

The PDI Real World Test

The Resin Reconditioning Study is part of an ongoing industry study on the reuse of plastic drums that once contained hazardous materials. No part of this study should be used to determine compliance with any part of the Department of Transportation's Hazardous Materials Regulations. While the SPI Plastic Drum Institute, its members and the study participants have attempted to conduct this study using industry-accepted test methods, use of a method does not warrant the validity of a particular method.

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Plastic Drum Fabricators

Russell-Stanley Corporation
Sonoco Plastic Drum

Resin Manufacturers

Fina Corporation
Mobil Chemical Company
Novacor Chemical, Ltd.
Phillips 66 Company
Quantum Chemical Corporation

Reconditioners

National Container Services Inc.
Restoration Plastics Inc.

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John Malik	Sonoco Plastic Drum

PDI REAL WORLD TEST PART 2
“RESIN RECONDITIONING STUDY”

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EXECUTIVE SUMMARY

INTRODUCTION

Two prior Plastic Drum Institute (PDI) reports have been issued on studies of HDPE drums to determine their ability to be reused and the effect of lading contamination. These studies were written up as the following reports:

In 1983 the PDI issued a report entitled "The 55-Gallon All Plastic Drum Reuse Study." Its main purpose was to investigate whether the lading or reconditioning of the plastic drum affected the structural integrity of the plastic drum for a second use. It was found that the lading or drum reconditioning did not affect the structural integrity of the plastic drum. It was noticed that minimal residue from the previous loadings packaged remained in the reconditioned drums. This residue was broadly discussed and prompted a second study.

The PDI issued a second report in 1990 that was entitled "The Real World Test." This study was to establish the suitability of 55-gallon all-plastic drums, which had previously seen service with another lading, for the storage and shipping of a second lading. The primary objective was acceptability of the loadings determined through quality control analysis upon the loadings after a three month storage in used containers. A secondary objective was reconfirmation of the results from the 1983 study regarding the integrity of the all-plastic drums following service and reconditioning. A third objective was the determination of the second lading absorbed into the polyethylene drum wall and remaining after reconditioning.

The results of this second study reconfirmed that minimal residue from previous loadings packaged remained in the reconditioned drums. Most shippers concluded that the reconditioned plastic drums were acceptable for the loadings used. Also reconfirmed was that the loadings and reconditioning did not affect the structural integrity of the plastic drum, as seen in the 1983 study.

DISCUSSION

It was shown in the "Real World Test" that 55-gallon all-plastic drums can be successfully reused in selected secondary service. A possible conclusion to the life of a polyethylene drum would be as a recycled material going into a totally unrelated application. As the recycled polyethylene currently available goes through a special cleaning process, above and beyond that used to recondition a drum for secondary use, it is reasonable that an investigation be made into continuing the PDI work to measure the amount of residual lading detectable after the polyethylene has been subjected to such a process.

OBJECTIVES

The primary objective of the “Resin Reconditioning Study” was to quantify the residual of six selected ladings remaining absorbed into the HDPE used to make a drum after both a drum reconditioning step and the treatment of the resin through a typical post consumer polyethylene plant.

A secondary objective was to conduct investigative work into the usefulness of a Microtox® test that could determine toxicity level of the residual lading in the high density polyethylene (HDPE) drum samples during the various stages of the study. This test consisted of leaching the drum samples to try and remove any residual ladings in them. The leachate was then mixed with the pure Microtox® reagent. The Microtox® reagent is a living bacteria which emits light. If the lading or concentration of the lading from the leachate is toxic to the living bacteria, then some of the bacteria will perish. The amount of light emitted will, therefore, be reduced. An instrument measures the light and a relative value is reported on the toxicity of the sample.

CONCLUSIONS

The results of the “Resin Reconditioning Study” showed:

- Residual lading absorbed into the HDPE of the drum could be reduced for some of the ladings through the drum reconditioning, secondary flake washing and repelletization steps.
- The Microtox® test was able to give some measurement of toxicity and changes in toxicity during steps of the cleaning process.
- It is believed that failure in the Microtox® test does not mean it precludes recycling of the tested drum sample.

SUMMARY

The Real World Test - Part 2, “Resin Reconditioning Study” involved obtaining in-service drums with six ladings. The ladings were similar to those used in the “Real World Test”. The ladings were

Hydraulic Oil	Acrylic Acid
Acetic Acid	Sulfuric Acid
Mineral Spirits	Methanol

The drums were emptied of their contents and sent to a drum reconditioner where the drums were cleaned under normal procedures for plastic HDPE drums.

As a next step the drums were then granulated into a flake. The flake was sent to a pilot reconditioning system where the flakes were washed in a secondary operation for further possible removal of residual lading that absorbed into the HDPE.

After this secondary washing step the flakes were sent to a facility for repelletization.

Samples which had been saved at several step of the cleaning process were forwarded to four laboratories for analytical testing of levels of residual lading in the samples. Physical property testing was also conducted on the HDPE to determine if any loss in properties was observed. Also at this time some of the samples were forwarded to a laboratory to conduct a Microtox® test to determine its ability to be used for testing of toxicity of residual lading and differences in level of toxicity of the various stages of the cleaning process of the study.

PART I

RESIN RECONDITIONING

**Analytical Procedures
and Results**

PART I - RESIN RECONDITIONING

Analytical Procedures and Results

OBJECTIVE

The primary objective of the "Resin Reconditioning Study " was to quantify the residual amounts of six ladings remaining absorbed into the HDPE used to make a drum after both drum reconditioning and the treatment of the resin through a typical post consumer polyethylene plant.

PROCEDURE

Drums that contained the desired ladings were acquired from field service for the study. One drum was acquired for each of the ladings with the available color(s). This would allow for a 20 pound sample to conduct the flake wash and later sample pulls. In the initial stages of the testing the methanol lading drums were not available and subsequently dropped from the testing matrix.

The drums were emptied of their ladings and sent to a reconditioning facility for cleaning. The drum reconditioning company used for this step was National Container Services Inc. The reconditioning procedure involved several steps *. Initially the drums were preflushed with a caustic or acid solution for neutralization of any remaining lading. The exterior of the drums was then cleaned. A series of internal washes were then conducted which consisted of a hot caustic wash, clean rinse, lower level caustic wash, water rinsing and a final fresh cold water wash. The drums were then flame treated and inspected.

The drums of the five lading types were then chopped by National Container Services Inc. in a granulator as would normally be done during a operation when reclaiming the HDPE from the drum at the end of its useful life. The flakes generated from each lading type were kept segregated for further work.

Samples for analysis were obtained from the generated flakes of each individual lading type drum. The analysis samples of each individual lading type drum were held for later testing that would be conducted once all testing samples were available. These flake samples were designated with an integer number. The remaining, major portion of the flake samples were then sent on for a secondary cleaning step to determine if any additional amount of the lading could be removed during that cleaning step.

* - The procedure used for washing drums could have an effect on the residual lading present. Use of a different washing procedure or process may give different results.

The remaining, major portion of the flake samples for each individual lading type drum was sent to Restoration Plastics Inc. of Casselberry, Florida. They processed the individual lading drum flake samples through their pilot reconditioning system. This system provided a second cleaning step in which further removal of the lading absorbed into the HDPE could occur. This cleaning step would be very similar to the steps used by post-consumer recyclers of plastic bottles. The process consisted of washing each flake sample in a 165° F proprietary solution for approximately 60 seconds followed by an ambient temperature rinse for 30 seconds. The flake samples were then dried and packaged for shipment. A letter stating the procedure used is shown in Appendix A. After running the flake samples through their process Restoration Plastic Inc. sent the flake samples to Quantum Chemical for repelletization.

The pelletization process that Quantum conducted on the flake samples from Restoration Plastics Inc. completed the process that normally would be conducted by a post-consumer recycler of plastic bottles. They compounded individually flake samples on a one inch diameter, non-vented Killion extruder and produced a strand cut pellet. Once completed with the pelletization of these flake samples Quantum designated each of them with a integer number followed by the letter A. These samples were then sent with the earlier flake samples collected after the granulation, designated with an integer, to the respective analytical testing laboratories for analysis of the residual lading that may remain in the specific lading HDPE drum samples.

The identification of laboratories and the samples sent to those laboratories for the analytical analysis of residual loadings in the HDPE samples are shown in Table 1.

ANALYTICAL TESTING PROCEDURES

Four test laboratories volunteered to conduct testing procedures for the determination of residual lading in the high density polyethylene samples from the lading drums. Only five loadings were tested for because of the inability to acquire a lading drum of methanol. The full procedures and test results of the individual laboratories is shown in Appendix A.

The testing techniques used by the laboratories are as follows:

<u>Lading</u>	<u>Test Method</u>	<u>Laboratory</u>
Hydraulic Oil	Extraction GC	Mobil Chemical
Acrylic Acid	GC Chromotography	Fina Corp.
Acetic Acid	Extraction GC	Mobil Chemical
Sulfuric Acid	X-Ray Florescence	Phillips Chemical & Fina Corp.
Mineral Spirits	GC-MS	Novacor Chemical

The testing laboratories also tested the samples for physical properties. Test conducted were HLMI, density and TGA.

RESULTS & DISCUSSION

Physical property testing of samples, Table 2, showed no changes in the characteristic properties of the HDPE due to lading or any of the reconditioning or repelletization steps.

The results of the analytical testing for the ladings are shown in Table 3. Results are reported in parts per million for all ladings.

Mineral Spirits and hydraulic oil showed the highest levels of residual of the five tested ladings. Both of these ladings show a significant drop in the absorbed levels of lading in the HDPE between the drum reconditioning step and the secondary cleaning/repelletization step. The measured level of the mineral spirit decreased by 33 percent between these steps and the hydraulic oil decreased by 20 percent.

Sulfuric acid reacts with the inside surface of the polyethylene drum, forming a surface layer in a process called sulfonation. The testing analysis showed a higher level of sulfur in the black pigmented samples than the blue pigmented samples. It is believe that the carbon black used in the black pigmented sample is either affecting the testing procedure used or provided for a higher level of absorption of the sulfuric acid. There was no change in the level of the sulfur measured between the drum reconditioning step and the secondary cleaning/repelletization step.

Acrylic acid and acetic acid were not found in measurable quantities for any of the samples with the testing techniques used.

TABLE 1**LEGEND OF DRUM SAMPLES FOR ANALYTICAL TESTING**

Sample #	Form	Unwashed/ Washed	Lading	Color	Testing Lab
1	Flake	Unwashed	Hydraulic Oil	Natural	Mobil
1A	Pellet	Washed	Hydraulic Oil	Natural	Mobil
2	Flake	Unwashed	Acrylic Acid	Black	Fina
2A	Pellet	Washed	Acrylic Acid	Black	Fina
3	Flake	Unwashed	Acrylic Acid	Black	Fina
3A	Pellet	Washed	Acrylic Acid	Black	Fina
4	Flake	Unwashed	Acetic Acid	Black	Mobil
4A	Pellet	Washed	Acetic Acid	Black	Mobil
8	Flake	Unwashed	Acetic Acid	Blue	Mobil
8A	Pellet	Washed	Acetic Acid	Blue	Mobil
5	Flake	Unwashed	Sulfuric Acid	Blue	Fina
5A	Pellet	Washed	Sulfuric Acid	Blue	Fina
6	Flake	Unwashed	Sulfuric Acid	Black	Fina
6A	Pellet	Washed	Sulfuric Acid	Black	Fina
5	Flake	Unwashed	Sulfuric Acid	Blue	Phillips
5A	Pellet	Washed	Sulfuric Acid	Blue	Phillips
6	Flake	Unwashed	Sulfuric Acid	Black	Phillips
6A	Pellet	Washed	Sulfuric Acid	Black	Phillips
7	Flake	Unwashed	Mineral Spirits	Blue	Novacor
7A	Pellet	Washed	Mineral Spirits	Blue	Novacor

TABLE 2

RESIN PHYSICAL PROPERTY MEASUREMENTS

Sample #	Lading	Flow Rate ASTM 1238 HLMI 21.6 kg	Density ASTM D792 g/cc	TGA * % Wt. Loss at 250 C
1	Hydraulic Oil	6.06	0.953	0.24
1A	Hydraulic Oil	6.80	0.954	0.27
2	Acrylic Acid	6.46	0.956	—
2A	Acrylic Acid	6.29	0.956	—
3	Acrylic Acid	6.18	0.956	—
3A	Acrylic Acid	6.64	0.956	—
4	Acetic Acid	5.96	0.955	0.27
4A	Acetic Acid	6.66	0.954	0.26
8	Acetic Acid	5.28	0.952	0.20
8A	Acetic Acid	6.00	0.952	0.19
5 **	Sulfuric Acid	2.1(2.08)	0.953(0.953)	0.03
5A **	Sulfuric Acid	3.6(2.98)	0.953(0.954)	0.11
6 **	Sulfuric Acid	10.4(11.08)	0.956(0.955)	0.17
6A **	Sulfuric Acid	11.0(10.46)	0.956(0.956)	0.26
7	Mineral Spirits	5.99	0.9548	0.40
7A	Mineral Spirits	6.62	0.9552	0.40

* - TGA testing was conducted on natural HDPE used in 55 gallon drums. This was to act as a control to determine % weight loss at 250 C in HDPE samples not exposed to any ladings. TGA test results on the natural HDPE from Novacor and Phillips were 0.20% and 0.15%, respectively.

** = Phillips Results(Fina Results).

TABLE 3

LADING ADSORPTION RESULTS

	Natural Flake (Unwashed)	Natural Flake (Washed)	Natural Pellet (Washed)	Blue Flake (Unwashed)	Blue Pellet (Washed)	Black Flake (Unwashed)	Black Pellet (Washed)
Hydraulic Oil	250 PPM *	200 PPM *	---	---	---	---	---
Acrylic Acid	---	---	---	---	---	None	None
Acetic Acid	---	---	---	None	None	None	None
Sulfuric Acid (Fina) **	---	---	---	150 PPM	58 PPM	49 PPM	157 PPM
Sulfuric Acid (Phillips)	---	---	---	157 PPM	168 PPM	473 PPM	461 PPM
Mineral Spirits	---	---	---	900 PPM	600 PPM	---	---

* Based on density of 1 gram/ml.

** The sulfuric acid results from Fina are report as forwarded. However because of discrepancies and the inability to clarify the results they were not used in the final analysis.

Part II
MICROTOX® TESTING

**Analytical Procedures
and Results**

Part II - Microtox® Testing

Analytical Procedures and Results

OBJECTIVE

The secondary objective of the study was to investigate the Microtox® test as to its utility as a procedure to determine the level of toxicity of any residual lading that remained in HDPE drum samples at various stages of the study.

The Microtox® test consisted of refluxing a twenty gram drum (resin) sample in 250 ml of deionized water for a 24 hour period. This allowed any residual lading the opportunity to leach from the HDPE drum sample. The leachate was then mixed with the pure Microtox® reagent in a standard 2:1 dilution for testing. The Microtox® reagent is a living bacteria which emits light. If the lading or concentration of the lading in the leachate was toxic to the living bacteria then some would perish and the amount of light emitted would be reduced. A light measuring instrument measures this reduction and reports a relative value on the toxicity of the sample. Further information on the Microtox® test appears in Appendix B.

PROCEDURE

Testing was conducted on four of the five lading HDPE drum samples. The test samples were obtained at three different stages of the study for testing in the Microtox® test. The three stages of when the samples were obtained were during the grinding operation after the drums had been reconditioned, after the secondary cleaning operation and finally after the repelletization. It was considered that in obtaining samples at these three stages of the study possible differences in the ability of the cleaning steps on the residual lading in the HDPE may be observed. The Microtox® test was conducted by Mobil Chemical.

RESULTS & DISCUSSION

The results of the testing are shown in Table 4. The test was conducted at both a five minute and fifteen minute interval. The fifteen minute test is a longer test but gives more complete results. The unit value of the results are given in percent with 100 percent showing no toxicity and toxicity increasing as the percent number decreases.

In the Microtox® test the samples taken from the acrylic acid and sulfuric acid lading drums were toxic throughout all three stages of the reclaiming process. The reconditioning step, secondary washing or repelletization did not show any effect on decrease of the level of toxicity through the Microtox® test. It was observed that the acrylic acid was slightly more toxic than the sulfuric acid.

The acetic acid samples showed no toxicity throughout the three stages of the reclaiming process.

The mineral spirits showed a significant change in toxicity during the repelletization step. After the repelletization it showed no toxicity.

Analysis of the results showed that the washing and repelletization steps did have an improving effect on the toxicity of the mineral spirits and acetic acid. The acrylic acid and sulfuric acid showed no improvement in toxicity with the Microtox® test during any of the steps of the washing and repelletization. It is believed that failure in the Microtox® test does not mean it precludes recycling of the tested drum sample.

Table 4

Microtox® Test on Lading HDPE Samples

Sample #	Form	Unwashed /Washed	Lading	Color	Microtox® 5 min	EC50 15 min
2	Flake	Unwashed	Acrylic Acid	Black	46	23
2	Flake	Washed	Acrylic Acid	Black	59	37
2A	Pellet	Washed	Acrylic Acid	Black	68	42
6	Flake	Unwashed	Sulfuric Acid	Black	76	60
6	Flake	Washed	Sulfuric Acid	Black	86	62
6A	Pellet	Washed	Sulfuric Acid	Black	66	52
7	Flake	Unwashed	Mineral Spirits	Blue	45	32
7	Flake	Washed	Mineral Spirits	Blue	45	26
7A	Pellet	Washed	Mineral Spirits	Blue	>100	>100
8	Flake	Unwashed	Acetic Acid	Blue	>100	96
8	Flake	Washed	Acetic Acid	Blue	86	>100
8A	Pellet	Washed	Acetic Acid	Blue	>100	>100
Water Blank (5 Samples)				—	>100	>100
Virgin + Color				White	>100	88
Virgin Re grind				Blue	>100	>100

Part IV

Technical Appendices

APPENDIX A

Part I - Resin Reconditioning

Analytical Procedures and Results

Restoration Plastics International**FAX Transmission**

From: Richard Dunkel
To: John Bergerhouse
Company: Quantum

Date: January 22, 1993
Time: 1:03 PM
FAX #: 513-530-4267

Message:

The granulate will be washed in a 165 degrees Fahrenheit proprietary solution for approximately 60 seconds followed by an ambient temperature rinse for 30 seconds. The granulate will be dried and packaged for shipment.

We employ a special waste filtration and recycling system to minimize water usage and to guarantee that any discharge is in compliance with the sanitary sewer district limits,

Best regards,

/ Richard Dunkel

VOICE: 407-767-2076 FAX: 407-767-5022

950 South Winter Park Drive, Suite 325, Casselberry, FL 32707

P D I RECYCLE STUDY

SAMPLE#	FORM	LADING	COLOR
1	FLAKE	HYD. OIL	NAT.
1A	PELLET	HYD OIL	NAT
4	FLAKE	ACETIC ACID	BLACK
4A	PELLET	ACETIC ACID	BLACK
8	FLAKE	ACETIC ACID	BLUE
8A	PELLET	ACETIC ACID	BLUE

FLAKE = UNWASHED

PELLET = WASHED

SAMPLE#	HLMI	DEN.
1	6.06	0.953
1A	6.80	0.954
4	5.96	0.955
4A	6.66	0.954
8	5.28	0.952
8A	6.00	0.952

K.T.FORD

MOBIL
KIPLESSE.TC

SAMPLE ID NAME	C#	ONSET TEMP (C)	WT. LOSS AT 250C (%)
P.D.I.			
FLAKE#1	C11080-1		
RUN#1		508	0.24
RUN#2		504	0.25
RUN#3		505	0.24
AVG		506	0.24
PELLET#1	C11080-2		
RUN#1		505	0.31
RUN#2		504	0.24
RUN#3		507	0.27
AVG		505	0.27
FLAKE#4	C11080-3		
RUN#1		507	0.21
RUN#2		510	0.27
RUN#3		505	0.33
AVG		507	0.27
PELLET#4	C11080-4		
RUN#1		508	0.27
RUN#2		508	0.26
RUN#3		512	0.25
AVG		509	0.26
FLAKE#8	C11080-5		
RUN#1		509	0.20
RUN#2		515	0.17
RUN#3		516	0.23
AVG		513	0.20
PELLET#8	C11080-6		
RUN#1		534	0.20
RUN#2		533	0.18
RUN#3		544	0.19
AVG		537	0.19

K. YOCK
7/20/92

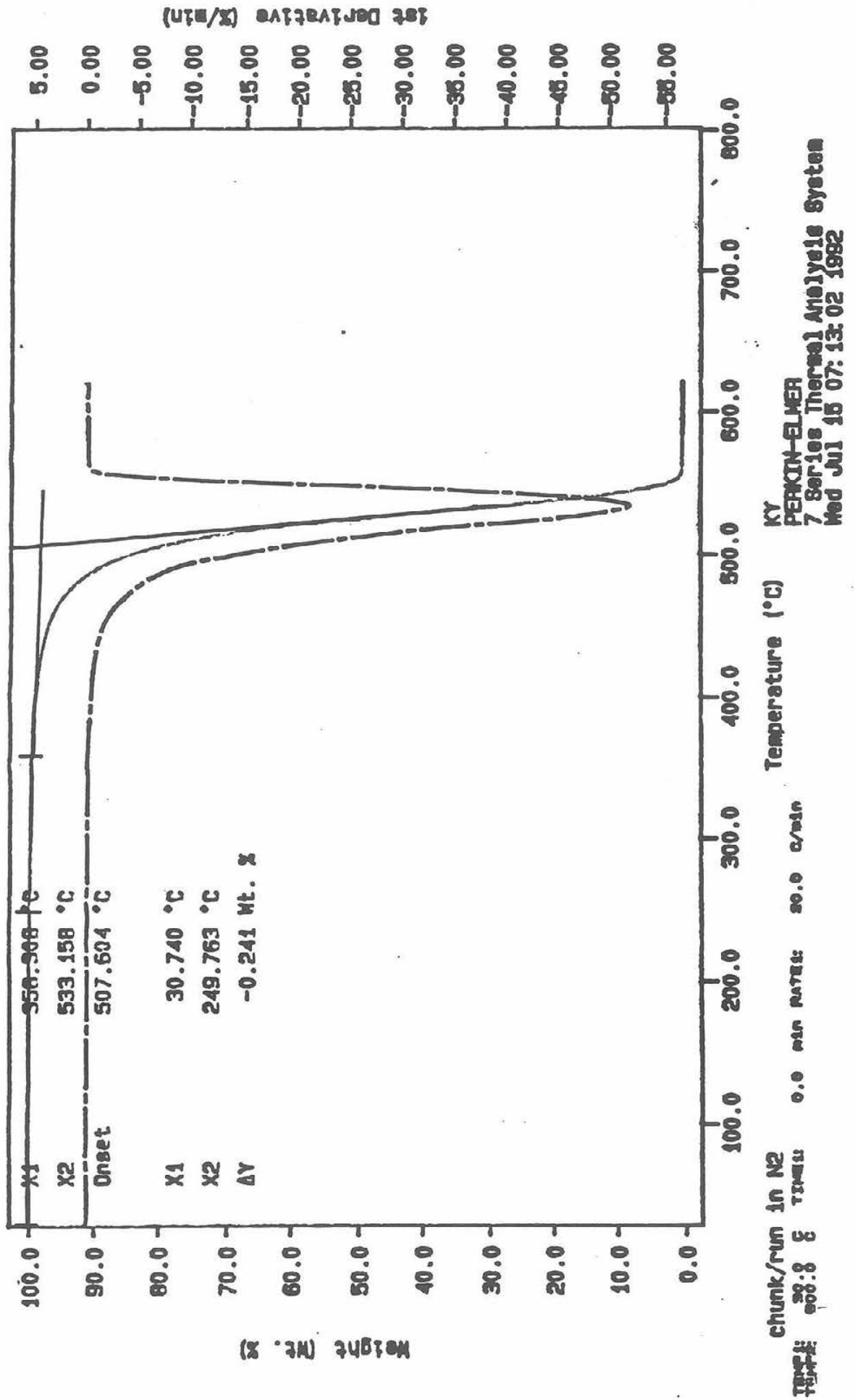
Curve 1: TGA

File info: c110801a Tue Jul 14 11:22:30 1992

Sample Weight: 11.502 mg

P.D.I. flakes C11089-1

P.D.I. #10671 C11080-1



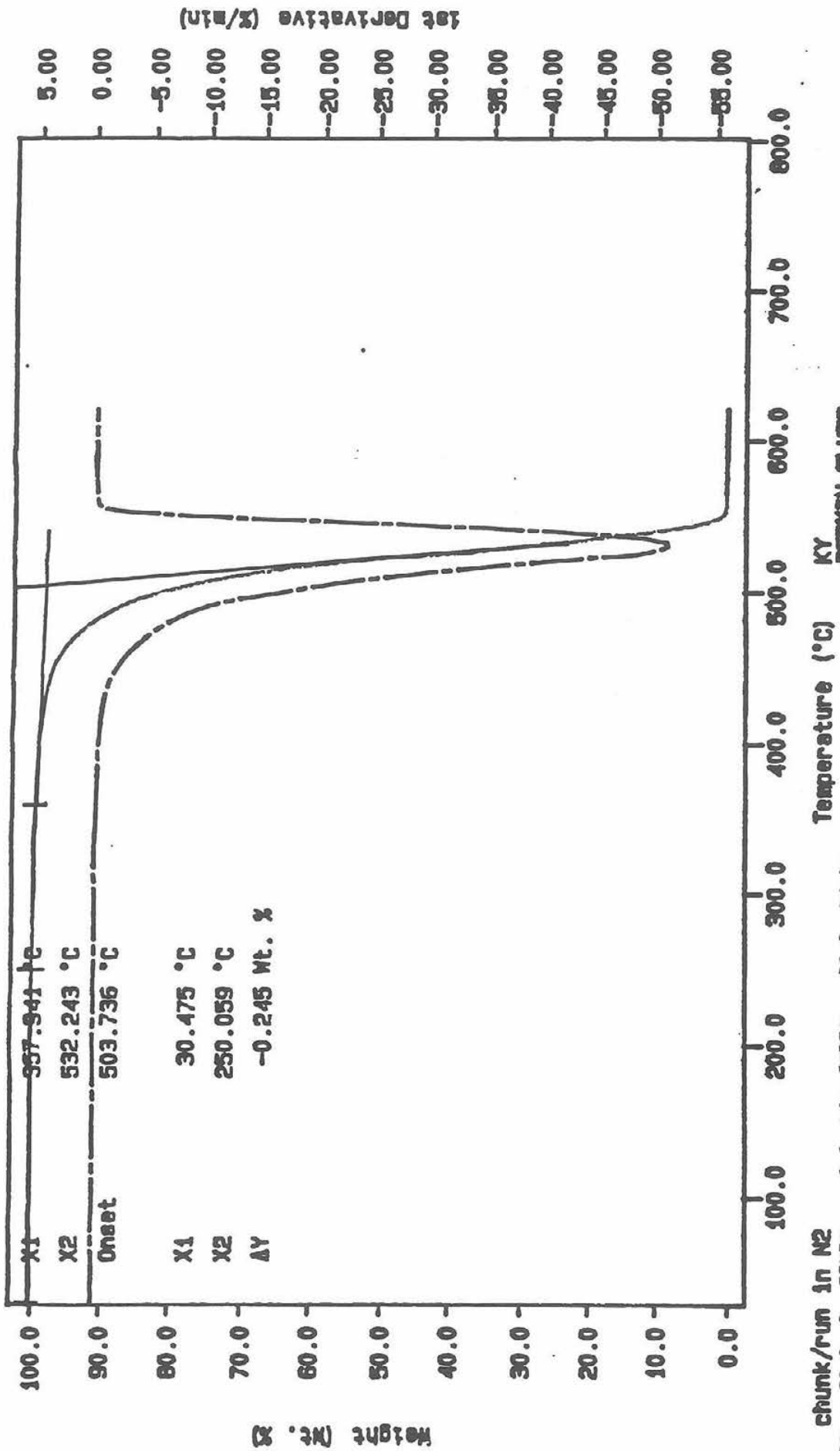
Curve 1: TGA

File Info: C110801b Tue Jul 14 12:57:15 1992

Sample Weight: 9.957 mg

P.D.I. flake#1 C11080-1

P.D.I. flake#1 C11080-1



chunk/run in N2
 TGA# 28.8 g
 TIME: 0.0 min RATE: 20.0 C/min
 KY PERKIN-ELMER
 7 Series Thermal Analysis System
 Wed Jul 15 07:10:26 1992

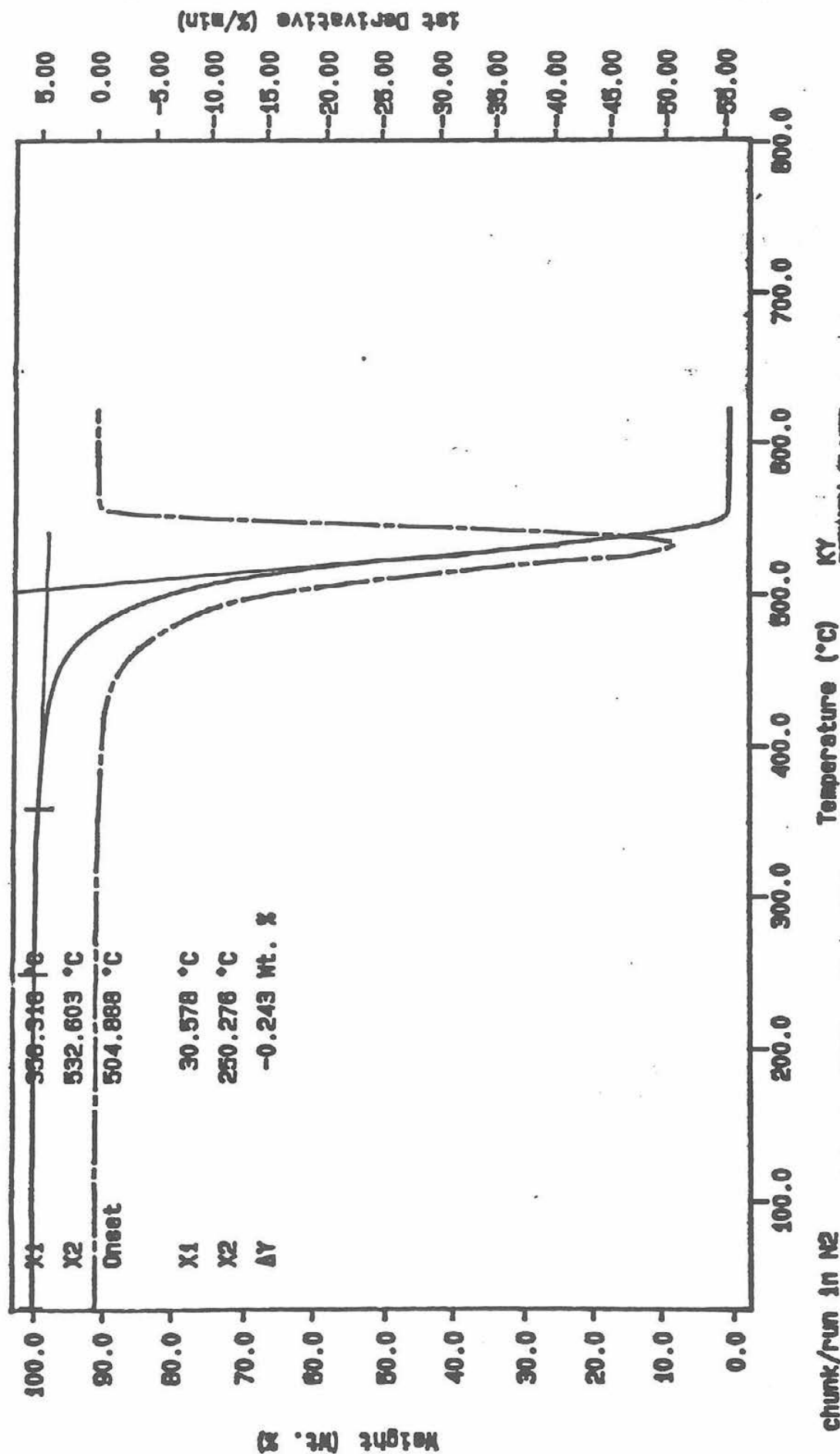
Curve 1: TGA

File Info: C110801c Tue Jul 14 14:59:10 1992

Sample Weight: 9.335 mg

P.D.I. flakes#1 C11080-1

P.D.I. flakes#1 C11080-1



chunk/run in N2
 TGA# 20.8 g
 TIME: 0.0 min RATE: 20.0 °C/min
 KY PERKIN-ELMER
 7 Series Thermal Analysis System
 Wed Jul 15 07:01:00 1992

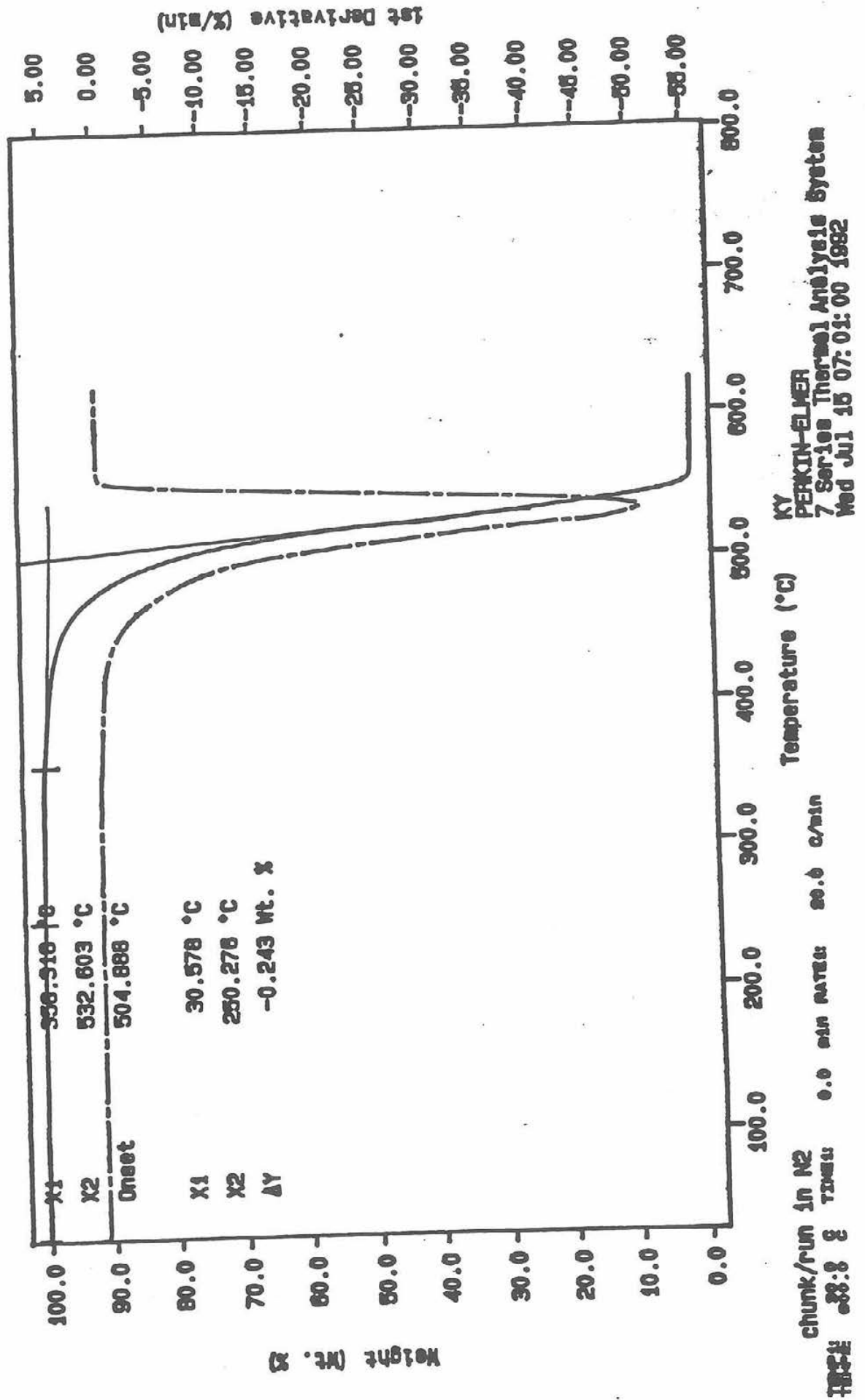
Curve 1: TGA

File Info: c110801c Tue Jul 14 14:59:20 1992

Sample Weight: 9.335 mg

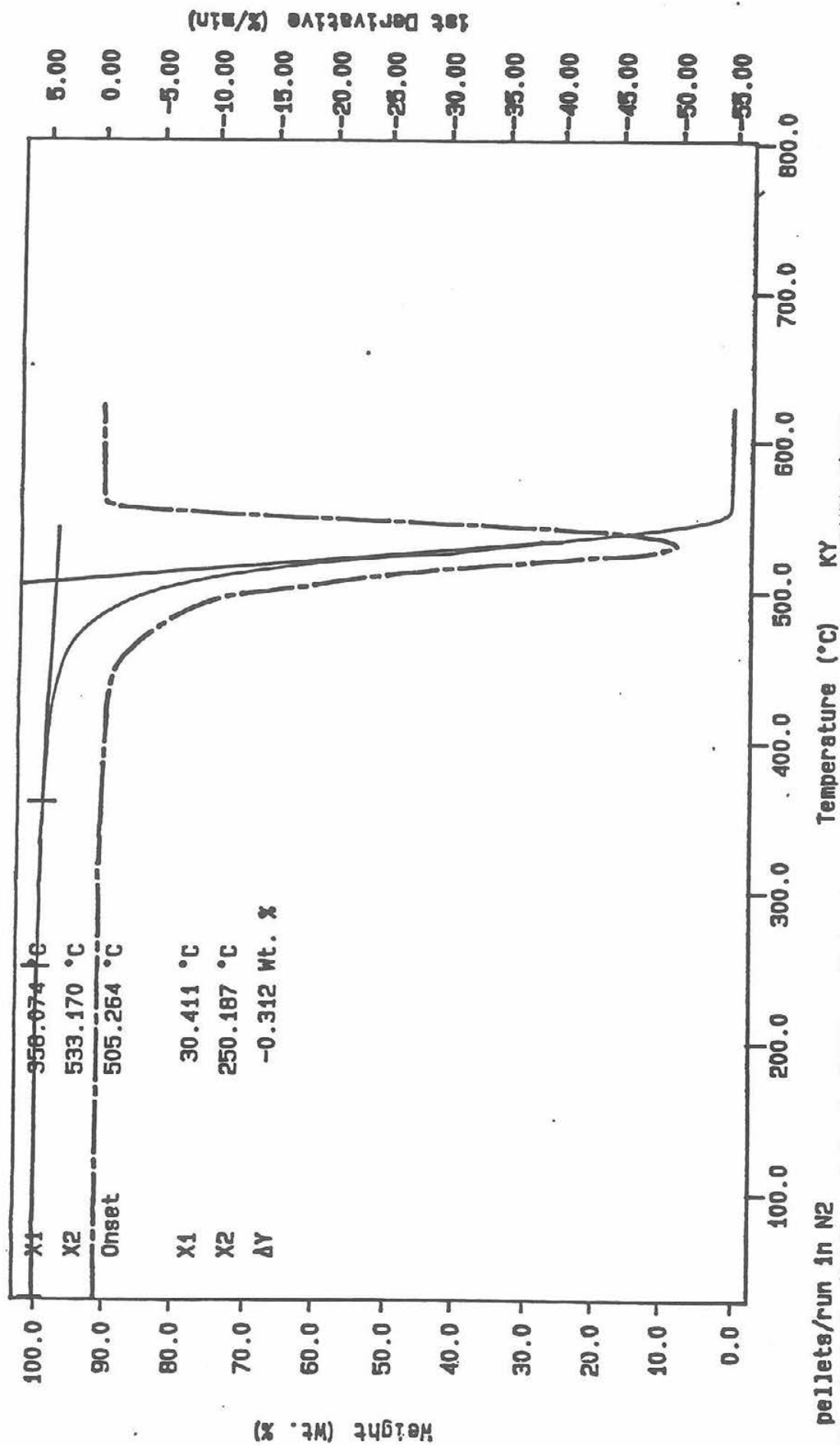
P.D.I. flakes#1 C11080-1

P.D.I. flakes#1 C11080-1



Curve 1: TGA
 File info: c110802a Wed Jul 15 08:00:11 1992
 Sample Weight: 10.650 mg
 P.D.I.pellet#1 C11080-2

P.D.I.pellet#1 C11080-2



pellets/run in N2
 temp: 30.0 °C
 time: 0.0 min
 rate: 20.0 °C/min
 KY
 PERKIN-ELMER
 7 Series Thermal Analysis System
 Wed Jul 15 12:33:14 1992

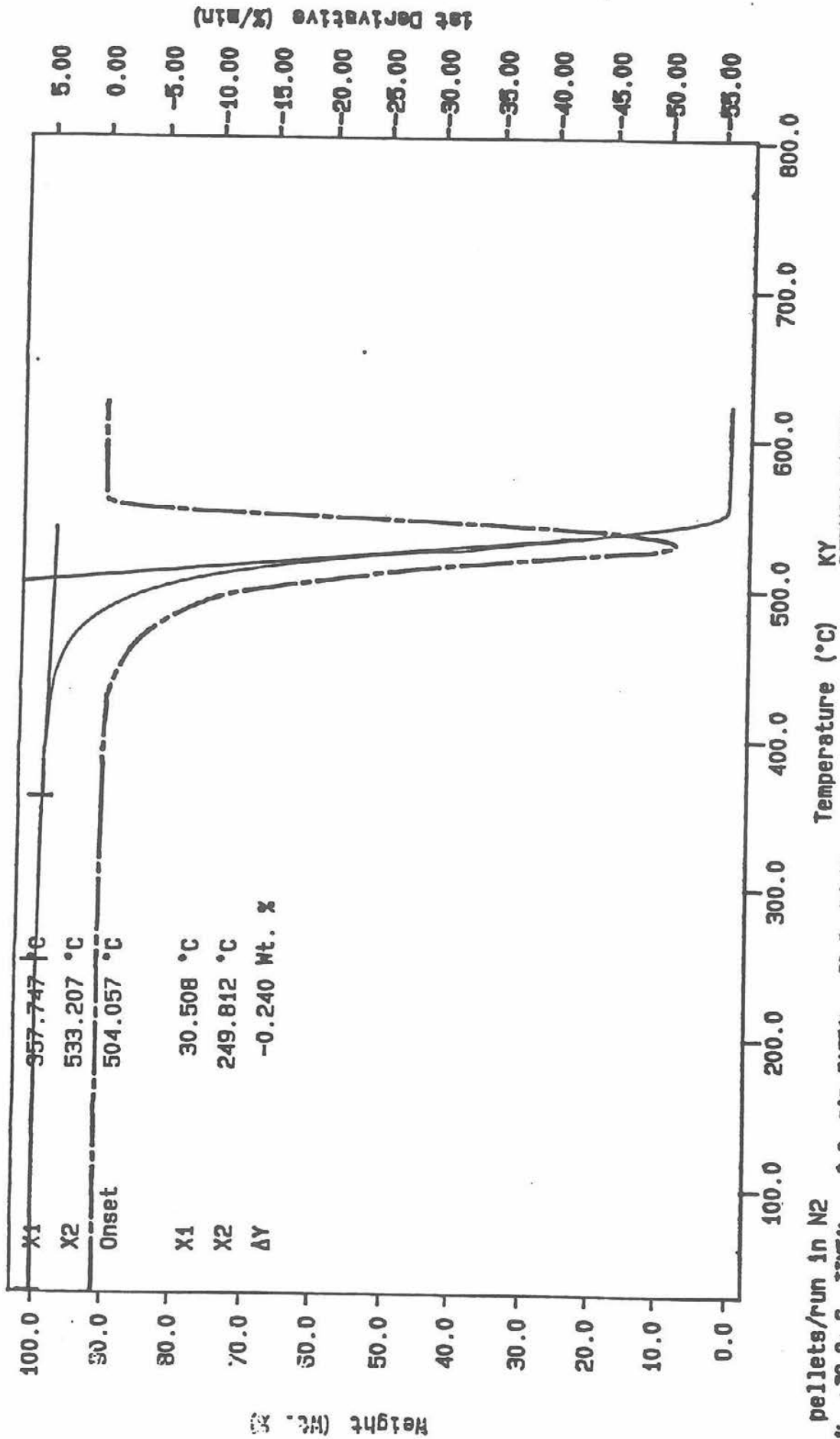
Curve 1: TGA

File info: c110802b Wed Jul 15 09:15:18 1992

Sample Weight: 9.929 mg

P.D.I.pellet#1 C11080-2

P.D.I.pellet#1 C11080-2



pellets/run in N2

TEMP: 30.0 °C TIME: 0.0 min RATE: 20.0 °C/min

 KY PERKIN-ELMER
 7 Series Thermal Analysis System
 Wed Jul 15 12:28:03 1992

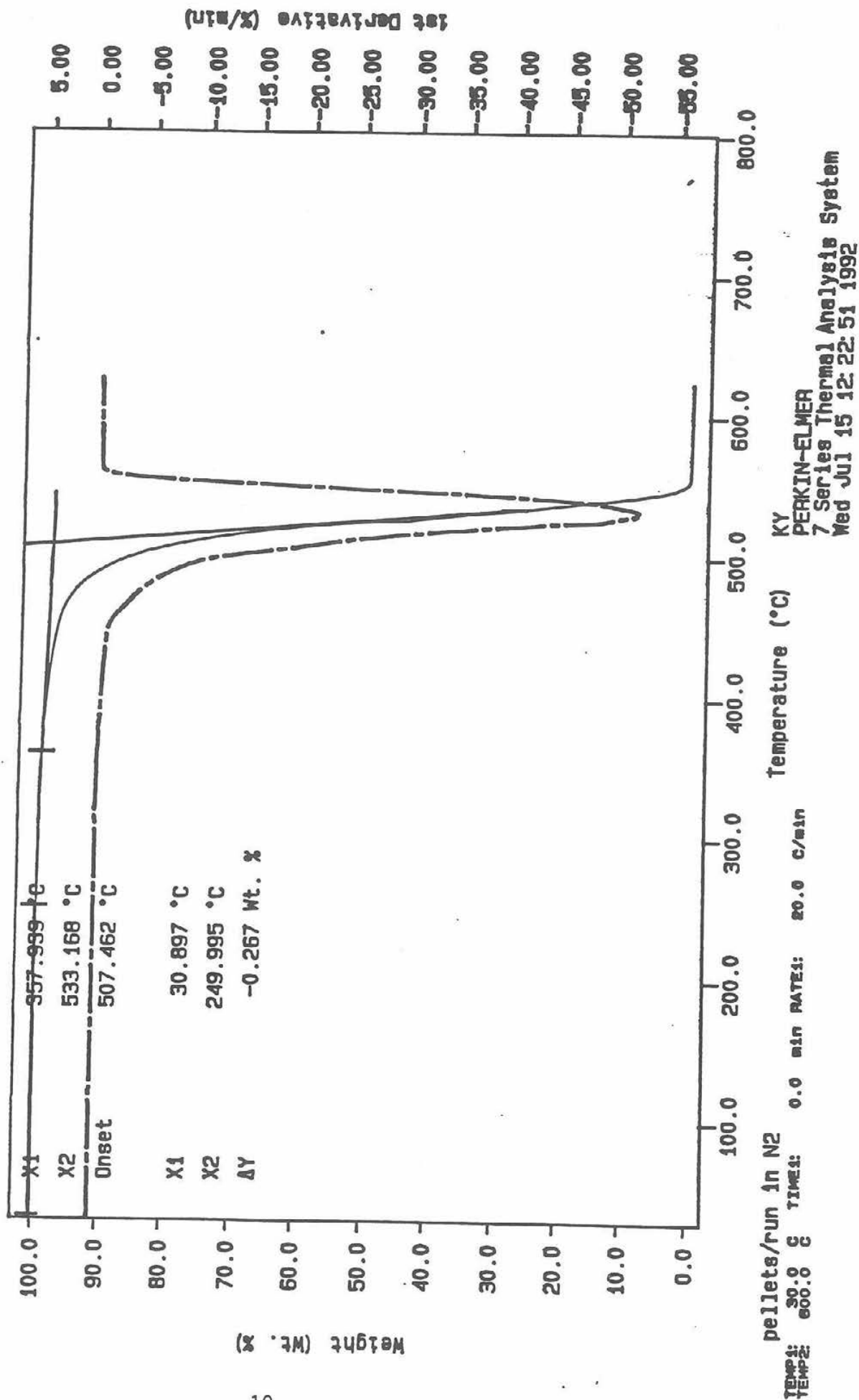
Curve 1: TGA

File Info: c110802c Wed Jul 15 10:27:25 1992

Sample Weight: 10.563 mg

P.D.I. pellet#1 C11080-2

P.D.I. pellet#1 C11080-2



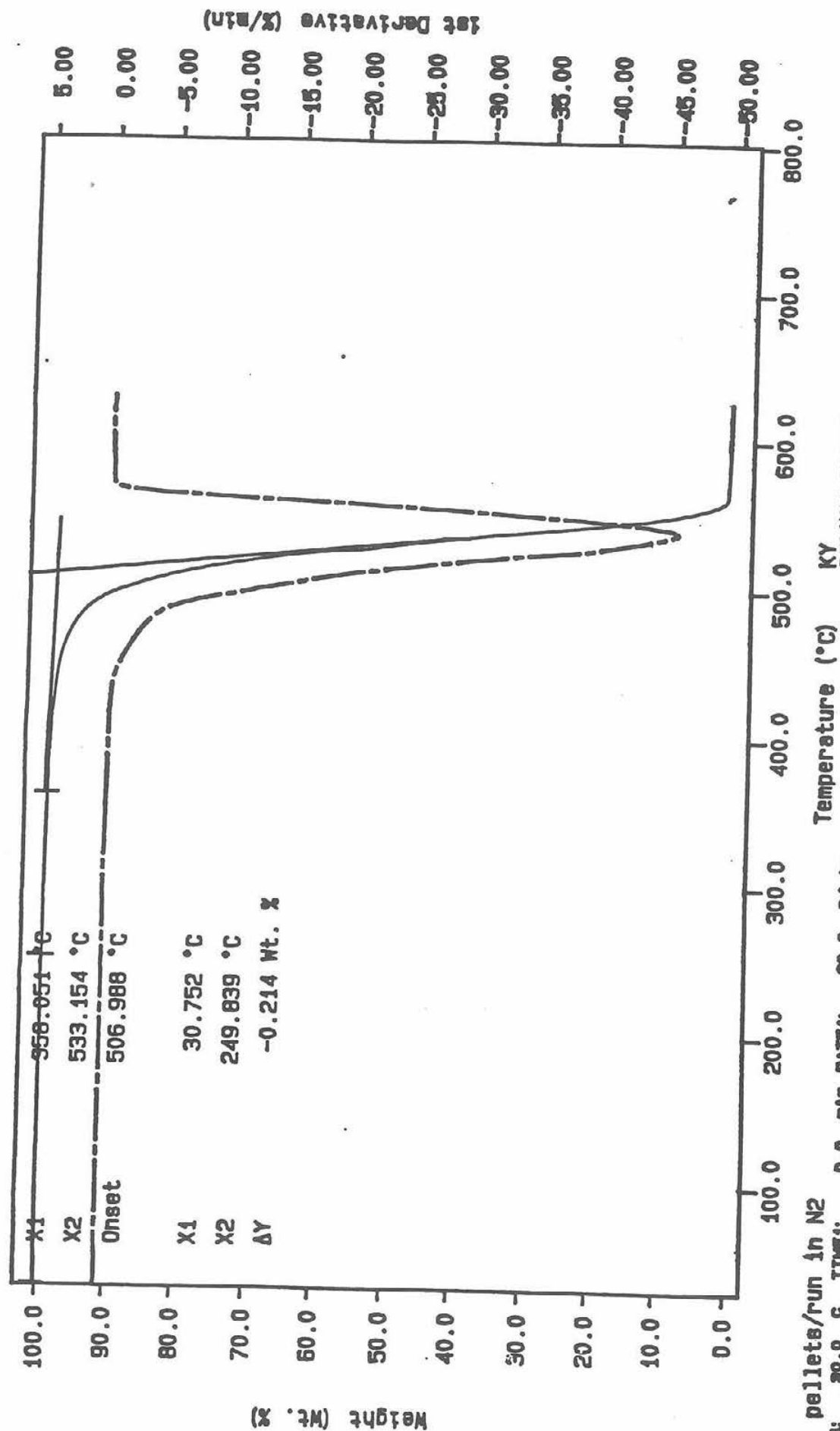
Curve 1: T6A

File Info: c110803a Wed Jul 15 13:14:18 1992

Sample Weight: 11.656 mg

P.D.I. flake #4 C11080-3

P.O.I. flake #4 C11080-3



pellets/run in N2

TEMP1: 30.0 C TIME1: 0.0 min RATE1: 20.0 C/min
TEMP2: 600.0 C

Temperature (°C)

42

PERKIN-ELMER

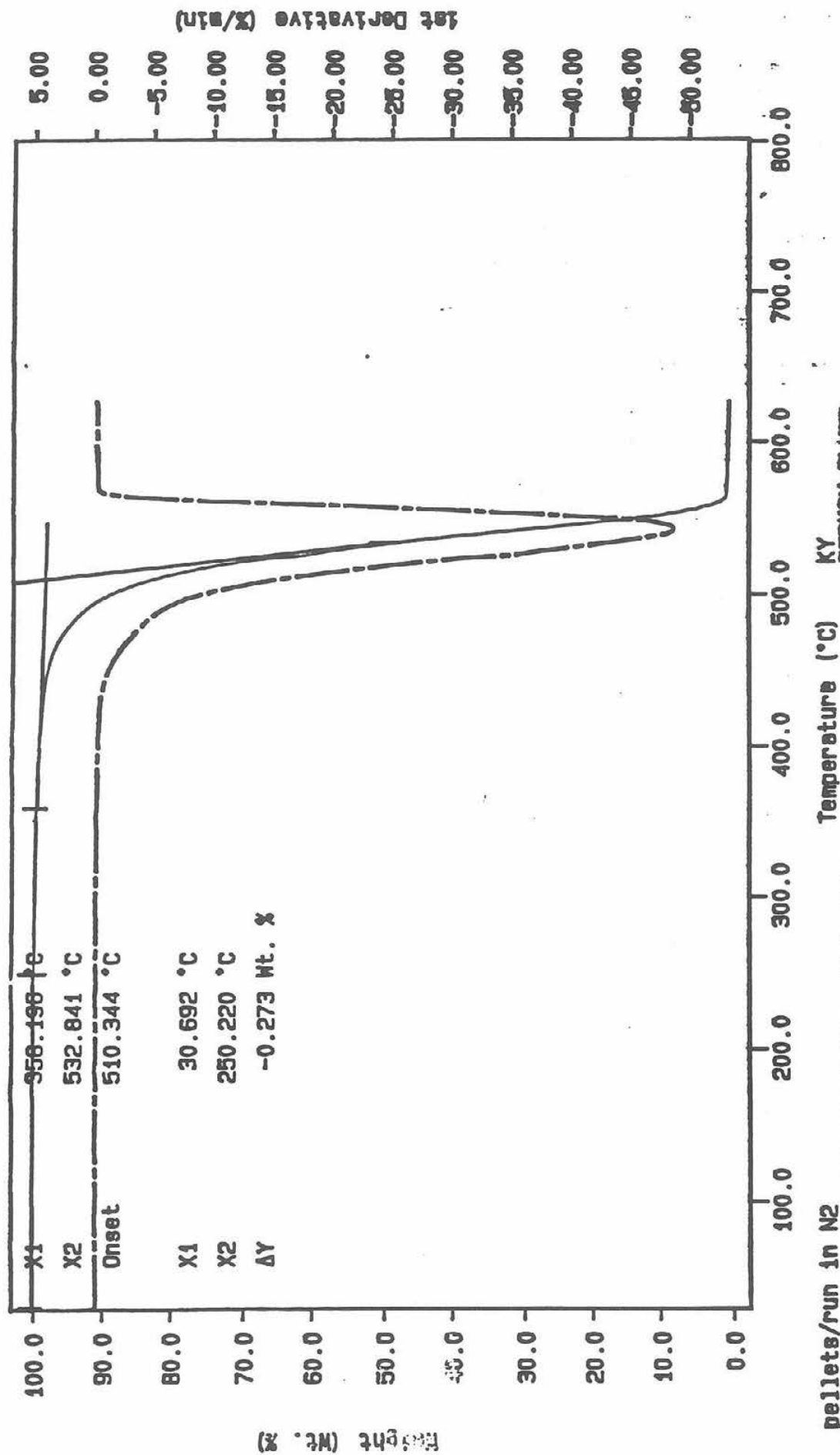
7 Series Thermal Analysis System

Thu Jul 16 07:10:38 1992

20.

Curve 1: TGA
 File Info: c110803b Wed Jul 15 14:24:17 1992
 Sample Weight: 8.897 mg
 P.D.I. flake #4 C11080-3

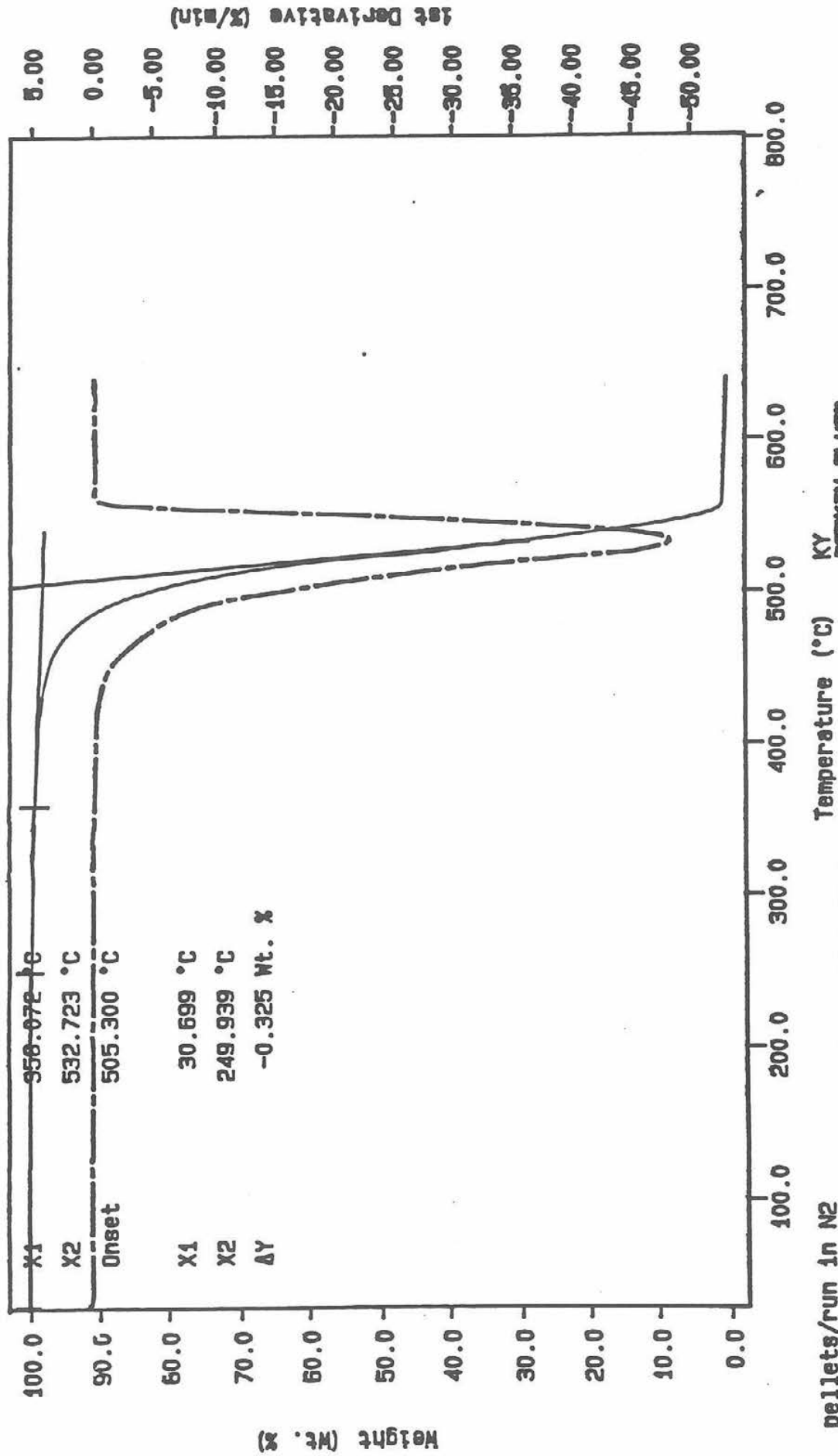
P.D.I. flake #4 C11080-3



pellets/run in N2
 Temp: 90.0 °C
 Time: 0.0 min
 Rate: 20.0 C/min
 KY PERKIN-ELMER
 7 Series Thermal Analysis System
 Thu Jul 16 07:03:09 1992

Curve 1: TGA
 File info: c110803c Thu Jul 16 09:31:51 1992
 Sample Weight: 7.709 mg
 P.D.I. flake #4 C11080-3

P.D.I. flake #4 C11080-3



PERKIN-ELMER
 7 Series Thermal Analysis System
 Thu Jul 16 10:27:25 1992

pellets/run in N2
 TEMP: 30.0 °C
 TIME: 0.0 min RATE: 20.0 °C/min

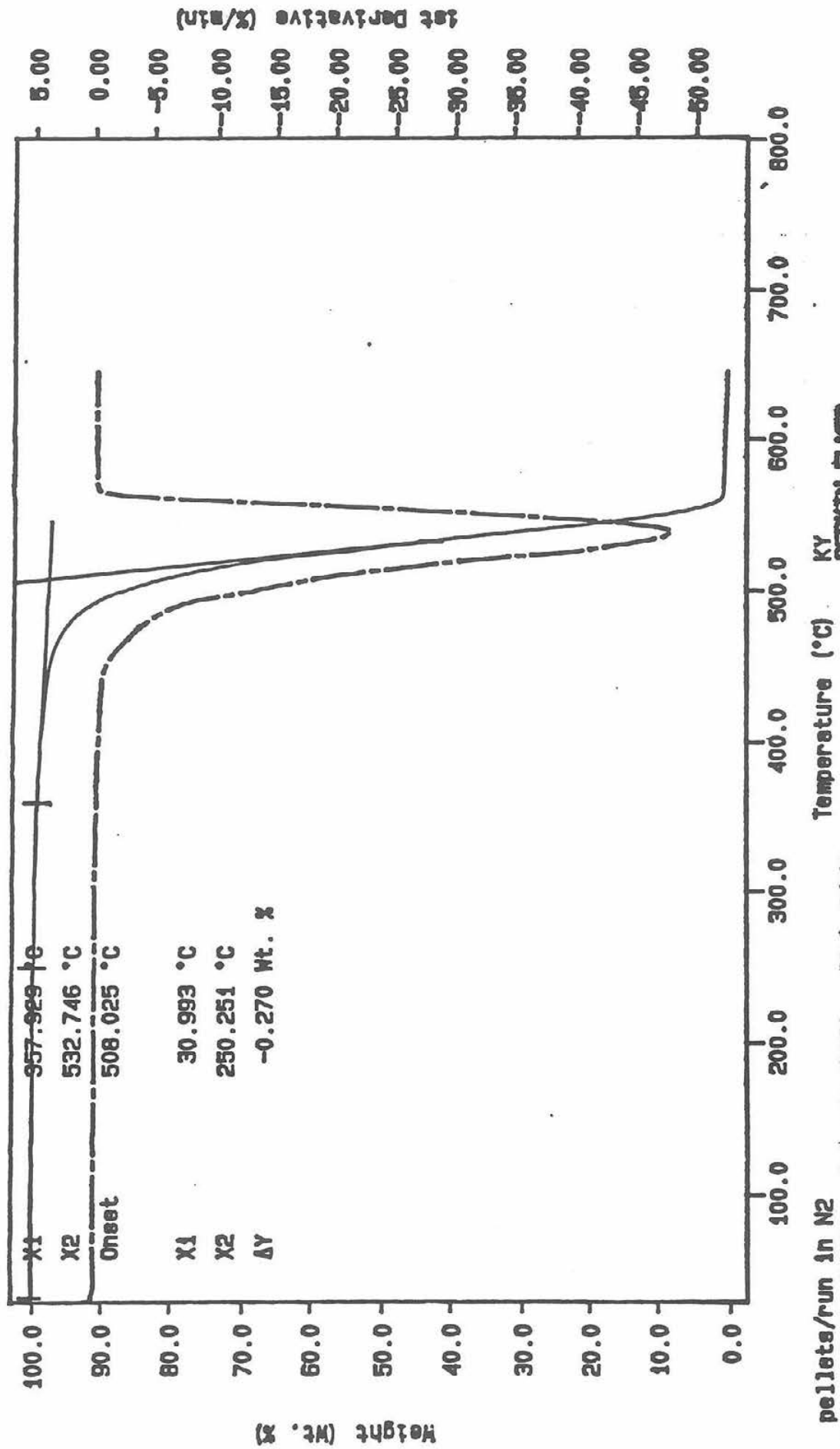
Curve 1: TGA

File Info: C110804a Thu Jul 16 11:12:37 1992

Sample Weight: 9.091 mg

P.D.I. pellet #4 C11080-4

P.D.I. pellet #4 C11080-4



PERKIN-ELMER
7 Series Thermal Analysis System
Mon Jul 20 08:18:33 1992

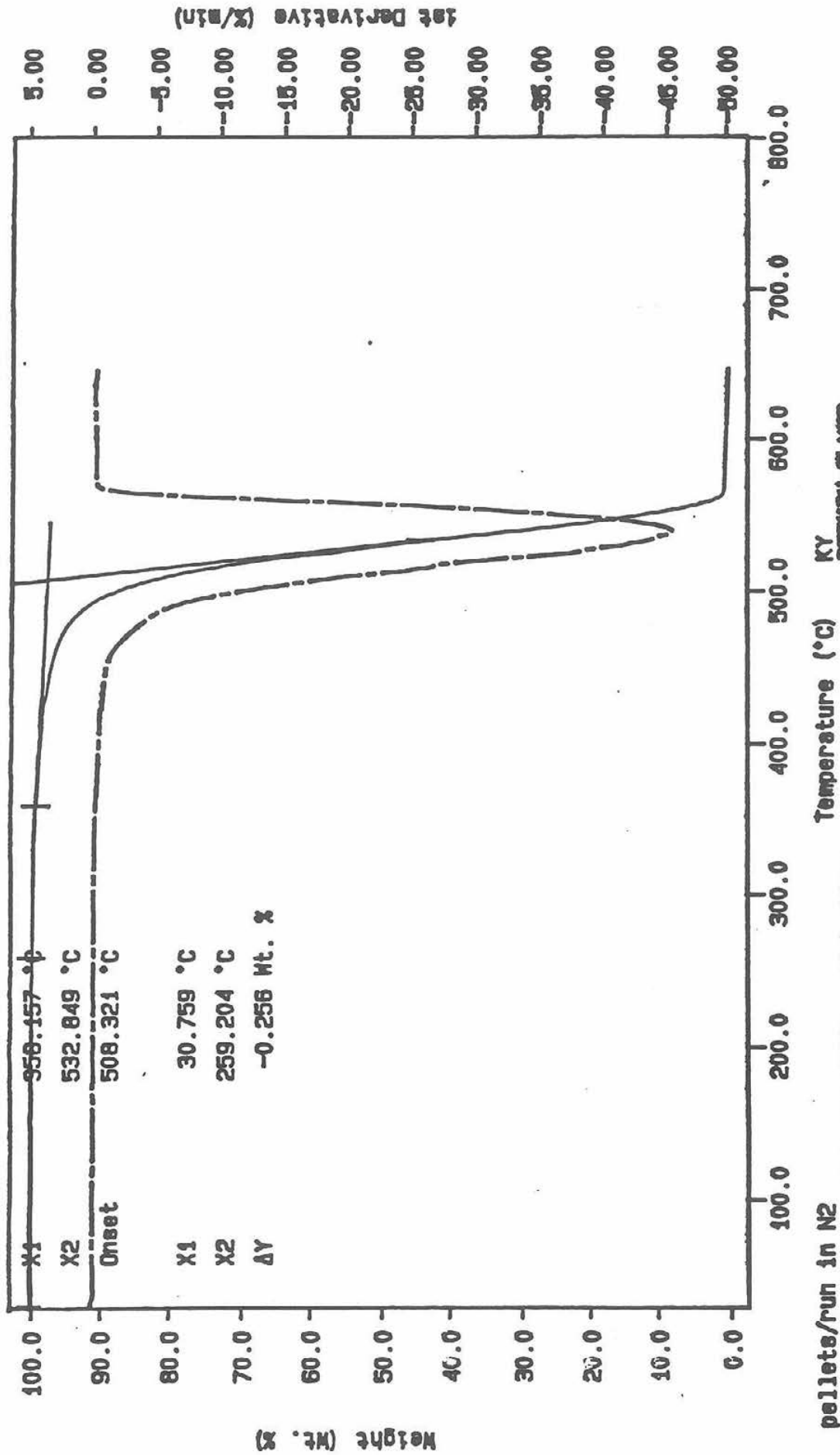
Curve 1: TGA

File Info: cc110804b Thu Jul 16 13:31:35 1992

Sample Weight: 10.176 mg

P.D.I. pellet #4 C11080-4

P.D.I. pellet #4 C11080-4



pellets/run in N2

TEMP: 30.8 °C

TIME: 6.0 min RATE: 20.0 °C/min

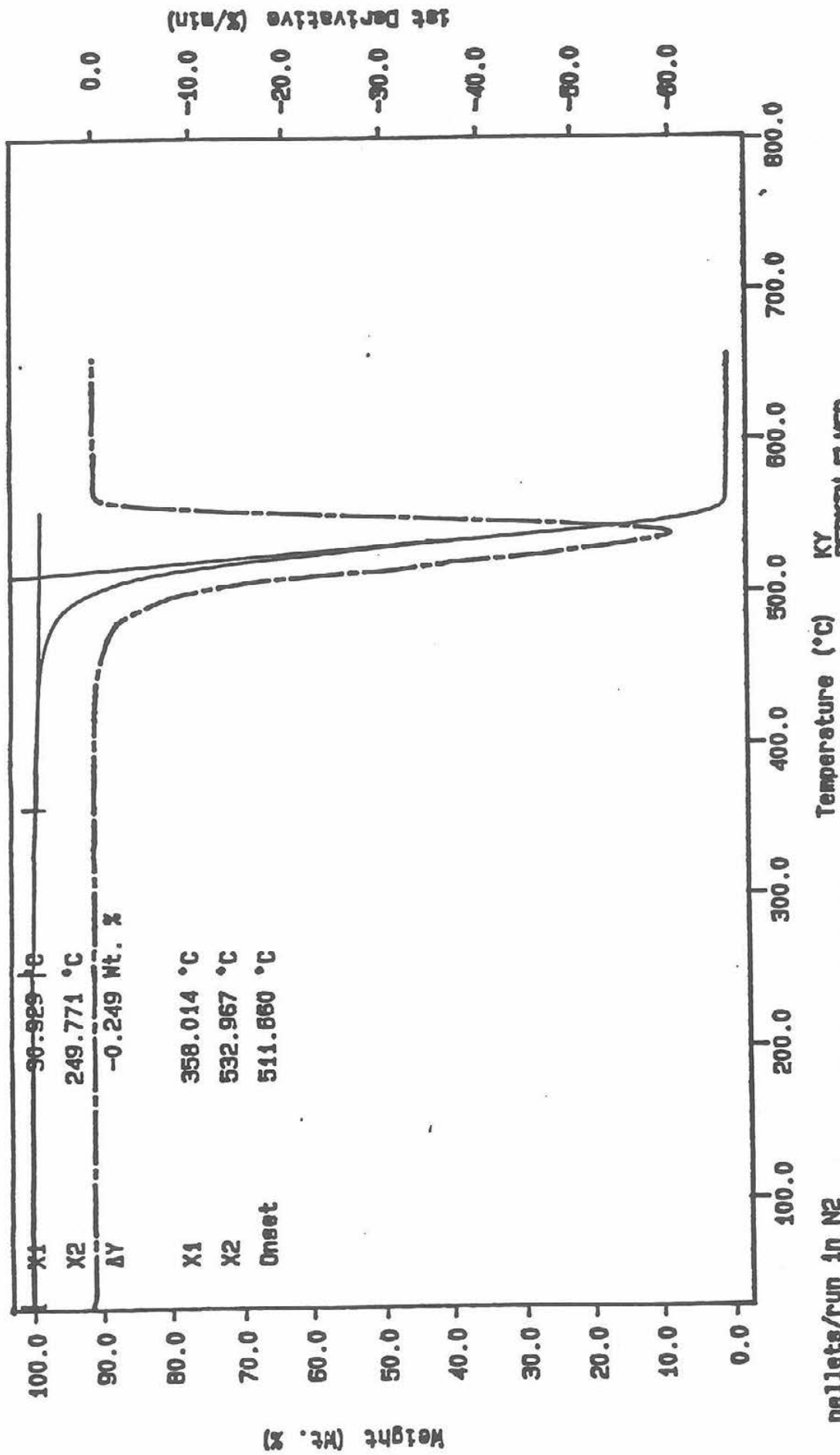
KY PERKIN-ELMER

7 Series Thermal Analysis System

Mon Jul 20 08:37:40 1992

Curve 1: TGA
 File Info: c110804c Fri Jul 17 07:41:22 1992
 Sample Weight: 8.918 mg
 P.D.I. pellet #4 C11080-4

P.D.I. pellet #4 C11080-4



PERKIN-ELMER
 7 Series Thermal Analysis System
 Mon Jul 20 08:41:28 1992

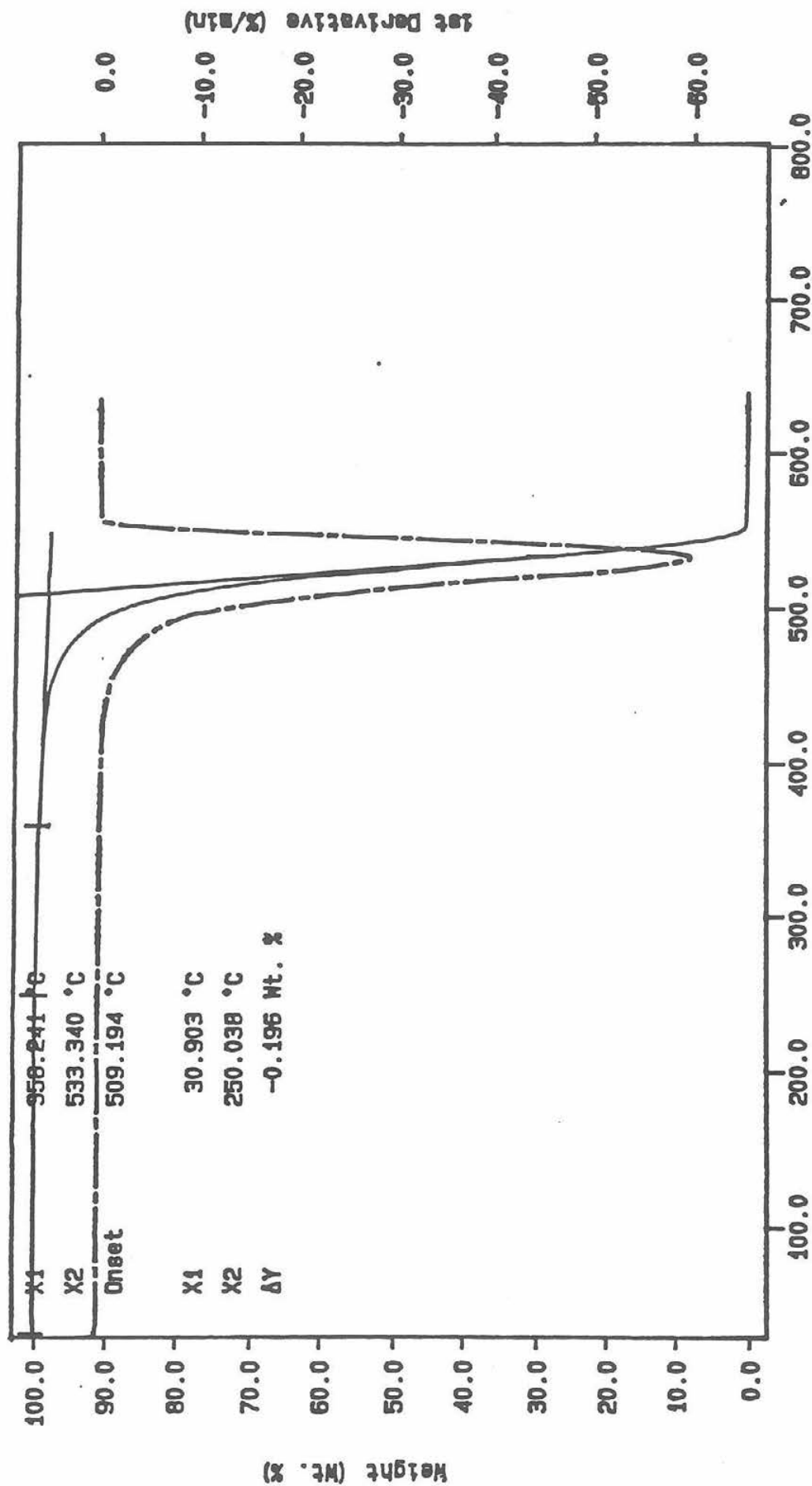
Curve 1: TGA

File info: C110805a Fri Jul 17 14:55:02 1992

Sample Weight: 10.383 mg

P.D.I. flake #8 C11080-5

P.D.I. flake #8 C11080-5



chunks/run in N2

TEMP: 30.0 °C

TIME: 0.0 min

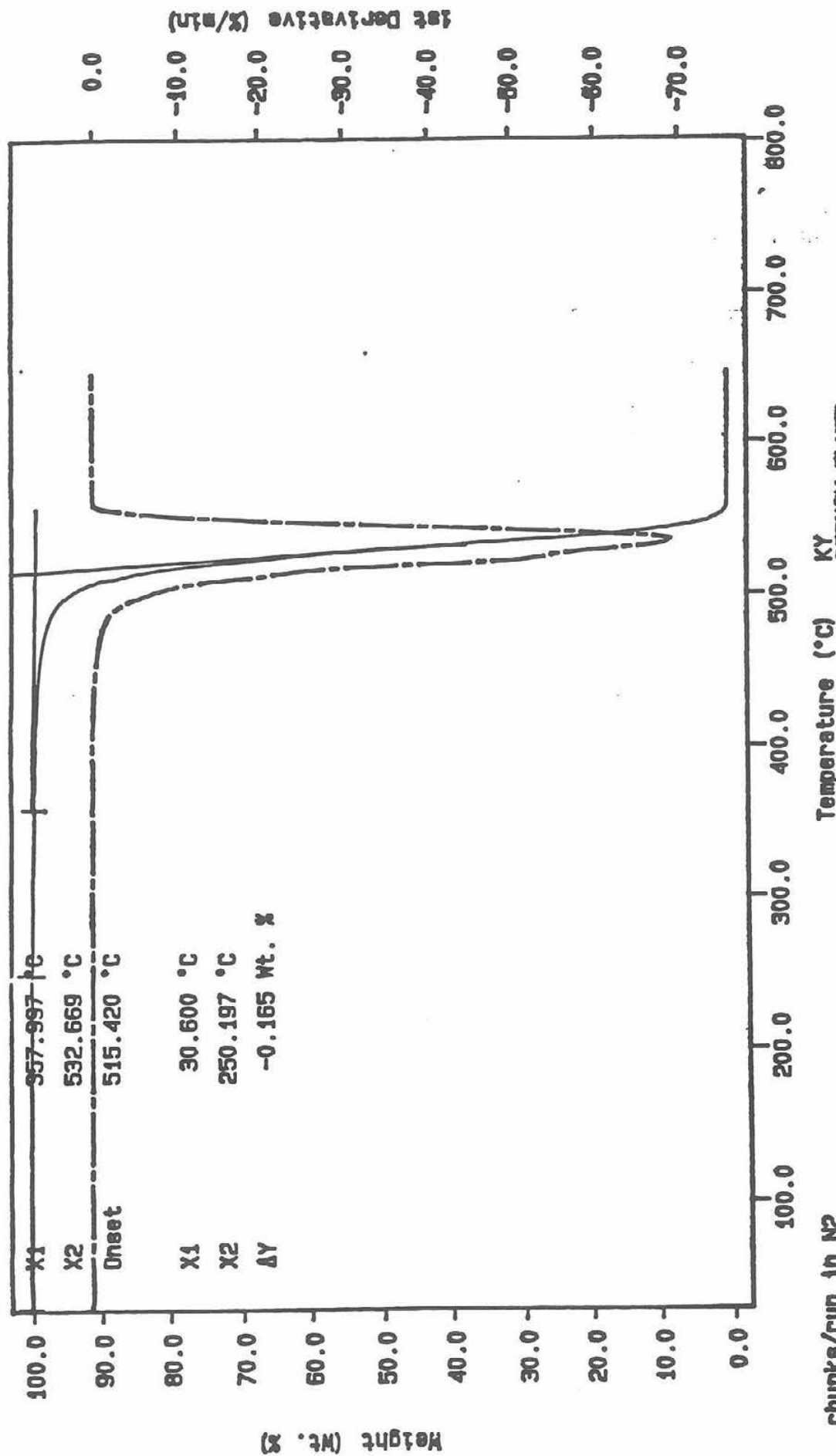
RATE: 20.0 °C/min

KY PERKIN-ELMER

7 Series Thermal Analysis System

Mon Jul 20 07:20:50 1992

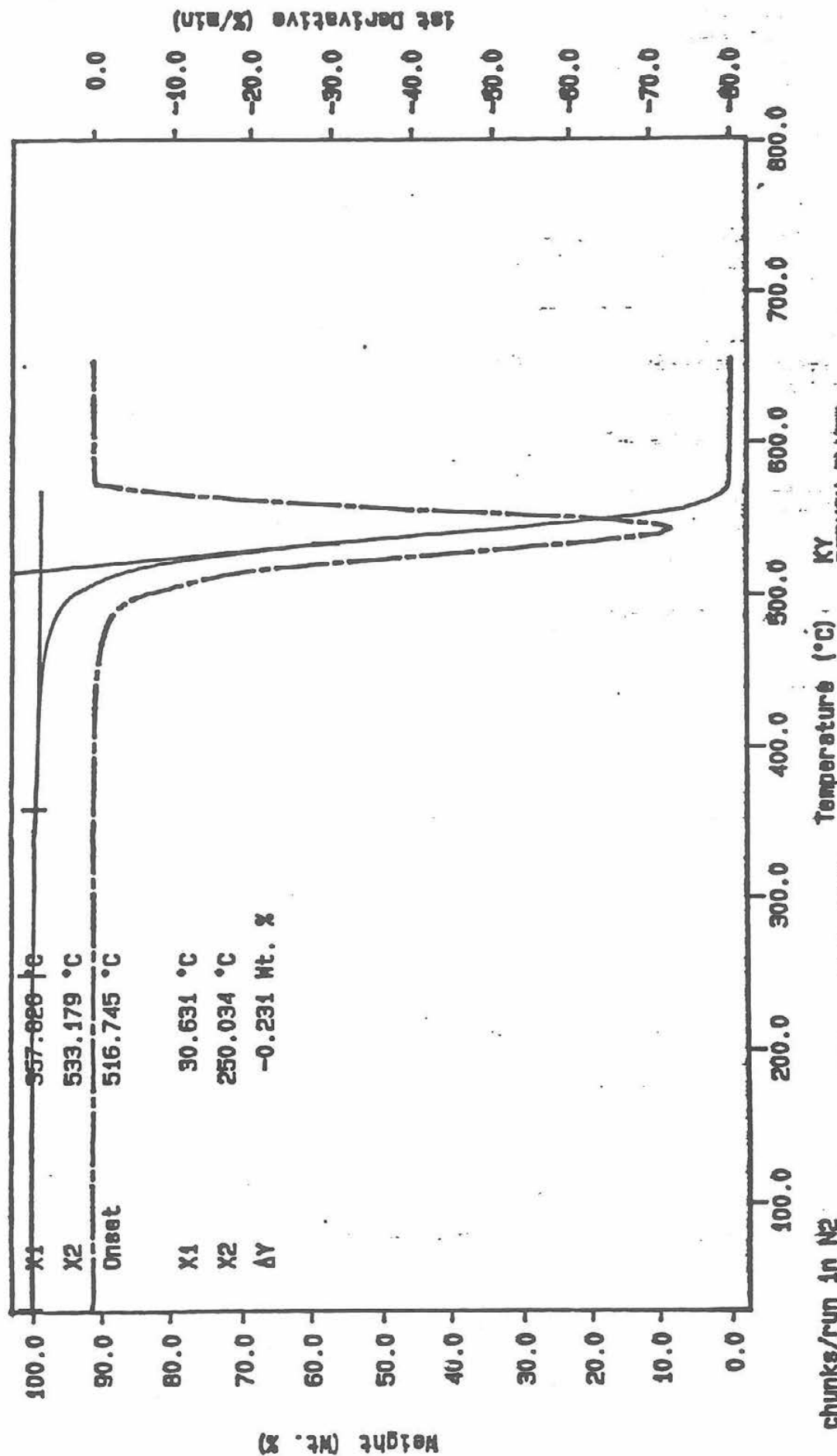
Curve 1: TGA
 File Info: c110805b Sat Jul 18 11:51:23 1992
 Sample Weight: 10.958 mg
 P.D.I. flake #8 C11080-5



chunks/run in N2
 30.0 g
 0.0 min RATE: 50.0 c/min
 TIME: 000.0 g
 KY PERKIN-ELMER
 7 Series Thermal Analysis System
 Mon Jul 20 07:23:16 1992

Curve 1: TGA
 File Info: C110805c Sat Jul 18 13:26:35 1992
 Sample Weight: 7.224 mg
 P.D.I. flake #8 C11080-5

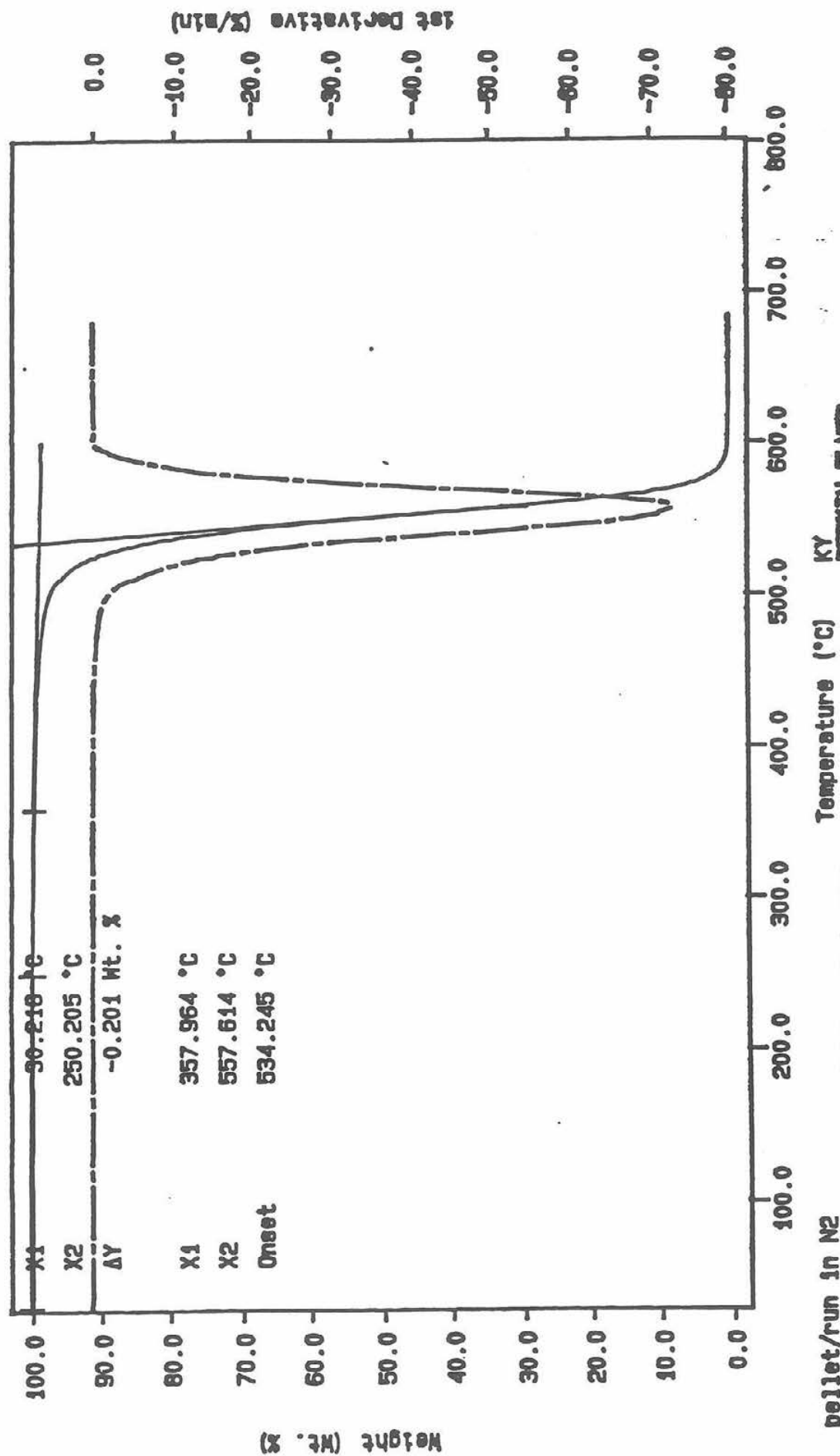
P.D.I. flake #8 C11080-5



chunks/run in N2
 28.8 g
 0.0 min RATE: 20.0 °C/min
 PERKIN-ELMER
 7 Series Thermal Analysis System
 Mon Jul 20 08:13:13 1992

Curve 1: TGA
 File Info: C110806a Mon Jul 20 09:20:41 1992
 Sample Weight: 10.993 mg
 P.D.I. pellet #8 C11080-6

P.D.I. pellet #8 C11080-6



pellet/run in N2
 TGA: 38.8 g
 Time: 0.0 min
 Rate: 50.0 °C/min
 KY
 PERKIN-ELMER
 7 Series Thermal Analysis System
 Mon Jul 20 13:18:16 1992

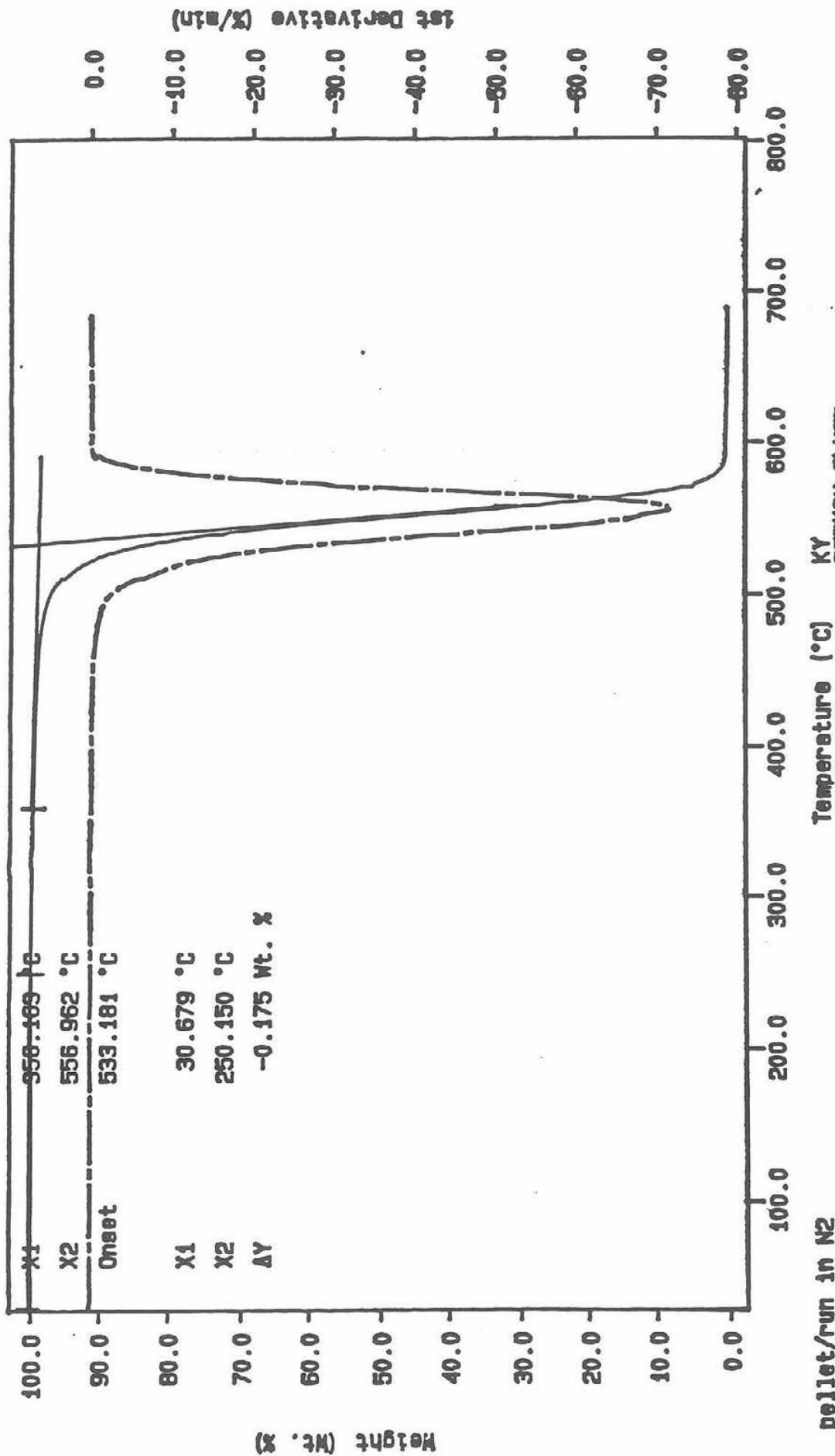
Curve 1: TGA

File info: c110806b Mon Jul 20 10:33:04 1992

Sample Weight: 11.535 mg

P.D.I. pellet #8 C11080-6

P.D.I. pellet #8 C11080-6



pellet/run in N2
 TGA: 38.8 g times: 0.0 min RATE: 20.0 C/min
 KY PERKIN-ELMER
 7 Series Thermal Analysis System
 Mon Jul 20 13:12:21 1992

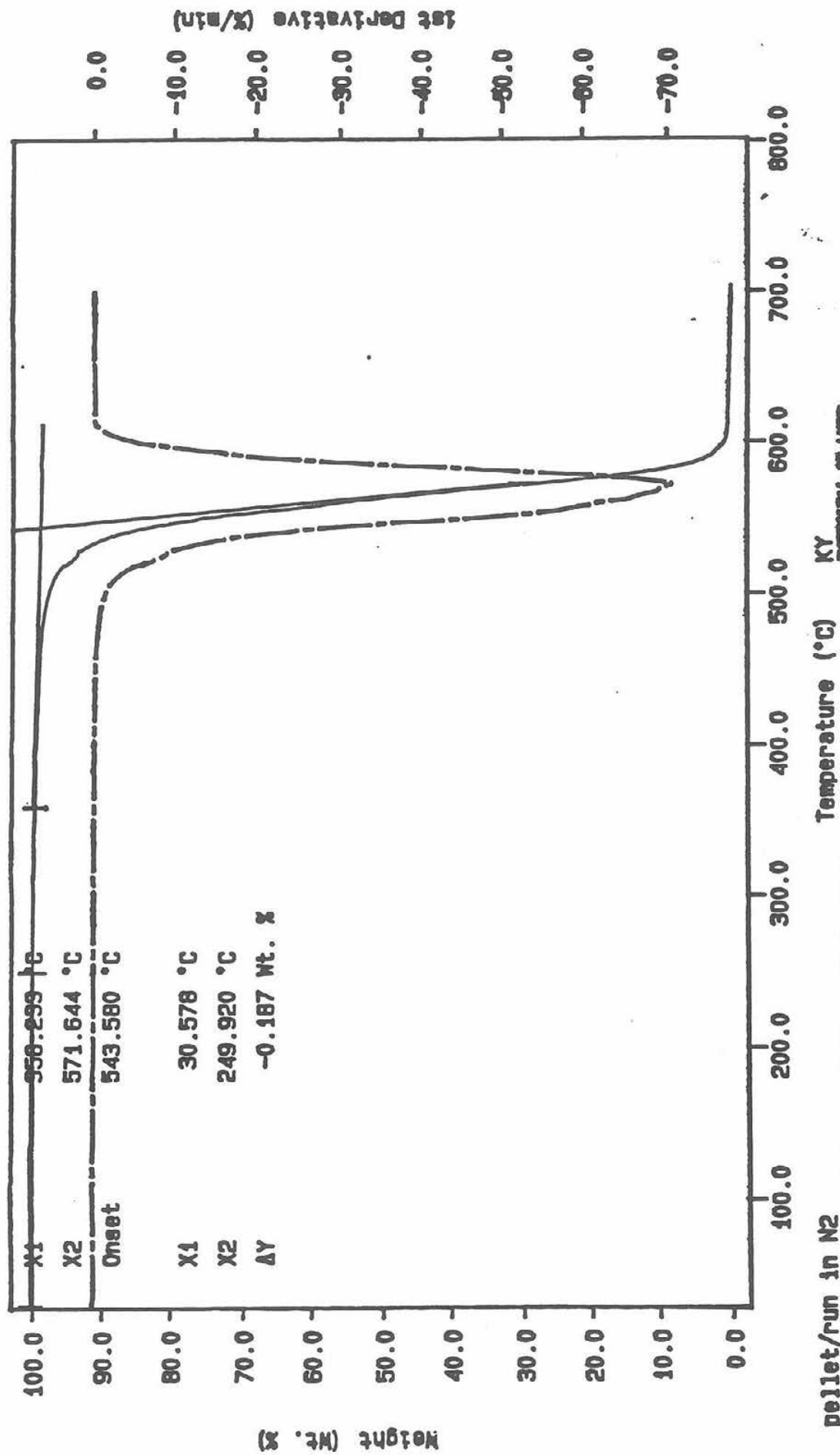
Curve 1: TGA

File info: c110806c Mon Jul 20 12:54:46 1992

Sample Weight: 10.530 mg

P.D.I. pellet #8 C11080-6

P.D.I. pellet #8 C11080-6



pellet/run in N2
 TGA# 00.8 C TGA# 0.0 min RATE: 50.0 C/min
 KY PERKIN-ELMER
 7 Series Thermal Analysis System
 Mon Jul 20 13:07:32 1992

PDI Recycle Study, Acetic Acid and Hydraulic Oil Ladings

I. Acetic Acid Lading

A. Procedure

1. Grind sample to 30 mesh.
2. Place 3 g of ground sample and 4 ml of dichloromethane in a 25 ml screw-top bottle with a polyethylene-lined cap. Shake the mixture to disperse the sample in the dichloromethane, and allow it to stand at ambient temperature for 72 hours.
3. Analyze the extract solution by gas chromatography.
 - a. Column: Supelcowax 10 fused silica, 60 m x 0.32 mm id, 0.25 um film thickness.
 - b. Temperature Program: 80 deg.C to 240 deg.C, 10 deg/min.
 - c. FID temperature: 200 deg.C; injector temperature: 150 deg.C.
 - d. Sample size: 1 uL + 0.5 uL solvent flush.
 - e. The retention time of the acetic acid is ca. 14 min.
4. External calibration with dichloromethane solutions of acetic acid established a detection limit of 10 mg/L.

B. Results and Discussion

1. Under the above conditions, acetic acid is not detected in any of the extracts.
2. A set of representative chromatograms is appended. An acetic acid standard (770 mg/L is shown in each chromatogram as a reference.

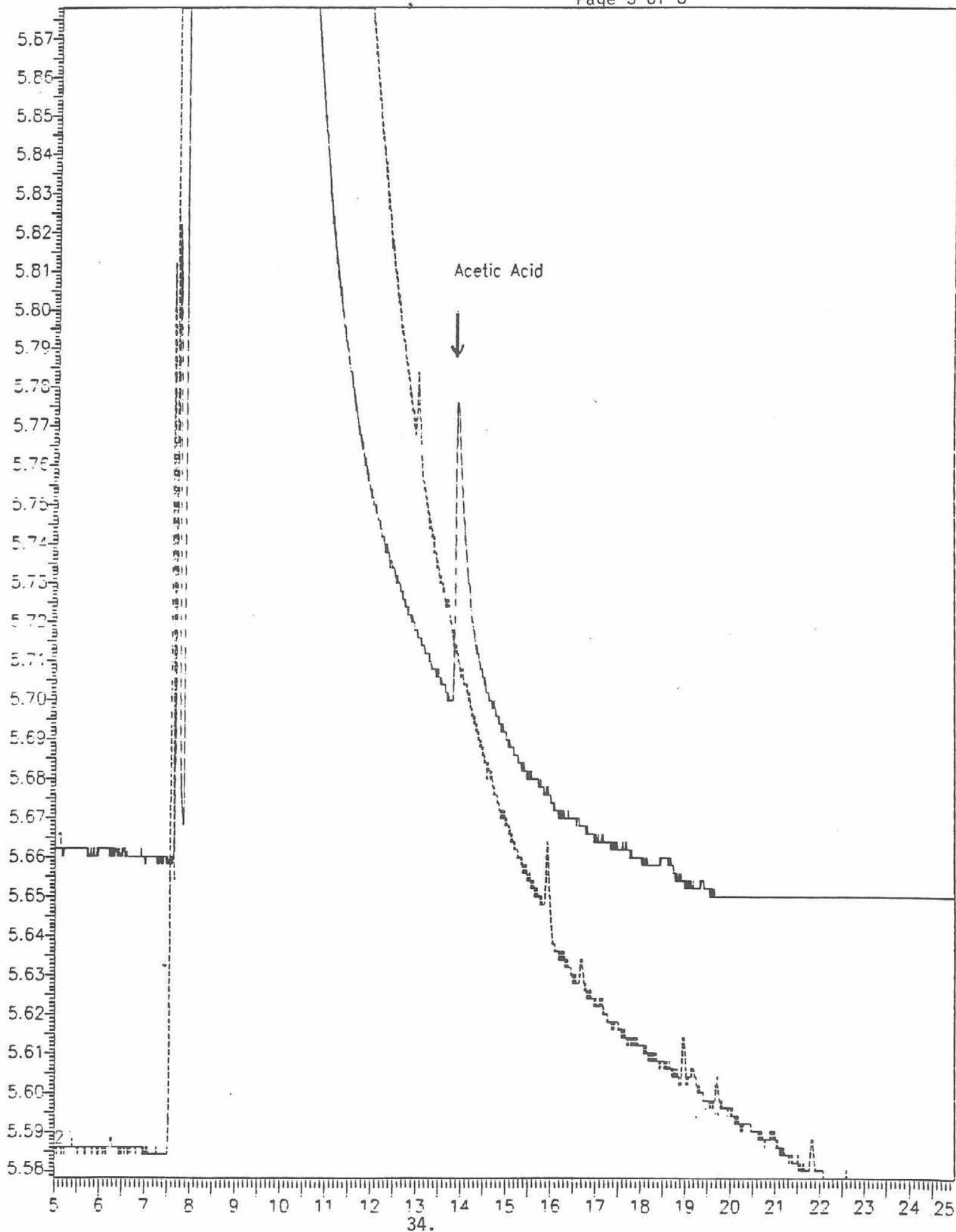
II. Hydraulic Oil Lading

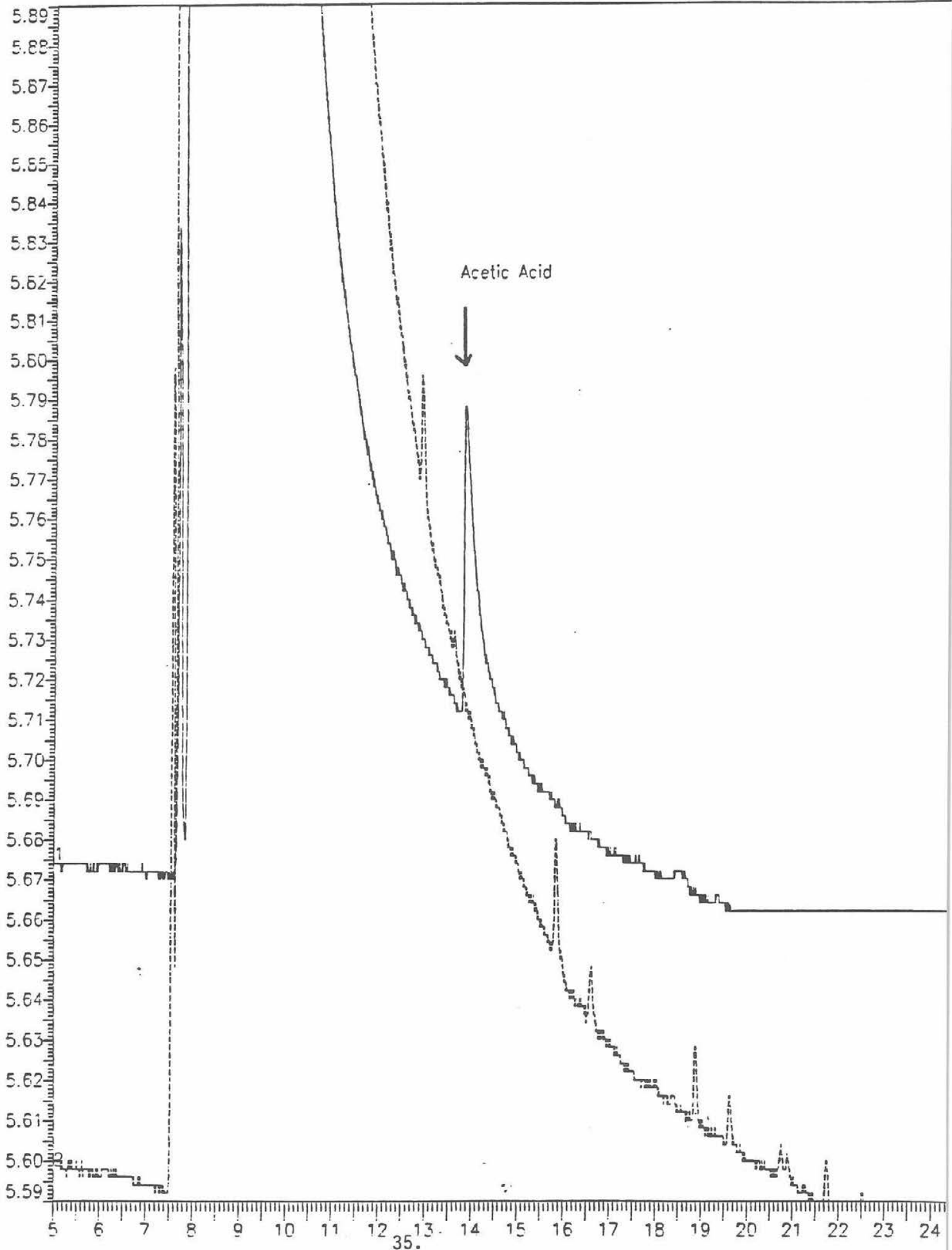
A. Procedure

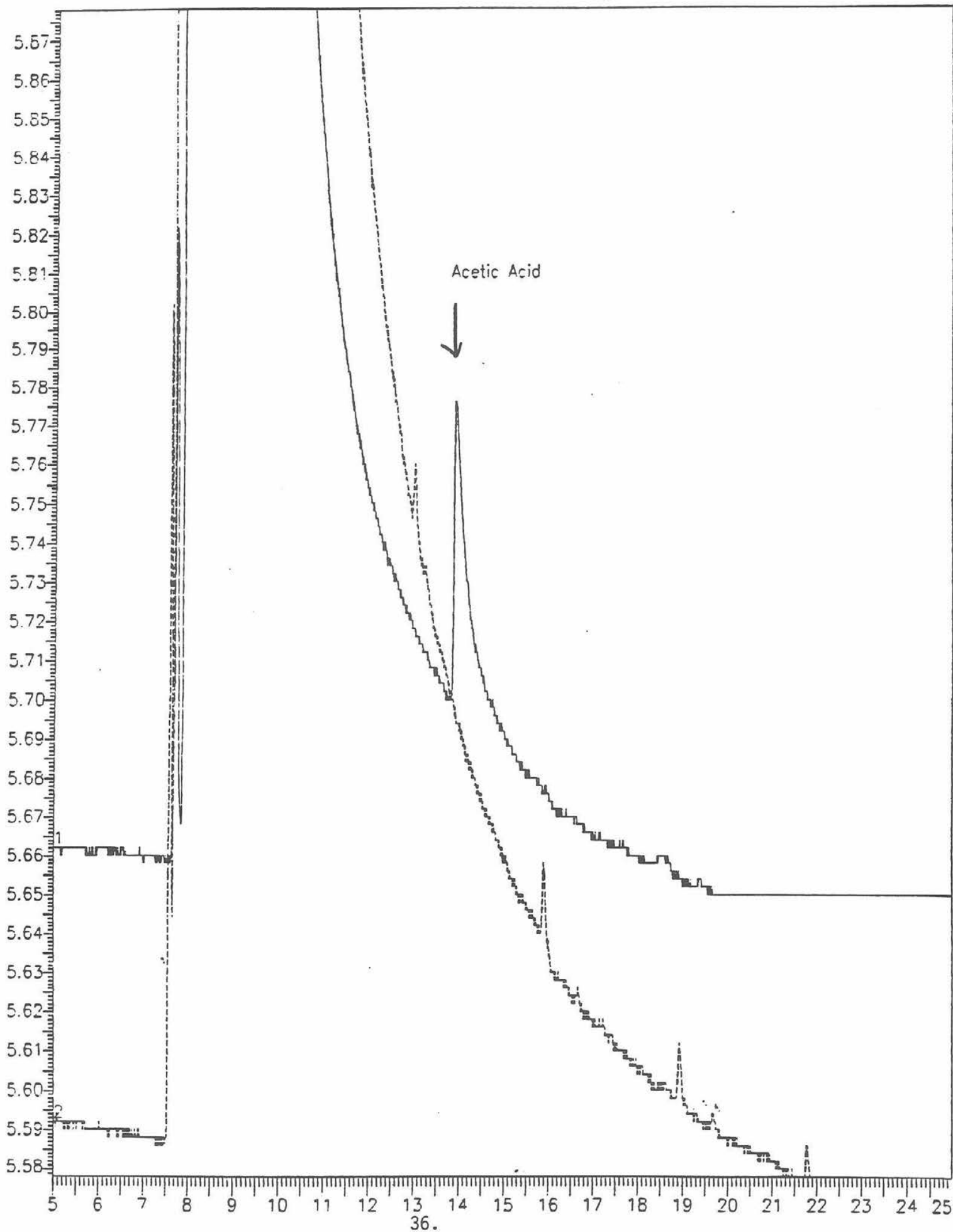
1. Grind sample to 30 mesh.
2. Place 2 g of ground sample and 10 of dichloromethane in a 25 ml screw-top bottle with a polyethylene-lined cap. Shake the mixture to disperse the sample in the dichloromethane, and allow it to stand at ambient temperature for 72 hours. (The oil extraction was done at greater dilution because we expected a positive result in this case.)
3. Analyze the extract solution by gas chromatography.
 - a. Column: Supelco SPB5 fused silica, 30 m x 0.53 mm id, 0.50 μ m film thickness.
 - b. Temperature Program: 70 deg.C for 2 min, program to 280 deg.C at 16 deg/min.
 - c. FID temperature: 350 deg.C; injector temperature: 250 deg.C.
 - d. Sample size: 3.0 μ L.
 - e. The oil elutes in an envelope centered at ca. 14 min.
4. The hydraulic oil provided as a standard was apparently not the same as the oil used in the lading, since it eluted in an envelope centered at ca. 16 min. However, since the response factors for most hydrocarbons are very similar, this difference was ignored for the calibration, which used dichloromethane solutions of the oil.
5. Polyethylene oligomers and additives are also extracted along with the oil; some of these co-elute. To obtain a separate area for the oil in each chromatogram, its peak was integrated in two ways:
 - a. Complete peak, including oligomers, etc. protruding from the oil envelope.
 - b. Oligomers, etc. only, obtained by setting the chromatography data system to treat the envelope as if it were baseline.

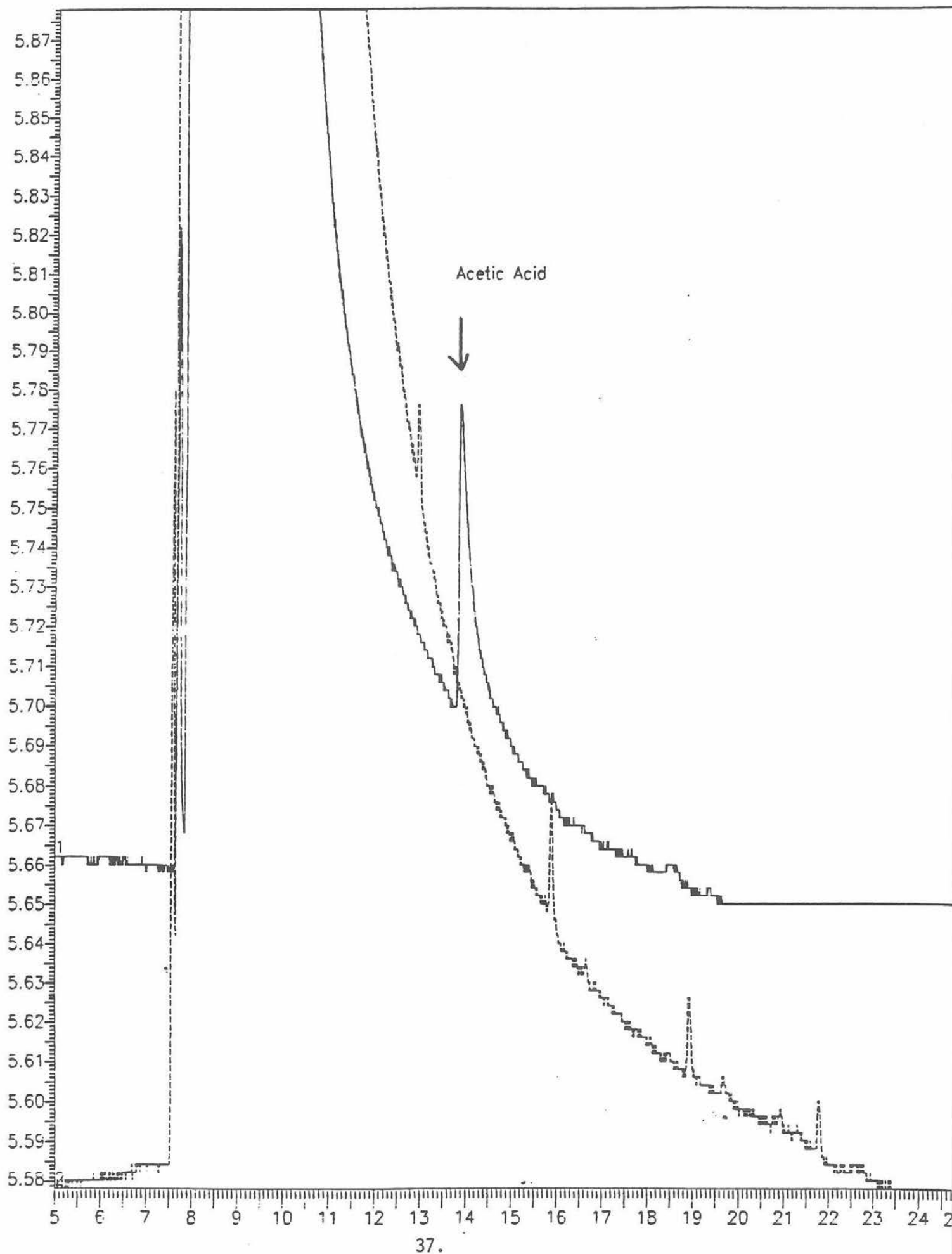
B. Results and Discussion

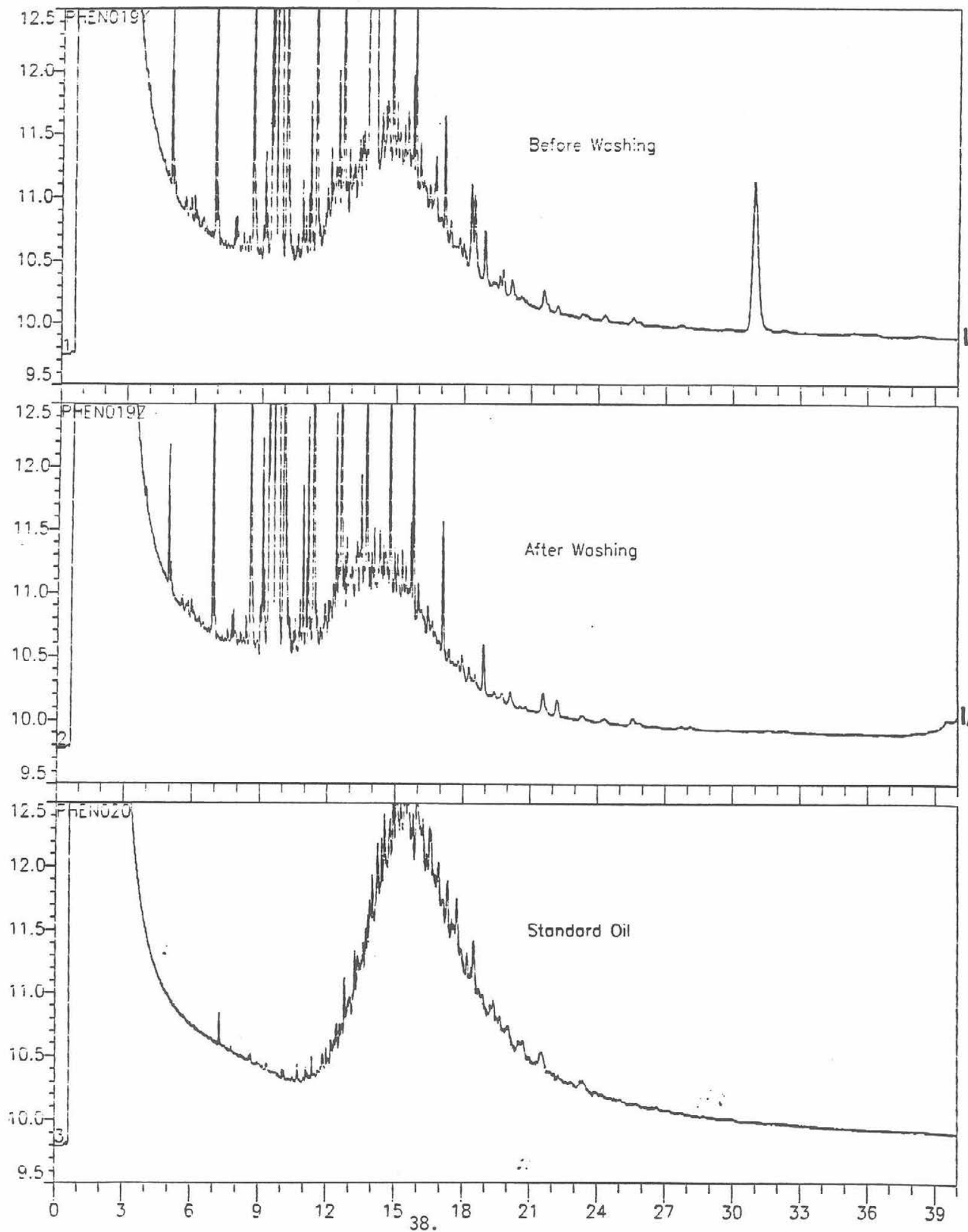
1. There was significant oil remaining in the polyethylene. The oil concentrations in the dichloromethane extract solutions (external standard calibration):
 - a. Extract of unwashed sample: 0.2₅ g/L.
 - b. Extract of washed sample: 0.2 g/L.
2. Thus approximately 20% of the oil in/on the polyethylene is removed by the washing procedure. Other components were also removed during washing. The representative chromatograms accompanying this summary indicate significant decreases in the concentrations of components eluting near 14 and 30 min.

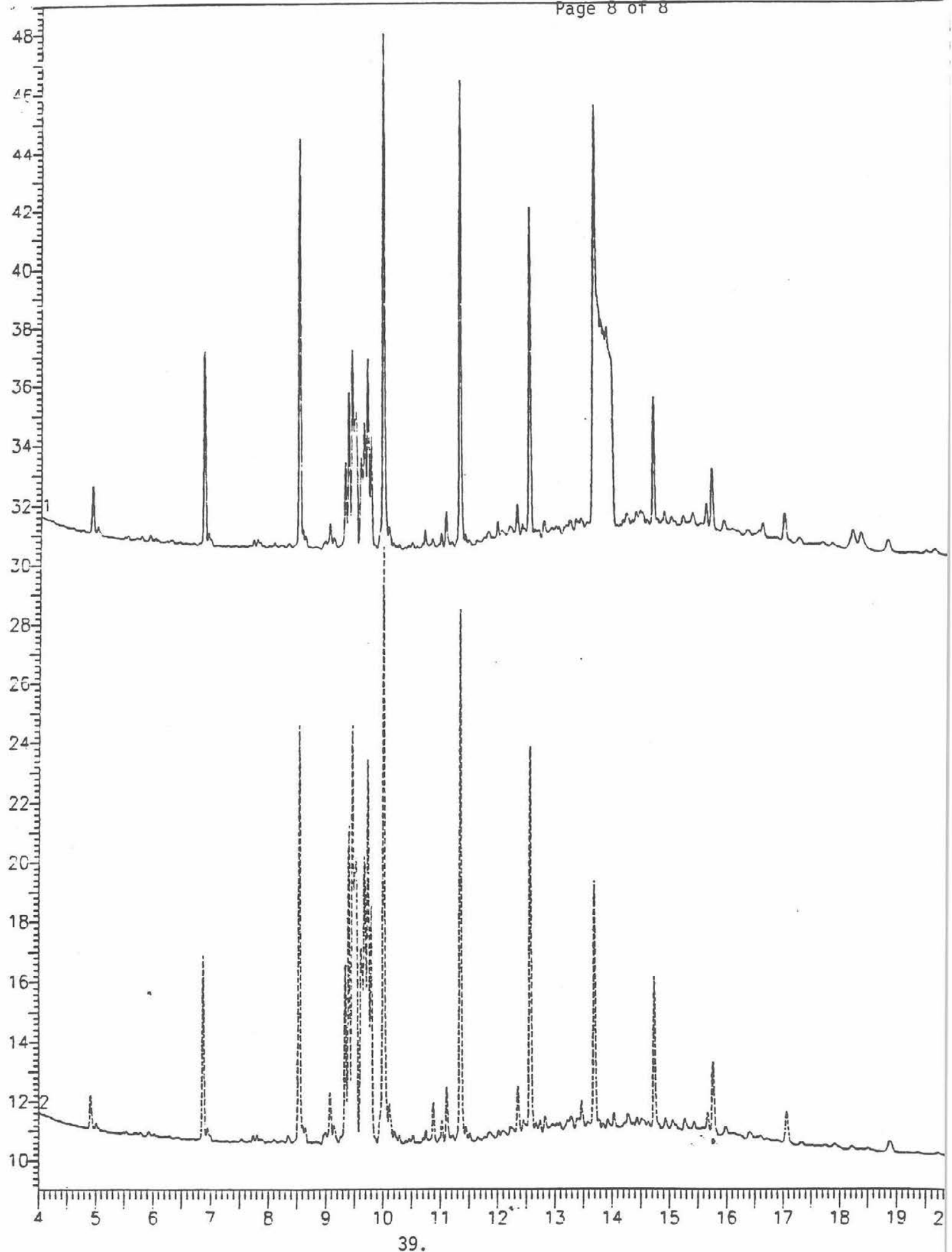














**Fina Oil and Chemical
Company**
Research and Technology Center
Post Office Box 1200
Deer Park, Texas 77536-1200
(713) 884-0500

DATE : September 23, 1992

TO: Mr. John Bergerhouse,
Chairman - Technical Committee
Plastic Drum Institute

FROM: Ms. D'Jaris M. Richardson - Technical Service Engineer

SUBJECT: Test Results for the Drum Recycling Project

	Flow Rate ASTM 1238 <u>HLMI 21.6 kg</u>	Flow Rate ASTM 1238 <u>MI 5.0 kg</u>	Density ASTM D 792 <u>g/cm</u>
SAMPLE 2 ACRYLIC ACID UNWASHED	6.46	0.22	0.956
SAMPLE 2A ACRYLIC ACID PELLETIZED	6.29	0.24	0.956
SAMPLE 3 ACRYLIC ACID UNWASHED	6.18	0.21	0.956
SAMPLE 3A ACRYLIC ACID PELLETIZED	6.64	0.23	0.956
SAMPLE 5 SULFURIC ACID UNWASHED	2.08	0.05	0.953
SAMPLE 5A SULFURIC ACID PELLETIZED	2.98	0.09	0.954
SAMPLE 6 SULFURIC ACID UNWASHED	11.08	0.32	0.955
SAMPLE 6A SULFURIC ACID PELLETIZED	10.46	0.35	0.956

The Fina Laboratory was able to perform an X ray florescence test on the samples containing sulfuric acid and the GC chromatography test on the samples containing acrylic acid. Results are expected in early October, and will be sent to you by Ms. Liesl Massey 713-474-6016.

Best Regards,

D'Jaris Richardson



**Fina Oil and Chemical
Company**
Research and Technology Center
Post Office Box 1200
Deer Park, Texas 77536-1200
(713) 884-0500

October 16, 1992

Mr. John Rathman
Phillips 66 Co.
Plastics Technical Center
Bartlesville, OK 74004

Dear Mr. Rathman,

As requested by D'Jaris Richardson, the following information is
in regard to a PDI Technical Committee request for analysis.

<u>Sample</u>	<u>acrylic acid</u>	<u>sulfuric acid</u>
A	none	
B	none	
C	none	
E	none	
F		157 ppm
G		58 ppm
H		150 ppm
I		49 ppm

Please note the detection limit for the acrylic acid is 50 ppm.

Sincerely,

FINA Oil and Chemical Company

Liesl K. Massey
Technical Service Specialist

cc: P. Blackett


Attachment to Fina Letter dated October 16, 1992

In phone conversations with Liesl Massey of Fina, it was asked that the samples shown in her letter dated October 16, 1992 be further identified. The samples were identified as shown in the table below. On review of the results, some discrepancies were noted and further clarifications could not be obtained. (JRR)

October 16 Letter Identification

PDI Report Identification

A	2A
B	2
C	3
E	3A
F	6A
G	5A
H	5
I	6

<p>FINA OIL AND CHEMICAL COMPANY</p> <p>LA PORTE PLANT</p> <p>QUALITY ASSURANCE METHODS</p>		<p>QA-C-015-4</p> <hr/> <p>Page 1 of 4</p> <hr/> <p>Issue Date: 10/28/80</p> <hr/> <p>Revision Date: 10/01/92</p> <hr/> <p>Approval: <i>[Signature]</i></p>
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**Additives and Metal in Polypropylene
By X-Ray Fluorescence Spectrometer**

I. Purpose


The purpose of this analytical procedure is to provide a method for determining certain additives and metals in Polypropylene by X-Ray Fluorescence Spectroscopy.

II. Equipment

1. Two (2) hydraulic presses, one (1) for molding samples at a temperature of $430 \pm 20^{\circ}\text{F}$, and one (1) for cooling samples.
2. Siemens sequential X-Ray Fluorescence Spectrometer, Model SRS-303 or the equivalent.
3. Molding assembly composed of:
 - a. Two $8\frac{1}{2}" \times 12" \times \frac{1}{8}"$ aluminum backup plates
 - b. Two $8\frac{1}{2}" \times 12"$ chrome ferrotype plates
 - c. Two sheets of mylar film, 1.5 mil thick
 - d. One mold, $8\frac{1}{2}" \times 12" \times \frac{1}{8}"$, with four (4) evenly spaced holes, 46 mm in diameter.
4. Dry-film lubricant, fluoroglide CP.

III. Sample Preparation

1. Mold assembly arrangement is as follows:
 - a. Aluminum backup plate
 - b. Chrome ferrotype plate
 - c. Mylar film
 - d. Mold
 - e. Mylar film

<p>FINA OIL AND CHEMICAL COMPANY</p> <p>LA PORTE PLANT</p> <p>QUALITY ASSURANCE METHODS</p>		<p>QA-C-015-4</p>
		<p>Page 2 of 4</p>
		<p>Issue Date: 10/28/80</p>
		<p>Revision Date: 10/01/92</p>
		<p>Approval:</p>

- f. Chrome ferrotype plate
 - g. Aluminum backup plate
- ffm 10/29/92*
Note: Both mylar sheets may be placed on same side of mold with sample in between as an alternate prep method.
2. Spray lightly the chrome ferrotype plates with dry-film lubricant. Use fresh Mylar with each plaqueing.
 3. Fill the holes in the mold with samples (2 holes per sample).
 4. Place the mold assembly in the heated press and close platens to contact pressure for two (2) minutes.
 5. Pressure to 10 tons for an additional two (2) minutes.
 6. Remove mold assembly from hot press, place in cold press and immediately pressure to 20,000 psi until cool.
 7. Remove mold assembly from cold press, dismantle mold, remove plaques and trim away overflow.
 8. Put plaques into coin envelope bearing the lot number and product type.

VI. Sample Analysis

1. Put sample into bottom half of holder.
2. Behind sample place backup disc.
3. Put on top half of holder.
4. Place sample into spectrometer and close lid.
5. Input sample I.D., position number, and program into the computer. Table I lists the available programs.



FINA OIL AND CHEMICAL COMPANY LA PORTE PLANT QUALITY ASSURANCE METHODS		QA-C-015-4
		Page 3 of 4
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		Approval:

Table I

<u>Program</u>	<u>Elements</u>
Mgl.Qan	Mg
Al.Qan	Al
Ni.Qan	Ni
Fe.Qan	Fe
Ti.Qan	Ti
Cl.Qan	Cl
S.Qan	S
Si.Qan	Si
Ca.Qan	Ca
P.Qan	P
Zn.Qan	Zn
Met.Qan	Al,Ni,Fe,Ti,Cl
Add.Qan	S,Si,Ca,P,Zn
All.Qan	Al,Ni,Fe,Ti,Cl, S,Si,Ca,P,Zn

6. The computer will printout the results in the following units:

Mg	ppm
Al	ppm
Ni	ppm
Fe	ppm
Ti	ppm
Cl	ppm
S	percent
Si	percent
Ca	percent
P	percent
Zn	percent

FINA OIL AND CHEMICAL COMPANY LA PORTE PLANT QUALITY ASSURANCE METHODS		QA-C-015-4
		Page 4 of 4
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		Revision Date: 10/01/92
		Approval:

7. The computer also calculates the percent of additives from the appropriate elements and lists them after the elemental results.

V. Calibration

The calibration will be performed by the person responsible for the instrument.
 (Refer to X-Ray Calibration Procedure QCC-C-001 in Quality Control Calibration Manual.)

VI. Statistical Process Control

At the beginning of each shift, a check sample is analyzed. The results must be input into a log book, the values checked versus the stated upper and lower control limits and initialed by the technician. If results are outside of the stated limits, ~~inform the person responsible for maintaining the equipment.~~ *retest the standard.*

If the same element is still out of limits, inform the person responsible for maintaining the equipment.

JLM 2/5/93

*Detection limit of S is 1 ppm.
 MKG 1/8/93*

September 10, 1992

John Bergerhouse
QUANTUM CHEMICAL
11500 Northlake Drive
PO Box 429550
Cincinnati, Ohio
45250

Sample 7 7A

Dear John,

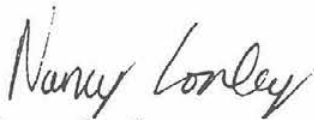
Testing has been completed on the two samples of recycled drum resin which were from drums used to store Mineral Spirits. The test results for the two samples were as follows:

	FLAKED SAMPLE	PELLET SAMPLE
Flow Index, g/10 min (190/21.6)	5.99	6.62
Melt Flow Ratio	20.0	21.0
Density, g/cc	.9548	.9552
Ash, %	.158	.162
OIT @ 200 C, min	158	141
TGA Residue, %	99.6	99.6
DSC melting point, C	133.4	134.7

Page 2
September 10, 1992

Both samples appear to be adequately stabilized and the flow properties are consistent. The thermal gravimetric testing "TGA" showed no difference in volatiles between the two samples. Retains of both samples have been saved at the Novacor Technical center in Calgary should further testing be required.

Sincerely

A handwritten signature in cursive script that reads "Nancy Conley".

Nancy Conley
Technical Service Specialist

cc: Don Parks
Chris Gick

NC/sm

**Novacor****NOVACOR CHIMIE (CANADA) LTÉE**

3100, Côte Vertu, Bureau 460

Saint-Laurent, Québec

H4R 2J8

Téléphone: (514) 745-1414

Fax: (514) 745-0874

FEUILLE DE TRANSMISSION/TRANSMISSION SHEET

DATE:	
POUR/TO:	JOHN RATHMAN
ENTREPRISE/COMPANY:	PHILLIPS
VILLE/CITY:	
FAX:	

REMARQUES/COMMENTS:	TGA'S have been completed on natural HB-W555-A. The average value is 99.8 % residue. Three TGA curves are attached

ENVOYEUR/SENDER:

Nancy Conley

Nombre de pages incluant celle-ci/Number of pages including this one:

4

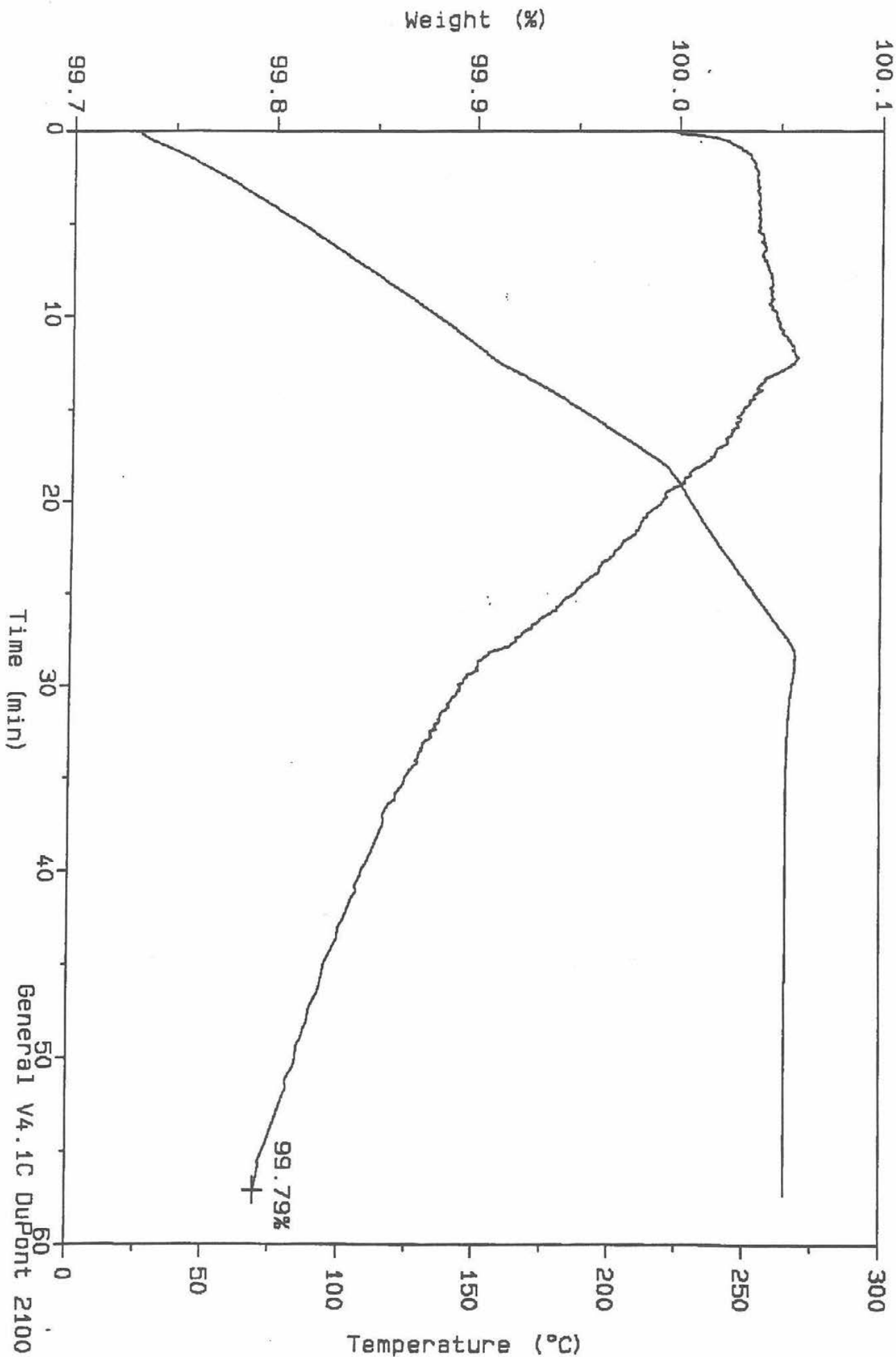
Si vous éprouvez des difficultés de transmission, veuillez appeler le (514) 745-1414.

If you experience transmission difficulties, please call (514) 745-1414.

Sample: 93-564 HBW555A R24844
Size: 57.0290 mg
Method: TGA
Comment: IN N2 RAMP 10C/MIN TO 200C THEN 5C/MIN TO 250C

TGA

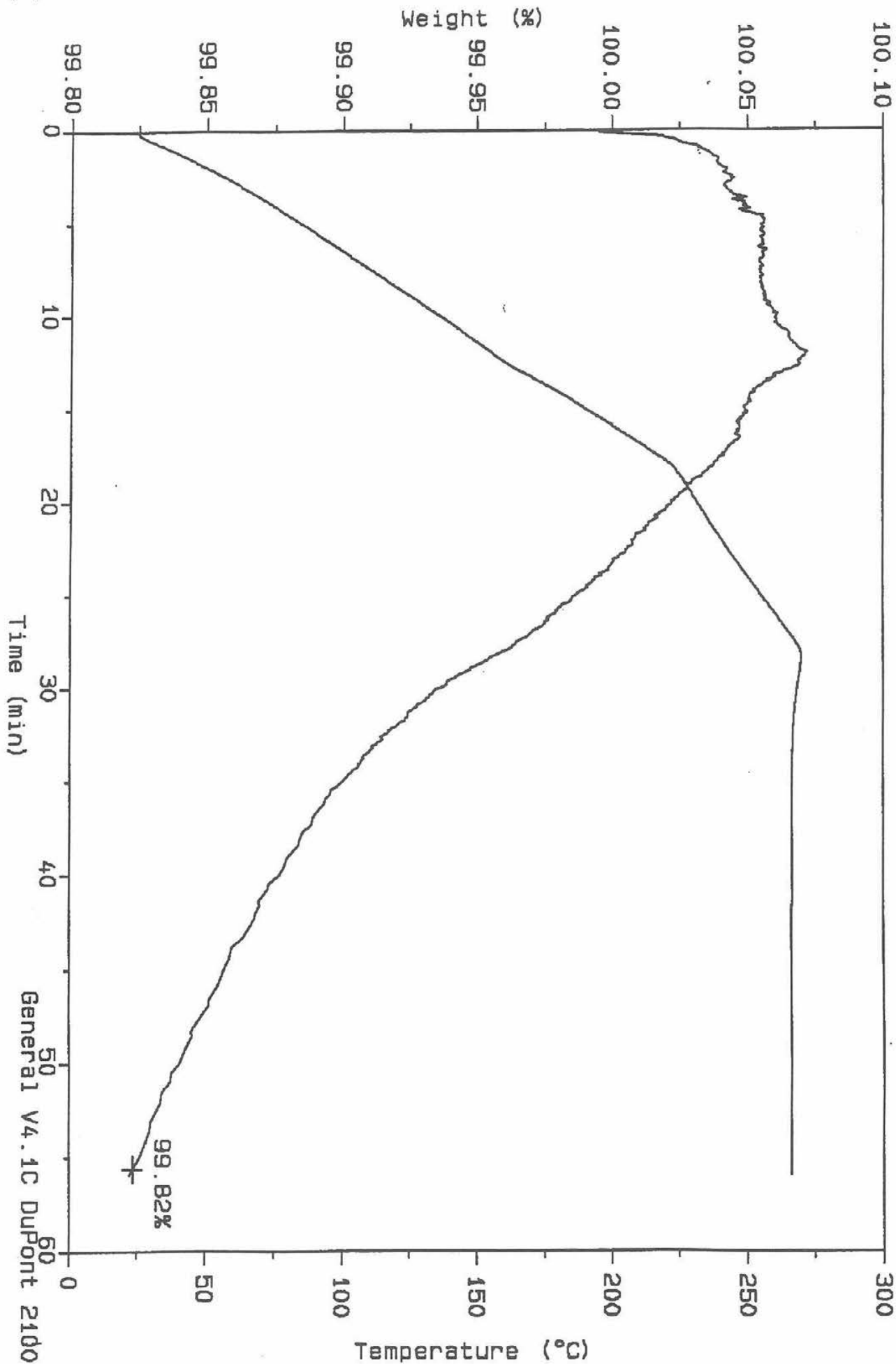
File: A: 93-564.01
Operator: VD
Run Date: 10-Mar-93 11:06



Sample: 93-564 HBW555A R24844
Size: 59.8940 mg
Method: TGA
Comment: IN N2 RAMP 10C/MIN TO 200C THEN 5C/MIN TO 250C

TGA

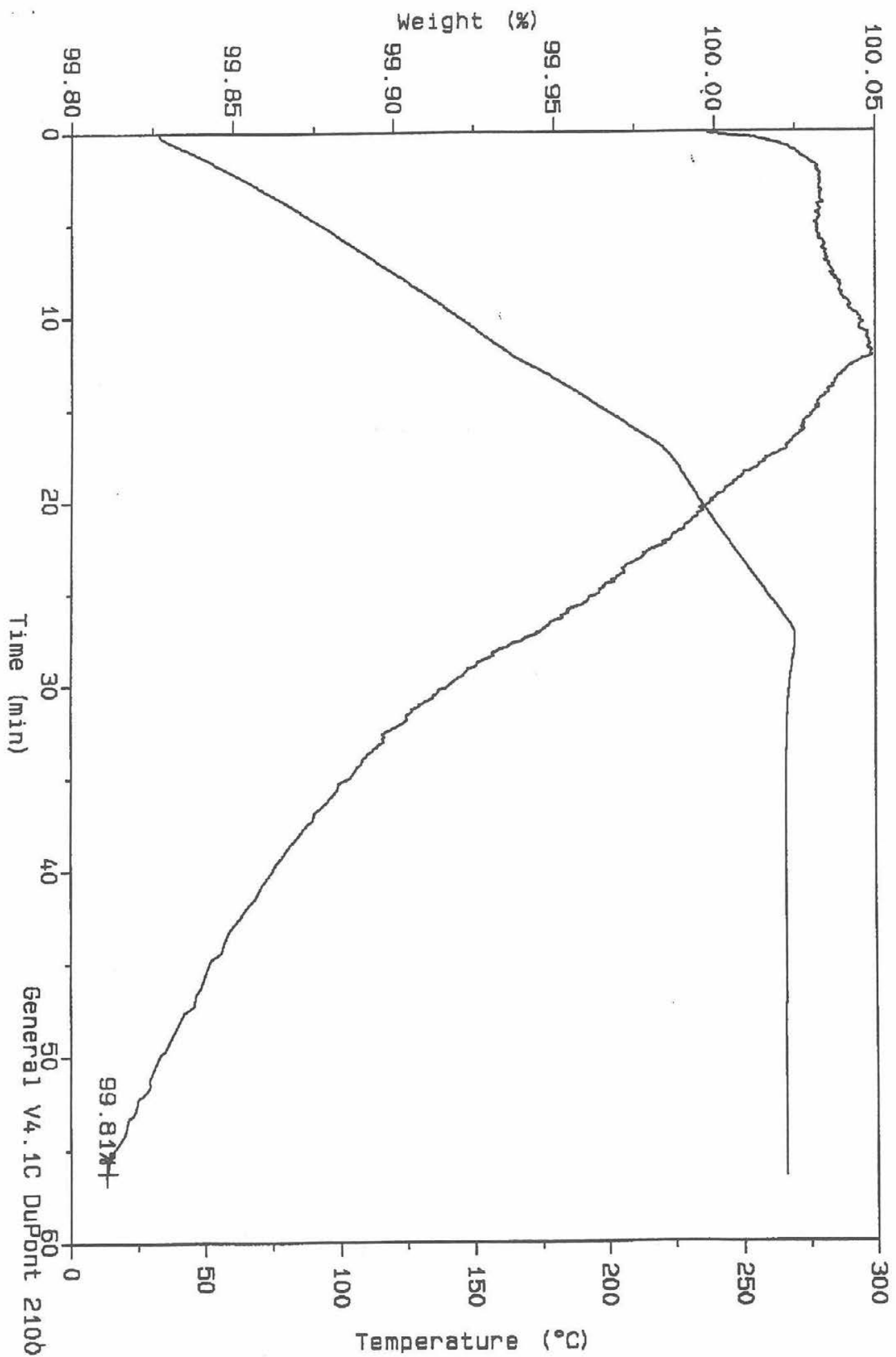
File: A:93-564.02
Operator: VD
Run Date: 10-Mar-93 13:41



Sample: 93-564 HBW555A R24844
Size: 67.2980 mg
Method: TGA
Comment: IN N2 RAMP 10C/MIN TO 200C THEN 5C/MIN TO 250C

TGA

File: A:93-564.03
Operator: VD
Run Date: 10-Mar-93 15:33



DATE: December 4, 1992
TO: N. Conley
FROM: K. Sonnenberg, T. Tikuisis
RE: GC-MS Analysis of Recycled PDI Drum Samples
CC: D. Parks, M. Olynyk, L. Hamielec

Two samples of a recycled drum which contained mineral spirits, one washed and one unwashed, have been analyzed by thermal-desorption cold-trap GC-MS to determine the level of residual spirits. The samples were cut into small pieces, transferred into a glass tube, and heated to 140°C. Desorbed volatiles were purged from the samples with helium gas, trapped on a capillary cold trap at -170°C, and injected onto the analytical column for subsequent separation and identification. Analytical conditions are given in Table 1; the total ion chromatograms of the samples are shown in Figures 1 and 2.

Figure 1 shows the chromatogram of the unwashed sample. Two groups of components are present, one centered around 28 minutes and the other around 49 minutes. The first comprises a large number of nine-to-twelve-carbon olefins and paraffins, most of which are unresolved, resulting in a large "envelope" in the chromatogram. Also present are ten-carbon benzene derivatives such as tetramethylbenzene. The second group is made up of phenols with nine-carbon alkyl side chains. The first group most likely represents residual mineral spirits, the large hump being characteristic of such a hydrocarbon fraction.

The chromatogram of the washed sample is shown in Figure 2. A similar pattern of components is observed in this sample. Based on relative peak areas, the levels of the phenol derivatives are similar to those in the unwashed sample, while those of the C9-C12 hydrocarbons appear to be lower. This result is to be expected, assuming that the phenol derivatives arise from the polymer and the hydrocarbons represent residual mineral spirits.

A sample of the high-density resin (HB-W555-A) presumed to be similar to the type of resin from which the drums were made, was analyzed under identical conditions. The resulting chromatogram is shown in Figure 3. A regular series of even-carbon-number olefins from C10 to C24 is present; also observed is a group of C9-phenol derivatives eluting between 47 and 50 minutes. The presence of the C9-phenol derivatives in this

sample validates the assumption that they arise from the polymer itself. This implies that the PDI drum resin contains an additive package similar to that found in HB-W555-A. The absence of the hydrocarbon envelope centered around 28 minutes supports the inference that those hydrocarbons represent mineral spirits.

The level of mineral spirits in the drum samples was determined as follows. A commercial sample of mineral spirits (Stoddard's reagent) was used to prepare calibration standards. The representative base resin (HB-W555-A) was spiked with aliquots of Stoddard's reagent diluted to varying concentrations in carbon disulphide, and analyzed as before.

Figure 4 shows the chromatogram obtained from the base resin spiked with 72 ug of Stoddard's reagent. The pattern of components is similar to that shown in Figures 1 and 2, validating the use of Stoddard's reagent. (The large peak at nine minutes represents the carbon disulphide solvent.) Three desorptions were required to achieve quantitative recovery of the added reagent; less than 2% of the total Stoddard's reagent recovered was obtained from the third desorption. A similar result was obtained from the base resin spiked with 15 ug of Stoddard's reagent (chromatogram not shown).

The total areas from the standard runs (corrected for co-eluting components from the base resin) were used to construct a calibration curve of area versus micrograms of Stoddard's reagent.

Repeat desorptions were also carried out on the drum samples to obtain quantitative recovery of the absorbed mineral spirits. Figures 5 to 8 show four successive desorptions of the unwashed sample; less than 1% of the mineral spirits recovered was obtained from the fourth desorption. The washed sample yielded similar results (chromatograms not shown). The total areas for each sample were used to determine micrograms of Stoddard's reagent (representing mineral spirits) from the calibration curve. The unwashed sample was found to contain approximately 900 ug/g mineral spirits, the washed sample approximately 600 ug/g.

In conclusion, thermal-desorption cold-trap GC-MS analysis was used to determine quantitatively the concentrations of mineral spirits adsorbed into the polyethylene drums. Washing of the drums appeared to remove approximately 30% of the adsorbed material; more complete cleaning may be required to further reduce carry-over or memory effects in the resin. This technique can also be applied to the analysis of other leachates in molded polyethylene containers.



Tony Tikuisis

TT:sd

TT: 144.mem

TABLE 1

**Chromatographic Conditions for the GC-MS Analysis of
PDI Drum Samples**

Instrument:	HP GC-MSD 5890/5970
Column:	DB-5ms, 30 m length x 0.25 mm i.d., d _f 0.5um
Carrier Gas:	UHP Helium
Column Head Pressure:	140 kPa
Temperature Program:	1) 3 minutes @ -20°C 2) -20°C to 300°C @ 5°C/minute 3) 3 minutes @ 300°C
Injector Temperature:	250°C
Detector Temperature:	250°C
MSD Scan Range:	1) 0-30 minutes: 20-200 amu 2) 30-50 minutes: 30-300 amu 3) 50-70 minutes: 40-450 amu
TCT	
Desorption:	140°C for 10 minutes
Cold Trap	1) -170°C for 10 minutes 2) 270°C for 3 minutes

FIGURE 1: TOTAL ION CHROMATOGRAM OF THE UNWASHED SAMPLE

Sample weight: 169 mg

TIC of 9210-13-3.d

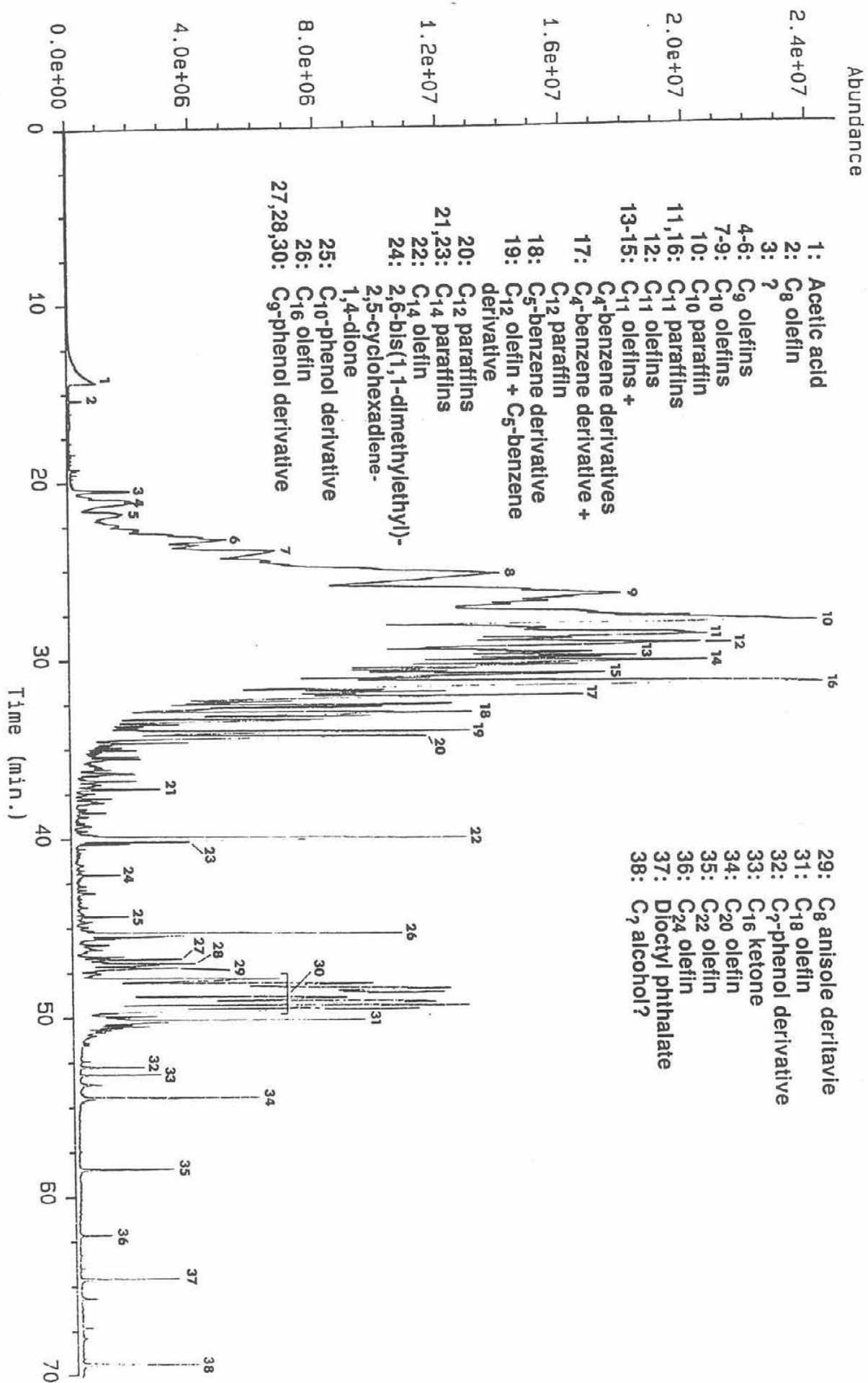


FIGURE 2: TOTAL ION CHROMATOGRAM OF THE WASHED SAMPLE

Sample weight: 163 mg

TIC of 9210-13-2.d

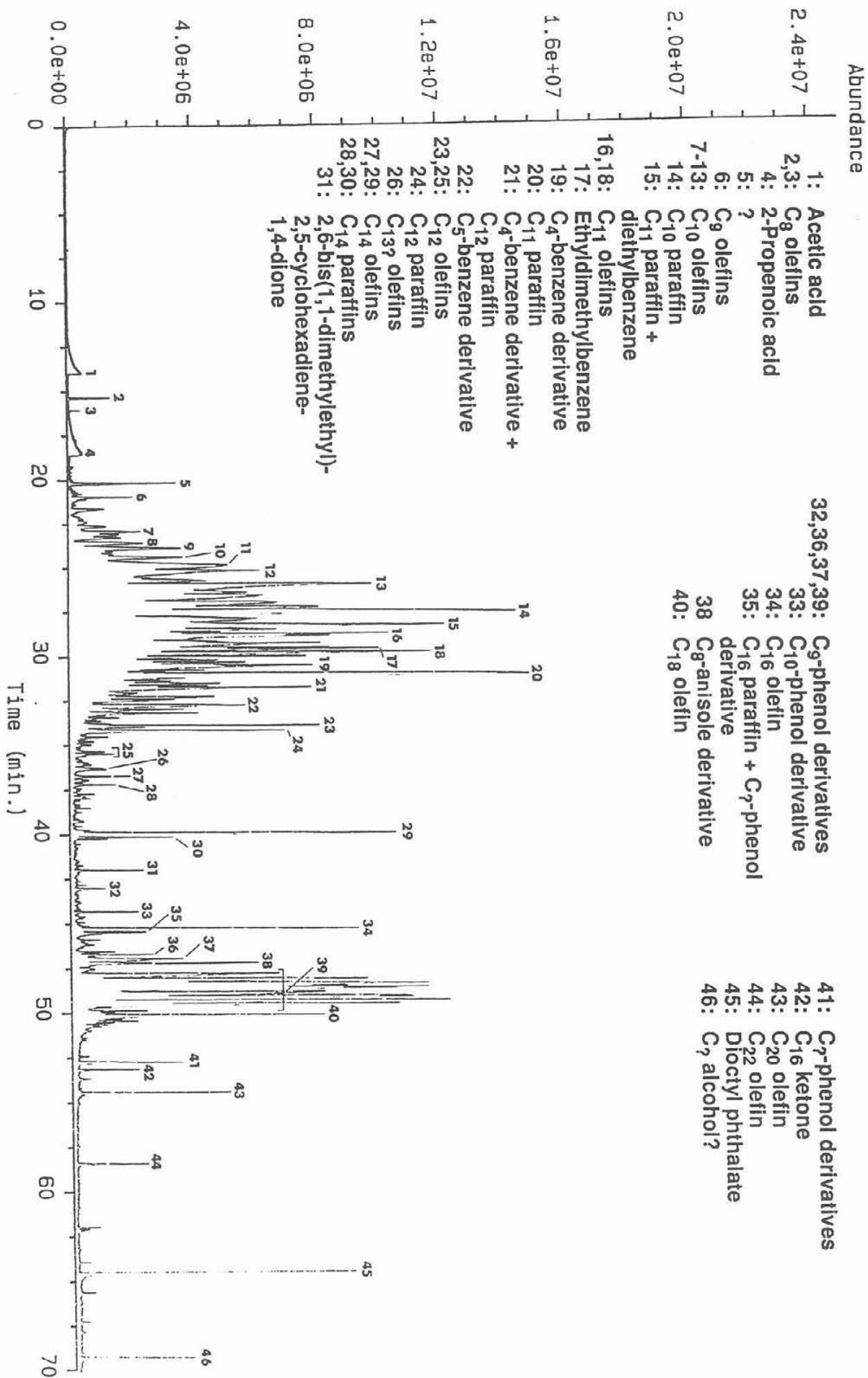


FIGURE 3: TOTAL ION CHROMATOGRAM OF THE HB-W555-A RESIN SAMPLE

Sample weight: 192 mg

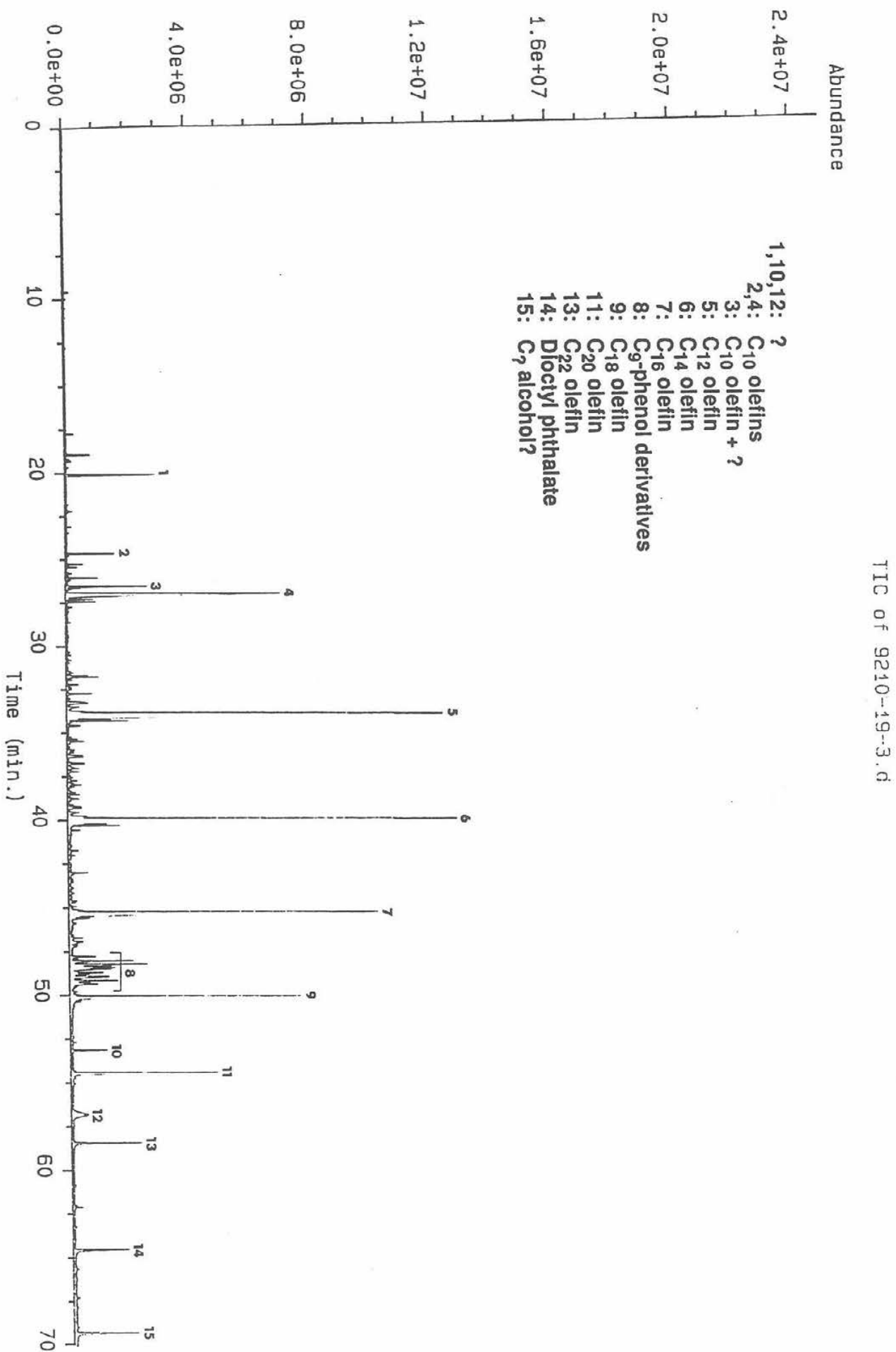


FIGURE 4: TOTAL ION CHROMATOGRAM OF HB-W555-A RESIN SPIKED WITH 72 UG OF STODDARD'S REAGENT (FIRST DESORPTION)

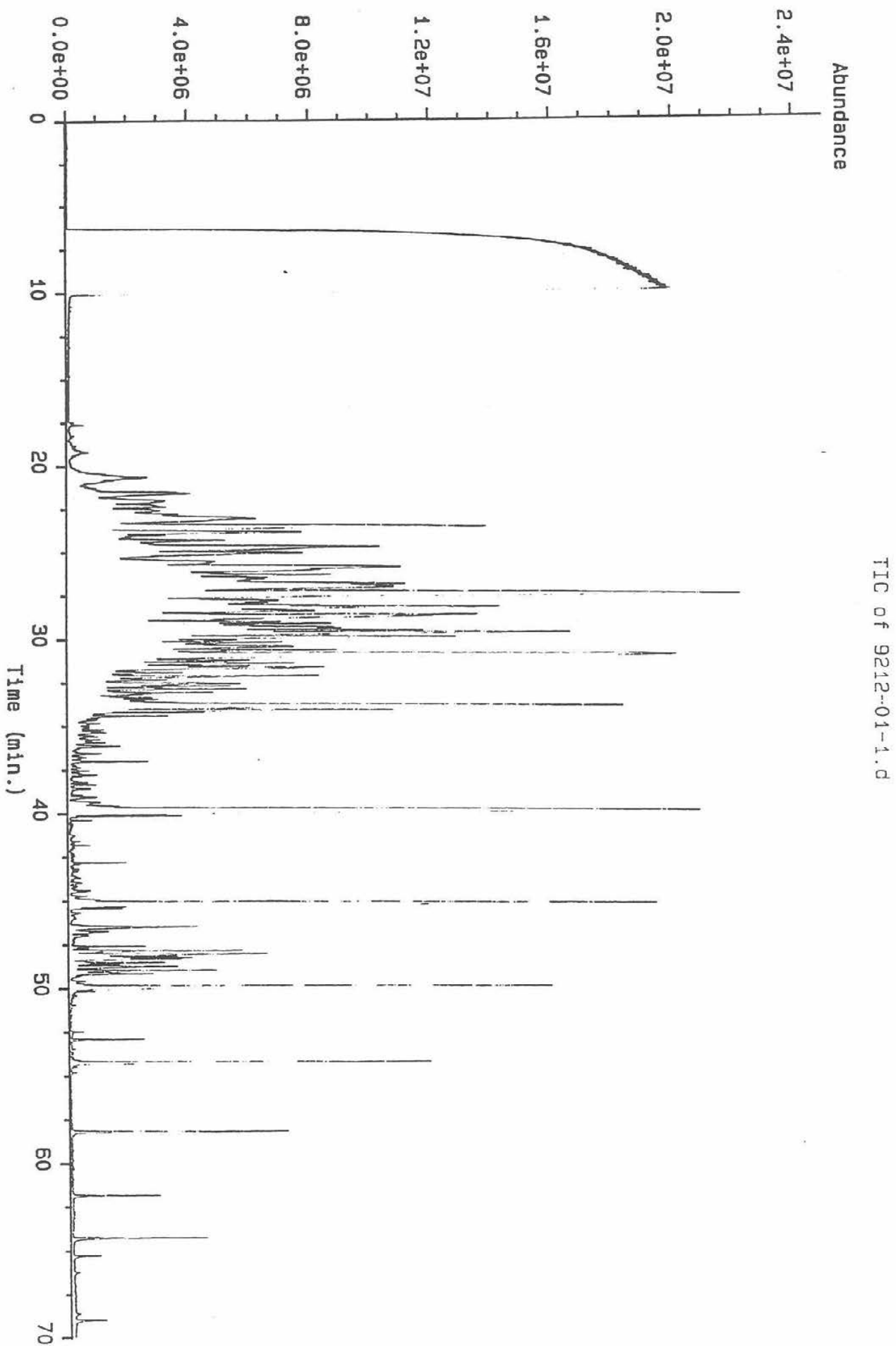


FIGURE 5: TOTAL ION CHROMATOGRAM OF THE UNWASHED SAMPLE (FIRST DESORPTION)

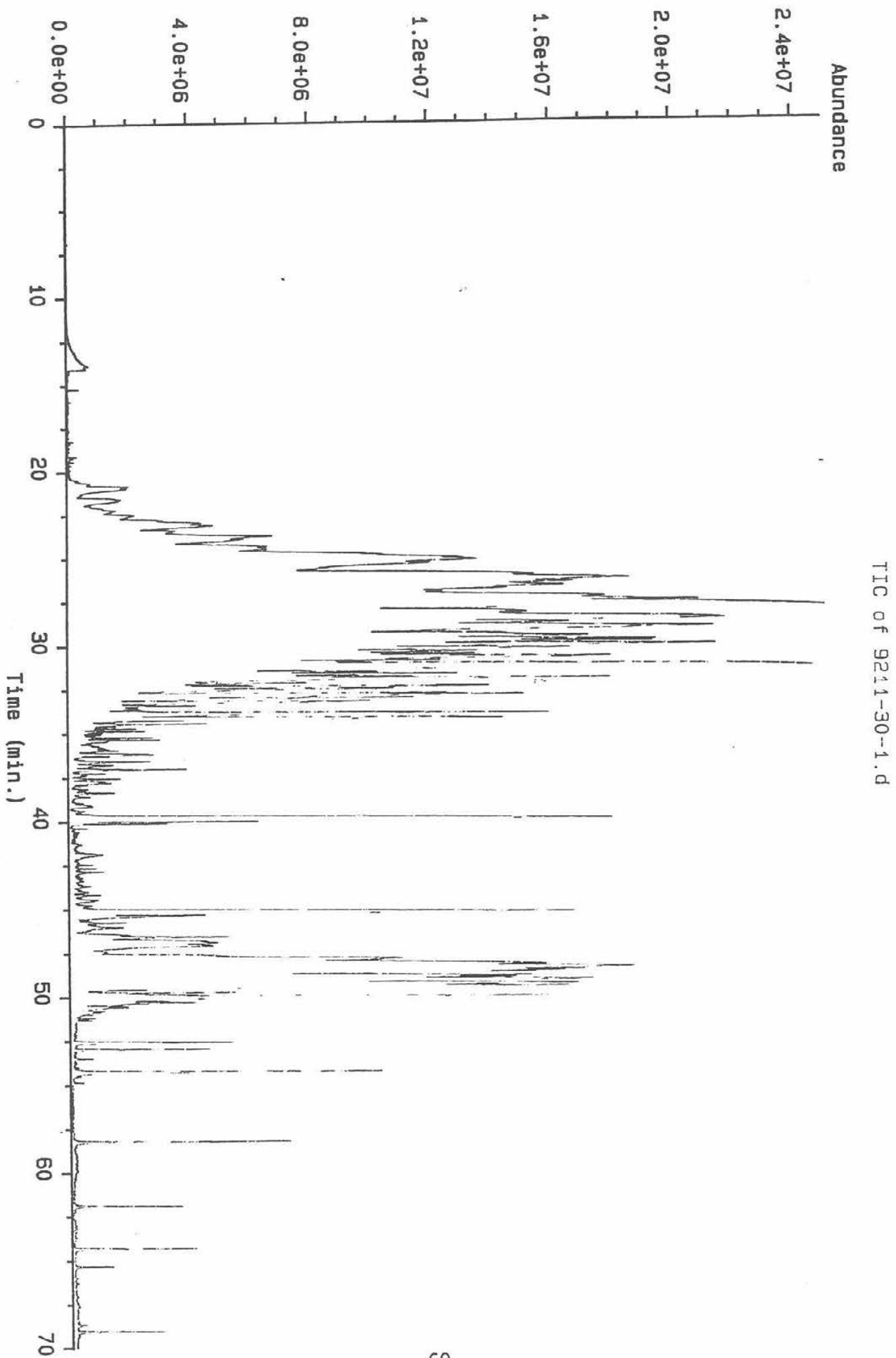


FIGURE 6: TOTAL ION CHROMATOGRAM OF THE UNWASHED SAMPLE (SECOND DESORPTION)

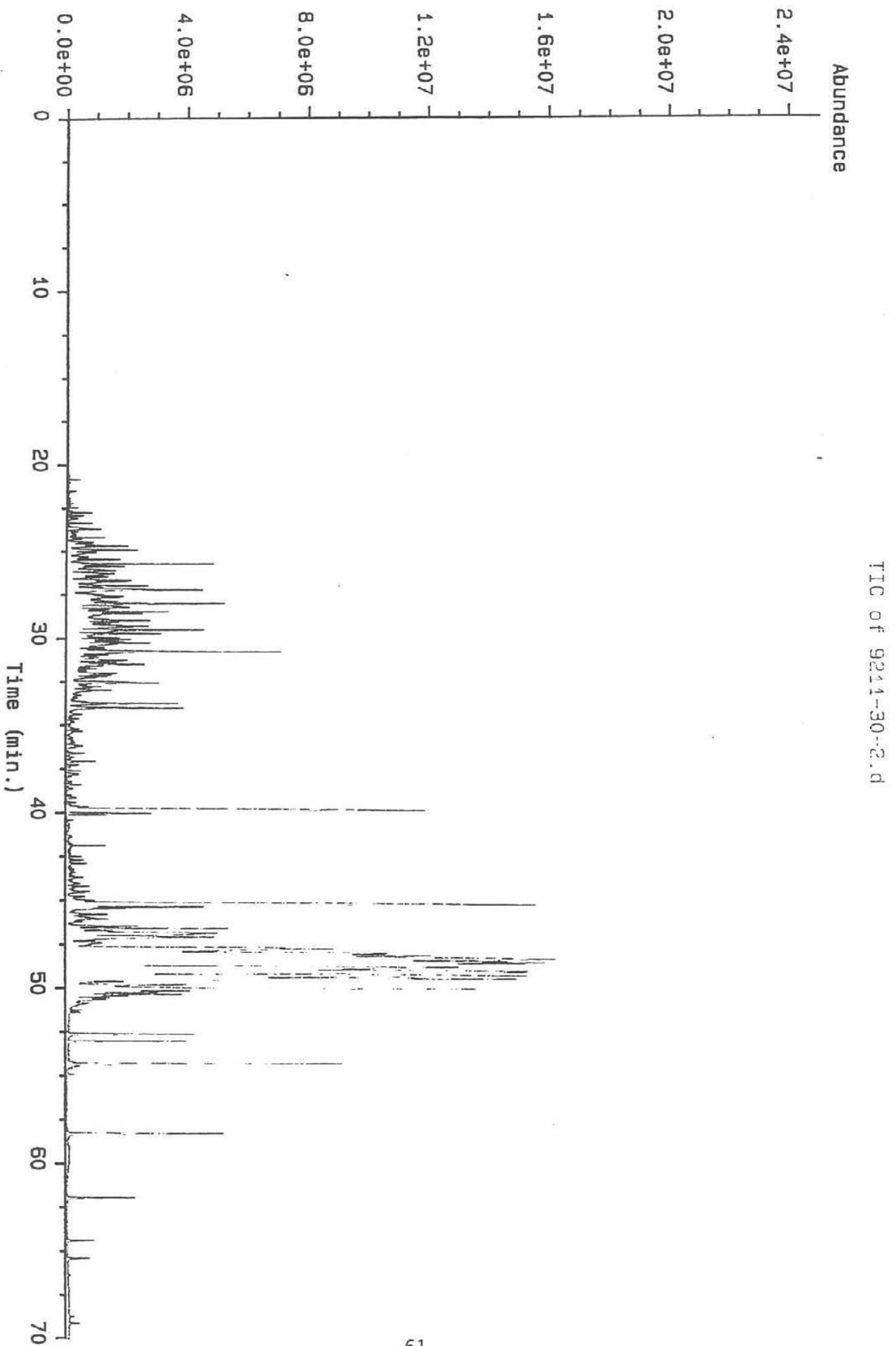
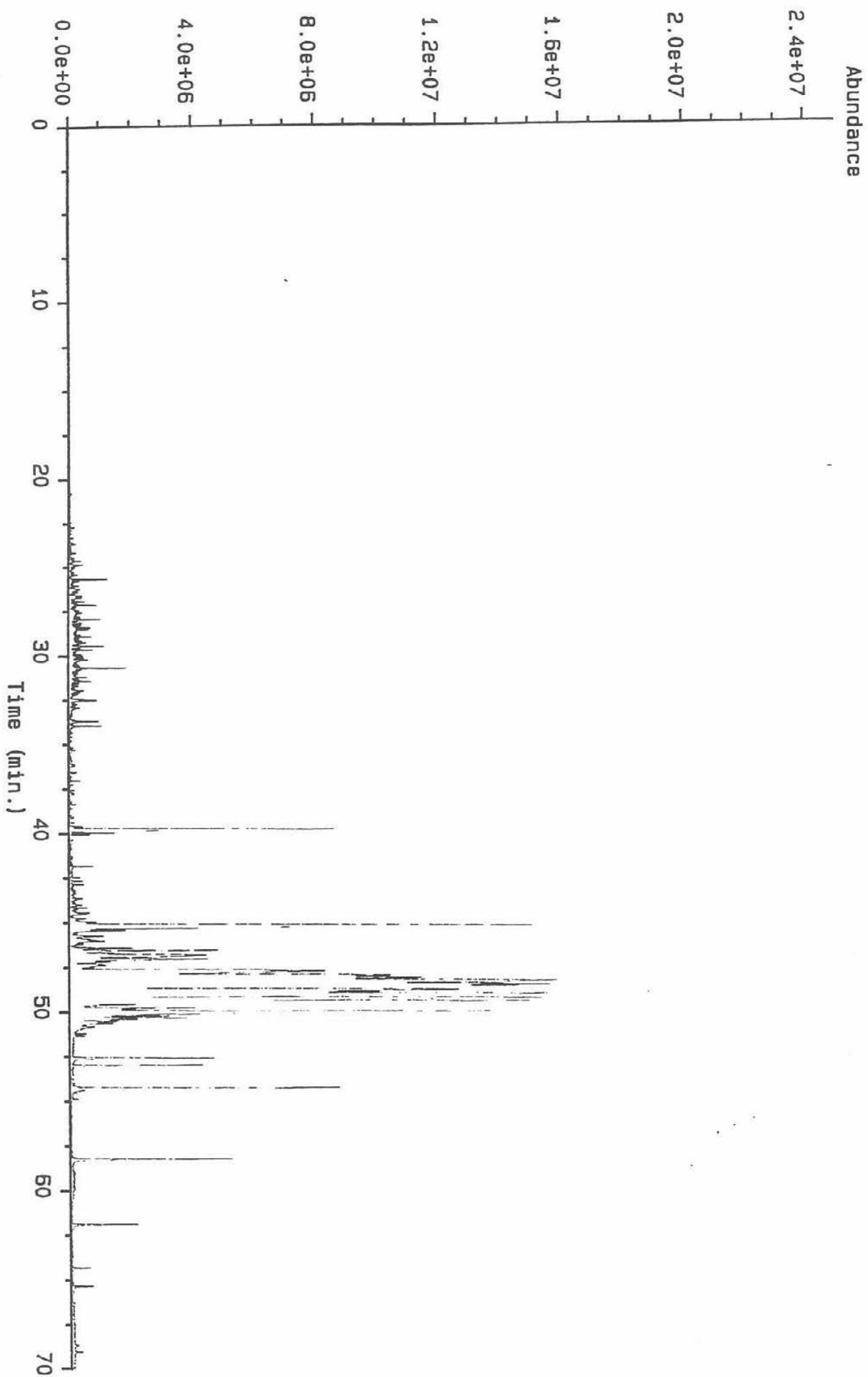


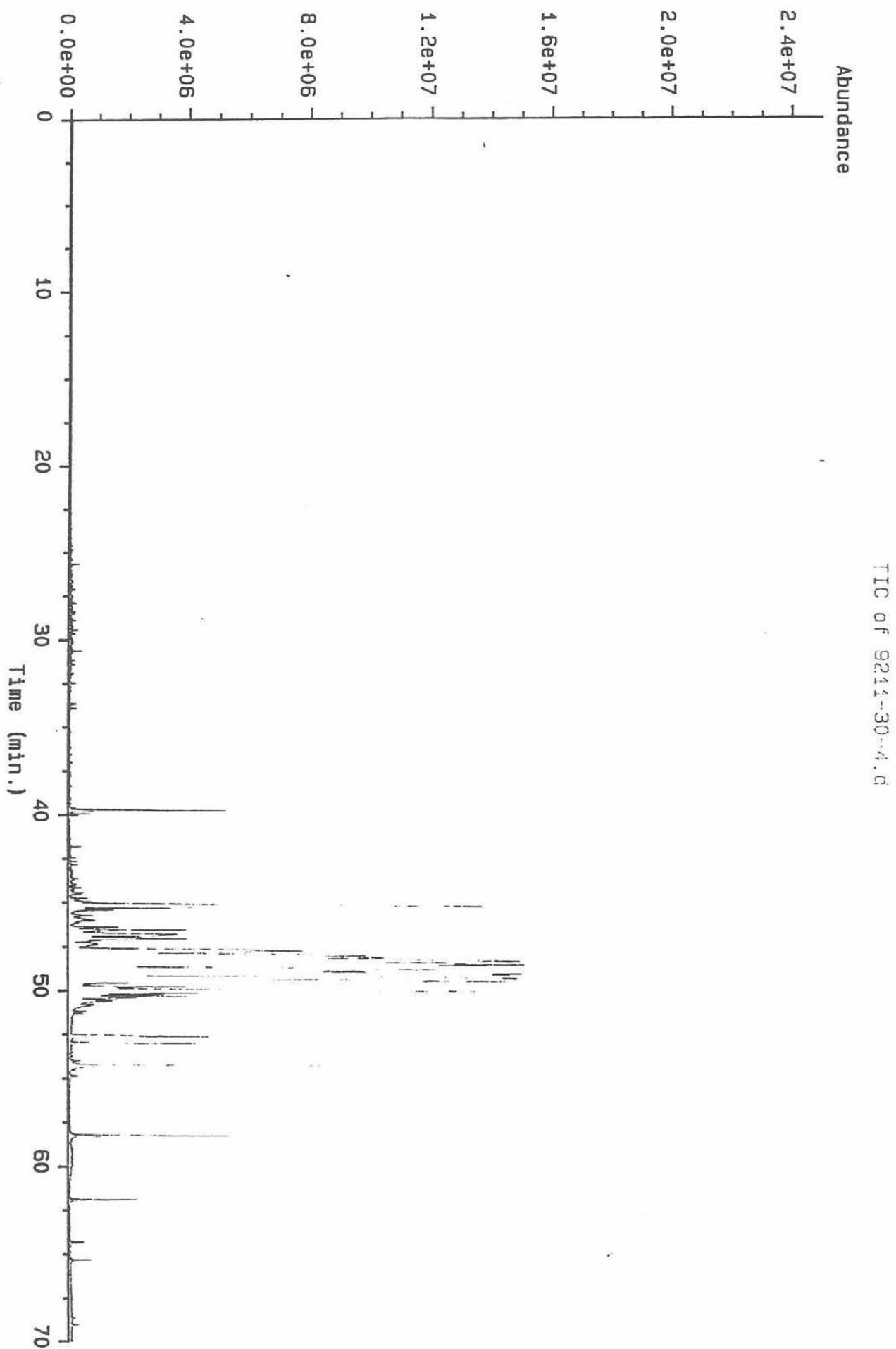
FIGURE 7: TOTAL ION CHROMATOGRAM OF THE UNWASHED SAMPLE (THIRD DESORPTION)

TIC of 9211-30-3.d



TI: 144.mem

FIGURE 8: TOTAL ION CHROMATOGRAM OF THE UNWASHED SAMPLE (FOURTH DESORPTION)





PHILLIPS 66 COMPANY

BARTLESVILLE, OKLAHOMA 74004 918 661-6600

PLASTICS DIVISION
Plastics Technical Center

January 18, 1992

PDI Drum Samples

Mr. John Bergerhouse
Quantum Chemical
11500 Northlake Drive
P. O. Box 429550
Cincinnati, Ohio 45250

Dear John:

Testing has been completed on the four recycle HDPE drum samples sent for analysis of the residual sulfuric acid, HLMI, density and TGA. Attached are the results of the X-ray fluorescence and TGA. The results of the HLMI and density testing are as follows:

Sample #	Form	Color	HLMI	Density
5	Flake	Blue	2.1	0.953
5A	Pellet	Blue	3.6	0.953
6	Flake	Black	10.4	0.956
6A	Pellet	Black	11.0	0.956

Samples have been retained for further work if necessary.

Sincerely,

John Rathman

JRR:dkc

Attach.

NOTEGRAM

September 4, 1992

To: John Rathman
Curtis Tevebaugh, PTC

From: W.W. Tseng
154, CPL, x0335

RE: TGA Analysis for Recycled PE

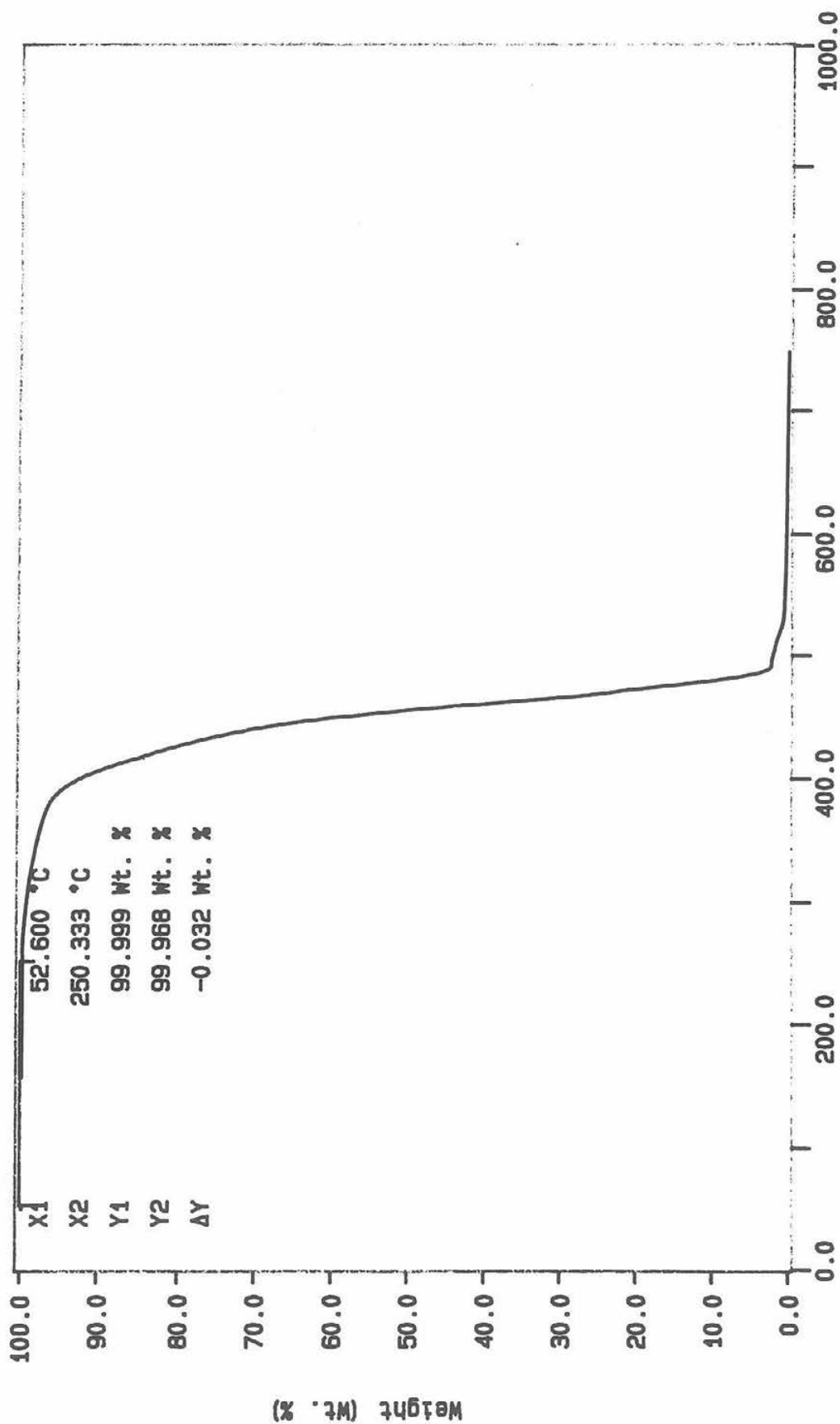
Attached are TGA data on the recycled PE flake and pellet. The experiments were performed using a dry nitrogen purge at a heating rate of 20 C/min for a temperature range of 50 to 900 C. The temperature was calibrated using an Alumel and a Nicoseal metal standard, according to the instructions in the TGA operating manual provided by the manufacturer.

I leave interpretation of the results⁵ to you, since you know what you want from this. However, I will be glad to answer any questions you might have.

cc: J. Janzen

92note8.doc

Curve 1: TGA
 File Info: 1023 Mon Aug 24 09:24:29 1992
 Sample Weight: 17.389 mg
 PE #5 Blue Flake (PTC)



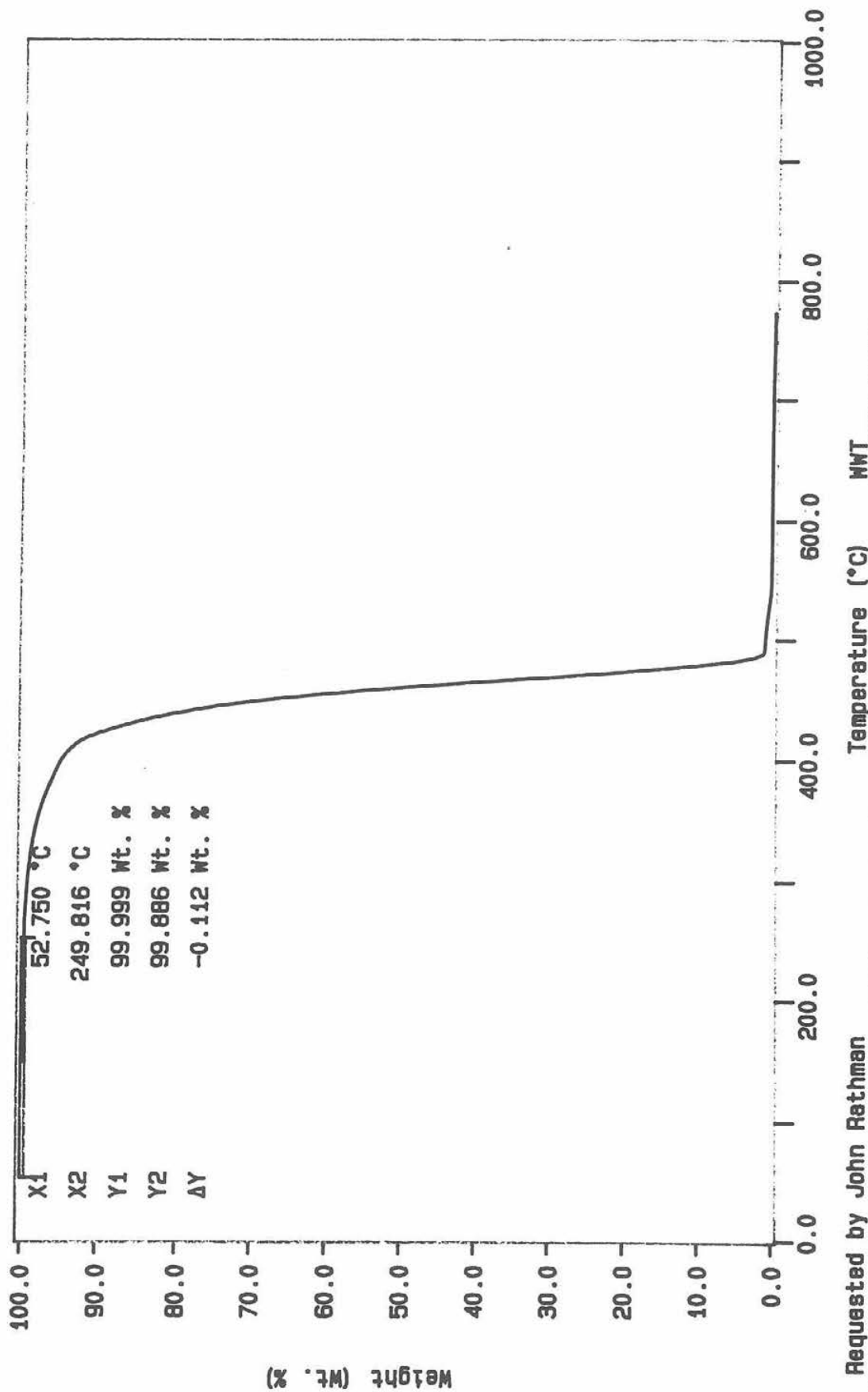
Requested by John Rathman
 TEMP: 30.8 °C TIME: 0.0 min RATE: 20.0 °C/min
 WWT PERKIN-ELMER
 7 Series Thermal Analysis System
 Thu Feb 25 07:47:46 1993

Curve 1: TGA

File info: 1024 Mon Aug 24 10:38:34 1992

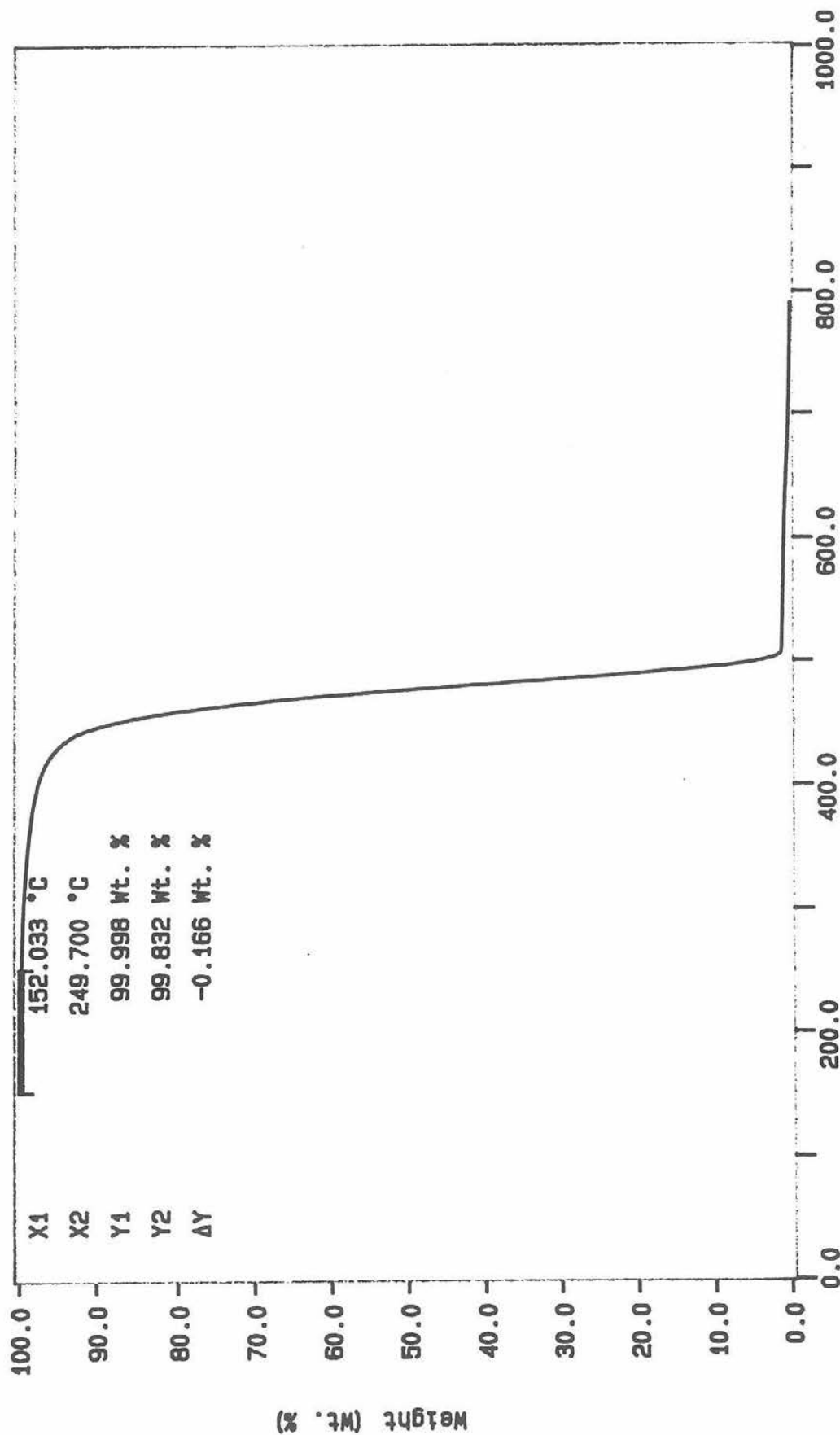
Sample Weight: 9.047 mg

PE #5A Blue Pellet (PTC)



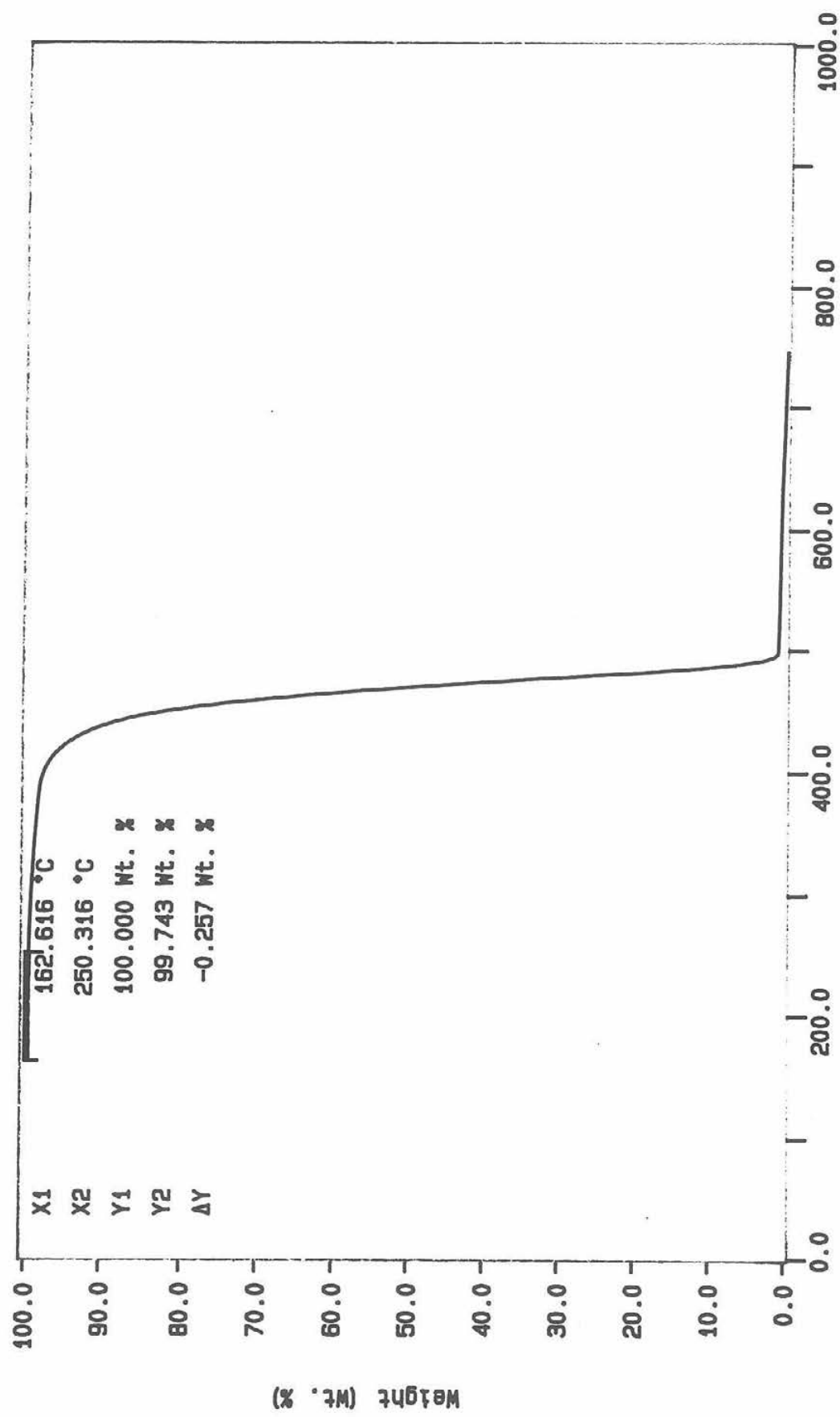
Requested by John Rathman
TEMP: 50.0 °C
TIME: 00.0 min
RATE: 20.0 °C/min
WWT
PERKIN-ELMER
7 Series Thermal Analysis System
Thu Feb 25 08:01:44 1993

Curve 1: TGA
 File info: 1025 Mon Aug 24 12:02:56 1992
 Sample Weight: 16.403 mg
 PE #6 Black Flake (PTC)



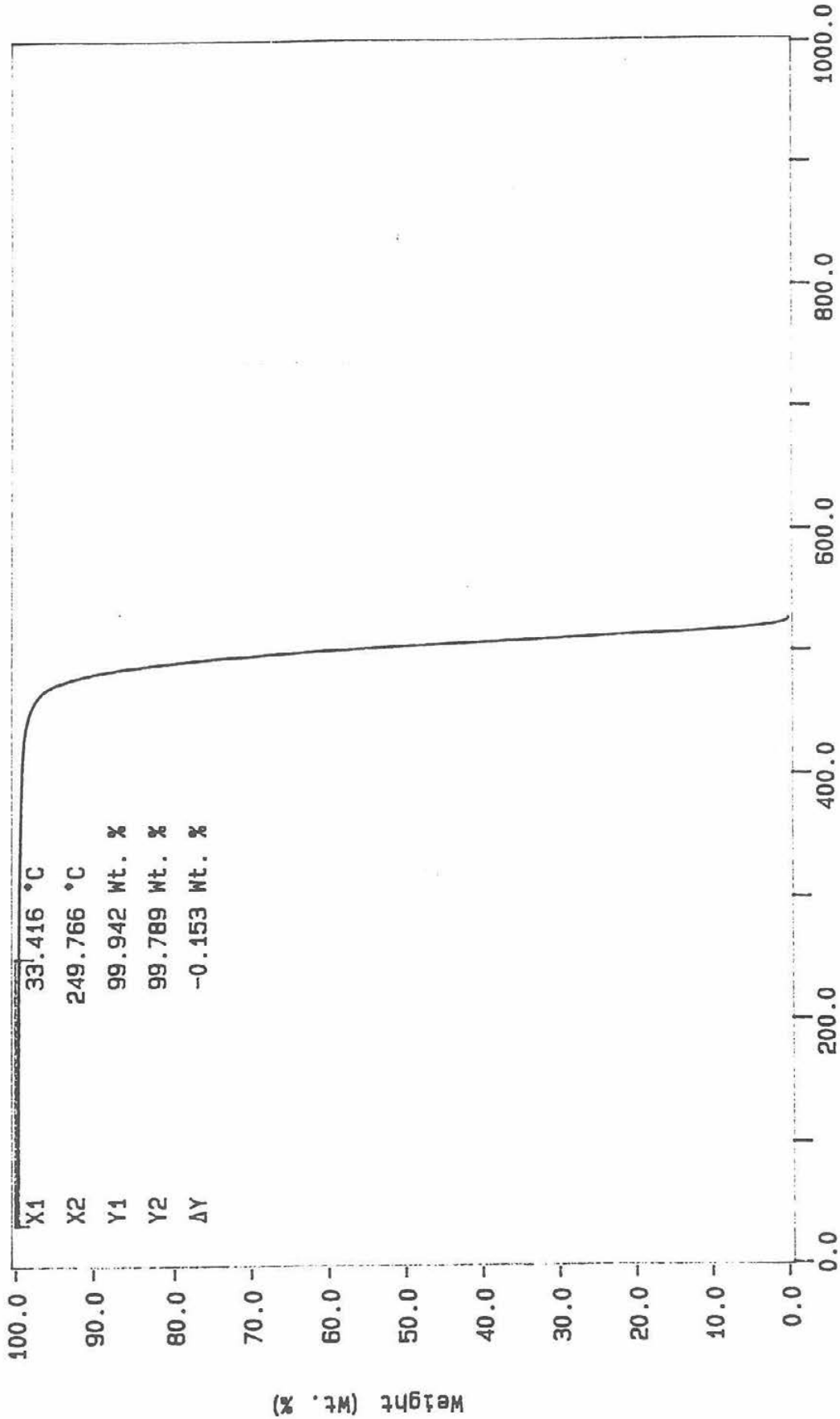
Requested by John Rathman
 TEMP: 50.0 °C TIME: 0.0 min RATE: 20.0 °C/min
 MWT PERKIN-ELMER
 7 Series Thermal Analysis System
 Thu Feb 25 08:00:35 1993

Curve 1: TGA
 File info: 1026 Mon Aug 24 13:53:55 1992
 Sample Weight: 7.263 mg
 PE #6A Black Pellet (PTC)



Requested by John Rathman
 TEMP: 80.0 °C TIME: 0.0 min RATE: 20.0 °C/min
 WWT PERKIN-ELMER
 7 Series Thermal Analysis System
 Thu Feb 25 08:00:09 1993

Curve 1: TGA
 File Info: 08-27 Tue Mar 16 11:56:00 1993
 Sample Weight: 9.370 mg
 TR570 lot 61-2-1204



for John Rathman
 TEMP1: 50.0 °C TIME1: 0.0 min RATE1: 20.0 °C/min
 TEMP2: 900.0 °C
 JJ PERKIN-ELMER
 7 Series Thermal Analysis System
 Tue Mar 16 11:56:36 1993



Notegram

September 4, 1992

To: John Rathman
PTC

From: Kathy Swallows *KSwallows*
Analytical Services
225A PL, 1-9523

Subject: *Residual Sulfuric Acid in Drums Used in the Drum Recycle Study*

In order to determine the residual sulfate content of washed and unwashed high density polyethylene samples, the sulfur content of the flaked and pelletized samples was determined by X-ray fluorescence and a sulfate value calculated assuming all the sulfur was in the form of sulfate. The four polymer samples were also extracted with two different solvent systems and the extract analyzed for sulfate by ion chromatography (Phillips Method Number 8909-AR). X-ray fluorescence yielded the highest values (Table 1), the results obtained from the extracts are significantly lower. The observed results appear to indicate that washing the polymer does lower the sulfate content, however, these results may be biased due differences in the exposed surface area and homogeneity of the pellets versus the flaked material. In addition, the possible presence of sulfate as a contaminate in the polymer additives should not be ignored. Such contamination would yield inflated experimental sulfate values. Any conclusions based on these results should be used with caution. Rerunning the X-ray and TCLP leach experiments on ground flake and pellets is recommended in order to reduce the biases due to the differences in homogeneity and exposed surface area. A polymer sample from a virgin container would be needed in order to account for any sulfate originating from the additives.

SAMPLE DESCRIPTION	ANALYSIS BRANCH NUMBER	X-RAY FLUORESCENCE	TCLP LEACHATE	METHANOL/WATER EXTRACT
Blue flake unwashed	9215657	390 ppm SO ₄	170 ppm SO ₄	20 ppm SO ₄
Blue pellet washed	9215658	195 ppm SO ₄	159 ppm SO ₄	13 ppm SO ₄
Black flake unwashed	9215659	1000 ppm SO ₄	203 ppm SO ₄	44 ppm SO ₄
Black pellet washed	9215660	465 ppm SO ₄	119 ppm SO ₄	11 ppm SO ₄

The results from X-ray fluorescence were obtained by direct comparison to sulfur in mineral oil standards as outlined in Phillips Method 7105-AM. The sulfate values reported in Table I were calculated from the sulfur values assuming that all the sulfur is present as sulfate. Bob Morton has

indicated that the sulfur values may be high due certain physical effects inherent in the measurement of sulfur. In spite of this, the X-ray results do provide a reasonable estimate of the sulfate content which is useful when evaluating the effectiveness of the extraction procedures. X-ray also noted significant levels of titanium, iron, copper, and zinc in the blue flake and pellet samples. Calcium, titanium, and iron were present in the black flake and pellet. Substances containing these metals are often added as fillers and sulfate is a common contaminant. If sulfate was present as a contaminant, any results obtained from the polymers (including the results from the extracts) may be biased. In addition, both the black and blue flake samples exhibited inhomogeneity (6 replicate measurements were used to calculate an average sulfur content). Both pellet samples appeared to be relatively homogenous. (The original x-ray results are attached).

Two different extraction procedures were evaluated. A modified form of the EPA's Toxicity Characteristic Leaching Procedure (TCLP) and a methanol/water extraction at an elevated temperature were utilized. Approximately 100 grams of the flakes or pellets (no sample preparation was performed) was extracted into 2000 mL of an acetic acid/sodium hydroxide solvent or 300 mL of a methanol/water mixture. Both types of extracts were then analyzed for sulfate content by ion chromatography. More detailed outlines of both procedures are attached. As shown in Table I, the TCLP procedure yielded lower results than reported by X-ray fluorescence, but higher than those obtained from the methanol/water leach. Several factors may have affected the extraction results. The flaked material has surfaces which were exposed directly to the sulfuric acid and which are in direct contact with the solvent. Once the sample is pelletized, any residual sulfuric acid is evenly distributed throughout the pellet and may not be extracted unless the extraction solvent totally permeates the polymer. The permeability of the polymer to the TCLP solvent is unknown, thus the observed decrease in the residual sulfate in the pellets may be related to how much of the originally exposed polymer is in contact with the solvent and not due to losses from the washing procedure. It is also possible that part or all of the sulfate measured in the extracts is due to contamination introduced by the additives.

Several other extraction schemes were considered, but were not implemented due to time constraints. More experiments would be needed to determine the optimum extraction media and conditions. The pelletization of the polymer and the possibility of sulfate contamination from the additives introduces other variables. Grinding the samples before analysis should reduce any biases in the extraction and X-ray results due to the differences in the origin of the exposed surfaces. Samples obtained from a container which was not used for sulfuric acid could be used to account for any sulfate due to additive contamination. If you are interested in pursuing any of these experiments, please contact me (918-661-9523) or John Paxson (918-661-4292).

I will be happy to answer any questions concerning these results or the attached procedures.

References: AB# 9215657-9215660
Notebook 36103-47, 34440-94, 36467-30

cc: J. R. Paxson
C. M. Fu
T. H. Backhouse
R. W. Morton

TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP)

Determination of Appropriate Extraction Fluid

1. Weigh 5g of sample into 500 ml beaker or flask (samples were weighed in submitted from: pellets or flakes).
2. Add 96.5 mL of water to the beaker, cover with a watchglass, and stir with a magnetic stirrer.
3. Measure and record the pH.
4. If pH is less than 5, use extraction fluid #1
5. If the pH is greater than 5, add 3.5 mL of 1.0N hydrochloric acid, cover with a watchglass, heat to 50°C for 10 minutes, let cool to room temperature.
6. If the pH is now less than 5, use extraction fluid #1.

pH of samples was less than 5, so extraction fluid #1 was used.

Extraction Fluid #1: 5.7 mL glacial acetic acid to 500 mL water + 64.3 mL 1.0N sodium hydroxide brought to 1L. The resultant pH will be 4.93 ± 0.05 .

Procedure for Leaching

1. Quantitatively transfer 100 g of sample into the extractor vessel. Samples were extracted as submitted: in pellet or flake form.
2. Slowly add Extraction Fluid #1 in the amount of 20X the sample's weight (2000 mL was added).
3. Close extractor bottle tightly, secure in rotary extractor device and rotate at 30 ± 2 rpm for 16 to 20 hours.
4. Contents of the extractor vessel were filtered and the filtrate submitted for ion chromatographic determination of the sulfate content.

NOTE: The final step in the EPA protocol is for acidification to a pH of <2 with nitric acid. Excessive amounts of nitric acid would have interfered with the ion chromatographic determination and thus this step was omitted.

WATER/METHANOL EXTRACTION

Extraction Solvent: Add 500 mL of methanol to a 2L volumetric flask and dilute to the mark with water.

Procedure

1. Weight out 100g of polymer (samples analyzed as is: flake or pellet form).
2. Add 300 mL of extraction solvent.
3. Extract overnight (24 hours) at 55-60°C
4. Decant extract into 500 mL volumetric flask. Wash polymer residue 2-3 times with 50 mL portions of distilled, deionized water. Add the rinses to the volumetric flask. Dilute to the mark with water.
5. Extract samples submitted for determination of sulfate by ion chromatography.

 *
 * MAIL TO: K A Swallows *
 * 225A PL *
 *
 *

[** FORWARD TO: John R. Rathman PTC **]

ANALYSIS BRANCH RESULT FORM

ATTACHED ARE THE RESULTS AND/OR ORIGINAL DATA FOR SAMPLE(S)
 SUBMITTED TO THE ANALYSIS BRANCH. REMINDER: NO ORIGINAL DATA ARE
 KEPT BY THE ANALYSIS BRANCH.

ANALYST: L C Patterson

DATE COMPLETED: 8/26/92

SAMPLE NUMBER /DESCRIPTION DATE IN AB NUMBER TEST DESCRIPTION TO NEAREST 5 ppm S
 /METHOD

BLUE FLAKE UNWASHED 8/14/92 9215657 SULFUR +
 /5 BLUE FLAKE UNWASHED /X-RAY

130 ppm S

COMMENT FOR S BY X-RAY ON 9215657: DIRECT COMPARISON TO S IN MINERAL OIL S
 DS.. X-RAY FLUPRESCENCE. APPROX. ~~WT. %~~ ^{ppm} SULFUR ONLY!!!!!!! LCP

A BLUE PELLET WASHED 8/14/92 9215658 SULFUR +
 /5A BLUE PELLET WASHED /X-RAY

65

COMMENT FOR S BY X-RAY ON 9215658: DIRECT COMPARISON TO S IN MINERAL OIL S
 DS.. X-RAY FLUPRESCENCE. APPROX. ~~WT. %~~ ^{ppm} SULFUR ONLY!!!!!!! LCP

BLACK FLAKE UNWASHED 8/14/92 9215659 SULFUR +
 /6 BLACK FLAKE UNWASHED /X-RAY

335

COMMENT FOR S BY X-RAY ON 9215659: DIRECT COMPARISON TO S IN MINERAL OIL S
 DS.. X-RAY FLUPRESCENCE. APPROX. ~~WT. %~~ ^{ppm} SULFUR ONLY!!!!!!! LCP

A BLACK PELLET WASHED 8/14/92 9215660 SULFUR +
 /6A BLACK PELLET WASHED /X-RAY

155

COMMENT FOR S BY X-RAY ON 9215660: DIRECT COMPARISON TO S IN MINERAL OIL S
 DS.. X-RAY FLUPRESCENCE. APPROX. ~~WT. %~~ ^{ppm} SULFUR ONLY!!!!!!! LCP

INDICATES PRIORITY REQUEST FOR TESTS.

*** END OF REPORT ***

SOL-FOR 4

P.P.F.I.L.M

MOLDED SPEC. 3 EACH.
WATERS

AVG.
mm S

RMAC
Correction

CORRECTED
AVG mm S

REPORTED
AS
TO NEAREST 5 mm

STOE "A"

130.22

1.0170

132.43

130 mm S

62.32

1.0173

63.40

65 mm S

STOE "B"

STOE "A"

336.2

1.0024

337.01

335 mm.

156.73

1.0021

157.06

155 mm S

STOE "B"



Notegram

November 23, 1992

To: John Rathman
PTC

From: Kathy Swallows *JS*
Analytical Services
234B PL, 1-3418

Subject: *Residual Sulfuric Acid in Drums Used in the Drum Recycle Study*

In response to several questions which arose during the initial attempts to determine the residual sulfate content in high density polyethylene samples (see notegram dated 09/04/92), a new series of experiments was conducted. In the original study, the samples were analyzed as is (pellet or flake form) by X-ray fluorescence and acetic acid extracts of the flaked or pelletized polymer samples were analyzed by ion chromatography (IC). This time all samples were ground using a 1 um screen before analysis to avoid any biases due to the difference in the exposed surface area of the pellets versus the flakes. The IC work is not yet completed, however, the X-ray fluorescence results are summarized below. The results obtained from the ground polymer samples indicate that there is no difference in the sulfate content of a sample steamed washed once versus a sample steamed washed twice.

SAMPLE DESCRIPTION	ANALYSIS BRANCH NUMBER	X-RAY RESULTS (pellets/flakes)	X-RAY RESULTS (ground)	95% CONFIDENCE LIMITS (ground)
Blue Flake unwashed	9215657	390 ppm	157 ppm	± 12 ppm
Blue Pellet washed	9215658	195 ppm	168 ppm	± 2 ppm
Black Flake unwashed	9215659	1000 ppm	473 ppm	± 62 ppm
Black Pellet washed	9215660	465 ppm	461 ppm	± 11 ppm

The x-ray results obtained from the ground pelletized samples compare well with those reported for the x-ray analysis of the pellets themselves. This is expected since the processing of polymer into pellets tends to homogenize the samples. The biases due to sample inhomogeneity are significant, as the results from the ground flaked versus flaked material indicate.

Results obtained from the IC analysis of the acetic acid extracts on the ground material are inconclusive. However, a review of the original data discussed in the first letter has yielded some new insights. The original IC analysis indicated significant differences between the washing

process for only the black flake and pellet. In the time since the report on the original IC work, we have found problems in determining trace amounts of anions in several different matrices. Unfortunately, the levels of sulfate we observed in these samples fall in this region, leading us to question our original results. We are conducting additional experiments in order to clarify the issue. The results from these experiments and any necessary corrections will be forwarded to you as soon as they become available.

Grinding the samples eliminated the biases due to exposed surface area and inhomogeneity of the samples. However, the source of the measured sulfate is still suspect. Only the analysis of material from a virgin container can tell us whether the sulfate present is due to residual sulfuric acid, contaminants in the additive packages, or a combination of both. However, the X-ray fluorescence results clearly indicate that there is no difference between the sulfate content in samples cleaned by different procedures.

I will be happy to answer any questions concerning these results.

References: AB#9215657-9215660
Notebook 36467, 34440, 36103

cc: J. R. Paxson
C. M. Fu
R. W. Morton

APPENDIX B

Part II - Microtox® Testing

Analytical Procedures and Results -

INTEROFFICE CORRESPONDENCE
ENVIRONMENTAL HEALTH AND SAFETY DEPARTMENT
ENVIRONMENTAL AND HEALTH SCIENCES LABORATORY

August 26, 1992

TO: K.T. Ford
MCC, Edison

CC: Kumar T.
C.R. Mackerer
A.E. Melnarian
P.A. Naro *PN*
N.J. Novick

FROM: M.T. BenKinney

RE: Microtox Results for Plastic Drum Resins

As you requested, we have evaluated the toxicity of various plastic drum resins before and following washing and repelleting. Summarized below are the results of this program. The resins from drums which held acrylic acid and sulfuric acid remained toxic throughout the cleaning process, although the sulfuric acid resins were less toxic than the acrylic acid resins. The resins from drums which held mineral spirits became less toxic during the cleaning process and showed no toxicity in the pelleted form. The resins from drums which held acetic acid remained essentially non toxic throughout the cleaning process.

Sample Preparation

All samples were evaluated in the same manner. Twenty grams of resin were added to 250 ml deionized water in a round bottom flask. The contents of each flask were then refluxed for 24 hours. A water blank was run with each set of samples as a control for the procedure. After cooling, each sample was evaluated in the Microtox assay, using a standard 2:1 dilution approach to prepare the test concentrations. The 5 and 15 minute EC50 values obtained for each sample are summarized in Attachment 1.

Summary

Based on the preliminary Microtox results, the prior contents of the drum and the stage of the cleaning process both influenced the results obtained. Resins contaminated with acrylic acid and sulfuric acid remained comparable in toxicity throughout the cleaning process, while resins contaminated with mineral spirits and acetic acid were nontoxic with Microtox in pelleted form.

Please let me know if you have questions or need additional information.


Marie T. BenKinney

Attachment 1

Summary of Microtox Data for Plastic Drum Resins

Sample No.	Label	Color	Microtox EC50	
			5 min.	15 min.
	Water Blank (5 samples)	—	>100	>100
1	Virgin + Color	White	>100	88
2	Virgin Regrind	Blue	>100	>100
3	Acrylic Acid, Drum #2	Black	46	23
4	Acrylic Acid, Washed #2A	Black	59	37
5	Acrylic Acid, Pellets #2B	Black	68	42
6	Sulfuric Acid, Drum #6	Black	76	60
7	Sulfuric Acid, Washed #6A	Black	86	62
8	Sulfuric Acid, Pellets #6B	Black	66	52
9	Mineral Spirits, Drum #7	Blue	45	32
10	Mineral Spirits, Washed #7A	Blue	45	26
11	Mineral Spirits, Pellets #7B	Blue	>100	>100
12	Acetic Acid, Drum #8	Blue	>100	96
13	Acetic Acid, Washed #8A	Blue	86	>100
14	Acetic Acid, Pellets #8B	Blue	>100	>100

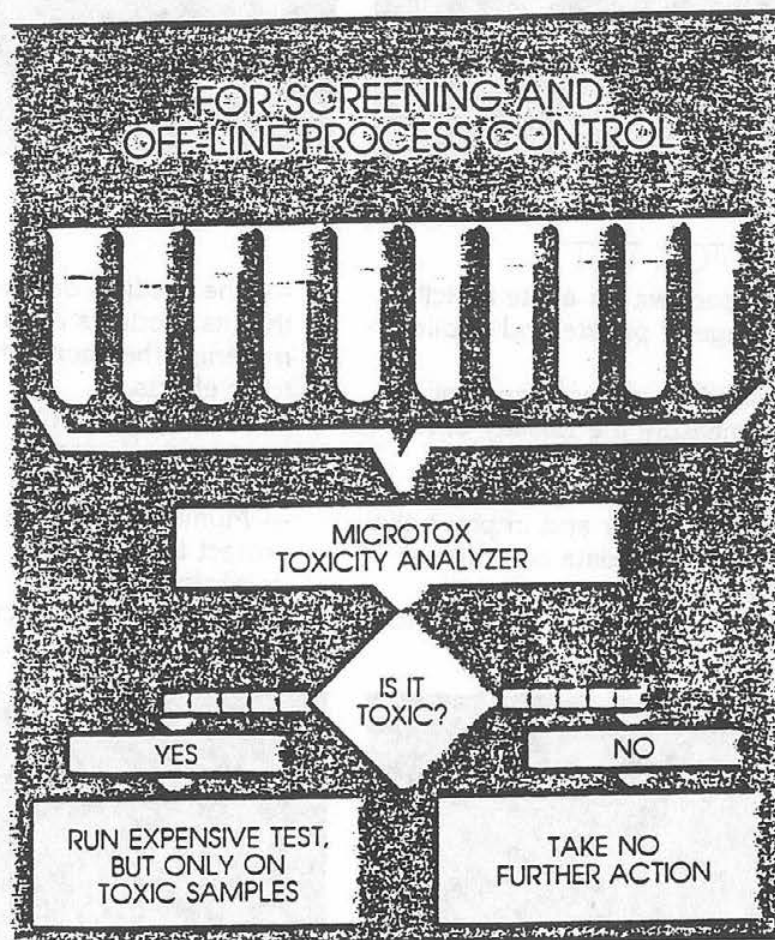
MICROTOX® ...THE FAST TOXICITY TEST™ T.M.

WHAT IS TOXICITY?

A given amount of any chemical is said to be toxic when it causes harm to a living thing that absorbs, swallows, or breathes it. Everything we consume or come into contact with is theoretically hazardous; only the concentration matters. Many substances are absolute requirements for life at certain levels, but are lethal at both higher and lower levels. For example, normal diets for laboratory rats contain about .15 mg/Kg per day of selenium. A selenium-free diet is lethal. At 1 mg/Kg, growth and health are much better than normal; at 3 mg the rats get sick; at 5 mg they die.

HOW DOES MICROTOX DETECT TOXICITY?

Pure Microtox Reagent (living bacteria) emits light. When toxic material is added to the reagent, the light output drops. The higher the concentration of toxicant, the greater the light loss. The Microtox Instrument measures the light level before and after a sample of unknown toxicity is added to the reagent. The amount of decrease in the light level tells us how toxic the sample is.



WHAT DO PEOPLE USE MICROTOX FOR?

Microtox is used chiefly in two ways:

- 1.) As an inexpensive, fast screening tool, Microtox quickly quantitates the relative toxicity of samples. Slower, more expensive qualitative tests need be run only on samples determined to be toxic.
- 2.) As an off-line process control test, Microtox lets system operators check toxicity levels frequently at many different points so they can adjust systems to operate most efficiently.

WILL MICROTOX TELL ME WHAT THE TOXICANT IS?

No. Microtox tells us the degree of toxicity of a sample.

WHAT IS THE COST OF A MICROTOX TEST?

The materials and supplies for a single Microtox test cost between \$2 and \$20, depending on the number of tests being run and on the test procedure.

WHAT TYPES OF TOXICANTS CAN MICROTOX DETECT?

In general, Microtox reagent is sensitive to the full range of chemicals we consider harmful to life. Microtox detects the toxic effects of rare, unexpected chemicals as well as common chemicals like PCB's, cyanide, mercury and formaldehyde.

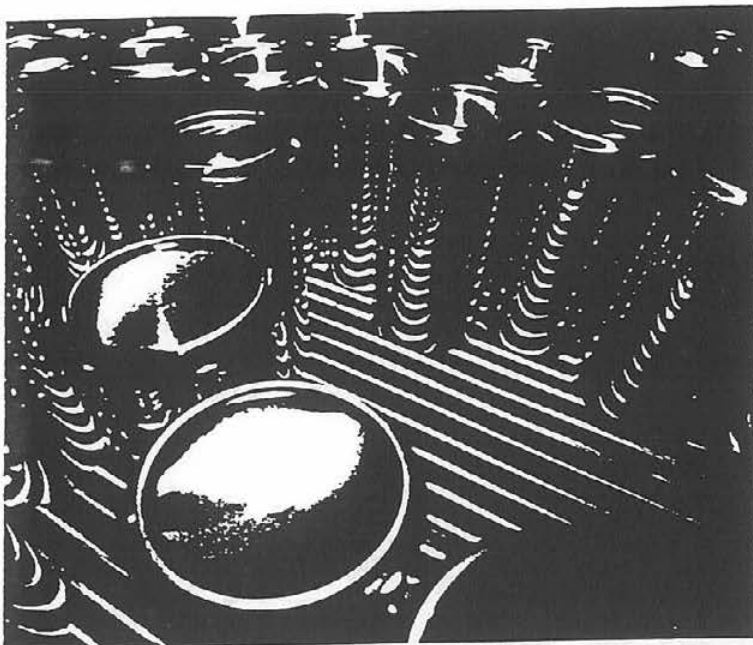
Microtox reagent also integrates the effects of mixtures of toxicants, providing an overall toxicity index for complex samples.

HOW DO YOU RUN A MICROTOX TEST?

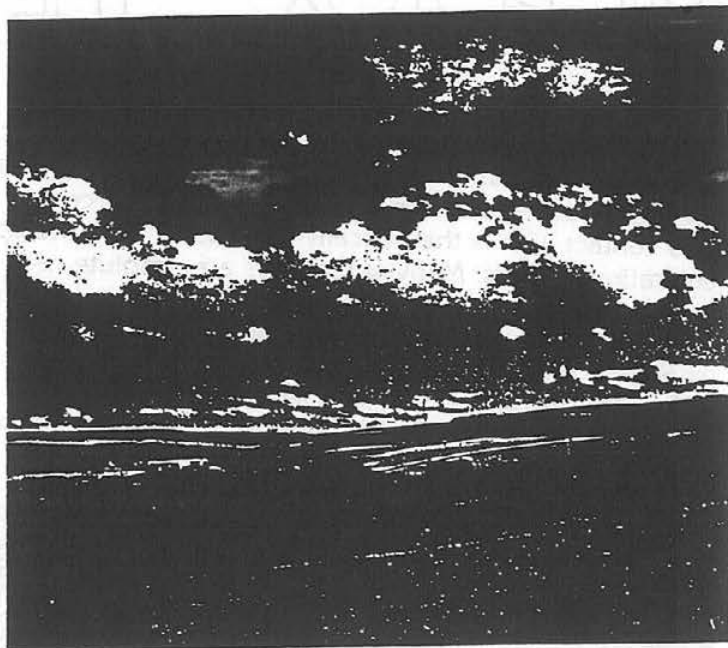
There are five steps in a Microtox test.

- Step 1: Reconstitute reagent
- Step 2: Dilute sample
- Step 3: Add sample to reagent
- Step 4: Measure results
- Step 5: Calculate toxicity

It is easy to run 10 or more tests per hour. The simple procedure can be learned in less than a day.



In the Beverage Industry.



In Environmental Protection.

WHO USES THE MICROTOX TEST?

As the most commonly used test for acute toxicity, Microtox works for a wide range of private and public organizations. Some examples:

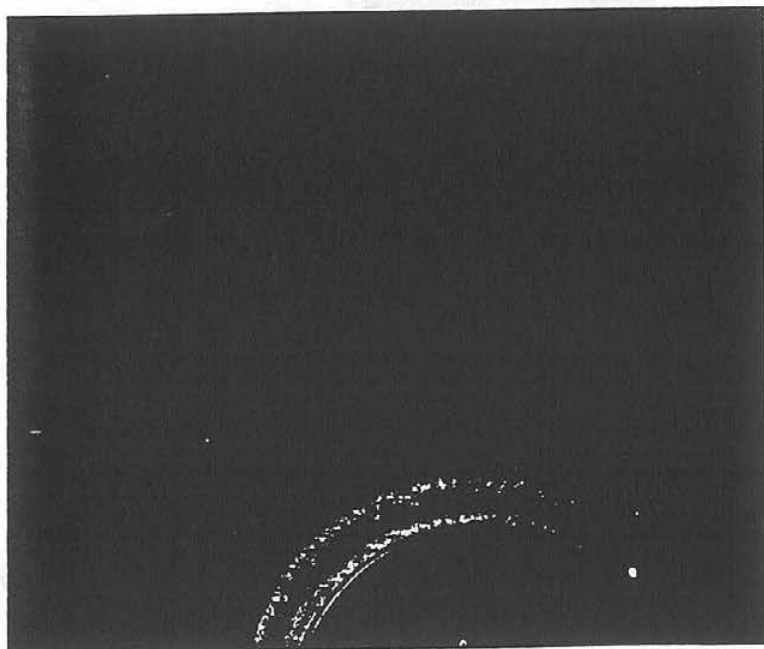
- Federal and state monitoring and enforcement agencies use Microtox to measure the toxicity of hazardous waste, accidental spills and waste water.
- Publicly owned and industrial waste water treatment plants use Microtox to monitor and improve the treatment process, as well as to validate compliance with EPA and state regulations.
- The food and beverage industries use Microtox to assure product safety.

— The medical devices industry uses Microtox to see that its products don't leach toxic chemicals into the materials they contact, or react with them to produce toxic effects.

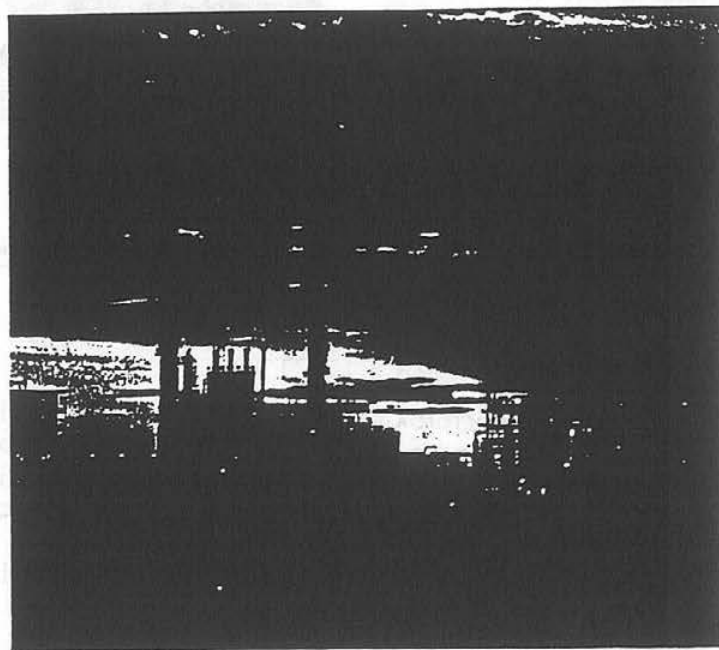
— Offshore oil drillers check the toxicity of drilling muds with Microtox, protecting shrimp, fish and other animals.

— Municipal drinking water suppliers use Microtox to protect the consumer from accidental or intentional poisoning.

— Chemical manufacturers screen new product candidates with Microtox.

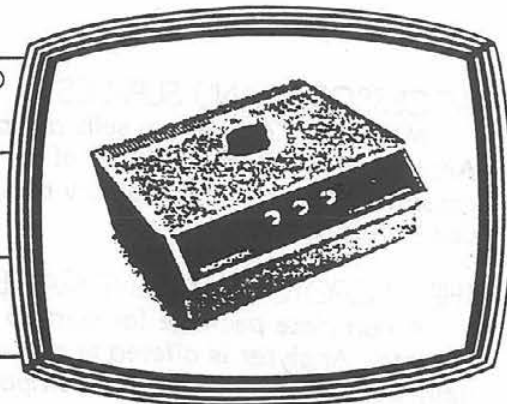


In the Medical Device Industry.



In Hazardous Waste Monitoring and Clean-up.

THE MICROTOX[®] INSTRUMENT ACCESSORIES AND SUPPLIES



*Cost ~ \$5000.00 for system
Cost ~ \$25.00 per test.*

WHAT MICROTOX DOES

The Microtox system measures the toxicity of aqueous solutions. In the test, a light emitting reagent is exposed to samples of material in small cuvettes. If the samples are toxic, the light output of the reagent is reduced in proportion to the toxicity of the samples. The light loss is a simple measure of toxicity. The significance and applications of Microtox, The Fast Toxicity Test[™], are treated at length in other literature.

THE MICROTOX INSTRUMENT

The Microtox Model 2055 Toxicity Analyzer employs a sensitive photomultiplier to measure the light emitted by small volumes of glowing reagent.

Separate refrigeration units maintain samples and reagent at precise temperatures.

A patented turret mechanism allows the cuvettes containing test materials to be exposed conveniently to the photomultiplier in a brightly lighted lab without ambient light leakage.

USE OF THE INSTRUMENT

The Microtox Instrument serves both as an experimental laboratory instrument and as a "production" tool. It can be operated easily over a broad range of sensitivities and temperatures for research. It can be used to run large numbers of tests reliably, day after day, month after month.

THE CHART RECORDER

Microbics Corporation offers an accessory chart recorder matched to the analyzer: a ten-inch, single-pen, manual lift, 10mv fixed (17 speed) device in either 120 or 220 volt versions.

Though the Microtox instrument is equipped with a three-digit display that shows temperatures and light levels, most laboratories rely on the chart recorder for permanent records of the tests. Use of the recorder

simplifies the testing process, (serving as an automatic timer, for example), greatly reduces the probability of transcription errors, and aids in final data reduction.

SPECIFICATIONS

Power Requirements: The standard unit operates on nominal 100-120 volts AC at 50/60 Hz. It is fused for 2 amps. (At startup, when the coolers are working to bring temperatures from ambient to their set points, the unit draws about 150 watts. Later, when the coolers need only maintain the set temperature, the unit draws about 60 watts.) An export model, running on nominal 220-240 volts AC at 50/60 Hz. is fused for one amp.

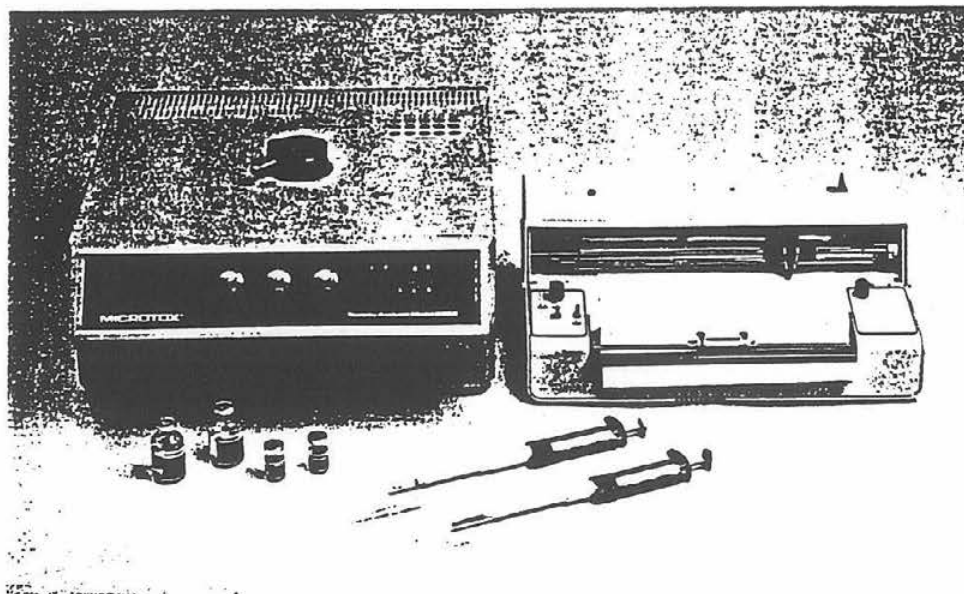
Height to the top of the turret: 10 inches.

Width: 18.3 inches.

Depth: 20.7 inches.

Weight: 48 pounds. (about 55 pounds, packaged for shipment.)

The unit was designed to sit on a laboratory bench. Even so, people move Microtox instruments around freely, using them wherever they are needed. The system can easily be carried into the field, and operated in the back of a station wagon or van, using a small portable generator for power.

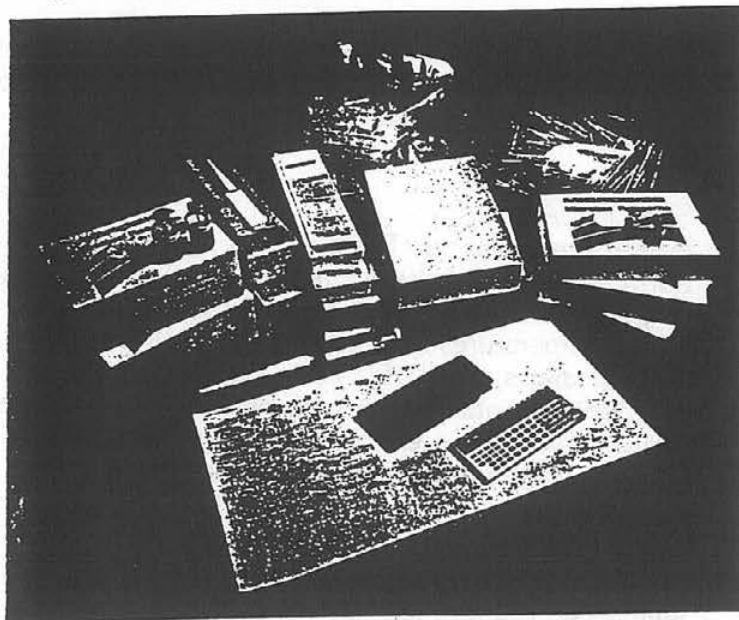


ACCESSORIES AND SUPPLIES

Microbics Corporation sells not only the Microtox Analyzer and reagents, but all of the supplies and accessories necessary for ordinary testing. A single phone call brings quick delivery.

THE MICROTOX REAGENT STARTER PACK

A complete package for start-up operation of the Microtox Analyzer is offered at a price which is substantially below that of individual components.



- Microtox Reagent - 40 vials, each sufficient for testing ten to fifty samples. Shelf life is one year.
- Microtox Reagent Diluent - 2% salt solution for sample dilution, 20 bottles of 50 ml.
- Microtox Osmotic Adjusting Solution - 22% salt solution for controlling the salinity of the sample, 10 vials of 5 ml.

- Microtox Reconstitution Solution - specially treated pure water for reconstitution of the lyophilized bacterial reagent, 2 bottles of 25 ml.
- Cuvettes - 720, boxed.
- 10 Microliter Pipette.
- 250 Microliter Pipette.
- 500 Microliter Pipette.
- Pipette tips, Type I - 1000.
- Pipette tips, Type II - 1000.
- Preprogrammed Calculator for data reduction.
- Microtox Data Reduction Supplies.

All items in the Starter Pack may be ordered individually from Microbics Corporation.

COLOR CORRECTION PROCEDURE SUPPLIES

Microtox Color Correction Cuvettes may be required for work with highly colored or turbid samples. These non-disposable double cuvettes are used in a special procedure, which also uses disposable aspirators available from Microbics Corporation.



CHART RECORDER SUPPLIES

Microtox Chart Paper is supplied in packs of six 100' rolls. Recorder Pens, Black or Red, are supplied in packs of four.

A NOTE ON SHIPPING:

The Microtox reagent is shipped in insulated container with artificial ice by expedient routes to insure that it arrives in good condition. It has a long shelf-life when stored at refrigerator or colder temperatures.

MICROBIC CORPORATION

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