

AGRICULTURAL CHEMICALS DIVISION - RESEARCH DEPARTMENT

12114

72114

100 Analysis W. B. L. Rins
 PRODUCT 245-T PREPARED BY SIGNATURE DATE 3-20-68

40) - 220° Sw - 190 mm
 TDD Sw. - 29.0 } Ave = 28.2 mm - 100 ppm
 27.4 }

52144-2 3.6 } 3.8
 4.0 }

52144-3.5 3.8 } 3.8
 3.8 }

52144-2 TDD (3.8) (0.1 ml) (10 ml)
 28.2

$\frac{.1347}{.13843 \times 10^4} = 1.0 \text{ ppm}$

52144-3.5 TDD (3.8) (0.1) (10)
 28.2

$\frac{.1347}{.155643 \times 10^4} = 0.8 \text{ ppm}$

EX. 56

057497

8317460

READ AND UNDERSTOOD BY

DATE

W. B. L. Rins

3/20/68

72118
 245-T
 PREPARED BY (SIGNATURE)
 DATE 3-2-68

62144-2 was concentrated stepwise to 2nd solution.
 TCA & TCB precipitates were added and redistilled to
 assay the benzene layer.

TDD - (9.0) (0.1 - 1/100) (2.0) - .0018 - 0.7 mm
 22.0
 1/218 x 10^4
 Sample loss in TDD
 from org. analysis

087501

531746

READ AND UNDERSTOOD BY

DATE

W. Binnard 2/21/69

10 Analyze Nitro Plant Samples

PRODUCT	PREPARED BY (SIGNATURE)	DATE
245T	James Lane	4-5-68
100ppm - 21.5, 20.3, 21.2, 20.6 → 20.8, 20.9 20ppm - 4.2, 4.4 10ppm - 2.0, 2.1 5ppm - 1.2		
134, Sample 1	2.5, 2.7 → 2.6	PPM
TPO = $\frac{(2.6)(0.1)(1)(35)}{20.8} = .4354$ / 1.2010×10^{-4} = 3.6		
134, Sample 2	2.5, 2.9 → 2.7	
TPO = $\frac{(2.7)(0.1)(1)(35)}{20.8} = .454$ / 1.0710×10^{-4} = 4.2		
134, Sample 3	4.0, 4.0 → 4.0	
TPO = $\frac{(4.0)(0.1)(1)(35)}{20.8} = .6698$ / 1.1610×10^{-4} = 5.8		
134, Sample 4	4.0, 4.1 → 4.2	
TPO = $\frac{(4.2)(0.1)(1)(35)}{20.8} = .7033$ / 1.0510×10^{-4} = 6.7		
134, Sample 5	4.5, 4.7 → 4.6	
TPO = $\frac{(4.6)(0.1)(1)(35)}{20.8} = .7703$ / 1.1010×10^{-4} = 7.0		
134, Final	15.4, 15.0 → 15.2	
TPO = $\frac{(15.2)(0.1)(1)(70)}{20.8} = 5.0909$ / 5.945×10^{-4} = 8.6		
READ AND UNDERSTOOD BY		DATE
W. D. Darnard		3/24

MONSANTO COMPANY

AGRICULTURAL CHEMICALS DIVISION - RESEARCH DEPARTMENT

72124

77123 Cont'd - TDO Analysis

PRODUCT

245-T

PREPARED BY (SIGNATURE)

James Sims

DATE

4-5-68

F.I.L. 136 Sample 1 4.0 4.0 → 4.0 PPM

TDO (4.0)(0.1 ml) (35 ml) 3253 / 20.9 = 3.7

F.I.L. 136 Sample 2 5.2 5.6 → 5.4

TDO (5.4)(0.1 ml) (35 ml) 9013 / 20.9 = 5.5

F.I.L. 136 Sample 3 6.3 6.7 → 6.5

TDO (6.5)(0.1 ml) (35 ml) 10885 / 20.9 = 5.5

F.I.L. 136 Sample 4 6.0 6.0 → 6.0

TDO (6.0)(0.1 ml) (35 ml) 10017 / 20.9 = 7.0

F.I.L. 136 Final 7.7 7.3 → 7.5

TDO (7.5)(0.1 ml) (50 ml) 17947 / 20.9 = 6.2

REF 500

EXHIBIT 56 cont.

5317363

READ AND UNDERSTOOD BY

DATE

W. L. Bismard 2/24/6

MONSANTO COMPANY

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72125

PRODUCT	PREPARED BY	DATE
2455	James J. Smith	4-5-68
130 Sample 1	2.6 7.8 → 2.7	WDB
100	(2.7)(1.01)(1.2) (18.2)	1325
	20.9	1325 / 10.5

Calc 138 Sample 2 48.54 → 5.1

$$\frac{500 - (5.1)(0.01 \text{ mol/L}) - (35 \text{ mL})}{20.9} = \frac{850}{111.10 \text{ g}} = 7.3$$

Calc 138 Sample 3 53.36 → 3.5

$$\frac{500 - (3.5)(0.01 \text{ mol/L}) - (35 \text{ mL})}{20.9} = \frac{986}{112.10 \text{ g}} = 7.6$$

Calc 138 Sample 4 54.54 → 5.4

$$\frac{500 - (5.4)(0.01 \text{ mol/L}) - (35 \text{ mL})}{20.9} = \frac{943}{114.10 \text{ g}} = 8.2$$

057510

8317465

READ AND VERIFIED BY W. B. Arnold DATE 3/30/11

AGRICULTURAL CHEMICALS DIVISION - RESEARCH DEPARTMENT

72166

Run #2 TDD Analysis
 PRODUCT 2,4,5-T PREPARED BY (SIGNATURE) DATE 7-19-68

TDD Std - 25.9 24.5 → 25.2

Run 2 Sample 2 TDD $\frac{(2.5)(0.1)(10)}{25.2} = .0990 \text{ mg}$ 9.6 ppm
 $\frac{1.03 \text{ mg}}{1.07 \text{ g}}$

Run 2 Sample 3 TDD $\frac{(3.7)(0.1)(10)}{25.2} = .1468 \text{ mg}$ 12.1 ppm
 $\frac{1.21 \text{ mg}}{1.00 \text{ g}}$

Run 2 Final Pwd TDD $\frac{(40)(0.1)(50)}{25.2} = 3.5714 \text{ mg}$ 28 ppm
 $\frac{4.539 \text{ mg}}{1.00 \text{ g}}$

Run 2 Close wash of salt cake TDD $\frac{(35)(0.1)(30)}{25.2} = .4166 \text{ mg}$ 9.3 ppm
 $\frac{4.47 \text{ mg}}{1.00 \text{ g}}$

857354
 8317366

READ AND UNDERSTOOD BY

DATE

W. B. Bismard 2/3/1/1.6

TEST	Run #3 - TDD	PREPARED BY (SIGNATURE)	DATE
7102	24.5T	<i>[Signature]</i>	7-20-68

TDD Std → 29.0 30.0 → 29.5
 40% 140mm
 220° in.

Run #3 Sample 1

$$TDD = \frac{(3.5)(0.1)(5)}{29.5} = \frac{0.593}{1.17 \times 10^4} = 5.1 \text{ ppm}$$

Sample 2

$$TDD = \frac{(4.3)(0.1)(5)}{29.5} = \frac{0.762}{1.56 \times 10^4} = 5.0 \text{ ppm}$$

Sample 3

$$TDD = \frac{(5)(0.1)(5)}{29.5} = \frac{0.897}{1.59 \times 10^4} = 5.5 \text{ ppm}$$

Final Sample Product

$$TDD = \frac{(28)(0.1)(50)}{29.5} = \frac{4.7457}{4.55 \times 10^5} = 10.5 \text{ ppm}$$

READ AND VERIFIED BY	DATE
<i>[Signature]</i>	2/24/6

AGRICULTURAL CHEMICALS DIVISION - RESEARCH DEPARTMENT

72174

Product: 745-T
Prepared by (Signature): James Sims
Date: 7-25-68

TDD Std → 27.6, 27.0 → 27.3
40% 190m
225 m

Run #4 Sample 1

TDD = $\frac{(4)(0.1)(5)}{27.3} = \frac{0.732}{8.5 \times 10^3} = 8.6 \text{ ppm}$

Run #4 Final Prod
TDD = $\frac{(31.3)(4)(0.1)(5)}{27.3} = \frac{22.914}{3.718 \times 10^5} = 61.7 \text{ ppm}$

Run #5 Sample 1
TDD = $\frac{(4.5)(0.1)(5)}{27.3} = \frac{0.674}{1.49 \times 10^4} = 5.5 \text{ ppm}$

Sample 2
TDD = $\frac{(17)(0.1)(5)}{27.3} = \frac{3.11}{1.55 \times 10^4} = 20.0 \text{ ppm}$

Sample 3
TDD = $\frac{(27)(0.1)(5)}{27.3} = \frac{4.45}{1.60 \times 10^4} = 31 \text{ ppm}$

Run #4 Final Prod
TDD = $\frac{(29.2)(4)(0.1)(5)}{27.3} = \frac{21.39}{4.05 \times 10^5} = 52.8$

READ AND INTERPRETED BY: W. Simard
DATE: 7/24/68

72183

MONSANTO COMPANY AGRICULTURAL CHEMICALS DIVISION - RESEARCH DEPARTMENT

MONSANTO COMPANY

00421

T00 Analysis -

2102

2451

PREPARED BY SIGNATURE

DATE

8-17-68

Round T00 analysis method has drawbacks due to sensitivity on thermal analysis and also the method is not quantitative below 4-5 ppm. A method is used whereby low levels of T00 can be detected qualitatively. Analysis by F10 is desirable.

Run #3 - Benzene layer (30ml) was acidified and then benzene layer transferred to 2 oz. bottle and benzene evaporated in a N_2 stream. CS_2 was added to the residue and bottle stopper dissolved in CS_2 would be acidified on a flow dispenser due to its insensitivity to a flame.

Revised chromatograph for T00 analysis of T00 - 2 - 1/2% CS_2 column, the amount in R-E 800 chromatograph for T00 analysis of T00 - A1 - 6' x 1/8" 5% polyd with 5% OCAF-1 on Chrom W (Hercs) B. 1' x 1/8" x 1/8"

Carb. 190° 500 - 0.5 x sample size 5-3 = 310° 104 - 250°

H₂ = 50 ppm (bulk) 5-15. R-15 5-500 L/min 30 ml/min 2 column with

6815159

DATE 8/31/69

72187

TOO *Schubert Reagent*
 PRODUCT *245-T* PREPARED BY *JAMES LIND* DATE *8-22-69*

G = 3056

T = 2933

0.103

g. TOO

(2,3,7,8-Tetrachloro-dibenzo-p-dioxin)

in 100 ml Benzene

(low)

= .103 mg/liter

Comb. time = TOO analysis on A' column (6'-5% Decaf-)
 on Chrom W (Hmax)

Inj - 310 Det - 250 Col - 150 Iso - (6-20)

H₂ - 50 ps. (back) Regulator - Sec - 15; R - 15

Air - 50 " " = 500 ml/min

N₂ - 50 " " 30 ml/min flow at column exit

Sample size - 0.5 µl Attenuation = x20

Standard - 72187-A

84.7 min

93.5 "

88.5 "

87.8 "

Ave = 88.6 min

DE 57577

5812370

READ AND RECORDED BY

DATE

W. Benward

2/24/69

AGRICULTURAL CHEMICALS DIVISION - RESEARCH DEPARTMENT

72189

TPD Analysis - Run #7
 PRODUCT 745-T PREPARED BY (SIGNATURE) DATE 8-23-66
 1102

Instrument conditions on 72187

Run #7 Sample #1

$$TDD = \frac{(4.5)(.103)(10)}{88.6} = .0355 \text{ mg} / 1.23 \times 10^4 = 2.9 \mu\text{m}$$

Sample #2

$$TDD = \frac{(5.0)(.103)(10)}{88.6} = .0581 \text{ mg} / 1.92 \times 10^4 = 3.0 \mu\text{m}$$

Sample #3

$$TDD = \frac{(5.5)(.103)(10)}{88.6} = .0639 \text{ mg} / 1.96 \times 10^4 = 3.3 \mu\text{m}$$

Sample #4

$$TDD = \frac{(5.5)(.103)(10)}{88.6} = .0639 \text{ mg} / 1.91 \times 10^4 = 3.3 \mu\text{m}$$

Final

$$TDD = \frac{(19.5)(.103)(50)}{88.6} = 1.1334 \text{ mg} / 4.218 \times 10^5 = 2.9 \mu\text{m}$$

- 7-1 -
- 7-2 - 3.4
- 7-3 - 4.3
- 7-4 - 4.9
- 7-F - 2.8

READ AND UNDERSTOOD BY

DATE

W. Bernard 2/24/69

Run #8 TDO Analysis
 2103 2.45-T James L. 9-24-69

TDO Std = 82.3, 88.0, 93.6, 92.0 → 89.0
 (0.5) 190°

Run #8 Sample 1

TDO = $\frac{(10)(.103)(10)}{89.0} = \frac{1.03}{89.0} = 1.157 \times 10^{-4} \text{ g} = 6.8 \text{ ppm}$

Sample 2

TDO = $\frac{(7.5)(.103)(10)}{89.0} = \frac{0.867}{89.0} = 9.74 \times 10^{-4} \text{ g} = 4.6 \text{ ppm}$

Sample 3

TDO = $\frac{(7.0)(.103)(10)}{89.0} = \frac{0.810}{89.0} = 9.10 \times 10^{-4} \text{ g} = 4.1 \text{ ppm}$

Sample 4

TDO = $\frac{(4.5)(.103)(10)}{89.0} = \frac{0.630}{89.0} = 7.08 \times 10^{-4} \text{ g} = 3.7 \text{ ppm}$

Final (20)

(15.7) (103) (50) TDO = $\frac{15.7 \times 103 \times 50}{89.0} = \frac{8011}{89.0} = 89.11 \times 10^{-4} \text{ g} = 2.8 \text{ ppm}$

- 8-1 - 3.1
- 8-2 - 4.8
- 8-3 - 5.2
- 8-4 - 5.1
- 8-F - 3.1

057506

READ AND INTERPRETED BY

DATE

W. Binnard 2/24/69

MONSANTO COMPANY

AGRICULTURAL CHEMICALS DIVISION - RESEARCH DEPARTMENT

72199

245-T
 PRODUCT
 TDD Analysis
 PREPARED BY SIGNATURE
 DATE 9-23-68

ionizing cond. changed from 72183 due to peak shape
 and analysis time. Peaks were broad at low levels
 and not dependable -

Cond. Column oven - 215° Inc. Det - 265° Inj - 325°
 Flows - same - N₂ - 40 psi inlet @ 30 ml/min -
 H₂ 15-R, 15-S - Air 50 psi inlet @ 500 ml/min -

Sample size 0.5 µl - Analysis on 'A' Column

Under these conditions - TDD peak was very sharp and
 resolved from other impurities, with an elution
 time of 3.6 min.

With lower concentrations samples will be concentrated
 and analyzed to see if this approach can be
 utilized to give more defined peak level.

887565

8317473

READ AND UNDERSTOOD BY

DATE

W. Benisard

3/24/69

MONSANTO COMPANY

AGRICULTURAL CHEMICALS DIVISION - RESEARCH DEPARTMENT

72200

TEST ANALYSIS (WV 52.49) Run #7
 PRODUCT 745-T PREPARED BY (SIGNATURE) DATE 9-13-68

Sig. Run = 76.1 mm, 77.4 mm, 77.8 mm → 77.8 mm
 (0.5 x) x 50
 215°C

52149-1 (7-1) 100 = $\frac{(13)(.103)(10)}{(77.8)(2.5)}$ = 5.7 ppm
 (17.1)

52149-2 (7-2) 100 = $\frac{(35)(.103)(10)}{(77.8)(2.5)}$ = 12.2 ppm
 (11.4)

52149-3 (7-3) 100 = $\frac{(53.7)(.103)(10)}{(77.8)(2.5)}$ = 20.6 ppm
 (12)

52149-4 (7-4) 100 = $\frac{(112.5)(.103)(10)}{(77.8)(2.5)}$ = 30.0 ppm
 (11)

Cont'd Book # 85301 - 85400

U.L. = (vol. ppm to 100)

7-1 - 16.2
 7-2 - 14.4
 7-3 - 6.1
 7-4 - 3.5

854500

8317-77.1

READ AND UNDERSTOOD BY

DATE

W. Deminard 2/34/68