

FILE NITRO
DIOXIN

on your files

MONSANTO COMPANY / DAYTON LABORATORY / DAYTON, OHIO 45407

ESC/DAYTON

(Department/Section)

ANALYTICAL

(Type of Report)

REPORT NO.: MDA-565

JOB/PROJECT NO.: 484.4420

DATE: 8 January 1985

PERIOD COVERED:

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.

TECHNICAL APPROVAL:

P. L. Sherman
P. L. Sherman
Sr. Research Group Leader

APPROVED BY:

R. F. Ivory
R. F. Ivory
Environmental Sciences Center
Service Project Manager

APPROVAL DATE:

1-8-85

RESTRICTED DISTRIBUTION: (see over)

COMPANY CONFIDENTIAL

This document is the property of Monsanto Company and the recipient is responsible for its safekeeping and disposition. It contains CONFIDENTIAL INFORMATION which must not be reproduced, revealed to unauthorized persons or sent outside Monsanto Company without proper authorization.

CONFIDENTIAL

SUBJECT TO PROTECTIVE ORDER.

C11059

AUTHORS: G. L. Thomas

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

REPORT NO.: MDA-565

COPY NO.: 4

DISTRIBUTION

COPY NUMBER

1-3 M. Townsend, Nitro Plant
4 F. Kearney, St. Louis-E3NA
5-7 T. Bistline, St. Louis-E2ND
8 G. Thomas
9 R. F. Ivory
10 Technical Reports Library - R2C
11 Central Files

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

COMPANY CONFIDENTIAL

CONFIDENTIAL

SUBJECT TO PROTECTIVE ORDER

C11060

DOCUMENT CONTENTS

	Page
Cover Sheet	1
Document Contents	3
Report	4
Document Inventory Form	9
Sample Receipt Form	10
Chain of Custody Form	11
Analysis Request Form	13
Monsanto Notebook Pages For:	
a. Extraction/Preparation	14
b. Analysis/Data Reduction	15
Instrument Logbook Pages	16
Computer Output and Chromatograms	17
Additional Documentation	70

00003

CONFIDENTIAL
SUBJECT TO COOPERATIVE ORDER

C11061

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 (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).

CONFIDENTIAL

00004

SUBJECT TO PROTECTIVE ORDER

C11062

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.

0000

CONFIDENTIAL
SUBJECT TO PROTECTIVE ORDER.

C11063

TOXIN DATA REPORT
MONSANTO COMPANY, DAYTON LABORATORY

GC COLUMN: SP2330
RF NATIVE: .998
RF SURROGATE: 1.121

SAMPLE NUMBER	EETR MTHD	CLEAN	ND TCDD	D.L.	ANALYTICAL DATE	TIME	320		322	334	SURR %		320	322	257	320+	332	334	M
							322	334			ACC								
1	J	RC	1.15	—	12/19	1041	.80	.80	108.				4210.	5238.	3604.	119652.	105329.	132009.	
2	J	RC	.94	—	12/19	1101	.82	.81	103.				3981.	4991.	3190.	130100.	121734.	149508.	
3	J	RC	ND	.27	12/19	1122	----	.81	108.				0.	0.	0.	87404.	72763.	90262.	X
4	J	RC	ND	.11	12/19	1142	----	.80	108.				0.	0.	0.	207749.	177694.	222731.	X
5	J	RC	ND	.18	12/19	1202	----	.80	104.				0.	0.	0.	115823.	104544.	129927.	X
6	J	RC	1.24	—	12/19	1242	.83	.80	107.				7473.	8978.	4848.	192439.	170592.	213347.	
7	J	RC	1.28	—	12/19	1302	.80	.81	108.				5934.	7449.	3684.	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 M/E 322)

NOTE CODES:

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

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

L=NATIVE TCDD SPIKES
ND=NOT DETECTED
DL=DETECTION LIMIT
J=JAR

FILENAME =TCDD033

CONFIDENTIAL
SUBJECT TO PROTECTIVE ORDER

C11064

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

00007
CONFIDENTIAL
 SUBJECT TO PROTECTIVE ORDER.

C11065

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.

GTT3X

00008

CONFIDENTIAL
SUBMIT IN WRITING ORDER

C11066

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, TES

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>1-84-12-18-01</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>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)

00009
CONFIDENTIAL

SUBJECT TO PROTECTIVE ORDER.

C11067

Sample Receipt Form

Monsanto Research Corporation
Dayton Laboratory

Customer Identification: NHRO
MRC Log Number: 1-84-12-18 01
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>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>8</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).

D) SEALED WITH PACKING TAPE.

Gary Thomas
(Signature)

00010

SUBJECT TO PROTECTIVE ORDER

C11068

CHAIN OF CUSTODY RECORD

MRC JOB NUMBER _____

CUSTOMER REPRESENTATIVE Keith 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>K. Miller</u>	Received by:			
Relinquished by:	Received by: <u>R. W. Webb</u>	<u>12-84</u>	<u>820</u>	
Logged-in-by:				
Received for Analysis by:				
Method of Shipment:		Number of Containers:		

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

Deliver to	<u>Mr. Roy Noble</u>
	<u>Monsanto Research Corp</u>
	<u>Denton Laboratory</u>
	<u>1515 Nicholas Rd</u>
	<u>Box 8</u>
	<u>Denton Ohio 45407</u>

SUBJECT TO PROTECTIVE ORDER

00011

00011

C11069

SAMPLE IDENTIFICATION LOG

MRC Job No. _____

Customer Representative KEITH MILLER

Contract No. _____

MRE Contract Manager

Plant Name or Code Nitro

Team Leader

Sampling Location	Run Number	Date Sampled	Type of Sample & No. of Containers	Analysis Required	Sample recovered By (Initials) and Comments
No. 1		12-14	LAB Blank NITRO PAD	2,3,7,8 - TCDD	
No. 2			LAR Blank NITRO PAD		
No. 3			LAR Blank DAYTON PAD		
No. 4			LAR Blank DAYTON PAD		
No. 5			79 Blank DAYTON PAD		
No. 6			TOP OF Valve house DAYTON PAD		
No. 7			TOP OF Valve house NITRO PAD		
No. 8			79 Blank NITRO PAD		

00012

C11070

Sub. Ref. No. 125075-125076

ANALYSIS REQUEST		DATE 18 DEC 84	LOG NO. 1-84-12-R-01	REPORT RESULTS TO FILES / KULIK
MONSANTO RESEARCH CORPORATION DAYTON LABORATORY		REQUESTER** NITRO / K. MILLER	CHARGE NO.** 459.44229	STORY
SAMPLE DESCRIPTION	NUMBER OF BOTTLES: 8	NUMBER OF SAMPLES ** 8		ANALYSIS REQUEST FILES STORY 100113
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 1/2 VALVE HOUSE (DAYTON) NO. 7 1/2 VALVE HOUSE (DAYTON) NO. 8 79 BLANK (NITRO)				SAMPLES FOR: 100113
RECORDED BY: Andy Thomas				SAMPLE LOCATION CSL

REQUIRED ANALYSIS**

2,3,7,8-TCDD

KNOWN HEALTH HAZARD PRESENTED BY SAMPLE: **			ANALYTICAL TECHNIQUE **	
REFERENCE TO PREVIOUS WORK (REQUEST #, JOURNAL, ETC.) **			☐ GAS CHROMATOGRAPHY	☐ NMR
			☐ ULTRAVIOLET	☐ HPLC
COMPLETION DATE _____			☐ VISIBLE	☐ XRD
			☐ INFRARED	☐ ICP
TIME ALLOTTED FOR ANALYSES			☐ EMISSION	☐ ATOMIC ABSORPTION
			☐ EDAX XRF	☐ MASS SPEC
ANALYST _____ HOURS _____ INST. TIME _____			☐ MICROSCOPY	☒ GC/MS
			☐ OTHER _____	☐ AUTO ANALYZER
SUBJECT TO PROTECTIVE ORDER.			REQUIRED QA/QC	

CONFIDENTIAL 00013

**SAMPLE DISPOSAL: ☐ DISCARD ☐ RETURN HOLD FOR ☐ 30 DAYS ☐ 60 DAYS ☐ OT

** THIS INFORMATION MUST BE SUPPLIED BY ANALYSIS REQUEST ORIGINATOR

C11071

Nº 2831620

ECT

Nitro Wipes

1 NO.

484.44229

PREPARED BY (SIGNATURE)

EXPANED BY (SIGNATURE)
Benet R. Curtis

DATE _____

19 Dec 84

Date 18 Dec 84

Nitro Wipes

weighted by

Log No. 424.44229
Tide No. 424.44229

Data Sheet

Spiked by

B. R. Curtis

[illegible]

CONFIDENTIAL

SUBJECT TO PROTECTIVE ORDER.

06014

READ AND UNDERSTOOD BY

DATE **C11072**