

Monsanto

COMPANY

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St. Louis, Missouri 63166
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May 9, 1973

Mr. Louis DiBello
W. R. Grace and Company
Cryovac Division
Duncan, South Carolina 29334

Re: Task Group for PCB Analysis
on Flexible Barrier Materials -
Sub Committee F2.30

Dear Mr. DiBello:

Enclosed are the results of our analyses of the four task group samples for polychlorinated biphenyls (PCBs). The samples were analyzed, as requested, using the tentative procedure and our usual laboratory procedure. The instrumental conditions are shown on attachments I and II.

Comparisons of the Aroclor 1242 and Aroclor 1254 standards sent to us with our standards are shown in Chromatograms I-IV (tentative procedure) and in Chromatograms V-VIII (Monsanto procedure). Minor differences were noted in the homolog distributions of the standards sent vs ours. The Aroclor 1242 and Aroclor 1254 standards sent contained less of the lower chlorinated homologs than ours, as witnessed by ratioing the early peak areas to the late peak areas. However, these differences were not significant enough to affect quantitation of the samples.

Shown on attachments III and IV are sample calculations for the tentative procedure and the Monsanto procedure, respectively. The tentative procedure calculation was modified to compensate for the different sample sizes injected into the instrument and our dilution of the sample to 25 ml. These changes were necessary because of the sensitivity of our detectors and the fact that we, as a matter of practice, use the mid-range of the syringe.

The results of our analyses are summarized in Table I. It appeared that some of sample #4 was lost during shipment. We assumed that the loss was due to volatility, and that the bulk of the PCBs remained in the vial. Chromatograms of the samples are enclosed: IX-XII (tentative procedure), XIII-XVI (Monsanto procedure).

In our opinion, the method of calculation used in the tentative procedure causes a small, but clearly observable, positive bias (see Table I). This bias occurs because the calculation ignores ~10-25% of the area from the standards. Furthermore, when dealing

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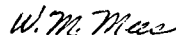
with a sample containing only Aroclor 1242, it is incorrect to ask how much Aroclor 1254 is present and vice versa. However, when one is clearly dealing with samples which contain detectable levels of more than one Aroclor, splitting the chromatogram into parts is a legitimate means of estimating the total amount of PCBs present.

For a number of good reasons, we also recommend the use of calibration curves prepared with one or more of the commercial PCB mixtures (Aroclor) which most closely match the residue, since the suggested ratio technique can, under certain conditions, lead to serious errors.

Very truly yours,



E. S. Tucker



W. M. Mees

db
Attach.

0270334

TOWOLDMON0054249

ATTACHMENT I

INSTRUMENTAL CONDITIONS

ASTM F2.30

Tentative Method for the Analysis of Polychlorinated Biphenyls

Instrument: H&P 5750 Research Gas Chromatograph
equipped with a High Temperature Ni-63
Electron Capture Detector

Column: 6' X 1/8" Stainless Steel, 10% DC-200
(12,500 cs) on 80/100 Mesh Chromosorb W H.P.

Column Temp: 220°C

Injection Temp: 325°C

Detector Temp: 325°C

Carrier Gas: Helium - 60 ml/min

Purge Gas: 10% Methane/Argon - 60 ml/min

Pulse Interval: 50 μ sec

Chart Speed: 1/2"/min

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TOWOLDMON0054250

ATTACHMENT II

INSTRUMENTAL CONDITIONS

Monsanto, Analytical Chemistry Method 71-35

Instrument: H&P 5750 Research Gas Chromatograph
equipped with a High Temperature Ni-63
Electron Capture Detector

Column: 2M X 4 mm Glass, 4% XE-60 on 80/100 Mesh
Chromosorb W H.P.

Column Temp: 170°C - Aroclor 1242
185°C - Aroclor 1254

Injection Temp: 220°C

Detector Temp: 250°C

Carrier Gas: Helium - 60 ml/min

Purge Gas: 10% Methane/Argon - 60 ml/min

Pulse Interval: 50 μ sec

Chart Speed: 1/2"/min

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TOWOLDMON0054251

ATTACHMENT III

SAMPLE CALCULATION

ASTM F2.30

Tentative Method for the Analysis of Polychlorinated Biphenyls

	<u>Aroclor 1242</u>	<u>Aroclor 1254</u>	<u>TFFB #4</u>
Area 96-234 sec	1794	203	231
235-888 sec	659	2888	2862
µl injected	5 µl	4 µl	7 µl
Std conc	1.014 µg/ml	0.977 µg/ml	-

$$\mu\text{g Aroclor 1242} = \frac{1.014 \mu\text{g/ml} \times 25 \text{ ml} \times 5 \mu\text{l} \times 231}{1794 \times 7 \mu\text{l}} = 2.3 \mu\text{g}$$

$$\mu\text{g Aroclor 1254} = \frac{0.977 \mu\text{g/ml} \times 25 \text{ ml} \times 4 \mu\text{l} \times 2862}{2888 \times 7 \mu\text{l}} = 13.8 \mu\text{g}$$

Total µg of PCB = 16.1 µg

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TOWOLDMON0054252

ATTACHMENT IV

SAMPLE CALCULATION

ASTM F2.30

Monsanto, Analytical Chemistry Method 71-35

See Calibration Curve

Area of Sample TFFB #4 - 3462

ng of Aroclor 1254 in Sample TFFB #4 from Calibration Curve - 2.9 ng

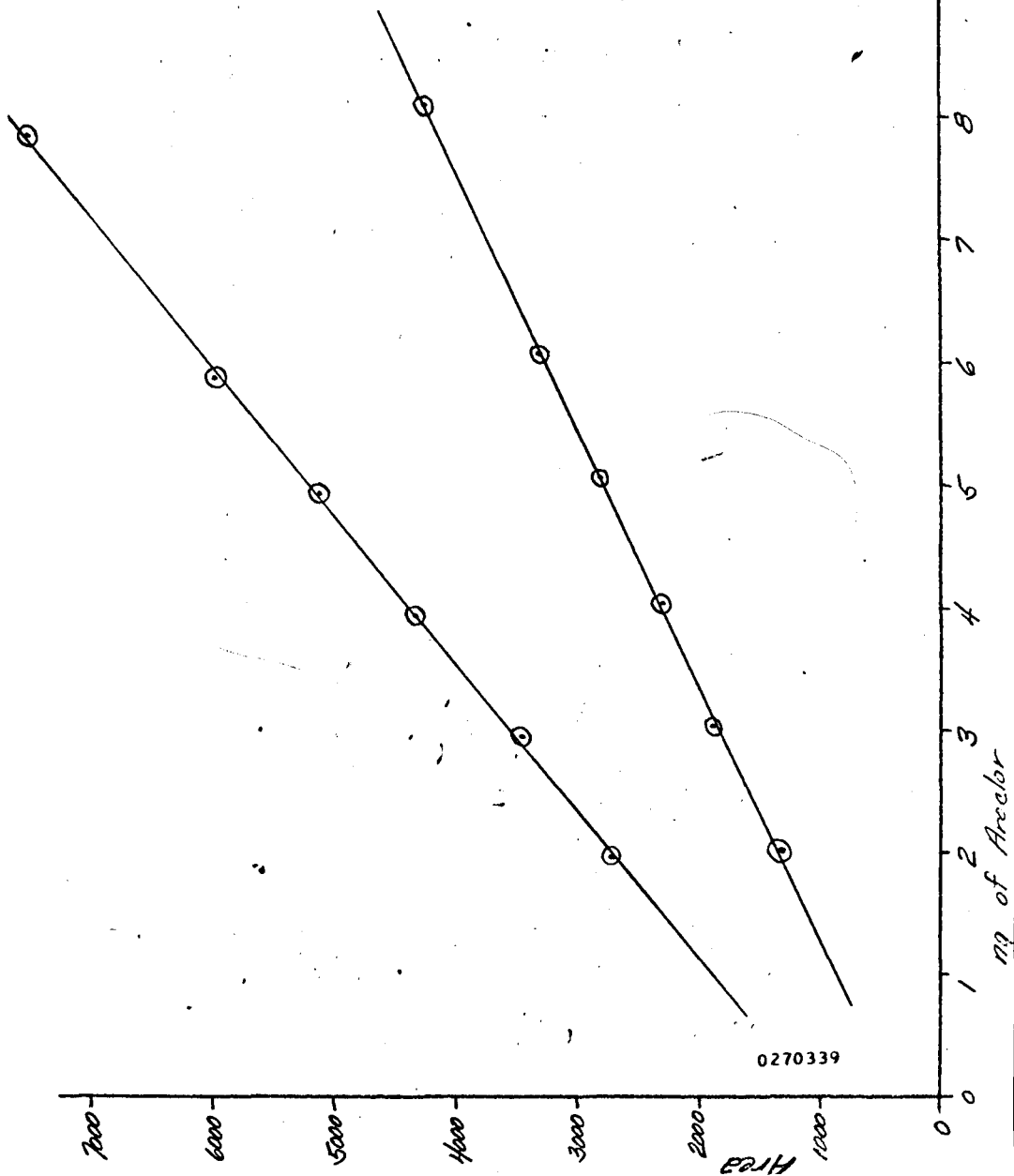
$$\frac{2.9 \text{ ng}}{5 \text{ } \mu\text{l}} \times \frac{10^3 \text{ } \mu\text{l}}{\text{ml}} \times \frac{\mu\text{g}}{10^3 \text{ ng}} \times 25 \text{ ml} = 14.5 \text{ } \mu\text{g of Aroclor 1254}$$

Total μg of PCB = 14.5

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TOWOLDMON0054253

Attachment IV a



0270339

TABLE I

<u>Sample No.</u>	<u>Task Group's Tentative Procedure</u>	<u>Monsanto Analytical Chemistry Method 71-35</u>
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1

31.8

26.7

31.2

26.6

2

25.6

20.3

25.2

21.1

3

35.0

31.9

35.5

30.8

4

16.7

14.3

16.1

14.5

*Arvelor 12422
12543*

mg?

Duplicate

0270340

TOWOLDMON0054255

Chromatogram I

*

$$1425 - (3.7 \times 3.6 \times 4.74) = 631$$

1794

~~2071~~

$$1527 - (3 \times 21 \times 4.74) = 899$$

559

~~1762~~

$$- (2.5 \times 48 \times 4.74) = 569$$

5% TFB Cauden 12H2 - 10/11/10

0 1 2 3 4 5 6 9

0 1 2 3 4 5 6 9

0270341

4x10

~~2 1 5~~

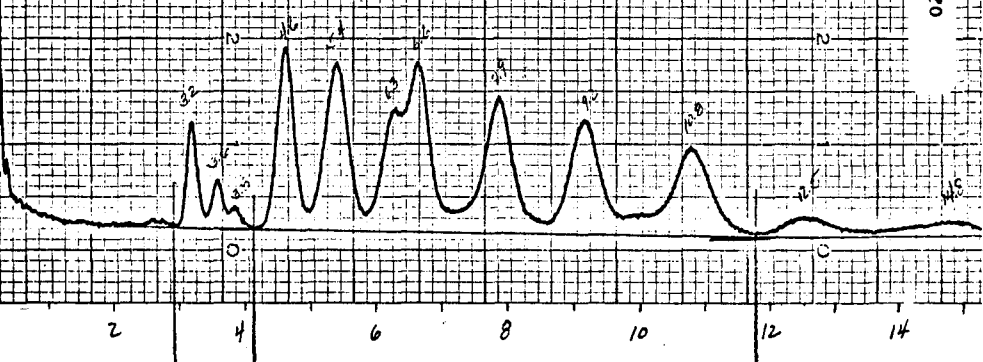
$$350 - (2 \times 15.5 \times 4.74) = 14$$

$$3917 - (1.6 \times 97 \times 4.74)$$

$$- (1.2 \times 51.5 \times$$

4.4 TFB Linear Plot

0270342



2283 - 524
 (21 x 27 x 4.74)
 1250 - 524
 (21 x 27 x 4.74)
 1250 - 524
 (21 x 27 x 4.74)

1459 2285 - (3.2 x 6.5 x 4.74) = 99
 153 1257 - (2.5 x 21 x 4.74) = 42
 1257 - (2 x 48 x 4.74) = 45

4th Order 12H2

1051 x 10⁶ / msc

4x10

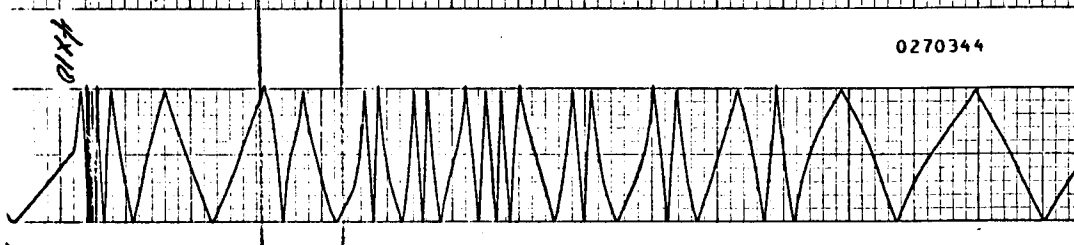
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4th Order 12H2

0.012 x 10⁶ / msc

4x10

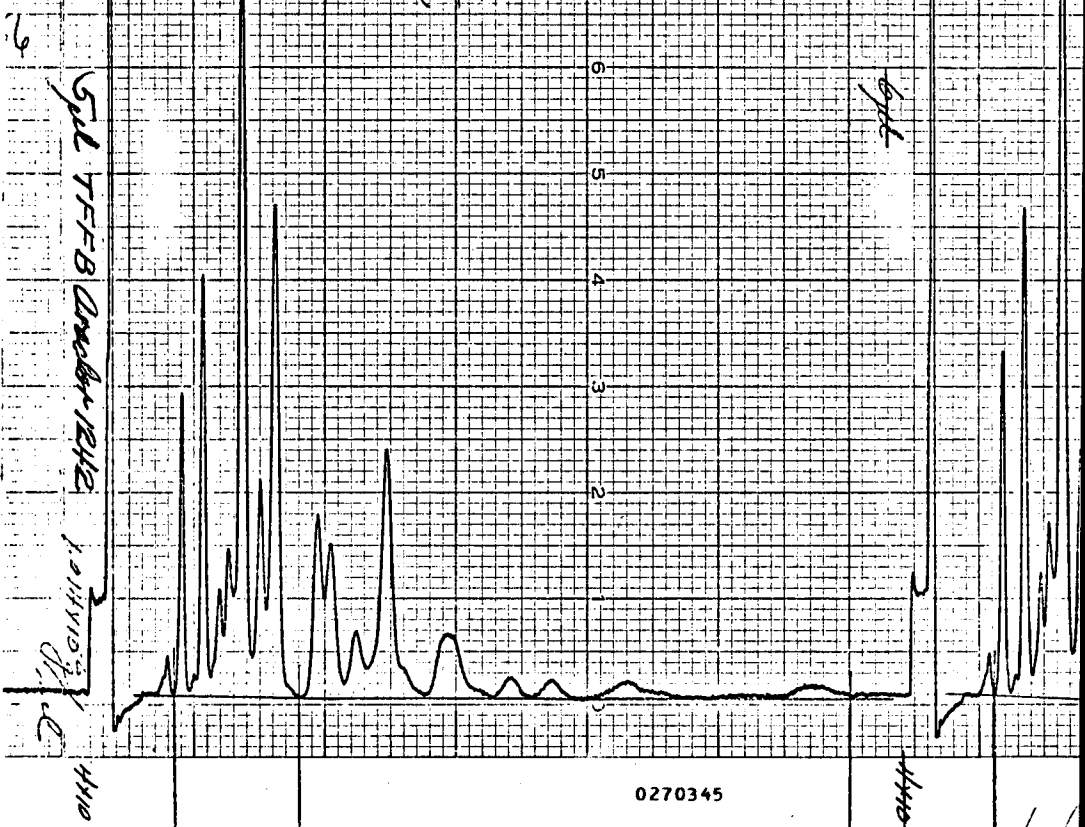
Chromatogram IV



2773

$$3118 - (8 \times 24 \times 4.74) = 91$$

$$- (5 \times 107 \times) = 254$$



Chem: F. n. vi. V

4697 - (40.5 x 1.1 x 4.74) 2.1
 (22.5 x 2.5 x 4.74) 2.67
 (65.2 x 0.6 x 4.74) 10.7

Hold

4 pul TFFB, Arcolex 1254

0.0001 sec

4x10

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4x10

TOWOLD MON 0054261

281.3

$$3110 - (1.3 \times 24 \times 4.74) = 148$$

$$- (1.3 \times 10.5 \times 4.74) = 149$$

4th Order 12/12

1.051 x 10⁻⁴

2nd 11-F-B - Order 12.54

0.997 x 10⁻⁶ 1/100

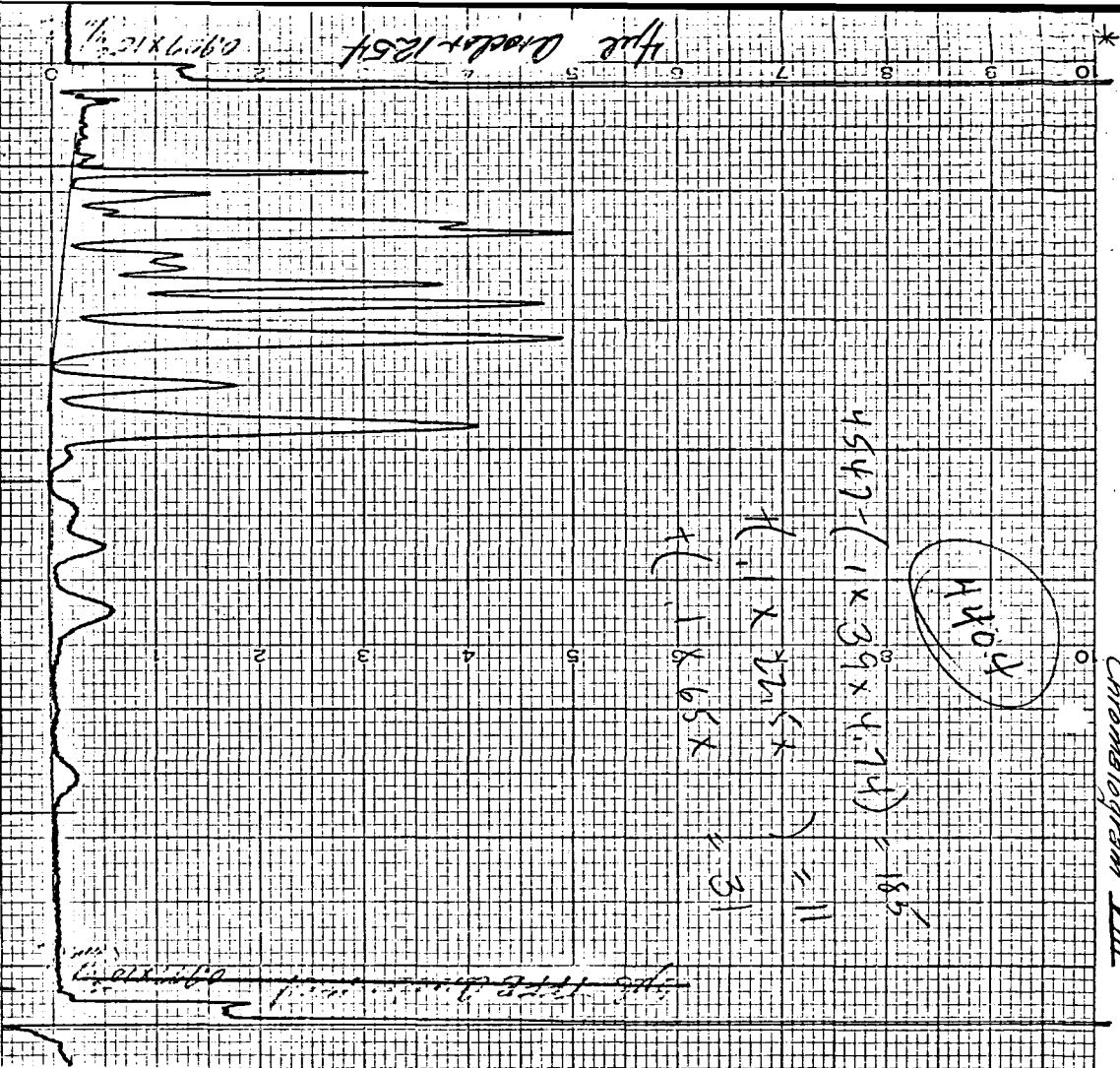
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H₂O

4547-(1 x 39 x 4.74) = 185

4(1 x 20.5) = 11

4(1 x 6.5) = 31



0.007815%

File 00001251

0270346

11/11

Chromatogram IX

* 0.25 ml

25 ml

$$2167 - (1.5 \times 36.5 \times 4.74) = 266 \quad (1907)$$

$$2180 - (1.5 \times 36.5 \times 4.74) = 266$$

$$743 - (1.6 \times 20.5 \times 4.74) = 58 \quad (722)$$

$$870 - (1.6 \times 20.5 \times 4.74) = 58$$

$$+ (1.1 \times 19 \times 4.74) = 9$$

$$+ (1.2 \times 30 \times 4.74) = 28$$

5 ml TFFB #1

5 ml TFFB #1

0270349

4x10

4x10

Chromatogram X

* 25ml

5µl TFFB #2

1820 - (35 x 1.65 x 4.74)

789 - (38 x 1 x 4.74)

= (32 x 0.3 x 4.74)

25ml

5µl TFFB #2

1544

560

607

570

0270350

4x10

4x10

Chromatogram XI

* 25 ml

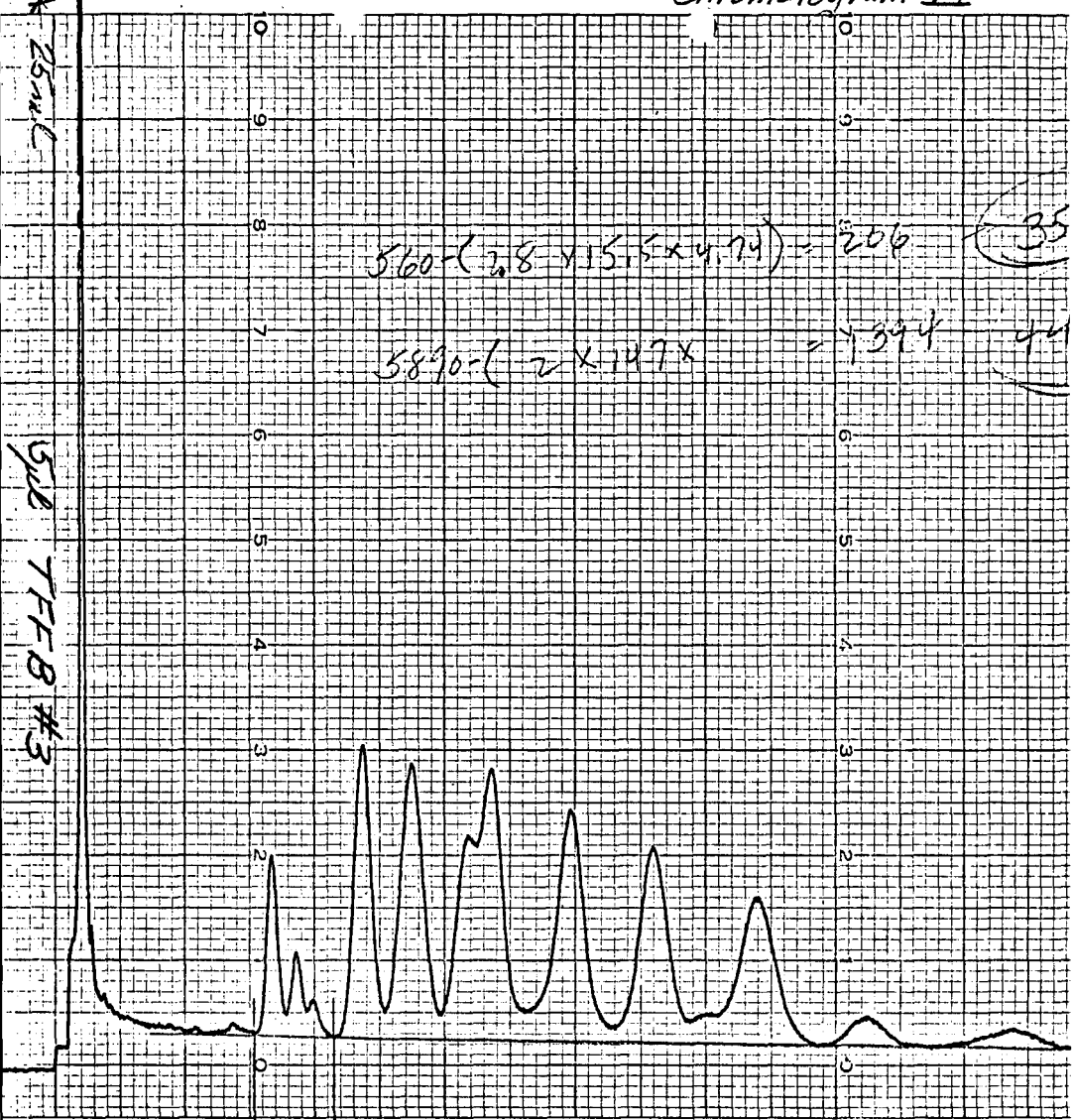
5.2 TFFB #3

$$560 - (2.8 \times 15.5 \times 4.74) = 206$$

(35)

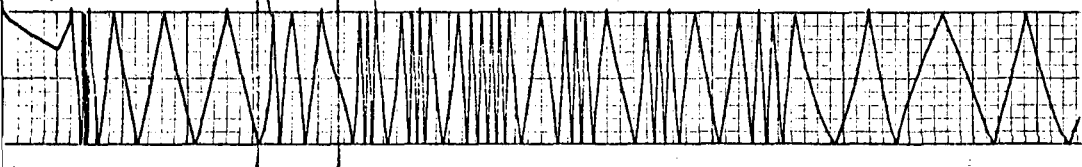
$$5890 - (2 \times 147 \times 4.74) = 5394$$

(44)



4x10

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Chromatogram XII

* 25.1

76c TFFB #4

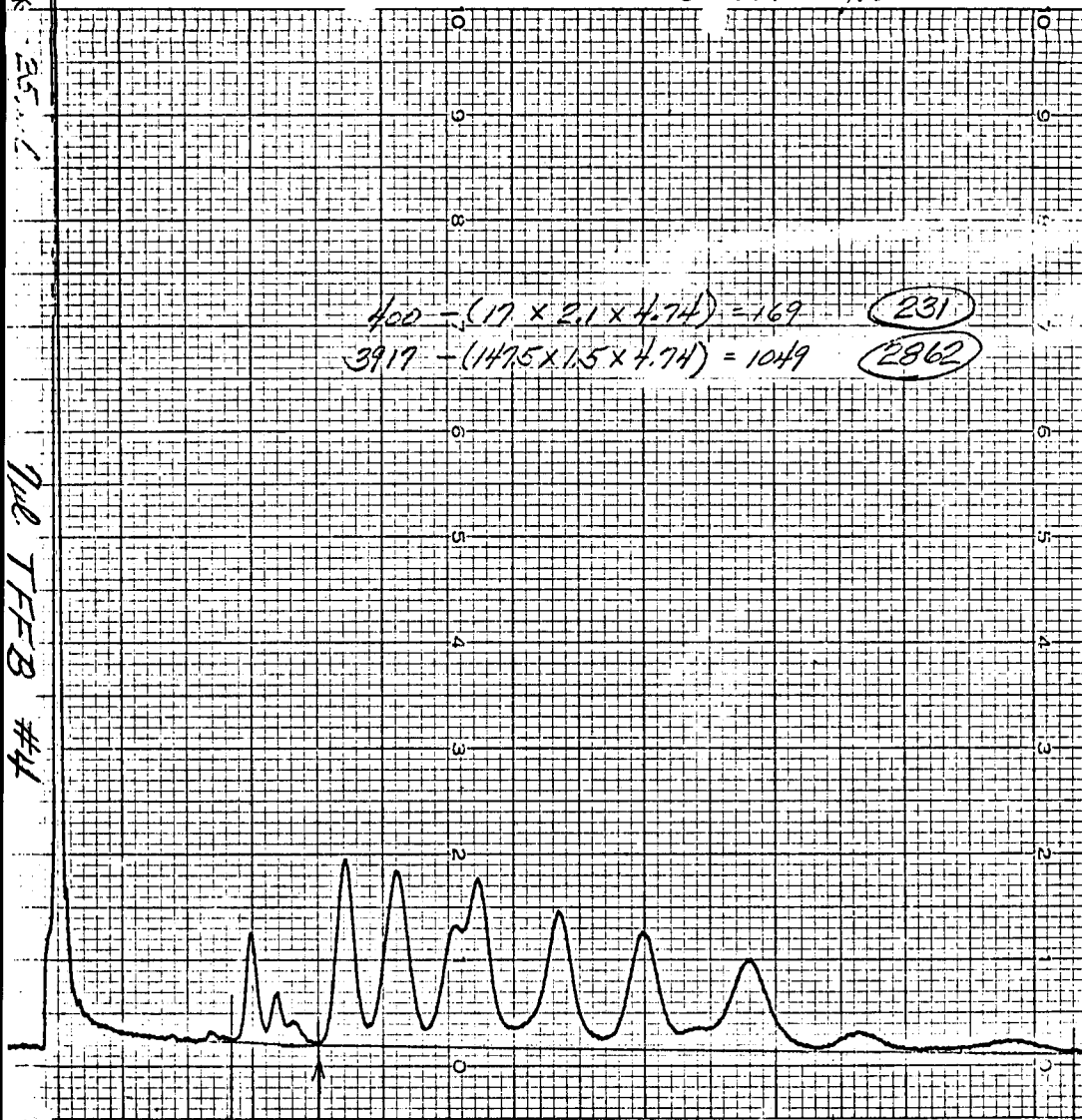
0110

$$400 - (17 \times 2.1 \times 4.74) = 169$$

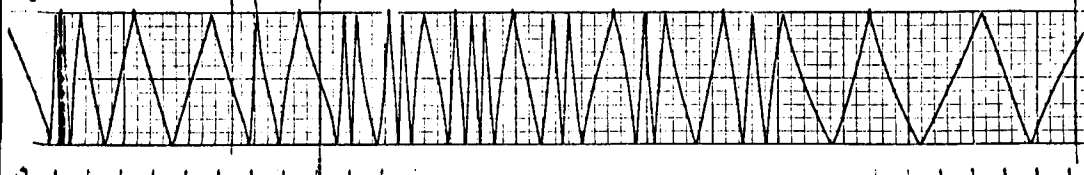
(231)

$$3917 - (1475 \times 1.5 \times 4.74) = 1049$$

(2862)



0270352



TOWOLDMON0054267

Chromatogram XIII

* 25 mL

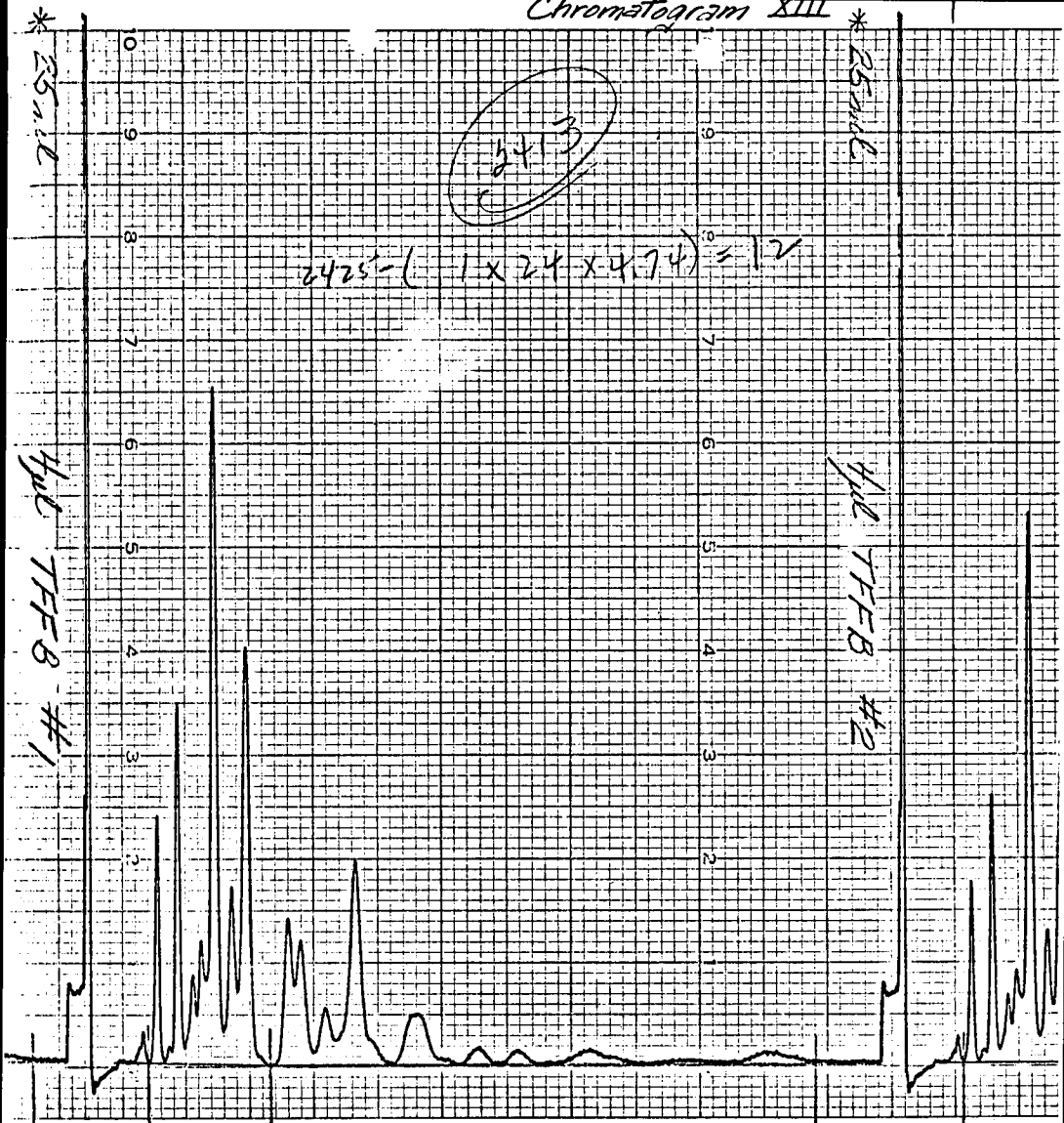
* 25 mL

2413

$$2425 - (1 \times 24 \times 4.74) = 12$$

4th TFFB #1

4th TFFB #2



4x4

0270353

4x4



Chromatogram XIV

25 ml

1/4 TFFB #2

4x10

1.892

2080-(3x.32x4.74) = 188

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* 25 ml

1/4 TFFB #2

4x10

Chromatogram XV

25 ml

4 μ l TFFB #4

1x10

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43.5 - (0.4 x 61.5 x 4.74) = 117
= (0.5 x 65.5 x 4.74) = 155

42.90
4.74

* 25 ml

3 μ l TFFB #3

4x10

Chromatogram XVI*

