

FILE NITRO
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MONSANTO COMPANY / DAYTON LABORATORY / DAYTON, OHIO 45407

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(Department/Section)

ANALYTICAL

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TITLE: Analyses of Wipes for 2,3,7,8-TCDD

AUTHORS: G. L. Thomas

ABSTRACT: Wipe samples from the Nitro plant were analyzed for possible concentrations of 2,3,7,8-TCDD. The results of these analyses are shown in this report.

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1-8-85

RESTRICTED DISTRIBUTION: (see over)

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AUTHORS: G. L. Thomas

TITLE: ANALYSES OF WIPES FOR 2,3,7,8-TCDD

REPORT NO.: MDA-565

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ABSTRACT ONLY

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ACKNOWLEDGEMENT

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

Analyses: G. L. Thomas

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

INTRODUCTION

On 18 Dec 84 eight 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 #1-84-12-18-01. 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 (WIPE)

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 μ L 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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TOXIN DATA REPORT
MORRIS COMPANY, DAYTON LABORATORY

GC COLUMN: SP2300
RF NATIVES: .898
RF SURROGATE: 1.121

SAMPLE NUMBER	EXTR MTHD	CLEAN	NR TCDD	D.L.	ANALYTICAL		320		332		SURR		320		322		257		323+		332		334		N
					DATE	TIME	322	334	ACC		320	322	320	322	257	323+	332	334			332	334			
1	J	RC	1.15	—	12/19	1041	.80	.80	109.		4210.	5238.	3606.	119652.	105329.	132909.									
2	J	RC	.94	—	12/19	1101	.82	.81	103.		3981.	4881.	3180.	130100.	121234.	149508.									
3	J	RC	ND	.27	12/19	1122	***	.81	108.		0.	0.	0.	87406.	72763.	90262.	X								
4	J	RC	ND	.11	12/19	1142	***	.80	108.		0.	0.	0.	202749.	177694.	222731.	X								
5	J	RC	ND	.18	12/19	1202	***	.80	106.		0.	0.	0.	115823.	104544.	129927.	X								
6	J	RC	1.24	—	12/19	1242	.83	.80	107.		7473.	8978.	4868.	192439.	170592.	213347.									
7	J	RC	1.28	—	12/19	1302	.80	.81	108.		5934.	7449.	3984.	151415.	134805.	164051.									
8	J	RC	.89	—	12/19	1322	.83	.81	107.		4525.	5437.	3918.	161143.	143664.	177982.									
9	J	RC	ND	.14	12/19	1342	***	.81	108.		0.	0.	0.	152741.	135676.	167033.	X								
HIGH SPIKE	J	RC	106.76	—	12/19	1442	.80	.79	109.		396327.	493503.	165039.	122085.	106076.	134320.									

+ CORRECTED FOR CONTRIBUTION BY NATIVE TCDD (SUBTRACT .009 OF N/E 322)

NOTE CODES:

XX ISOTOPE RATIO OUT OF ACCEPTABLE RANGE
Y% % ACCURACY OUT OF ACCEPTABLE RANGE

NR=METHOD BLANK
P=PARTIAL SCAN
R=SEPARATE WEIGHING AND WORKUP
D=DUPPLICATE (INTERLAB)
FB=FIELD BLANK

L=NONNATIVE TCDD SPIKES
ND=NOT DETECTED
DL=DETECTION LIMIT
J=JUR

FILENAME = TCDD033

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TABLE 2. CALIBRATION SUMMARY

ug/Kg NATIVE TCDD	RRF (NATIVE)	DATE	TIME	RRF (SURROGATE)
0.5010	0.9868	12/19	1001	1.123
5.010	0.8551	12/19	1021	1.224
50.10	0.8558	12/19	1222	2.174
1.2515	0.9019	12/19	1422	1.017
12.515	0.8881	12/19	1505	1.895
125.15	0.8551	11/27	1525	1.798

GTT2X

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TABLE 3. QUALITY CONTROL SUMMARY

ITEM	# DATA POINTS	MEAN $\pm$ S.D.
SURROGATE ACCURACY	10	107.2 $\pm$ 1.7
SPIKE RECOVERY	1	*

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

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SOLITARY TITRE EXOR

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DOCUMENTATION INVENTORY FORM

Monsanto Research Corporation
Dayton Laboratory

I. Sample Information

Sample Identification: NITRO

Number of Samples: 8

Analyte: 2,3,7,8-TCDD

Matrix: W, PES

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>Bmw</u>
(b) Internal Sample Tracking Form	<u>—</u>	<u>—</u>
(c) Sample Tags	<u>—</u>	<u>—</u>
(d) Airbill (Number <u>—</u>)	<u>—</u>	<u>—</u>
(e) Chain of Custody Form	<u>✓</u>	<u>Bmw</u>
(f) Analysis Request (Log Number <u>1-84-12-184D</u>)	<u>✓</u>	<u>Bmw</u>
(g) Extraction/Preparation notebook pages (NBP) <u>2831630</u>	<u>✓</u>	<u>Bmw</u>
(h) Analysis NBP <u>2273276</u>	<u>✓</u>	<u>Bmw</u>
Instrument Logbook pages <u>156907</u>	<u>✓</u>	<u>Bmw</u>
Computer Data Files hard copy output <u>—</u>	<u>✓</u>	<u>Bmw</u>
Chromatograms	<u>✓</u>	<u>Bmw</u>
(i) Data Reduction NBP <u>2273276</u>	<u>✓</u>	<u>Bmw</u>
(j) Final Report	<u>✓</u>	<u>Bmw</u>
(k) Additional Documentation	<u>✓</u>	<u>Bmw</u>

(note immediately upon acquisition)

Standards Preparation
NBP 2273264, 2273265

File Complete

Barbara M. Williams
(signature)

1-4-85
(date)

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Sample Receipt Form

Monsanto Research Corporation
Dayton Laboratory

Customer Identification: NHRO
MRC Log Number: 1-84-12-1801
Airbill Number: —
Date: 18 DEC 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>—</u>	<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>8</u>	<u>GT</u>
(e) Sample Containers	<u>intact</u>	damaged (describe below)	No. <u>3</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)

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C11068

CHAIN OF CUSTODY RECORD

MAR JOB NUMBER _____

CUSTOMER REPRESENTATIVE Keith Miller

CONTRACT NUMBER _____

MRC CONTRACT MANAGER

PLANT NAME OR CODE Morinda 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: K. N. J. J.	Received by:		
Relinquished by:	Received by: R. J. W. Webb	12 Aug 82	820
Logged-in-by:			
Received for Analysis by:			
Method of Shipment:	Number of Containers:		

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

Deliver to Mr. Roy Noble
Monsanto Research Corp
Dayton Laboratory
1515 Nicholas Rd
Box 8
Dayton Ohio 45407

CONVICT TO PROTECTIVE ORDER.

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C11069

SAMPLE IDENTIFICATION LOG

MRC Job No. _____

Customer Representative KEITH MILLER

Contract No. _____

MRC Contract Manager _____

Plant Name or Code Nitro

Team Leader _____

[illegible]

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C11070

ANALYSIS REQUEST

DATE

18 DEC 84

LOG NO.

1-84-12-R-01

MONSANTO RESEARCH CORPORATION
DAYTON LABORATORY

REQUESTER**

NITRO / K. MILLER

CHARGE NO.**

454.4229

SAMPLE DESCRIPTION

NUMBER OF BOTTLES:

8

NUMBER OF SAMPLES **

8

REPORT RESULTS TO
FILES / KULIKNITRO
- 202YANALYSIS REQUEST
FILESNITRO
- 202Y

NITRO

SAMPLES FOR:

NITRO

SAMPLE LOCATION

CSL

NO. 1 LAB BLANK (NITRO)

NO. 2 LAB BLANK (NITRO)

NO. 3 LAB BLANK (DAYTON)

NO. 4 LAB BLANK (DAYTON)

NO. 5 79 BLANK (DAYTON)

NO. 6 79 VALVE HOUSE (DAYTON)

NO. 7 79 VALVE HOUSE (DAYTON)

NO. 8 79 BLANK (NITRO)

RECORDED BY:

J. H. 1/6/84

REQUIRED ANALYSIS**

2,3,7,8-TCDD

KNOWN HEALTH HAZARD PRESENTED BY SAMPLE: **

REFERENCE TO PREVIOUS WORK (REQUEST #, JOURNAL, ETC.) **

COMPLETION DATE

TIME ALLOTTED FOR ANALYSES

ANALYST

HOURS

INST. TIME

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ANALYTICAL TECHNIQUE **

☐ GAS CHROMATOGRAPHY☐ NMR☐ ULTRAVIOLET☐ HPLC☐ VISIBLE☐ XRD☐ INFRARED☐ ICP☐ EMISSION☐ ATOMIC ABSORPTION☐ EDAX XRF☐ MASS SPEC☐ MICROSCOPY☒ GC/MS☐ OTHER☐ AUTO ANALYZER

QUAL _____ SEMIQUANT. _____ QUANT. _____

REQUIRED QA/QC

**SAMPLE DISPOSAL:
AFTER REPORT COMPLETE☐ DISCARD☐ RETURNHOLD FOR ☐ 30 DAYS☐ 60 DAYS

OT

** THIS INFORMATION MUST BE SUPPLIED BY ANALYSIS REQUEST ORIGINATOR

C11071

Nº 2831620

Nitro Wipes

B NO. 484.44229

PREPARED BY (SIGNATURE)
Benet R. Curtis

DATE
19 Dec 84

Date 18 Dec 84
Log No. 454.44229
Job No. 454.44229

Nitro Wipes
Data Sheet

weighted by _____.

Spined by B. R. Curtis

[illegible]

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READ AND UNDERSTOOD BY

DATE **C11072**