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ABSTRACT ONLY

H. A. Woltermann

NOTE: Distribution of this report is restricted. Requests for additional copies should be made through the principal contact at the Monsanto location involved or the Manager, Environmental Sciences Center, Dayton Laboratory, Dayton, Ohio.

ACKNOWLEDGEMENT

Contributions of the following Dayton Laboratory personnel to this project are gratefully acknowledged:

Analyses: G. L. Thomas

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C11131

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C11132

ANALYSIS REPORT FOR 2,3,7,8-TCDD IN NITRO SAMPLES

INTRODUCTION

On 27 Nov 84 eleven wipe samples were received for the analysis of 2,3,7,8-tetrachlorodibenzo-p-dioxin (2,3,7,8-TCDD). For internal purposes, the samples were assigned MRC Log #1-84-11-27-01. The samples were analyzed according to the September 1983 EPA Region VII method "Determination of 2,3,7,8-TCDD in Soil and Sediment". For quality control purposes MRC Method #07-QAQC-R3 was followed. Briefly this method requires that for every set of samples of the same matrix type a replicate sample, a low (9.6 ng in this case) spiked sample, a high (96 ng in this case) spiked sample, and a method blank must be analyzed.

PREPARATION AND ANALYSIS (WIPES)

The wipes were spiked with 24.0 ng of carbon-13 labeled 2,3,7,8-TCDD and 9.6 ng of chlorine-37 labeled 2,3,7,8-TCDD. Then ten grams of washed anhydrous sodium sulfate was added and the samples were stirred, allowed to stand and stirred again. Samples were mixed again immediately prior to addition of extraction solvents. The extraction process involved the addition of 15 mL of methanol (all solvents are Burdick and Jackson "distilled in glass" grade), stirring and the subsequent addition of 150 mL of hexane. The samples were then shaken for one hour on a wrist action shaker and the extraction solvent removed. The solvent was then successively extracted with 50 mL of water, concentrated sulfuric acid (until clear), water, 20% potassium hydroxide and water. The hexane was then dried with 10 grams of anhydrous sodium sulfate. Each sample was then placed on the head of a mixed phase column consisting of (top to bottom) 2 g of silica, 4 g of silica plus 40% sulfuric acid, 1 g of silica, 2 g of silica plus 36% potassium hydroxide and 1 g of silica. The column was eluted directly onto a column containing 2.5 g of Woelm basic alumina. The alumina column was first eluted with 10 mL of 2% methylene chloride/hexane (v/v) and this fraction discarded. An additional elution was made with 15 mL of a 50% methylene chloride/hexane (v/v) solution to elute the TCDD off the column.

All samples had 50 uL of dodecane keeper added to them and then were concentrated under a gentle stream of nitrogen and submitted for GC/MS analysis. The GC/MS analyses were conducted on a Hewlett/Packard 5987 GC/MS instrument. The analysis was conducted using a 60 m SP2330 capillary column. The mass spectrometer was operated in the selected ion monitoring mode analyzing for ions characteristic of both the native (unlabeled) 2,3,7,8-TCDD (m/z 257,320,322), chlorine-37 labeled 2,3,7,8-TCDD (m/z 328), and carbon-13 labeled 2,3,7,8-TCDD (m/z 332,334).

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STANDARDS

Standard solutions were prepared from standards that had been cross validated with a standard provided as a check sample by the EPA. Concentration of the internal standard conformed to the levels specified by the method.

RESULTS

The results of the analyses are given in tables 1-3. The tables also give all pertinent QA/QC information.

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TECO DATA REPORT
MINISAKTO COMPANY, DAYTON LABORATORY

CC OL/PM SP270
RF NATIVE: .914
RF SURROGATE: 1.283

SAMPLE NUMBER	EXTR MTHD	CLFAM	MG TCDD	D.L.	ANALYTICAL DATE	TIME	320		332		SUM Z ACC	320	322	257	328+	332	334	H
							322	334	322	334								
# 1	J	BC	1.10	—	11/27	1631	.75	.81	97.		9748.	13006.	5718.	304793.	244028.	323984.		
# 2	J	BC	.99	—	11/27	1651	.85	.78	98.		9759.	11749.	6709.	323444.	274021.	350377.		
# 3	J	BC	.87	—	11/27	1711	.73	.85	102.		9924.	13221.	8333.	417910.	352124.	412631.		
# 4	J	BC	.78	—	11/27	1734	.84	.81	96.		10119.	12107.	7351.	414253.	340867.	447126.		
# 5	J	BC	.73	—	11/27	1754	.69	.82	94.		7850.	11350.	5405.	391320.	335085.	409731.		
# 6	J	BC	1.84	—	11/27	1834	.85	.81	95.		21036.	24636.	11288.	360041.	314326.	390442.		
# 7	J	BC	.96	—	11/27	1853	.85	.81	97.		12595.	14812.	8796.	420294.	362987.	449408.		
# 8	J	BC	1.03	—	11/27	1913	.81	.80	96.		11422.	14072.	7241.	363177.	313714.	370643.		
# 9	J	BC	.82	—	11/27	1931	.87	.81	96.		11325.	13044.	6678.	431972.	377533.	467478.		
# 10	J	BC	.75	—	11/27	1953	.77	.80	96.		9381.	17118.	7399.	417418.	362765.	453825.		
# 11	J	BC	.78	—	11/27	2033	.74	.79	96.		8505.	11472.	4819.	349120.	317922.	404090.		
LOW SPIKE	J	BC	5.87	—	11/27	2053	.79	.83	112.		54951.	71786.	31878.	373563.	292007.	339968.		
HIGH SPIKE	J	BC	70.82	—	11/27	2113	.81	.84	102.		653026.	807944.	300140.	317883.	265313.	317249.		
B	J	BC	ND	.44	11/27	2133	.68	.80	101.		3780.	5594.	0.	321927.	265654.	331450.		

* CORRECTED FOR CONTRIBUTION BY NATIVE TCDD (SUBTRACT .009 OF M/E 322)

NOTE CODES:

11 ISOTOPE RATIO OUT OF ACCEPTABLE RANGE
Y1 % ACCURACY OUT OF ACCEPTABLE RANGE

MB-METHOD BLANK
P-PARTIAL SCAN
R-SEPARATE WEIGHING AND WORKUP
D-REPLICATE (INTERLAB)
FB-FIELD BLANK

L-MINIMATIVE TCDD SPIKES
ND=NOT DETECTED
PL-DETECTION LIMIT
J=NR

FILENAME =TCDD06

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TABLE 2. CALIBRATION SUMMARY

ug/Kg NATIVE TCDD	RRF (NATIVE)	DATE	TIME	RRF (SURROGATE)
0.5010	1.0199	11/27	1551	1.318
5.010	0.8902	11/27	1611	1.224
50.10	0.8713	11/27	1814	1.238
1.2515	0.9030	11/27	2013	1.240
12.515	0.9096	11/27	2153	1.329
125.15	0.8882	11/27	2213	1.350

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TABLE 3. QUALITY CONTROL SUMMARY

ITEM	# DATA POINTS	MEAN $\pm$ S.D.
SURROGATE ACCURACY	14	98.7 $\pm$ 4.5
SPIKE RECOVERY	2	*

* Not technically correct to report recoveries for internal standard calculated results.

GTT3X

ALL INFORMATION CONTAINED
HEREIN IS UNCLASSIFIED
EXCEPT WHERE SHOWN
OTHERWISE
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DOCUMENTATION INVENTORY FORM

Monsanto Research Corporation
Dayton Laboratory

I. Sample Information

Sample Identification: *Nitro*

Number of Samples: *11*

Analyte: *2,3,7,8-TCDD*

Matrix: *wipes*

Method: *Determination of 2,3,7,8-TCDD in soil and sediment*

II. Documentation Control

	<u>Copies Attached</u>	<u>Initials</u>
(a) Sample Receipt Form	<u>✓</u>	<u>BHW</u>
(b) Internal Sample Tracking Form	<u>—</u>	<u>—</u>
(c) Sample Tags	<u>—</u>	<u>—</u>
(d) Airbill (Number <u>PE0004877</u>) <u>PACKAGE EXPRESS</u>	<u>✓</u>	<u>BHW</u>
(e) Chain of Custody Form	<u>✓</u>	<u>BHW</u>
(f) Analysis Request (Log Number <u>1-24-11-27-ND</u>)	<u>✓</u>	<u>BHW</u>
(g) Extraction/Preparation notebook pages (NBP) <u>2831613</u>	<u>✓</u>	<u>BHW</u>
(h) Analysis NBP <u>2273277</u>	<u>✓</u>	<u>BHW</u>
Instrument Logbook pages <u>156700</u>	<u>✓</u>	<u>BHW</u>
Computer Data Files hard copy output	<u>✓</u>	<u>BHW</u>
Chromatograms	<u>✓</u>	<u>BHW</u>
(i) Data Reduction NBP <u>2273277</u>	<u>✓</u>	<u>BHW</u>
(j) Final Report	<u>✓</u>	<u>BHW</u>
(k) Additional Documentation	<u>✓</u>	<u>BHW</u>

(note immediately upon acquisition)

Standards Preparation

NBP 2273264, 2273265

File Complete

Barbara M. McElreath
(signature)

1-4-85
(date)

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Sample Receipt Form

Monsanto Research Corporation
Dayton Laboratory

Customer Identification: NITRO
MRC Log Number: 184.11.27.01
Airbill Number: PE 0004877
Date: 27 Nov 84

Sample Receipt Information:

				Initials
(a) Shipping Containers	<u>intact</u>	damaged (describe below)	No. <u>1</u>	<u>GT</u>
(b) Custody Seals	present	<u>absent</u> (describe sealing below)	intact	<u>GT</u>
(c) Chain of Custody Forms	<u>present</u>	absent	No. <u>2</u>	<u>GT</u>
(d) Sample Tags	<u>present</u>	absent	No. <u>11</u>	<u>GT</u>
(e) Sample Containers	<u>intact</u>	damaged (describe below)	No. <u>11</u>	<u>GT</u>
(f) Agreement of Sample Documentation	<u>yes</u>	no (describe discrepancies)		<u>GT</u>

Additional Description (if sample numbers not listed on chain of custody form, list here and contact SMO).

b) SEALED WITH PACKING TAPE.

Gary Thomas
(Signature)

packageexpress

UNIFORM STRAIGHT BILL OF LADING - ORIGINAL NOT NEGOTIABLE

PE0004877

PACKAGE EXPRESS ACCOUNT NUMBER TO BE BILLED		DATE 11/21/74	PAYMENT (CHECK ONE) ☒ PREPAID ☐ COLLECT ☐ THIRD PARTY ☐ CASH		TYPE OF SERVICE (CHECK ONE) ☐ LETTER ☐ AIR ☐ PAK ☐ BOX OR TUBE ☐ GROUND		ORIG. AIRPORT		DEST. AIRPORT		
FROM COMPANY NAME (CONSIGNOR) MOUNTAIN STATE ENGINEERING SUPPLY				TO COMPANY NAME (RECIPIENT) MOUNTAIN STATE LAB				SATURDAY DELIVERY SERVICE REQUESTED			
STREET ADDRESS				STREET ADDRESS 1515 ...				DB			
CITY		STATE		CITY		STATE					
P.O. OR REFERENCE NO.		ZIP CODE (REQUIRED)		THIRD PARTY NAME AND ADDRESS				ZIP OR POSTAL CODE (REQUIRED)			
SHIPPER'S SIGNATURE X				COURIER GUARD SIGNATURE X		ROUTE		DATE		TIME AM PM	
NON-NEGOTIABLE BILL OF LADING SUBJECT TO CONDITIONS OF CONTRACT SET FORTH ON REVERSE SIDE OF SHIPPER'S COPY.		DESCRIPTION OF CONTENTS 1 SMALL PLASTIC CASE		PIECES		WEIGHT		DECLARED VALUE		DIMENSIONS L W H	
FREIGHT		ADV.		VALUATION		SPECIAL CHGS		CD AMOUNT		TOTAL CHARGES	

MOUNTAIN STATE ENGINEERING SUPPLY

PLEASE DO NOT ENTER ANY INFORMATION IN THE BLUE SHADED AREAS. COMPLETE ALL OTHER INFORMATION. SEE REVERSE SIDE OF COPY NO. 4 FOR INSTRUCTIONS.

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CHAIN OF CUSTODY RECORD

MRC JOB NUMBER _____

CUSTOMER REPRESENTATIVE Keith S. Miller

CONTRACT NUMBER _____

MRC CONTRACT MANAGER _____

PLANT NAME OR CODE MONSANTO - NITRO

MRC TEAM LEADER _____

(Specific information on samples is available on attached "Sample Identification Log")

Signatures		Date	Time	Remarks
Team Leader Verification of Collected Samples:				
Relinquished by: <u>KS Miller</u>	Received by:	<u>11/26/04</u>	<u>3:00pm</u>	
Relinquished by:	Received by: <u>Shut Carter</u>	<u>11/26/04</u>	<u>7:30</u>	
Logged-in-by: <u>Ry W. Webb</u>				
Received for Analysis by:				
Method of Shipment:	Number of Containers:			

Special Instructions (Sample preservation, security, delivery to, etc.)

Deliver to MONSANTO DAYTON RESEARCH LAB
1515 Nicholas Rd
Dayton, OH 45176

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011141

SAMPLE IDENTIFICATION LOG

MRC Job No. _____

Customer Representative K.S. Miller

Contract No. _____

MRC Contract Manager

Plant Name or Code MOLSAINT - NITRO Team Leader

Team Leader

[illegible]

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C11142

ANALYSIS REQUEST		DATE <u>27 Nov 84</u>	LOG NO. <u>1-84-11-27-01</u>	REPORT RESULTS TO: FILES / <u>KULIK</u> <u>P. Sherman</u> <u>R. Noble</u> <u>R. Ivory</u>
MONSANTO RESEARCH CORPORATION DAYTON LABORATORY		REQUESTER** <u>Nitro / Ivory</u>	CHARGE NO.** <u>484,43919</u>	
SAMPLE DESCRIPTION	NUMBER OF BOTTLES: <u>11</u>	NUMBER OF SAMPLES ** <u>11</u>		
<u>#1 Blank</u> <u>#2 Dry Valve Haise</u> <u>#3 NE Wall 46-N</u> <u>#4 North Pillar B46N</u> <u>#5 S. WP. No. 46N</u> <u>#6 SE Corner 46-N</u> <u>#7 Main Room South Wall 46-N</u> <u>#8 Center Fire Wall 46N</u> <u>#9 East Fire Wall 46S</u> <u>#10 Center Fire Wall 2nd level 46S</u> <u>#11 Center Fire Wall 3rd level 46S</u>				
				ANALYSIS REQUEST FILES / <u>mees</u> <u>P. Sherman</u> <u>R. Noble</u> <u>G. Thomas</u>
				SAMPLES FOR: <u>R. Noble</u>
				SAMPLE LOCATION <u>2/20</u>
RECORDED BY: <u>Ry V. Rutte</u>				

REQUIRED ANALYSIS**

TCDD

KNOWN HEALTH HAZARD PRESENTED BY SAMPLE: ** Possible Toxic Possible Carcinogen	ANALYTICAL TECHNIQUE ** <table style="width:100%;"> <tr> <td>☐ GAS CHROMATOGRAPHY</td> <td>☐ NMR</td> </tr> <tr> <td>☐ ULTRAVIOLET</td> <td>☐ HPLC</td> </tr> <tr> <td>☐ VISIBLE</td> <td>☐ XRD</td> </tr> <tr> <td>☐ INFRARED</td> <td>☐ ICP</td> </tr> <tr> <td>☐ EMISSION</td> <td>☐ ATOMIC ABSORPTION</td> </tr> <tr> <td>☐ EDAX XRF</td> <td>☐ MASS SPEC</td> </tr> <tr> <td>☐ MICROSCOPY</td> <td>☒ GC/MS</td> </tr> <tr> <td>☐ OTHER _____</td> <td>☐ AUTO ANALYZER</td> </tr> </table>	☐ GAS CHROMATOGRAPHY	☐ NMR	☐ ULTRAVIOLET	☐ HPLC	☐ VISIBLE	☐ XRD	☐ INFRARED	☐ ICP	☐ EMISSION	☐ ATOMIC ABSORPTION	☐ EDAX XRF	☐ MASS SPEC	☐ MICROSCOPY	☒ GC/MS	☐ OTHER _____	☐ AUTO ANALYZER
☐ GAS CHROMATOGRAPHY	☐ NMR																
☐ ULTRAVIOLET	☐ HPLC																
☐ VISIBLE	☐ XRD																
☐ INFRARED	☐ ICP																
☐ EMISSION	☐ ATOMIC ABSORPTION																
☐ EDAX XRF	☐ MASS SPEC																
☐ MICROSCOPY	☒ GC/MS																
☐ OTHER _____	☐ AUTO ANALYZER																
REFERENCE TO PREVIOUS WORK (REQUEST #, JOURNAL, ETC.) ** 	REQUIRED QA/QC <u>MRC 407-040C-023</u>																
COMPLETION DATE _____ 	CONFIDENTIAL SUBJECT TO PROTECTIVE ORDER																
TIME ALLOTTED FOR ANALYSES <table style="width:100%;"> <tr> <th style="text-align: left;">ANALYST</th> <th style="text-align: left;">HOURS</th> <th style="text-align: left;">INST. TIME</th> </tr> <tr> <td colspan="3" style="height: 40px;"> </td> </tr> <tr> <td colspan="3" style="text-align: center;">00014</td> </tr> </table>	ANALYST	HOURS	INST. TIME				00014										
ANALYST	HOURS	INST. TIME															
00014																	

**SAMPLE DISPOSAL: ☐ DISCARD ☐ RETURN ☐ HOLD FOR 30 DAYS ☒ 60 DAYS 071
 AFTER REPORT COMPLETE

C111143

Nº 2831613

SUBJECT

Nitro Wipes - Dioxin Analysis

JOB NO.

740.42 484.43919

PREPARED BY (SIGNATURE)

Benet Curtis

DATE

27 Nov 8

Date 26 Nov 84

Nitro Wipes

Log No.

Job No.

Data Sheet

weighed by

Spiked by

B. R. Curtis

Code No.	Nitro ID	Final Wt.	Final Wt.	Final & Nitro	Co	CI	Comments
UT 3179	#1 Blank				✓	✓	
UT 3180	#2 Dry Valve House (71)				✓	✓	each sample received
UT 3181	#3 NEWall 46-N				✓	✓	100ul of internal std
UT 3182	#4 North Pillar B46-N				✓	✓	0.24 ug/l / 0.1 "5/8"
UT 3183	#5 S. Pillar 46-N				✓	✓	in 130-october
UT 3184	#6 SE Corner 46-N				✓	✓	
UT 3185	#7 Main Room South wall 46-N				✓	✓	
UT 3186	#8 Center Fire Wall 46-N				✓	✓	
UT 3187	#9 East Fire Wall 46-S				✓	✓	
UT 3188	#10 Center Fire Wall 2nd level 46-S				✓	✓	
UT 3189	#11 Center Fire Wall 3rd level 46-S				✓	✓	
UT 3190	Lo spike			40ul	✓	✓	Hi-lo spiked with
UT 3191	Hi spike			400ul	✓	✓	0.24 Active TCDD
UT 3192	Solvent Blank (ours)				✓	✓	in 130-october

1) Samples were spiked and prepared in the manner described on page 2831610. They were worked up in Lab 29 and CSL I using the most recent standards.

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READ AND UNDERSTOOD BY

DATE