bcc: R. E. Keller
W. B. Papageorge

Monsanto
COMPANY

800 N. Lindbergh Boulevard
St. Louis, Missouri 63166
(314) OXford 4-1000

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

E. S. Tucker

W. M. Mees

W. M. Mees

db
Attach.

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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: ~~200~~°C 200

Injection Temp: ~~325~~°C VVS

Detector Temp: ~~325~~°C 300 - 325

Carrier Gas: Helium - 60 ml/min

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

Pulse Interval: 50 µsec

Chart Speed: 1/2"/min

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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 µsec

Chart Speed: 1/2"/min

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ATTACHMENT III

SAMPLE CALCULATION
ASTM F2.30
Tentative Method for the Analysis of Polychlorinated Biphenyls

	Aroclor 1242	Aroclor 1254	TFFB #4
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	-

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

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

Total µg of PCB = 16.1 µg

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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

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

Total µg of PCB = 14.5

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Attachment IV a

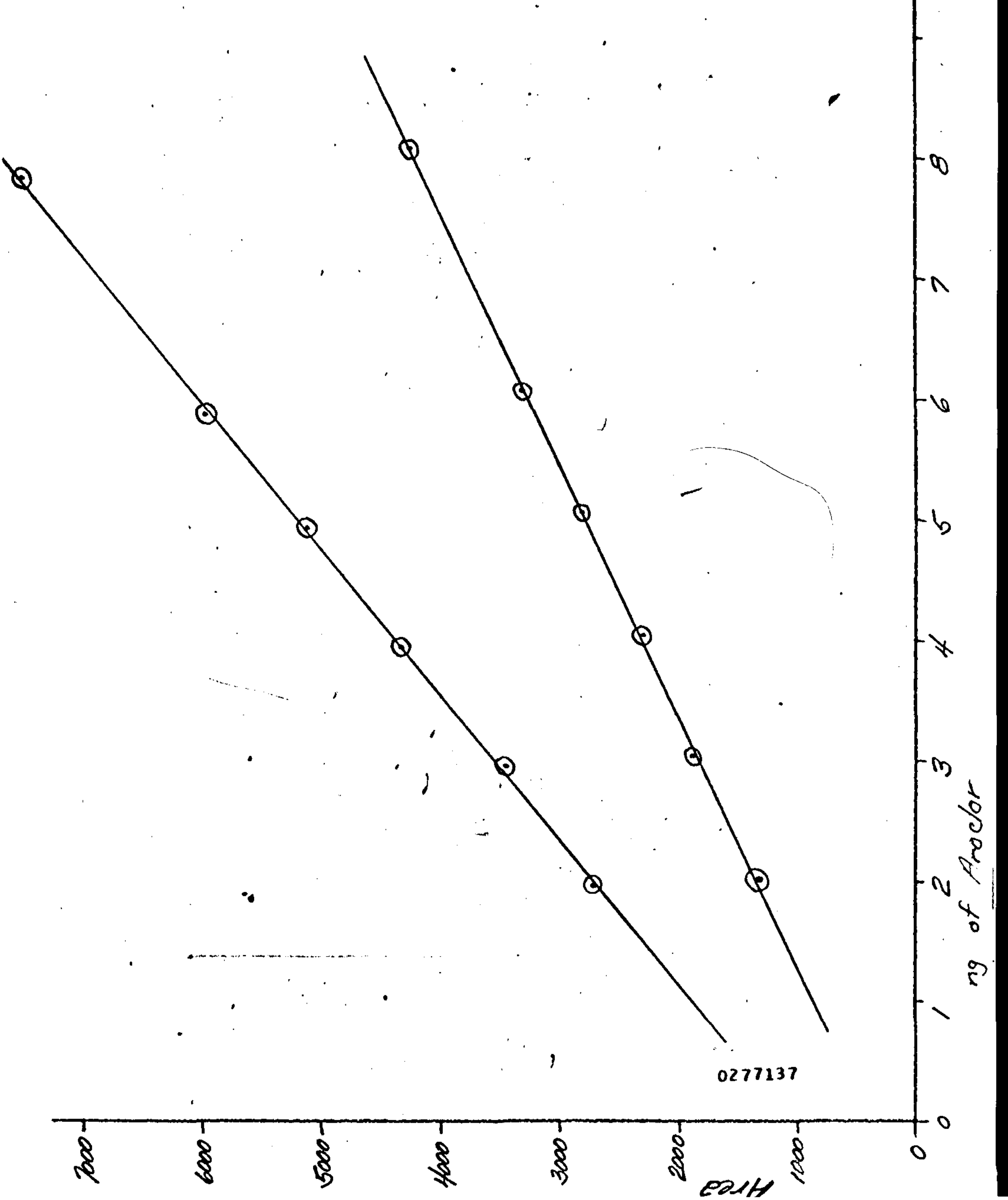

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TABLE I

Sample No.	Task Group's Tentative Procedure	Monsanto Analytical Chemistry Method 71-35
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

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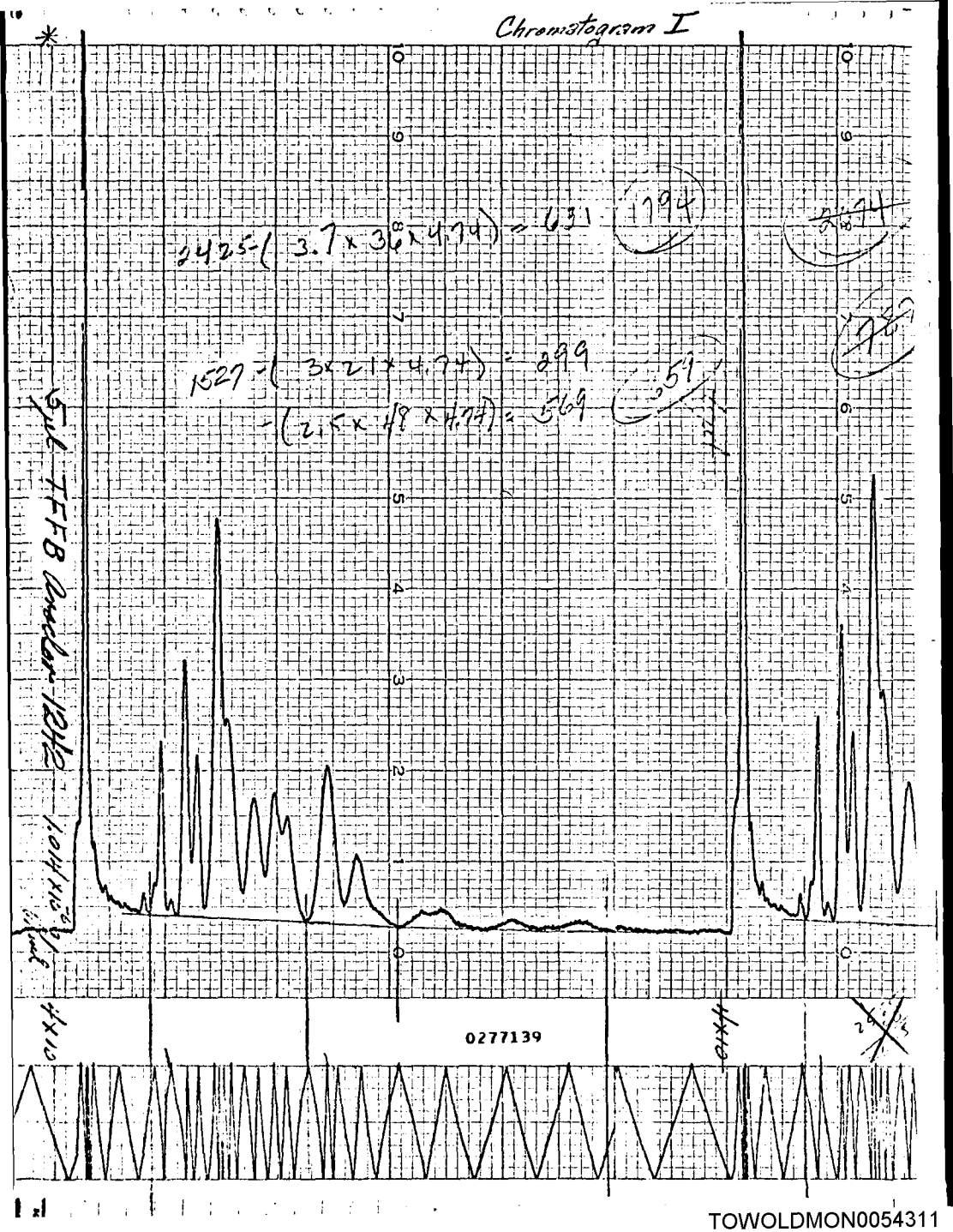

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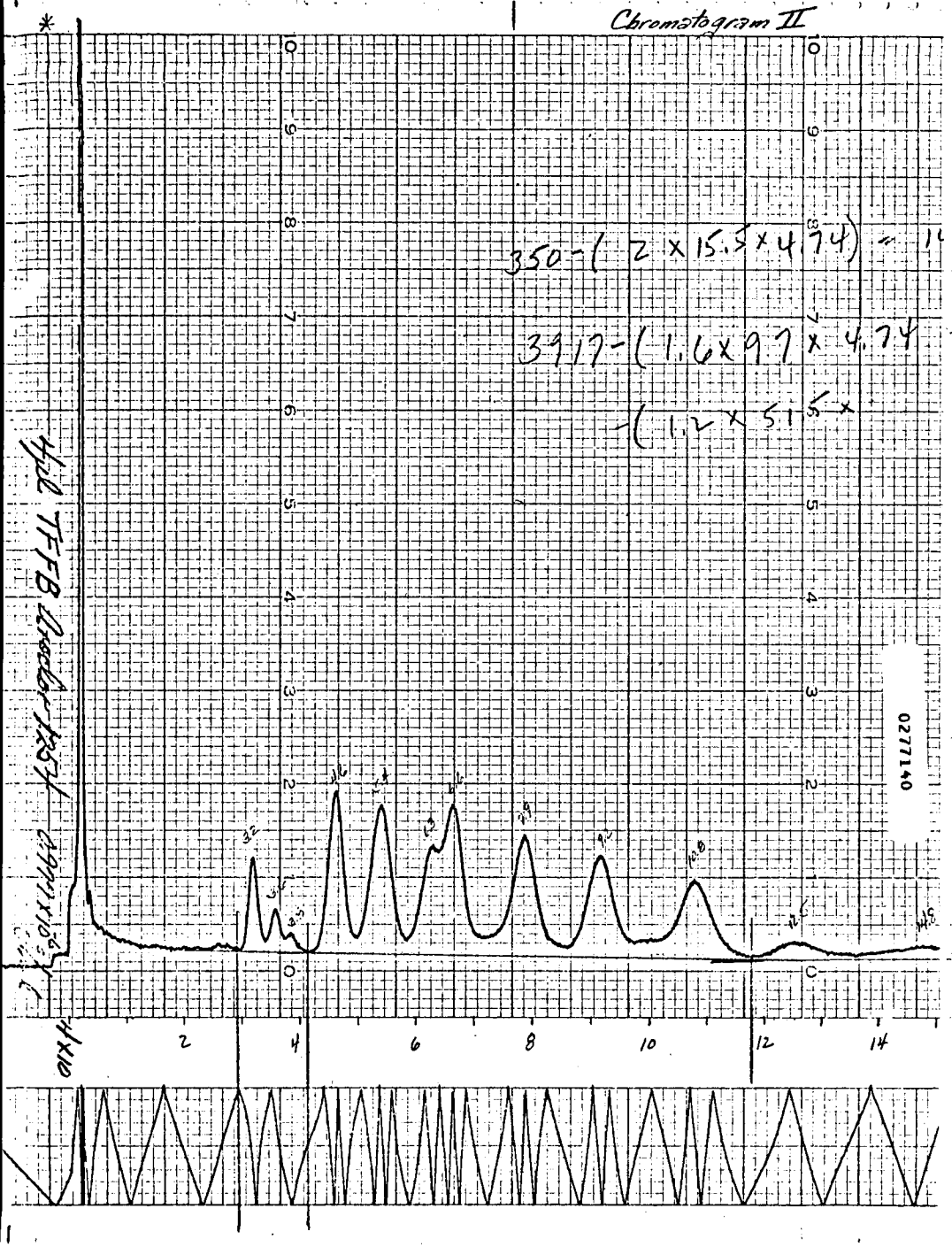
Chromatogram II



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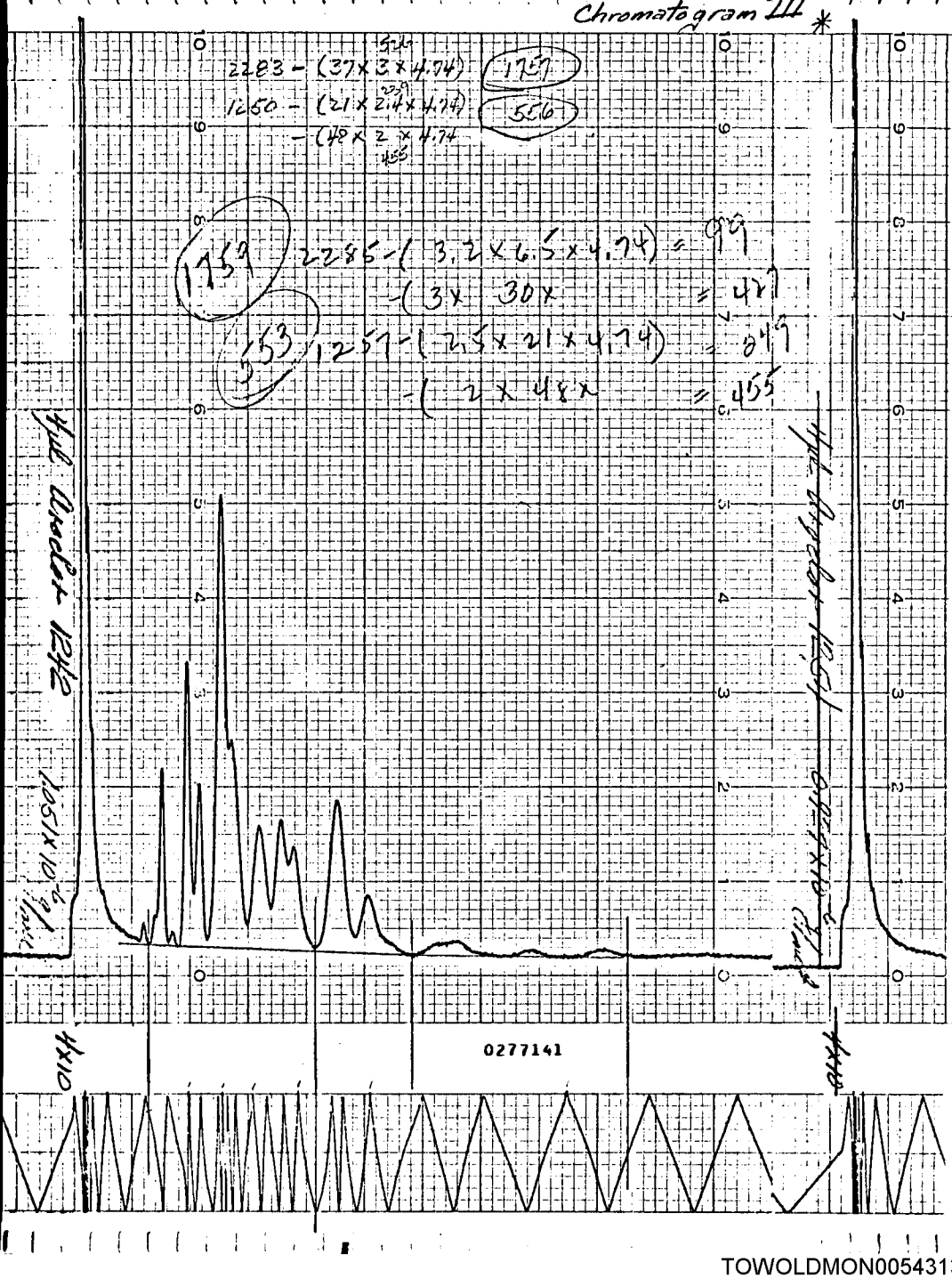

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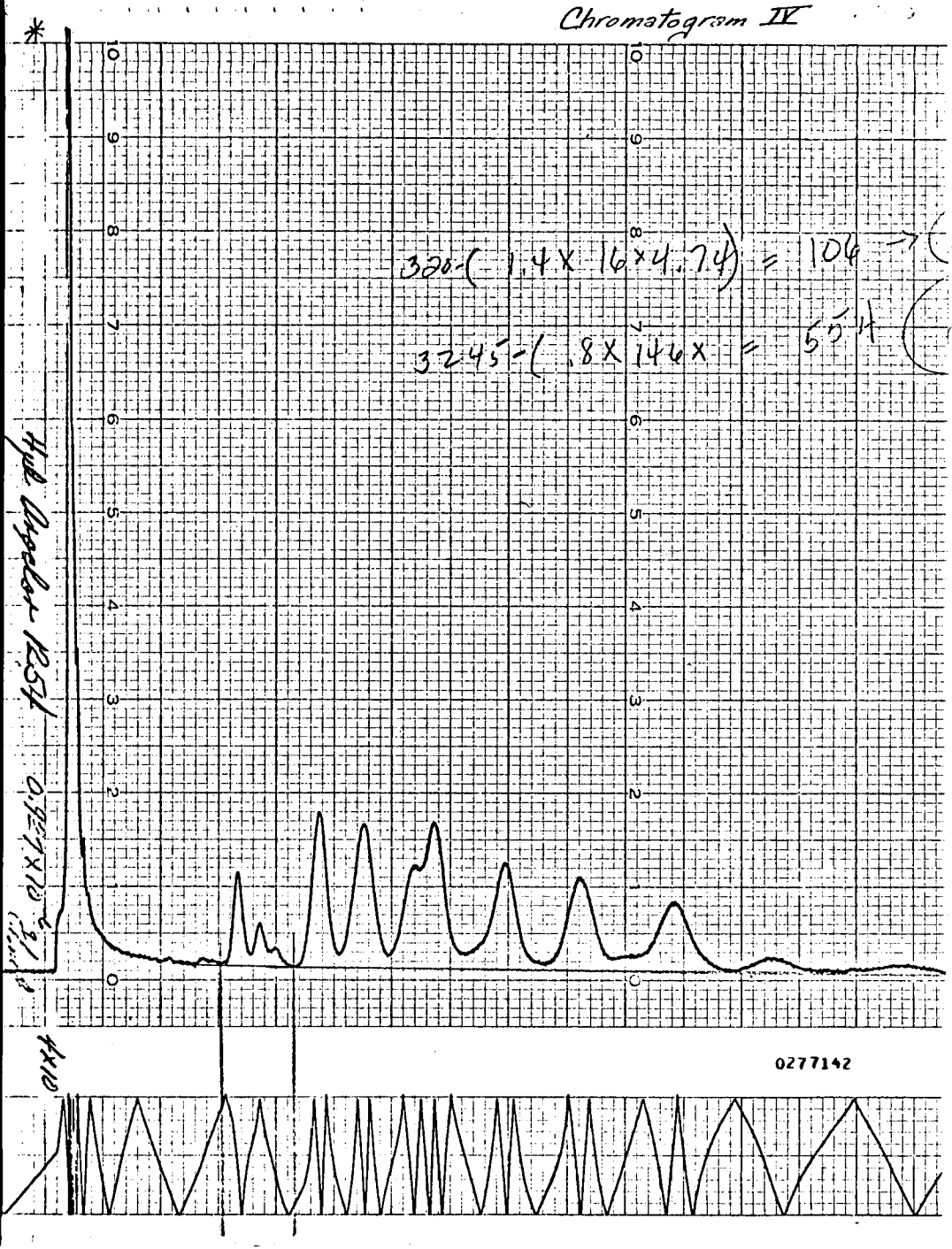
Chromatogram IV



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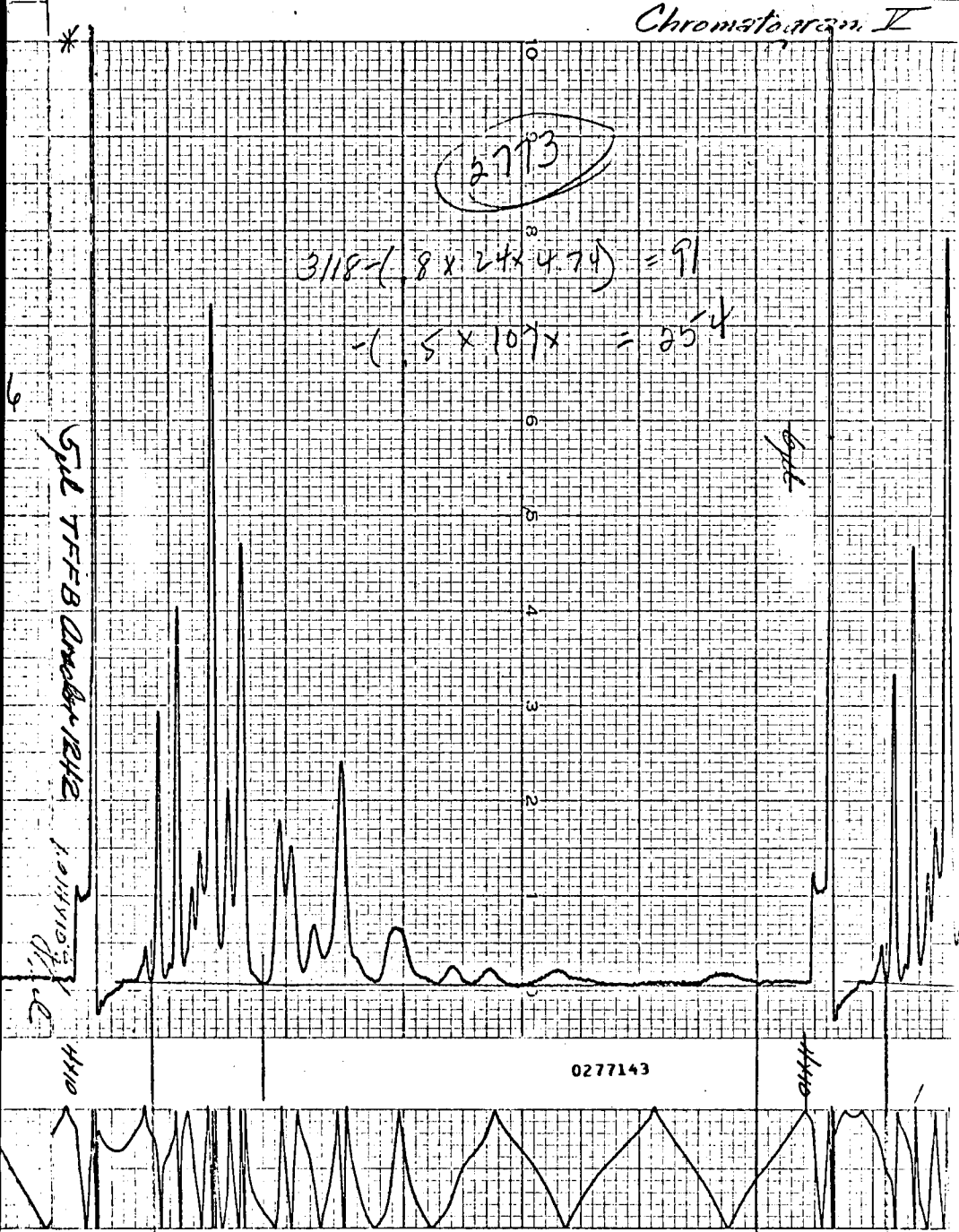
Chromatogram IV



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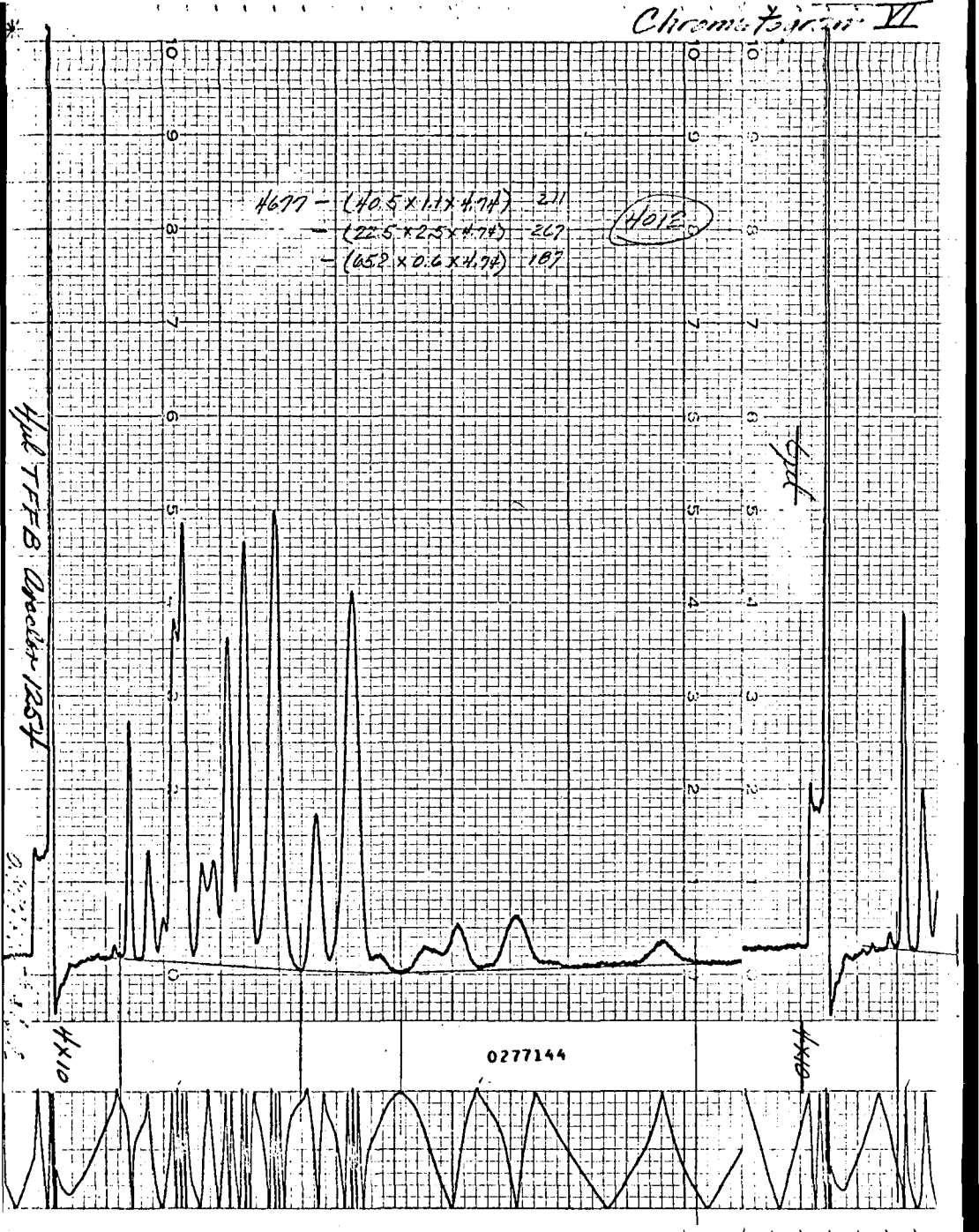

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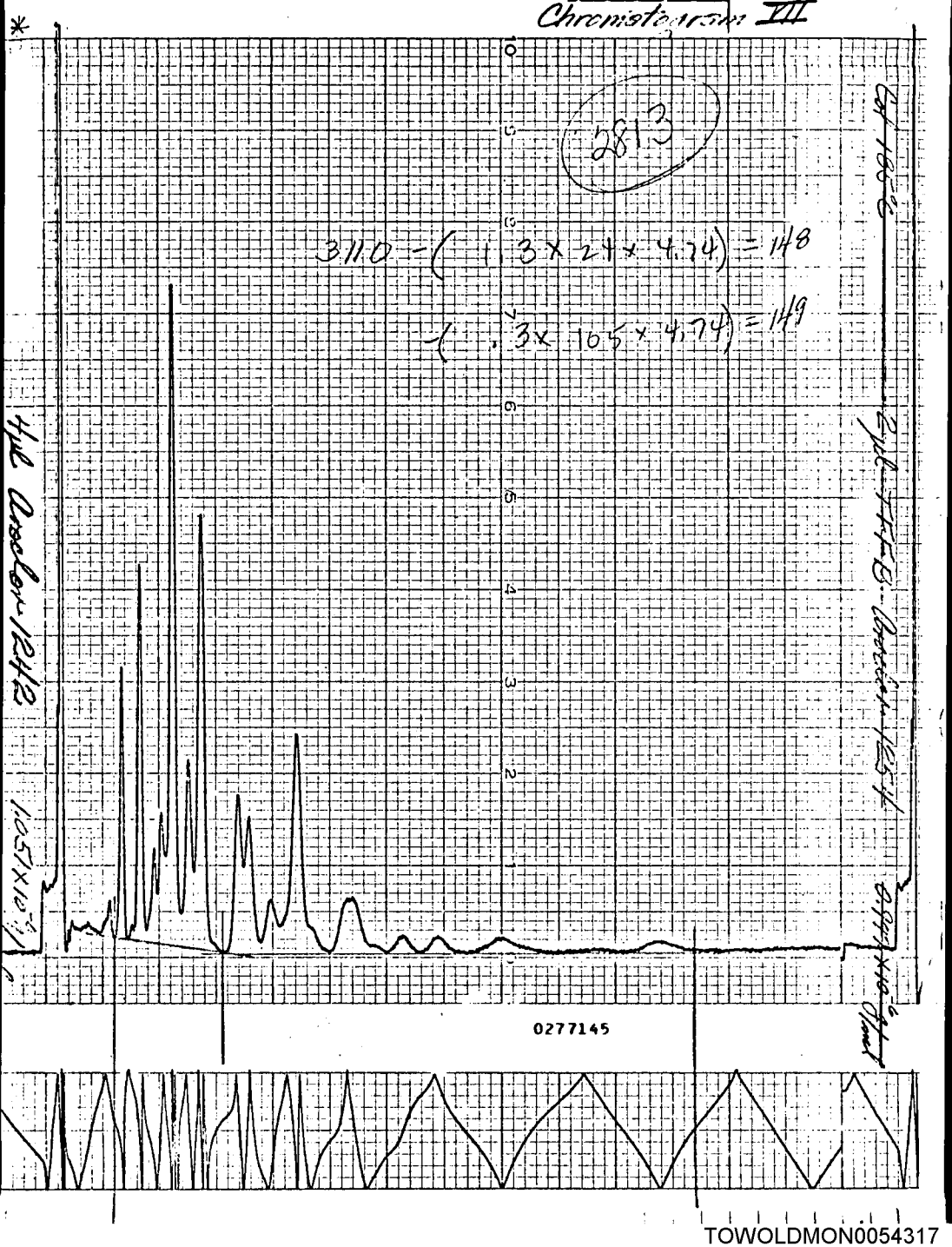

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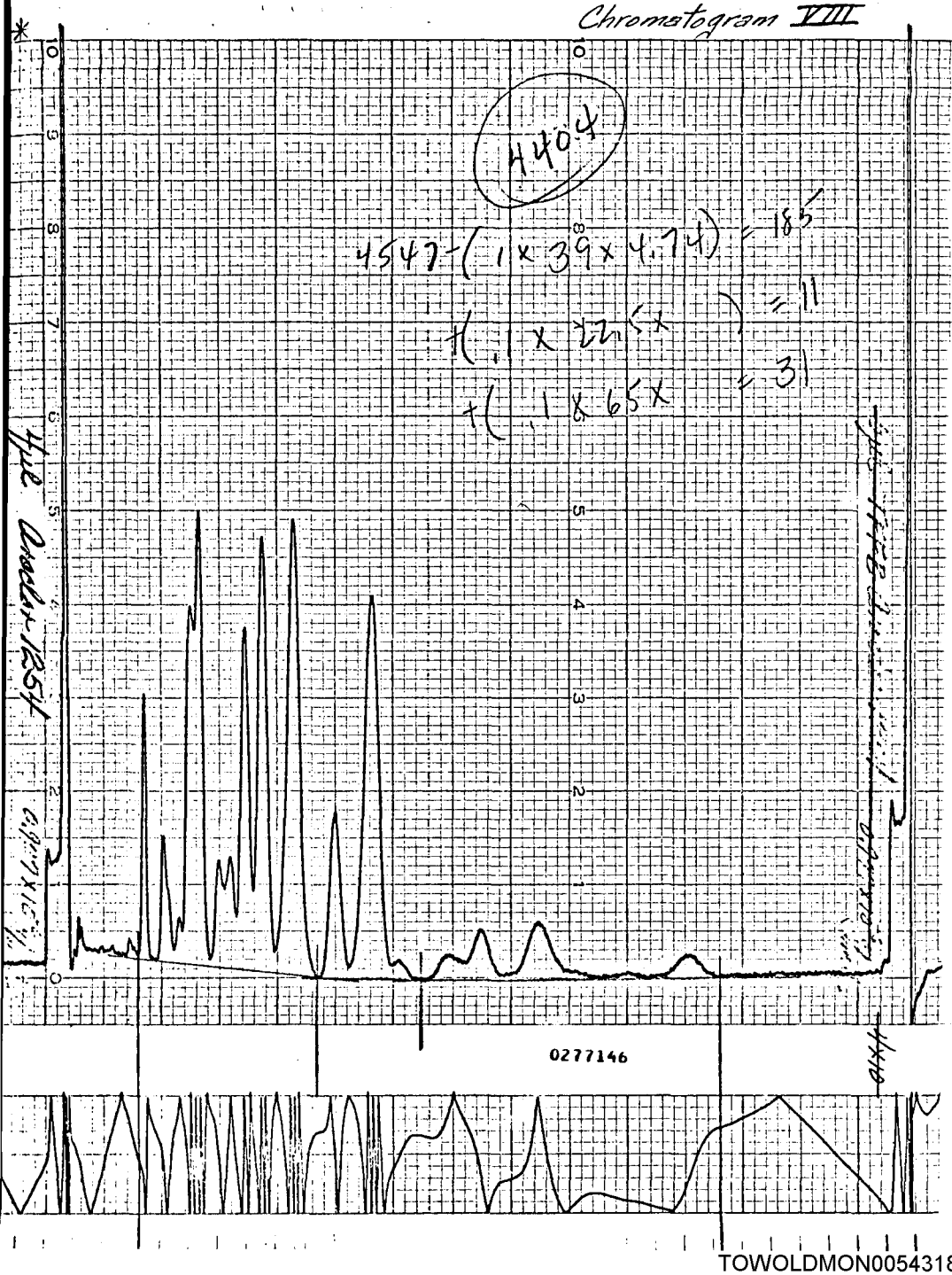
Chromatogram VIII



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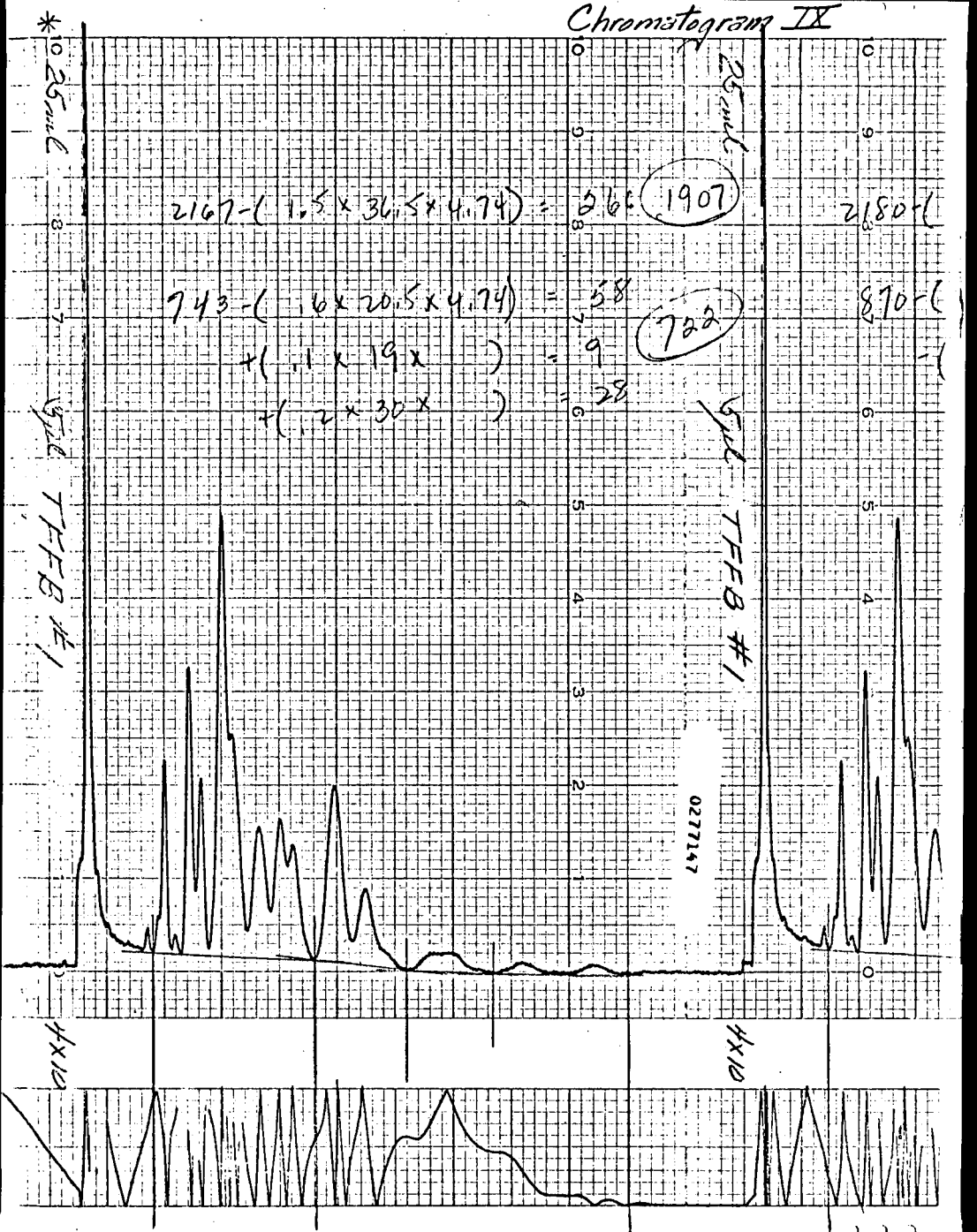
Chromatogram IX



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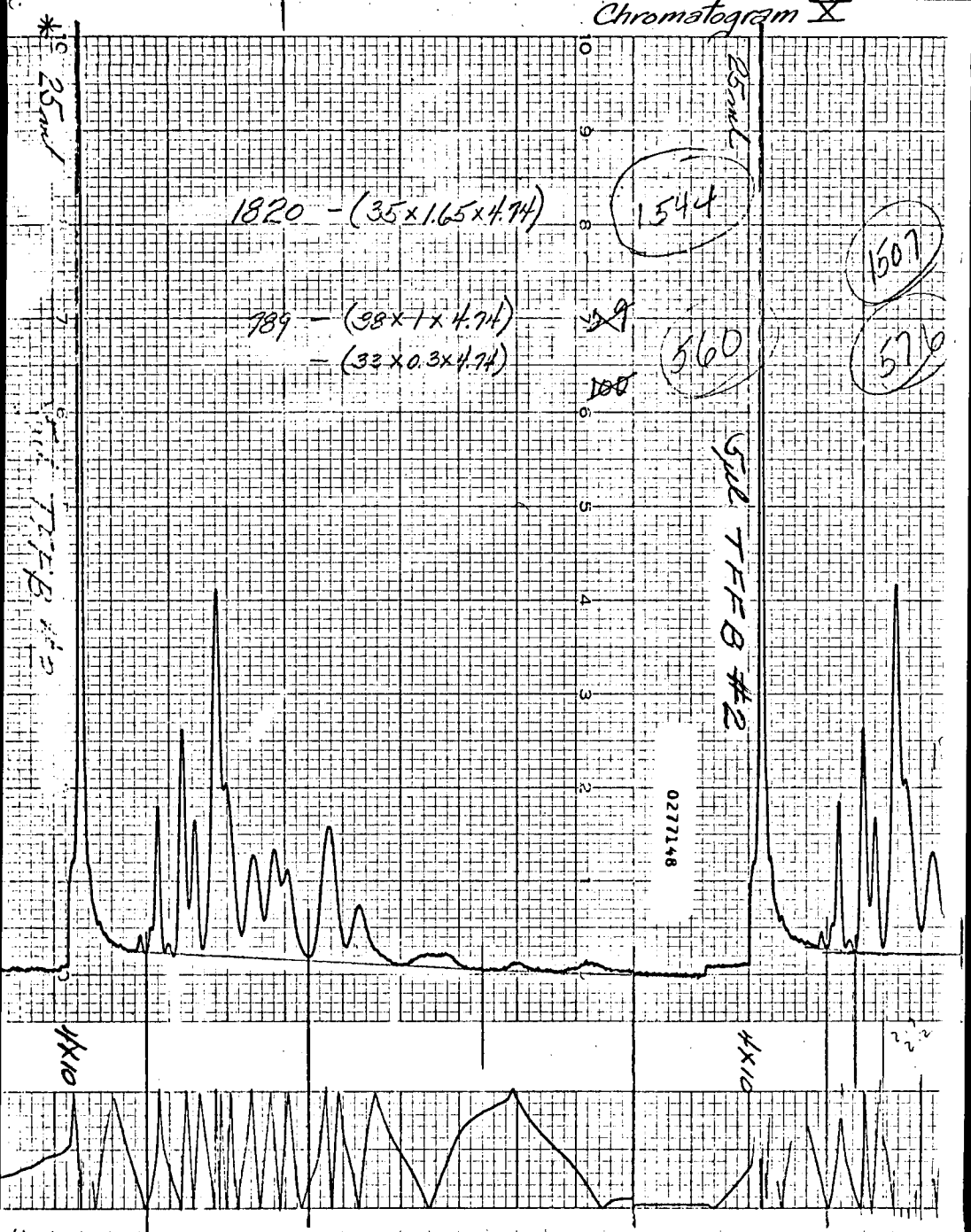

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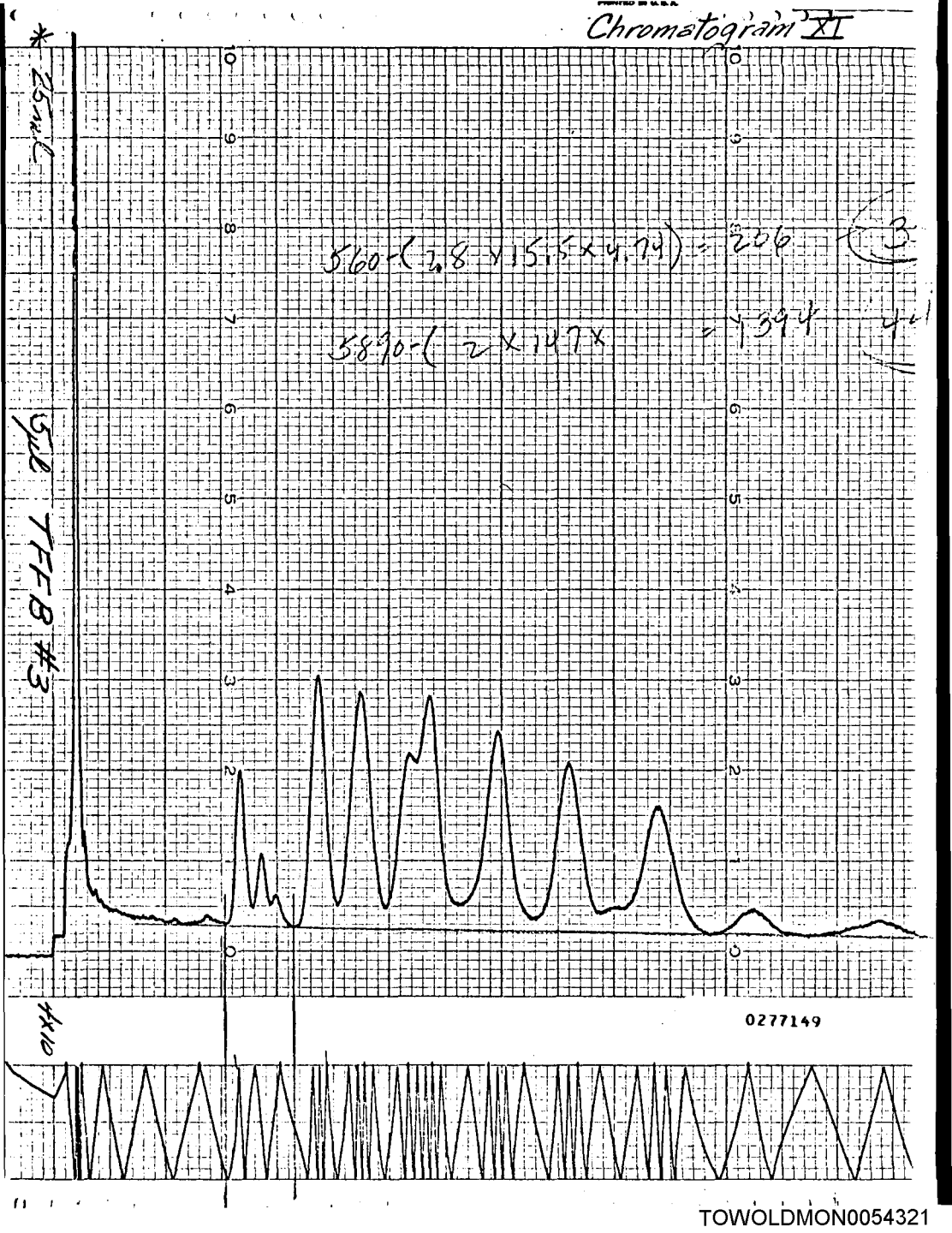
Chromatogram XI



560-(2.8 x 15.5 x 4.74) = 206 (3

580-(2 x 147 x = 394 4

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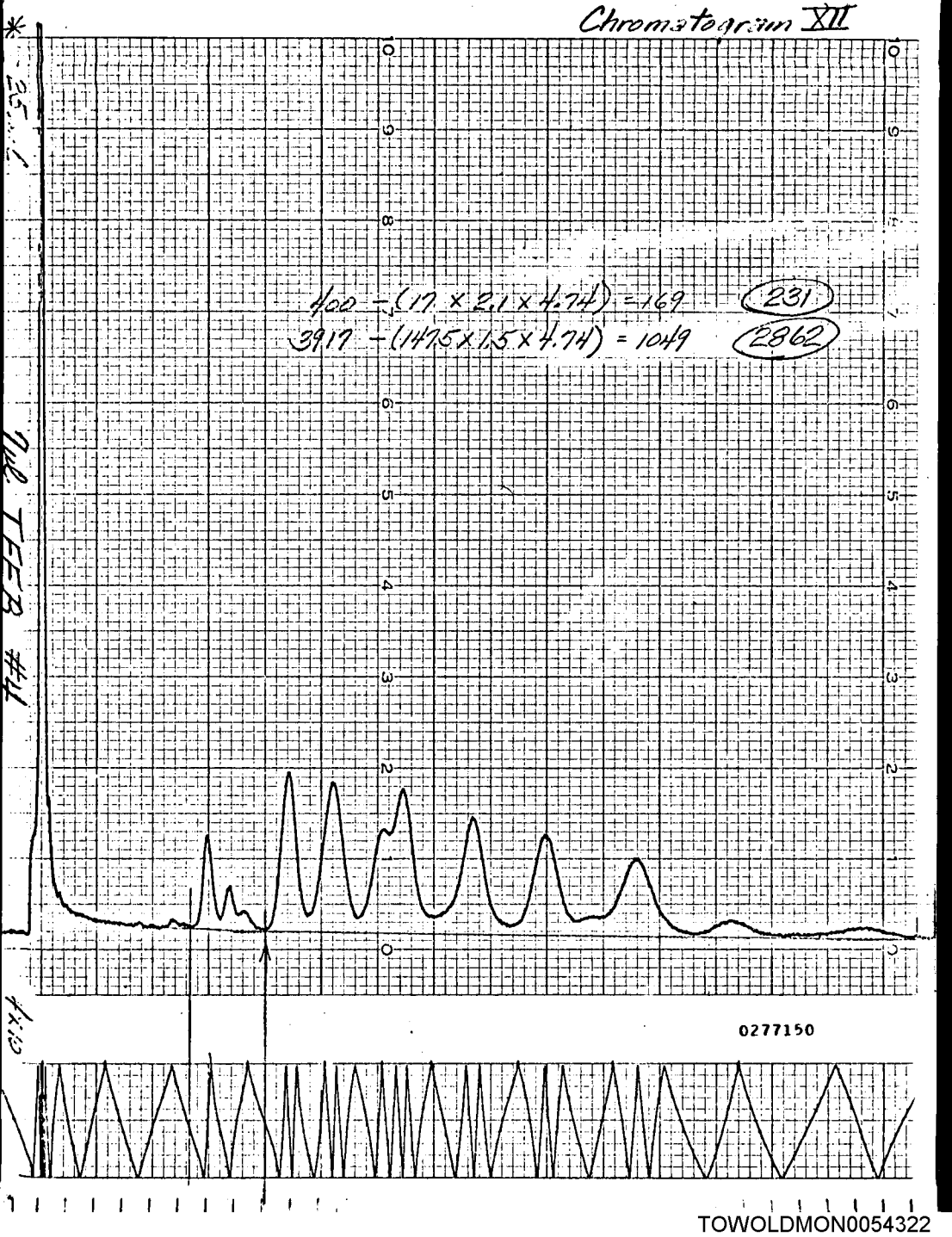
Chromatogram XII



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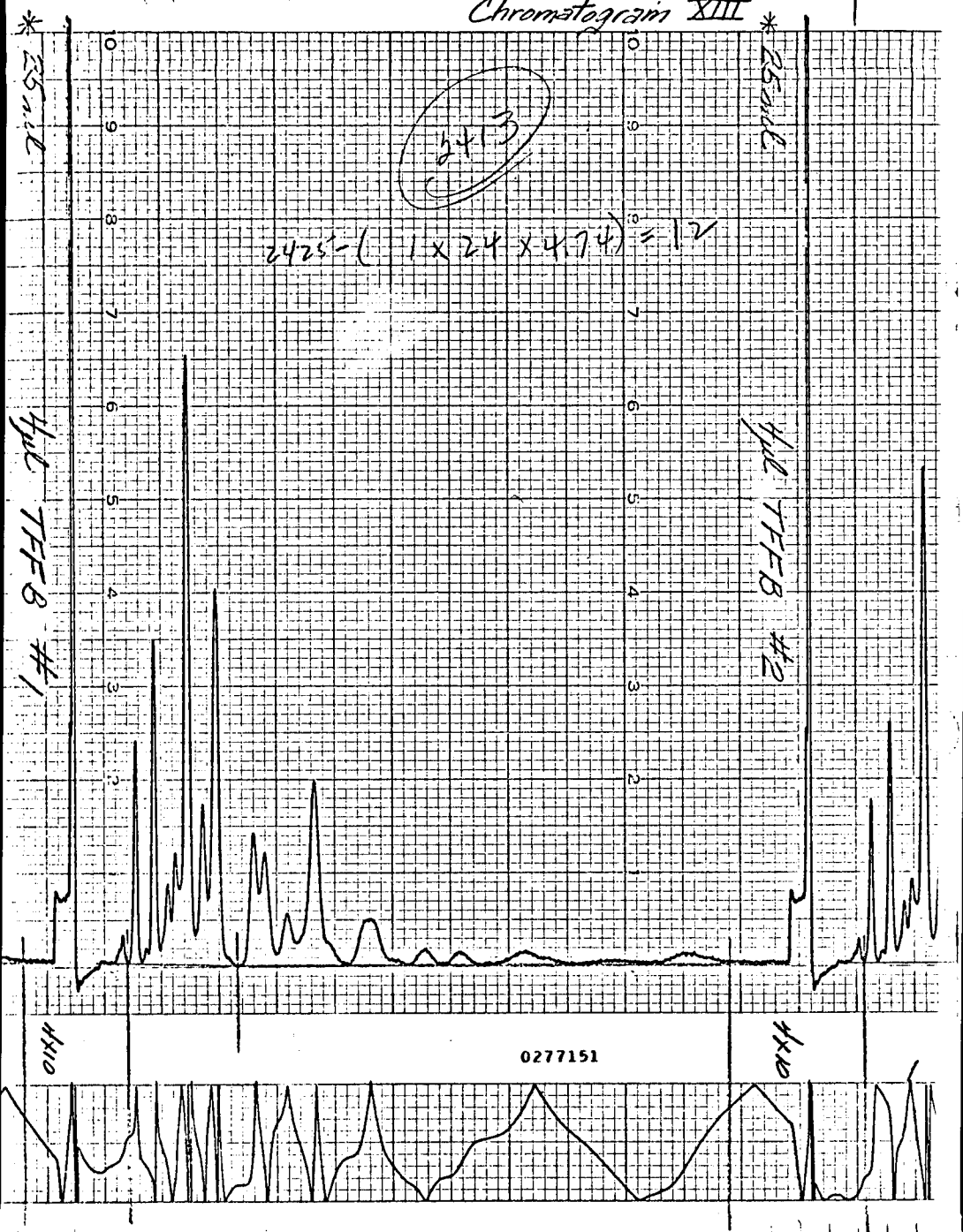
Chromatogram XIII



24-3

2425-(1x24x4.74) = 12

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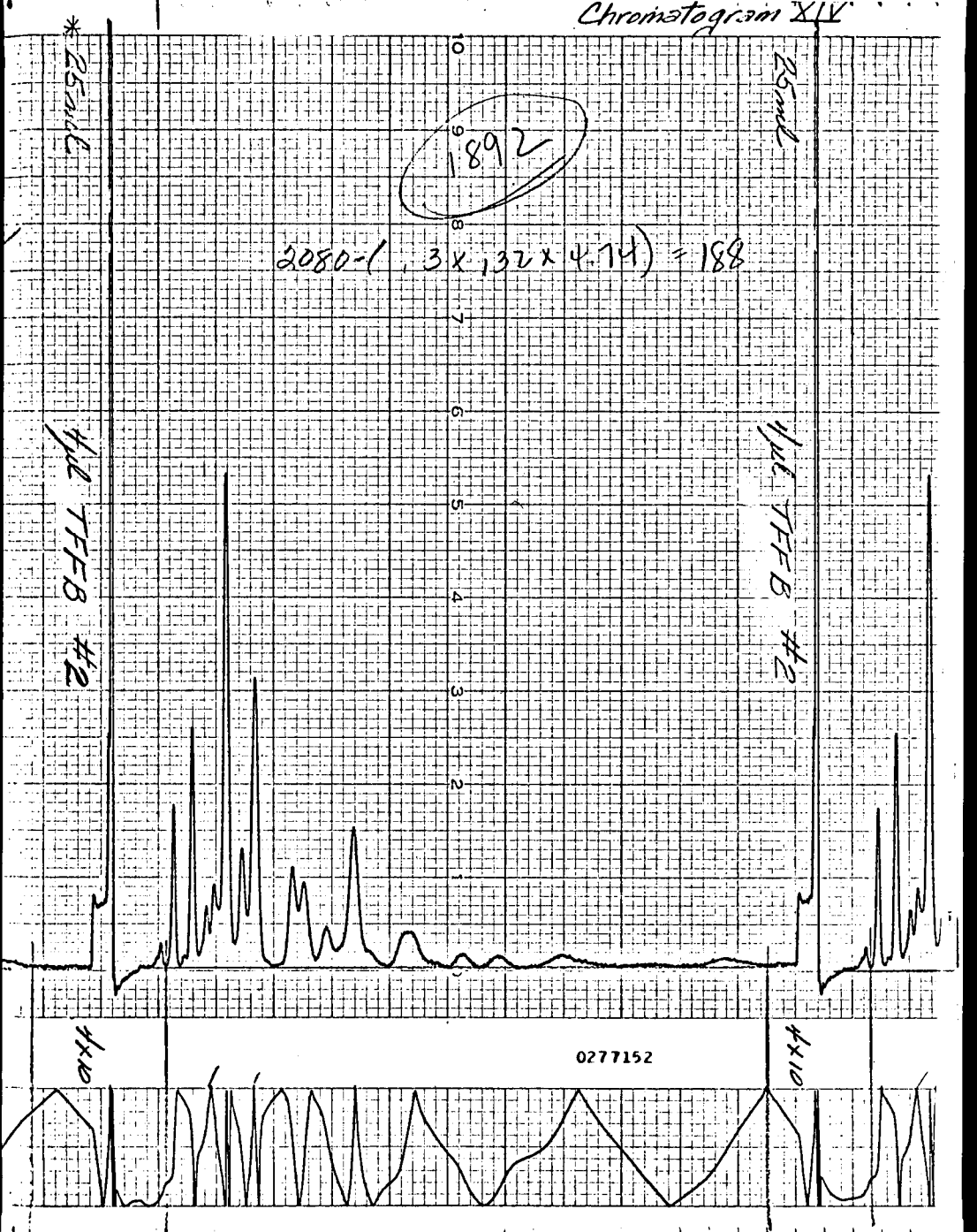
Chromatogram XIV



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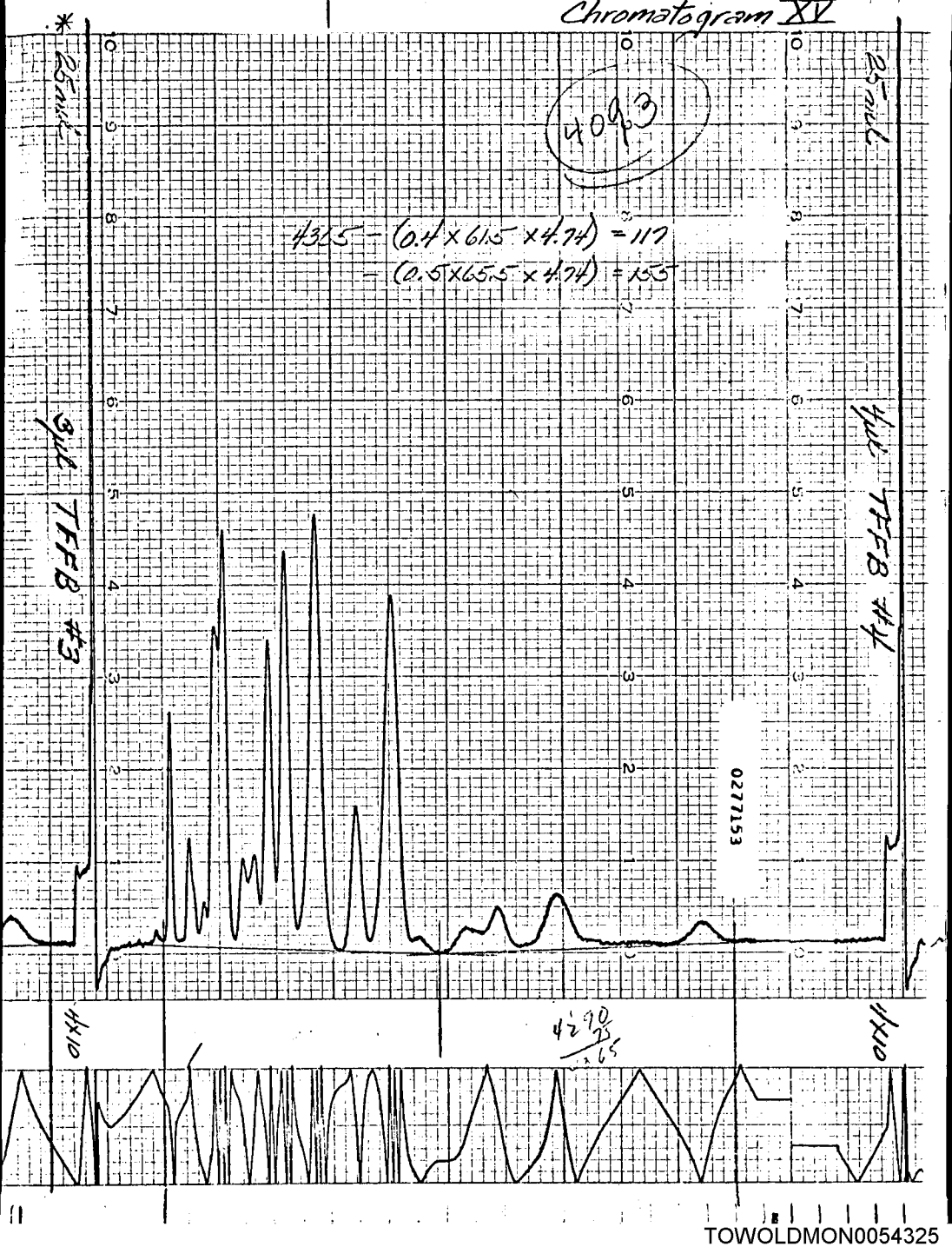
Chromatogram XV



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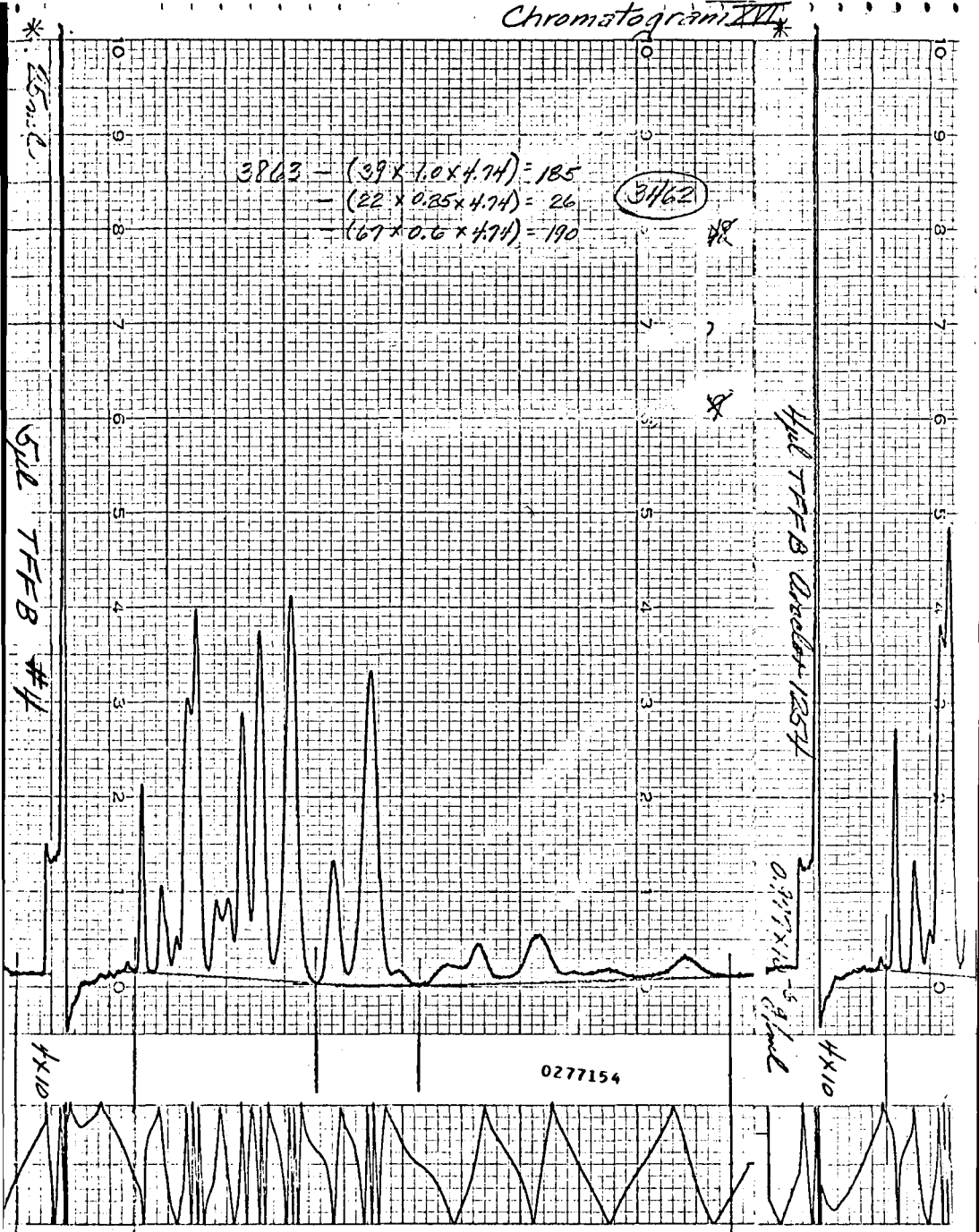
Chromatogram XVI



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