.A.

RECOVERY STANDARD ID DX005A-REC/18 20ul 14 SEPT. 02 3:01 J.A.  
 name quantity date time analyst

Final microvial volume: 20ul

Additional Work Action:	Reason:	GC Data File:	Requested by/date:	Completed in lab by/date:	Run on Instr. by/date:
<u>clean up ck EI &amp; combine with existing extract</u>	<u>LOW TCDF surr rec</u>	<u>3-243</u>	<u>EA 25-Sept-02</u>	<u>WJH 16 Oct 02</u>	<u>#2 16 Nov. 02</u>

Comments

F Carbon-Celite #107/1 Oct 02; Florisil #48A 15 Oct 02; Alumina #228 16 Oct 02. WJH  
Recombined GC EI with original microvial 16 Oct 02. WJH

0031

# DIOXIN/FURAN SAMPLE ANALYSIS RECORD

Method #: Pulp: DX-P-01/Ver.4 Sludge: DX-SL-01/Ver.4, DX-SL-8290 / Ver. 2 Filter/Pufs: DX-F-01/Ver.2  
 Sediment: DX-S-01/Ver.4 (DX-S-8290/Ver.2) Wood Chips: DX-WC-01/Ver.4 Black Liquor: DX-L-01/Ver.2

Level: DC \_\_\_\_\_ MDT <sup>15 DEPT 02</sup> ~~10 SEPT 02~~ HR \_\_\_\_\_ LR \_\_\_\_\_ Due Date: 15 SEPT 02  
 Sample ID: WG6796-103 MS <sup>10 SEPT 02</sup> Date: 10 SEPT 02 Analyst: JAS HUNDAL BATCH #: DXWG 6796  
 Blank: WG6796-10 BLK Rec'd: 15 Aug 02 Back-up: 7100g g Stored In: \_\_\_\_\_  
 Original Labelling: SS-21

Sample Type and Description: Solids - Black, tar-like, mushy matter. MATRIX SPIKE

Sample Pre-Treatment at Axys:  Date: 30 Aug 02 Homogenizer: (Q1)  
 at other: \_\_\_\_\_ Specifics:  homo, sur transfer

**AUTHENTIC STANDARD** DX012B-Ant/02 20 uL 11 SEPT 02 14:36 RAH  
 name volume date time analyst

Sample Weight: Wet 48.72 g Moisture: 77.8% Tare: 0.96 g  
 Dry + Tare: 1.30 g  
 Dry 10.83 g Wet + Tare: 2.49 g  
 Dry/Wet Ratio: 0.34/1.53 = 0.22

**SURROGATE STANDARD ID** DX013A-154/06 20 uL 11 SEPT 02 14:32 RAH  
 name quantity date time analyst

Extraction: Start: 11 SEPT 02 17:45 RAH End: 12 SEPT 02 9:00 RAH  
 date time analyst date time analyst

Cleanup: Base/Acid Wash: 2 B 3 W 10 A 1 W 12 SEPT 02 RAH  
 Chromatography: Cu rxn: 1 mag 2  
 Procedure No.

- (3x) Ag/A/B Silica Gel (Batch # 170) 12 SEPT 02 RAH
- ( ) Florisil (Batch # 47B) 13 SEPT 02 JH
- ( ) Carbon/celite (Batch # 106) 13 SEPT 02 JH
- ( ) Alumina (Batch # 226) 14 SEPT 02 RAH

**RECOVERY STANDARD ID** DX005A-REC/18 20 uL 14 SEPT 02 2:48 RAH  
 name quantity date time analyst

Final microvial volume: 20 uL

Additional Work Action:	Reason:	GC Data File:	Requested by/date:	Completed in lab by/date:	Run on Instr. by/date:
<u>Clean up E/C Et combine with existing extract</u>	<u>Low TCDF</u>	<u>3-243</u>	<u>EA 25 SEPT 02</u>	<u>WJ/16 OCT 02</u>	<u>13 OCT NOV 17 02</u>

Comments: - used large beasil columns to double the amount of silica powder used.  
- couldn't blow sample down to dryness after silica columns.

Protobead column # 80 16 SEPT 02 MBR. 0032



### DIOXIN/FURAN SAMPLE ANALYSIS RECORD

Method #: Pulp: DX-P-01/Ver.4 Sludge: DX-SL-01/Ver.4, DX-SL-8290/Ver.2 Filter/Pufs: DX-F-01/Ver.2  
Sediment: DX-S-01/Ver.4 DX-S-8290/Ver.2 Wood Chips: DX-WC-01/Ver.4 Black Liquor: DX-L-01/Ver.2

Level: DC \_\_\_\_\_ MDT <sup>12 Dec 02</sup> \_\_\_\_\_ HR \_\_\_\_\_ LR \_\_\_\_\_ Due Date: 15 SEPT 02  
 Sample ID: WG6796-104 MSD <sup>ENK2</sup> Date: 10 SEPT 02 Analyst: JAS HUNDAL BATCH #: DXWG 6796  
 Blank: WG6796-104 BLK Rec'd: 15 Aug 02 Back-up: 7 100g g Stored In: \_\_\_\_\_  
 Original Labelling: SS-21

Sample Type and Description: MATRIX SPIKE DUPLICATE - Solid. - Black, bar-like, mushy matter

Sample Pre-Treatment at Axys:  Date: 30 AUG 02 Homogenizer: QD  
 at other: \_\_\_\_\_ Specifics: Homo, Sur transfer

AUTHENTIC STANDARD DX013B-Aut102 20mL 10 SEPT 02 14:37 AH  
 name volume date time analyst

Sample Weight: Wet 47.58 g Moisture: 78.3% Tare: 0.96 g  
 Dry 10.32 g Dry + Tare: 1.32 g  
 Wet + Tare: 2.62 g  
 Dry/Wet Ratio: 0.36/1.66 = 0.21

SURROGATE STANDARD ID DX013A-Sur105 20uL 11 SEPT 02 14:33 NA  
 name quantity date time analyst

Extraction: Start: 11 SEPT 02 17:45 AH End: 12 Sept 02 9:00 A.A.  
 date time analyst date time analyst

Cleanup: Base/Acid Wash: 2 B 3 W 10 A 1 W 12 Sept 02 A.A.  
 Chromatography: Cu rxn: 1 mag. 2  
 Procedure No.

(3x) Ag/AB Silica Gel	(Batch # <u>170</u> )	<u>12 Sept 02</u>	<u>A.A.</u>
( ) Florisil	(Batch # <u>47B</u> )	<u>13 Sept 02</u>	<u>JH</u>
( ) Carbon/celite	(Batch # <u>106</u> )	<u>13 Sept 02</u>	<u>JH JH</u>
( ) Alumina	(Batch # <u>226</u> )	<u>14 Sept 02</u>	<u>A.A.</u>
		date	analyst

RECOVERY STANDARD ID DX005A-REC/18 20uL 14 Sept 02 2:50 A.A.  
 name quantity date time analyst

Final microvial volume: 20uL

Additional Work Action:	Reason:	GC Data File:	Requested by/date:	Completed in lab by/date:	Run on Instr. by/date:
<u>Clean up c/c EI &amp; combine with existing extract</u>	<u>Low TCM sur roc</u>	<u>3-243</u>	<u>EA 25-Sept-02</u>	<u>WJN/16 Oct 02</u>	<u>#2 KC MW/17</u>

Comments: - used large beasil columns to double the amount of silica powder used. 0034  
16 Sep 02 MBR - couldn't blow sample to dryness after silica columns.





# DIOXIN/FURAN SAMPLE ANALYSIS RECORD

Method #s: Pulp: DX-P-01/Ver.4 Sludge: DX-SL-01/Ver.4, DX-SL-8290 / Ver. 2 Filter/Pufs: DX-F-01/Ver.2  
Sediment: DX-S-01/Ver.4, DX-S-8290/Ver.2 Wood Chips: DX-WC-01/Ver.4 Black Liquor: DX-L-01/Ver.2

Level: DC \_\_\_\_\_ MDT \_\_\_\_\_ HR \_\_\_\_\_ LR \_\_\_\_\_ Due Date: 15 SEPT. 02  
 Sample ID: L4974-1 LNK2 Date: 10 SEPT. 02 Analyst: JAS HUNDAL BATCH #: DXWG 6796  
 Blank: WG6796-10 BLK Rec'd: 15 Aug. 02 Back-up: 7509 g Stored In: \_\_\_\_\_  
 Original Labelling: SS-21

Sample Type and Description: Solid Sediment - Black, tar-like, mushy matter

Sample Pre-Treatment at Axy's:  Date: 30 Aug. 02 Homogenizer: QD  
 at other: \_\_\_\_\_ Specifics: Homo, jar transfer

**AUTHENTIC STANDARD**

	name	volume	date	time	analyst
Sample Weight:	Wet <u>48.10</u> g	Moisture: <u>79.1%</u>		Tare: <u>0.96</u> g	
		Dry <u>10.03</u> g		Dry + Tare: <u>1.35</u> g	
				Wet + Tare: <u>2.83</u> g	
				Dry/Wet Ratio: <u>0.39/1.87 = 0.20</u>	

SURROGATE STANDARD ID	<u>DX03A-Sur106</u>	<u>20 uL</u>	<u>11 SEPT. 02</u>	<u>14:34</u>	<u>JA</u>
	name	quantity	date	time	analyst

Extraction :  
 Start: 11 SEPT. 02 17:45 JA End: 12 Sept. 02 9:00 JA  
 date time analyst date time analyst

Cleanup:  
 Base/Acid Wash: 3 B 3 W 10 A 1 W 12 Sept 02 J.A.  
 Chromatography: Cu rxn: 1 mg. 2  
 Procedure No.

( <u>3x</u> ) Ag/AB Silica Gel	(Batch # <u>170</u> )	<u>12 Sept 02</u>	<u>JA</u>
( ) Florisil	(Batch # <u>47B</u> )	<u>13 Sept. 02</u>	<u>JH</u>
( ) Carbon/celite	(Batch # <u>106</u> )	<u>13 Sept. 02</u>	<u>JH</u>
( ) Alumina	(Batch # <u>226</u> )	<u>14 Sept. 02</u>	<u>JA</u>
		date	analyst

RECOVERY STANDARD ID	<u>DX005A-REC/18</u>	<u>20 uL</u>	<u>14 Sept. 02</u>	<u>2:45</u>	<u>JA</u>
	name	quantity	date	time	analyst

Final microvial volume: 20 uL

Additional Work	GC Data	Requested	Completed in	Run on Instr.
Action:	File:	by/date:	lab by/date	by/date
<u>Clean up c/c Et; combine with existing extract</u>	<u>low TCDF sur rec</u>	<u>3-243</u>	<u>EA 25-Sept-02</u>	<u>WJH 16 Oct 02</u>
<u>1.0 NK (100%)</u>	<u>1.0 NK (100%)</u>	<u>2-418</u>	<u>25 Nov 02</u>	

**Comments**

Dioxin column # 79  
16 Sep. 02 MBR.  
 - used large basal columns to double the amount of silica used.  
 - couldn't blow samples to dryness after silica columns.



# DIOXIN/FURAN SAMPLE ANALYSIS RECORD

Method #: Pulp: DX-P-01/Ver.4 Sludge: DX-SL-01/Ver.4, DX-SL-8290/Ver.2 Filter/Pufs: DX-F-01/Ver.2  
Sediment: DX-S-01/Ver.4, DX-S-8290/Ver.2 Wood Chips: DX-WC-01/Ver.4 Black Liquor: DX-L-01/Ver.2

Level: DC \_\_\_\_\_ MDT \_\_\_\_\_ HR \_\_\_\_\_ LR \_\_\_\_\_ Due Date: 15 SEPT. 02  
 Sample ID: L4974-2L Date: 10 SEPT. 02 Analyst: JAS HUNDAL BATCH #: DXWG 6796  
 Blank: WG6796-10 BLK Rec'd: 15 Aug. 02 Back-up: 71009 g Stored In: \_\_\_\_\_  
 Original Labelling: SS-2A

Sample Type and Description: Sediment - Black/brown, thick, tar-like matter.

Sample Pre-Treatment at Axys:  Date: 30 Aug. 02 Homogenizer: ND  
 at other: \_\_\_\_\_ Specifics: Home, Jar transfer

**AUTHENTIC STANDARD**

	name	volume	date	time	analyst
Sample Weight:	Wet <u>25.69</u> g	Moisture: <u>59.4%</u>		Tare: <u>0.96</u> g	
		Dry <u>10.45</u> g		Dry + Tare: <u>1.68</u> g	
				Wet + Tare: <u>2.73</u> g	
				Dry/Wet Ratio: <u>0.72/1.77 = 0.40</u>	

SURROGATE STANDARD ID	name	quantity	date	time	analyst
<u>DX03A-Sur106</u>		<u>20ul</u>	<u>11 SEPT. 02</u>	<u>14:36</u>	<u>JA</u>

Extraction: Start: 11 SEPT. 02 17:45 JA End: 12 SEPT. 02 9:00 JA  
 date time analyst date time analyst

Cleanup: Base/Acid Wash: 3 B 3 W 6 A 1 W 12 SEPT. 02 JA  
 Chromatography: Cu rxn: 1 neg. 2  
 Procedure No.

( ) Ag/AB Silica Gel	(Batch # <u>170</u> )	<u>12 SEPT. 02</u>	<u>JA</u>
( ) Florisil	(Batch # <u>473</u> )	<u>13 SEPT. 02</u>	<u>JA</u>
( ) Carbon/celite	(Batch # <u>106</u> )	<u>13 SEPT. 02</u>	<u>JA</u>
( ) Alumina	(Batch # <u>226</u> )	<u>14 SEPT. 02</u>	<u>JA</u>

RECOVERY STANDARD ID	name	quantity	date	time	analyst
<u>DX05A-REC/18</u>		<u>20ul</u>	<u>14 SEPT. 02</u>	<u>2:45</u>	<u>JA</u>

Final microvial volume: 20ul

Additional Work Action:	Reason:	GC Data File:	Requested by/date:	Completed in lab by/date:	Run on Instr. by/date:
<u>Clean up c/c El &amp; combine with existing extract</u>	<u>Low TDF sur rec</u>	<u>3-243</u>	<u>EA 25-sept-02</u>	<u>WJN 16 Oct 02</u>	<u>#2 GN 16 Nov 02</u>
<u>reinject</u>	<u>carry-over</u>	<u>2-418</u>	<u>to 20 nov 02</u>		<u>#2 GN 05 Dec 02</u>
<u>Dil to 50ul</u>	<u>OCDA OLR</u>	<u>2-451</u>	<u>to 9 dec 02</u>	<u>AR 4 Dec 02</u>	<u>#2 R 12 Dec 02</u>

**Comments**

#1: Carbon-Celite #107 11 Oct 02; Florisil #48A 15 Oct 02; Alumina #228 16 Oct 02. WJN  
Recombined c/c El with original microvial. 16 Oct 02 WJN  
Recombined AgNO3/AB/Silica 17 Oct 02 WJN  
Topped up with Toluene CV = 50ul JA

**0038**

### DIOXIN/FURAN SAMPLE ANALYSIS RECORD

Method #s: Pulp: DX-P-01/Ver.4 Sludge: DX-SL-01/Ver.4, DX-SL-8290 / Ver. 2 Filter/Pufs: DX-F-01/Ver.2  
 Sediment: DX-S-01/Ver.4, DX-S-8290/Ver.2 Wood Chips: DX-WC-01/Ver.4 Black Liquor: DX-L-01/Ver.2

Level: DC \_\_\_\_\_ MDT \_\_\_\_\_ HR \_\_\_\_\_ LR \_\_\_\_\_ Due Date: 15-SEPT-02

Sample ID: L4974-3L Date: 10-SEPT-02 Analyst: JAS MUNDAL BATCH #: DXWG 6796

Blank: WG6796-10BLK Rec'd: 15-Aug-02 Back-up: 7/50 g Stored In: \_\_\_\_\_

Original Labelling: SS-23

Sample Type and Description: Sediment - Black/brown, fur-like appearance. Mushy matter.

Sample Pre-Treatment at Axy's:  Date: 4-SEP-02 Homogenizer: EP  
 at other: \_\_\_\_\_ Specifics: HOMO

**AUTHENTIC STANDARD**

	name	volume	date	time	analyst
Sample Weight:	Wet <u>25.53</u> g	Moisture: <u>54.7%</u>		Tare: <u>0.96</u> g	
		Dry <u>11.56</u> g		Dry + Tare: <u>1.83</u> g	
				Wet + Tare: <u>2.88</u> g	
				Dry/Wet Ratio: <u>0.87/1.92 = 0.45</u>	

SURROGATE STANDARD ID	name	quantity	date	time	analyst
<u>DX013A-SUR106</u>		<u>20 uL</u>	<u>11-SEPT-02</u>	<u>14:37</u>	<u>AM</u>

Extraction:

Start:	<u>11-SEPT-02</u>	<u>17:45</u>	<u>AM</u>	End:	<u>12-SEPT-02</u>	<u>9:00</u>	<u>AM</u>
	date	time	analyst		date	time	analyst

Cleanup:

Base/Acid Wash: 3 B 3 W 6 A 1 W 12-SEPT-02 AM

Chromatography: Cu rxn: 1 meg. 2

Procedure No.

(2x) Ag/AB Silica Gel	(Batch # <u>170</u> )	<u>12-SEPT-02</u>	<u>JH</u>
( ) Florisil	(Batch # <u>47B</u> )	<u>13-SEPT-02</u>	<u>AM</u>
( ) Carbon/celite	(Batch # <u>106</u> )	<u>13-SEPT-02</u>	<u>AM</u>
( ) Alumina	(Batch # <u>226</u> )	<u>14-SEPT-02</u>	<u>AM</u>
		date	analyst

RECOVERY STANDARD ID	name	quantity	date	time	analyst
<u>DX005A-REC/18</u>		<u>20 uL</u>	<u>14-SEPT-02</u>	<u>3:00</u>	<u>AM</u>

Final microvial volume: 20 uL

Additional Work	Reason:	GC Data	Requested	Completed in	Run on Instr.
Action:		File:	by/date:	lab by/date	by/date
<u>Clean up C/EI &amp; combine with existing extract</u>	<u>Low RDF sur rec</u>	<u>3-243</u>	<u>EA 25-SEP-02</u>	<u>LXH 16-SEP-02</u>	<u>KC #2 NOV-17-02</u>
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____

Comments

11-SEP-02 15-SEP-02

Fl: Carbon-Celite #107; Florisil #48A; Alumina #228 16-SEP-02 LXH

Recombined C/EI with original vial 16-SEP-02 LXH

Recolumn Ag/NO<sub>3</sub>/AB/Silica 17-SEP-02 LXH



# DIOXIN/FURAN SAMPLE ANALYSIS RECORD

Method #s: Pulp: DX-P-01/Ver.4 Sludge: DX-SL-01/Ver.4, DX-SL-8290 / Ver. 2 Filter/Pufs: DX-F-01/Ver.2  
 Sediment: DX-S-01/Ver.4, DX-S-8290/Ver.2 Wood Chips: DX-WC-01/Ver.4 Black Liquor: DX-L-01/Ver.2

Level: DC \_\_\_\_\_ MDT \_\_\_\_\_ HR \_\_\_\_\_ LR \_\_\_\_\_ Due Date: 15 SEPT. 02  
 Sample ID: L4474-4L Date: 10 SEPT. 02 Analyst: JAS HUNDAL BATCH #: DXWG 6796  
 Blank: WG 6796-10 BLK Rec'd: 15 AUG. 02 Back-up: 7 150 g Stored In: \_\_\_\_\_  
 Original Labelling: SS-24

Sample Type and Description: Sediment - Black/Brown, tar-like appearance, Mushy matter.

Sample Pre-Treatment at Axy's:  Date: 30 Aug. 02 Homogenizer: GD  
 at other: \_\_\_\_\_ Specifics: Homo

**AUTHENTIC STANDARD**

	name	volume	date	time	analyst
Sample Weight:	Wet <u>38.00</u> g	Moisture: <u>55.4%</u>		Tare: <u>0.95</u> g	
				Dry + Tare: <u>1.73</u> g	
		Dry <u>16.94</u> g		Wet + Tare: <u>2.70</u> g	
				Dry/Wet Ratio: <u>0.78/1.75 = 0.446</u>	

SURROGATE STANDARD ID DX03A-SUR406 20uL 11 SEPT. 02 14:38 JH

name	quantity	date	time	analyst
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Extraction : Start: 11 SEPT. 02 17:45 JH End: 12 SEPT. 02 9:00 JH  
 date time analyst date time analyst

Cleanup: Base/Acid Wash: 4 B 3 W 9 A 1 W 12 SEPT. 02 JH  
 Chromatography: Cu rxn: 1 mg. 2  
 Procedure No.

(3x) Ag/AB Silica Gel	(Batch # <u>170</u> )	<u>12 SEPT. 02</u>	<u>JH</u>
( ) Florisil	(Batch # <u>47B</u> )	<u>13 SEPT. 02</u>	<u>JH</u>
( ) Carbon/celite	(Batch # <u>106</u> )	<u>13 SEPT. 02</u>	<u>JH</u>
( ) Alumina	(Batch # <u>226</u> )	<u>14 SEPT. 02</u>	<u>JH</u>

RECOVERY STANDARD ID DX005A-REC/18 20uL 14 SEPT. 02 3:20 JH

name	quantity	date	time	analyst
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Final microvial volume: 20uL

Additional Work Action:	Reason:	GC Data File:	Requested by/date:	Completed in lab by/date:	Run on Instr. by/date:
<u>Clean up 40 EL; combine with existing extract</u>	<u>LONTCOF sur rec</u>	<u>3-243</u>	<u>EA 25-Sept-02</u>	<u>LN 16 Oct 02</u>	
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____

Comments: - couldn't blow sample to dryness after silica columns.  
EL: Carbon-celite #107; Florisil #48A 15 Oct 02; Alumina #228 16 Oct 02. LN  
Recombined GC EL with original microvial 16 Oct 02 LN





# DIOXIN/FURAN SAMPLE ANALYSIS RECORD

Method #s: Pulp: DX-P-01/Ver.4    Sludge: DX-SL-01/Ver.4, DX-SL-8290/Ver.2    Filter/Pufs: DX-F-01/Ver.2  
 Sediment: DX-S-01/Ver.4, DX-S-8290/Ver.2    Wood Chips: DX-WC-01/Ver.4    Black Liquor: DX-L-01/Ver.2

Level: DC \_\_\_\_\_ MDT \_\_\_\_\_ HR \_\_\_\_\_ LR \_\_\_\_\_ Due Date: 15 SEPT. 02  
 Sample ID: L4974 5 L Date: 10 SEPT. 02 Analyst: JAS HINDAL BATCH #: DXWG 6796  
 Blank: WG6796-10 BLK Rec'd: 15 AUG. 02 Back-up: 7150g g Stored In: \_\_\_\_\_  
 Original Labelling: SS-25

Sample Type and Description: Sediment. Black/brown, ear-like, mushy matter

Sample Pre-Treatment at Axys:  Date: 30 AUG. 02 Homogenizer: GD  
 at other: \_\_\_\_\_ Specifics:  homo # jar transfer

**AUTHENTIC STANDARD**

	name	volume	date	time	analyst
Sample Weight:	Wet <u>36.68</u> g	Moisture: <u>70.4%</u>		Tare: <u>0.95</u> g	
		Dry <u>10.87</u> g		Dry + Tare: <u>1.35</u> g	
				Wet + Tare: <u>2.30</u> g	
				Dry/Wet Ratio: <u>0.40 / 1.35 = 0.29</u>	

SURROGATE STANDARD ID	<u>DXOBA-SUR06</u>	<u>20ul</u>	<u>11 SEPT. 02</u>	<u>1439</u>	<u>AD</u>
	name	quantity	date	time	analyst

Extraction: Start: 11 SEPT. 02 17:45 AD End: 12 SEPT. 02 9:00 AD  
 date time analyst date time analyst

Cleanup: Base/Acid Wash: 4 B 3 W 9 A 1 W 12 SEPT. 02 AD

Chromatography: Cu rxn: 1 meg. 2

Procedure No.

(2x) Ag/AB Silica Gel	(Batch # <u>170</u> )	<u>12 SEPT. 02</u>	<u>JH</u>
( ) Florisil	(Batch # <u>47B</u> )	<u>13 SEPT. 02</u>	<u>AD</u>
( ) Carbon/celite	(Batch # <u>106</u> )	<u>13 SEPT. 02</u>	<u>AD</u>
( ) Alumina	(Batch # <u>226</u> )	<u>14 SEPT. 02</u>	<u>AD</u>

RECOVERY STANDARD ID	<u>DXOOSA-REC/18</u>	<u>20ul</u>	<u>14 SEPT. 02</u>	<u>3:21</u>	<u>AD</u>
	name	quantity	date	time	analyst

Final microvial volume: 20ul

Additional Work	GC Data	Requested	Completed in	Run on Instr.
Action:	File:	by/date:	lab by/date	by/date
<u>Clean up c/c El + combine with existing extract</u>	<u>Low TCDF sur rec</u>	<u>3-243</u>	<u>EA 25-sept-02</u>	<u>WH 16 Oct 02</u>

Comments

El: Carbon-Celite #107 11 Oct 02; Florisil #48A 15 Oct 02; Alumina #228 16 Oct 02 WH  
Recombined c/c El with original microvial 16 Oct 02 WH  
Recolumn Ag/AB/Silica 17 Oct 02 WH

0043



# DIOXIN/FURAN SAMPLE ANALYSIS RECORD

Method #: Pulp: DX-P-01/Ver.4 Sludge: DX-SL-01/Ver.4, DX-SL-8290 / Ver. 2 Filter/Pufs: DX-F-01/Ver.2  
 Sediment: DX-S-01/Ver.4, DX-S-8290/Ver.2 Wood Chips: DX-WC-01/Ver.4 Black Liquor: DX-L-01/Ver.2

Level: DC \_\_\_\_\_ MDT \_\_\_\_\_ HR \_\_\_\_\_ LR \_\_\_\_\_ Due Date: 15-SEPT-02  
 Sample ID: L4794-7L Date: 10-SEPT-02 Analyst: JAS HUNDAL BATCH #: DXWG 6796  
 Blank: WG6796-10 BLK Rec'd: 16 Aug 02 Back-up: 7150g g Stored In: \_\_\_\_\_  
 Original Labelling: DYP

Sample Type and Description: Sediment: Black/brown, tar-like appearance. Mushy matter.

Sample Pre-Treatment at Axys: X Date: 30 Aug 02 Homogenizer: (D)  
 at other: \_\_\_\_\_ Specifics: HOMO

**AUTHENTIC STANDARD**

	name	volume	date	time	analyst
Sample Weight:	Wet <u>24.02</u> g	Moisture: <u>56.1%</u>		Tare: <u>0.95</u> g	
		Dry <u>10.55</u> g		Dry + Tare: <u>7.85</u> g	
				Wet + Tare: <u>3.00</u> g	
				Dry/Wet Ratio: <u>0.90/2.05 = 0.43</u>	

SURROGATE STANDARD ID DX013A-Sur106 20mL 11-SEPT-02 14:41 JH

	name	quantity	date	time	analyst
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Extraction: Start: 11-SEPT-02 17:45 JH End: 12 Sept 02 9:00 d.a.

	date	time	analyst	date	time	analyst
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Cleanup: Base/Acid Wash: 3 B 3 W 6 A 1 W 12 Sept 02 d.a.  
 Chromatography: Cu rxn: 1 mag- 2  
 Procedure No.

- ( ) Ag/AB Silica Gel (Batch # 170) 12 Sept 02 JH
  - ( ) Florisil (Batch # 47B) 13 Sept 02 d.a.
  - ( ) Carbon/celite (Batch # 106) 13 Sept 02 d.a.
  - ( ) Alumina (Batch # 226) 14 Sept 02 d.a.
- |  |      |         |
|--|------|---------|
|  | date | analyst |
|--|------|---------|

RECOVERY STANDARD ID DX005A-REC/18 20ul 14 Sept 02 3:18 d.a.

	name	quantity	date	time	analyst
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Final microvial volume: 20ul

Additional Work	Reason:	GC Data File:	Requested by/date:	Completed in lab by/date:	Run on Instr. by/date:
<u>Cleanup c/c El; combine with existing extract</u>	<u>Low TCDF sur rec</u>	<u>3-243</u>	<u>EA 25-Sept-02</u>	<u>WN 16 Oct 02</u>	<u>KC #3 NOV 17 02</u>
<u>Reinject</u>	<u>closing cal late</u>	<u>2-419</u>	<u>to 25 Nov 02</u>		<u>#2 6/05/02</u>

**Comments**

El: Carbon/Celite #107 11 Oct 02; Florisil #48A 15 Oct 02; Alumina #228 16 Oct 02 WN  
Recombined c/c El with original μ-vial 16 Oct 02 WN  
Recolumn AgNO<sub>3</sub>/AB/Silica 17 Oct 02 WN

0045