The Plastic Drum Institute

The Real World Test

The Society of the
Plastics Industry, Inc.

THE PDI REAL WORLD TEST

While the SPI Plastic Drum Institute, its members, and others participating in this study have made their best efforts to provide reliable and accurate information, they individually and collectively disclaim all warranties, expressed or implied, including the warranties of merchantability and of fitness for a particular use. For a complete understanding of the study, it should be read in its entirety. Specific manufacturers' instructions should be consulted relative to the reuse of 55-gallon all-plastic drums for particular commodities.

Joint Reuse and Analytical
Protocol Subcommittees
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The Society of the Plastics
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PDI REAL WORLD TEST

ACKNOWLEDGEMENTS

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Ashland Chemical Company, Division of Ashland Oil, Inc.
Esso Petroleum, Canada
Exxon Chemical Americas
Hoechst Celenese Corporation
Phillips Petroleum
Quantum Chemical Corporation, USI Division
Shell Oil Company
Stanchem, A Unit of CIL
The Dow Chemical Company

Plastic Drum Fabricators

Container Corporation of America
Eastern/Plastic Division
Hunter Drums, Ltd.
Russell Stanley Corporation

Reconditioner

Bakerstown Container

The Plastic Drum Institute also acknowledges and thanks the members of the intra-industry task force:

Institute of Packaging Professionals *
Chemical Packaging Committee (CPC)
Petroleum Packaging Committee (PPC)

National Barrel and Drum Association (NABADA)

* The merger of the former Packaging Institute/International with the Society of Packaging Professionals

ACKNOWLEDGEMENTS

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PDI REAL WORLD TEST

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EXECUTIVE SUMMARY

INTRODUCTION

In 1983 the Plastic Drum Institute (PDI) issued a report entitled "The 55-Gallon All Plastic Drum Reuse Study." That report concluded:

- Ladings did not affect the structural integrity of the plastic drums.
- Reconditioning did not affect the structural integrity of the plastic drums.
- Minimal residue from previous ladings packaged remained in the reconditioned drums.

The significance of this residue to the chemical and petroleum packaging industry was broadly dicussed within the PDI.

As a result of these discussions, the PDI proposed a task force in conjunction with the Petroleum Packaging Committee (PPC) and the Chemical Packaging Committee (CPC) of the Institute of Packaging Professionals and the National Barrel and Drum Association (NABADA) to develop a second study to include the evaluation of shippers responsible for the purity of packaged ladings. The task force recommended a program outline which the PDI agreed to coordinate. This program has become known as the "Real World Test."

OBJECTIVES

The primary objective of the "Real World Test " 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 criteria for acceptability were the quality control analyses performed by the shippers upon their ladings following three months storage in the used containers.

A secondary objective was to reconfirm the results of the 1983 study regarding the integrity of all-plastic drums following service and reconditioning. The test criterion was a water-filled drop test.

A further objective was the determination of the residual second lading absorbed into the polyethylene drum wall and remaining after reconditioning. Five testing laboratories produced and performed analytical procedures on sections cut from walls of the test drums.

CONCLUSIONS

The results of the "Real World Test" reconfirmed:

- Ladings did not affect the structural integrity of plastic drums.
- Reconditioning did not affect the structural integrity of the plastic drums.
- Minimal residue from previous ladings packaged remained in the reconditioned drums.

And finally it was determined that:

- Most shippers concluded that reconditioned plastic drums were acceptable for the ladings used in the study.

SUMMARY

The "Real World Test" involved the filling and storage of seventy-two 55-gallon high density polyethylene containers with six selected ladings. The ladings were chosen by the joint Task Force as representative of classes of products currently shipped in all-plastic containers. The six ladings chosen by the joint Task Force were:

Methanol
Mineral Spirits
Acrylic Acid
Acetic Acid
Sulfuric Acid
Motor Oil

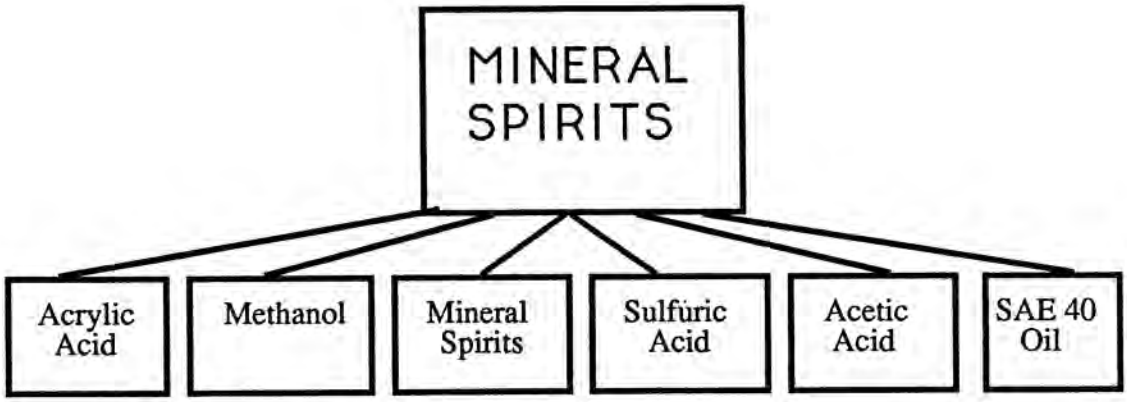
For the first phase of this test, twelve containers were filled with the ladings.

Before storage, the ladings were analyzed as a base reference. After the storage period of three months, the ladings would again be analyzed to determine what effect, if any, the storage in plastic containers had on the product in question.

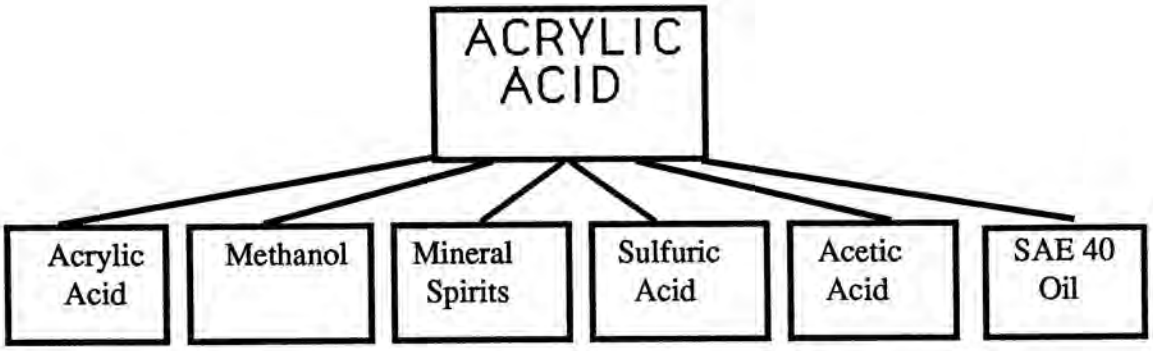
After three months storage at the shippers'/fillers' facilities, the drums were emptied and sent to the reconditioner where the drums were cleaned using normal procedures for plastic drum interior, exterior and closure and, were submitted to final inspection.

The next step involved twelve drums from each product being disbursed to the various other products and filled.

The chart below illustrates the 12 drums with Mineral Spirits being apportioned to the other chemicals in the test program



The following chart illustrates the apportionment of Acrylic Acid. This same procedure would be conducted with the remaining 4 chemicals until all 72 drums were in their proper categories.



After filling, the drums were stored by shippers/fillers for a period of three months. Before and after the three month storage period, the ladings were again analyzed for possible contaminates. Comments of the shippers/fillers concerning these assay results determined the suitability of reused drums for service with each lading.

After emptying the second lading, the drums were returned to the reconditioner and were again cleaned following normal procedures for plastic drums.

The drums were subjected to flat-side drop at an ambient temperature from a height of six feet. The drums were divided into sets pertaining to the second lading and forwarded to five laboratories while they developed techniques for quantifying the residual second lading absorbed into HPDE. The laboratories were asked to determine the residual second lading in sections cut from the drum walls. Lading concentrations were reported as an average value for each lading and as a function of the first lading.

Further, for drums that contained the same lading in both fillings, the laboratories determined a residual lading gradient by dividing the drum wall crossection into thirds (inner, middle, and outer) and performing the analysis on these sections.

TABLE 1

PDI REAL WORLD TEST

DRUM LADING KEY [1]

SECOND LADING: / FIRST LADING:	Acrylic Acid [2]	Methanol	Mineral Spirits	Sulfuric Acid	Acetic Acid	Motor Oil
Acrylic Acid	01, 02	03, 04	05, 06	07, 08	09, 10	11, 12
Methanol	13, 14	15, 16	17, 18	19, 20	21, 22	23, 24
Mineral Spirits	25, 26	27, 28	29, 30	31, 32	33, 34	35, 36
Sulfuric Acid	37, 38	39, 40	41, 42	43, 44	45, 46	47, 48
Acetic Acid	49, 50	51, 52	53, 54	55, 56	57, 58	59, 60
Motor Oil	61, 62	63, 64	65, 66	67, 68	69, 70	71, 72

1. Numbers refer to the numbered drum sets.

2. One drum set, which had contained acrylic acid as the second lading, was lost.

TABLE 3

PDI REAL WORLD TEST PROGRAM

SULFURIC ACID

FIRST LADING	ACRYLIC ACID	SULFURIC ACID	METHANOL	MINERAL SPIRITS	ACETIC ACID	MOTOR OIL
% SULFURIC ACID, 93.19% MIN	93.54	93.46	93.55	93.56	93.50	93.45
SPECIFIC GRAVITY	1.835	1.835	1.835	1.835	1.835	1.835
HAZEN COLOR, 40 UNITS MAX	20	25	25	25	20	20
IRON, 50 PPM MAX	8	8	11	11	11	8
SULFUR DIOXIDE, 50 PPM MAX	77	23	22	32	19	20
TRANSMITTANCE, 60% MIN	92	90	90	73	91	86

TABLE 4

PDI REAL WORLD TEST PROGRAM

METHANOL

FIRST LADING	CONTAMINATION, % BY VOLUME
ACRYLIC ACID	0.4
ACRYLIC ACID	.1
SULFURIC ACID	.1
SULFURIC ACID	.1
METHANOL	.1
METHANOL	.1
MINERAL SPIRITS	.2
MINERAL SPIRITS	.1
ACETIC ACID	.1
ACETIC ACID	.1
MOTOR OIL	.04
MOTOR OIL	.2

TABLE 5

PDI REAL WORLD TEST PROGRAM

MINERAL SPIRITS

FIRST LADING	CONTAMINATION, % BY VOLUME
ACRYLIC ACID	0.01
ACRYLIC ACID	.01
SULFURIC ACID	.01
SULFURIC ACID	.01
METHANOL	.01
METHANOL	.01
MINERAL SPIRITS	.001
MINERAL SPIRITS	.02
ACETIC ACID	.001
ACETIC ACID	.01
MOTOR OIL	.01
MOTOR OIL	.01

TABLE 6

PDI REAL WORLD TEST PROGRAM

MOTOR OIL

FIRST LADING	SECOND LADING	CONTAMINATION FROM FIRST LADING
ACRYLIC ACID	TRANSFORMER OIL	192 PPM
ACETIC ACID	MOTOR OIL	<30 PPM
ACETIC ACID	TRANSFORMER OIL	<30 PPM
SULFURIC ACID	MOTOR OIL	<80 PPM
SULFURIC ACID	TRANSFORMER OIL	<100 PPM
METHANOL	MOTOR OIL	<100 PPM
METHANOL	TRANSFORMER OIL	<100 PPM
MINERAL SPIRITS	MOTOR OIL	<1%
MINERAL SPIRITS	TRANSFORMER OIL	<1%
MOTOR OIL	MOTOR OIL	<100 PPM
MOTOR OIL	TRANSFORMER OIL	<100 PPM

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TABLE 7

PDI REAL WORLD TEST PROGRAM

ACRYLIC ACID

	CONTROL	ACRYLIC ACID	ACETIC ACID	METHANOL	SULFURIC ACID	MOTOR OIL	MINERAL SPIRITS
START (8/28/87)							
DIMER, WT.%	0.10	-	-	-	-	-	-
MEHQ, PPM	211						
9/16/87							
DIMER, WT.%	0.73	0.73	0.70	0.70	0.68	0.69	0.71
MEHQ, PPM	205	208	197	216	197	209	215
10/01/87							
DIMER, WT.%	1.08	1.16	1.11	1.12	1.09	1.07	1.14
MEHQ, PPM	205	211	209	208	207	207	208
11/05/87							
DIMER, WT.%	1.78	1.77	1.73	1.77	1.78	1.75	1.79
MEHQ, PPM	191	189	191	189	193	192	188
12/02/87							
DIMER, WT.%	1.94	2.01	1.92	1.86	1.93	1.95	1.93
MEHQ, PPM	186	190	189	189	185	187	190

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TABLE 8

PDI REAL WORLD TEST PROGRAM

ACRYLIC ACID

CONTAMINATION DETERMINATIONS

PREVIOUS LADINGS	CONTAMINATION BY GC	SULFUR BY MICROCOULOMETRY
ACRYLIC ACID	N/A	N/A
METHANOL	ND	N/A
MINERAL SPIRITS	50-100 PPM	N/A
SULFURIC ACID	N/A	5.8 PPM
ACETIC ACID	N/A	N/A
MOTOR OIL SAE 40	ND	1.4 PPM

N/A - TESTING NOT MEANINGFUL/OR APPLICABLE
ND - NOT DETECTED

2701E

PART II

STRUCTURAL INTEGRITY: Analytical Procedures And Results

TESTING & DISCUSSION

OBJECTIVE:

A secondary objective was to analyze the results of the "Real World Test" to reconfirm the structural integrity of all-plastic drums after fillings and reconditionings.

The 1983 Reuse Study had indicated good retention of structural integrity in the HDPE drums after service with most ladings and subsequent reconditioning. A random sampling of the drums was also drop tested to determine if the storage of two different lading had an affect on structural integrity.

The DOT -34 specificied drop test for 55-gallon all-plastic drums currently involves filling the drums with an ethylene glycol solution, cooling the filled drums to 0 deg. F. and dropping the drums from a height of four feet. In this test, however, ethylene glycol solution was not used because of the necessity for subsequent chemical analysis of the drum walls. Instead, the drums were filled with water at ambient temperature and dropped from an increased height of six feet. The drums were then examined for signs of leakage. Table 9 shows the drums that were dropped, their paired ladings, and the results of the drop tests.

CONCLUSIONS

All drums successfully passed the drop test. Temporary distortion of some drums did occur. The drums' shapes returned to normal within a short time. The results show that the multiple ladings, reconditionings and the drum color used in the study had no effect on the structural integrity of the all-plastic drums.

ANALYTICAL TESTING

OBJECTIVE:

Another objective of the "Real World Test" was to analyze and quantify the residual second lading absorbed into the durm wall and remaining after reconditioning.

TESTING PROCEDURES

Six test laboratories volunteered to develop and make available a testing procedure for determining the level of a residual lading in high density polyethylene. These same laboratories agreed to perform testing on the drum walls as provide by the "Real World Test". Only five procedures were produced because one set of drums which had contained acrylic acid as the second lading was lost. The procedures, as written by the testing laboratories, appears in Appendix B.

A summary of the test techniques used is as follows:

LADING	TEST METHOD	LABORATORY
Methanol	G. C. Headspace	Hoechst Celanese
Acetic Acid	Extraction G.C.	Quantum Chemical, USI Div.
Sulfuric Acid	X-Ray Fluorescence	The Dow Chemical Company
Mineral Spirits	Loss of Heating	Container Corp. of America
Motor Oil	Extraction G.C.	Phillips Petroleum

RESULTS & DISCUSSION

Table 10 summarizes the results of the analytical testing for the residual second lading. These results are averages of all tests without separating the possible effects of the first lading. Results are reported in parts per million residual in the drum wall except or sulfuric acid.

Sulfuric acid reacts with the inside surface of the polyethylene drum, forming a surface barrier layer in a process called sulfonation. Deeper penetration of the sulfuric acid was not expected.

Mineral spirits and motor oil showed the highest levels of residual of the five tested ladings. This is not unexpected as hydrocarbon permeation of polyethylene is well known.

Methanol tested the lowest. One theory is that the methanol volatility causes it to migrate out of the drum wall with time.

Tables 11 thru 15 separate the analytical results according to first lading. For the majority of cases, the level of second lading residual was not significantly affected by the nature of the first lading. Certain exceptions were noted: a higher level of motor oil was absorbed into drums that previously contained mineral spirits. Conversely, higher levels of mineral spirits were found in drums that had previously contained motor oil.

PARTITIONING

For drums that contained the same lading twice, four of the analytical testing laboratories agreed to partition a section of the drum wall.

The wall was partitioned into thirds in an attempt to determine a concentration gradient for the lading. Each third (inner, middle and outer) was analyzed for the residual lading. The results, determined for methanol, acetic acid, mineral spirits and motor oil, appear on Table 16.

With the exception of acetic acid, the ladings show highest concentration of residual in the inner third of the drum wall. This is expected as this is the point of direct contact with the lading.

Acetic acid showed the middle third of the drum wall to contain the highest level of residual. As acetic acid is quite soluble in water, it is speculated that the reconditioning process might have extracted some acetic acid from the inner third of the drum wall.

TABLE 9

PDI REAL WORLD TEST

DROP TEST RESULTS

DRUM NUMBER	FIRST LADING	SECOND LADING	PASS/FAIL
4	Acrylic Acid	Methanol	P
5	Acrylic Acid	Mineral Spirits	P
6	Acrylic Acid	Mineral Spirits	P
7	Acrylic Acid	Sulfuric Acid	P
8	Acrylic Acid	Sulfuric Acid	P
9	Acrylic Acid	Acetic Acid	P
10	Acrylic Acid	Acetic Acid	P
15	Methanol	Methanol	P
19	Methanol	Sulfuric Acid	P
20	Methanol	Sulfuric Acid	P
21	Methanol	Acetic Acid	P
22	Methanol	Acetic Acid	P
27	Mineral Spirits	Methanol	P
28	Mineral Spirits	Methanol	P
29	Mineral Spirits	Mineral Spirits	P
30	Mineral Spirits	Mineral Spirits	P
31	Mineral Spirits	Sulfuric Acid	P
32	Mineral Spirits	Sulfuric Acid	P
33	Mineral Spirits	Acetic Acid	P
34	Mineral Spirits	Acetic Acid	P
39	Sulfuric Acid	Methanol	P
43	Sulfuric Acid	Sulfuric Acid	P
44	Sulfuric Acid	Sulfuric Acid	P
45	Sulfuric Acid	Acetic Acid	P
46	Sulfuric Acid	Acetic Acid	P
51	Acetic Acid	Methanol	P
54	Acetic Acid	Mineral Spirits	P
55	Acetic Acid	Sulfuric Acid	P
56	Acetic Acid	Sulfuric Acid	P
58	Acetic Acid	Acetic Acid	P
64	Motor Oil	Methanol	P
66	Motor Oil	Mineral Spirits	P
67	Motor Oil	Sulfuric Acid	P
68	Motor Oil	Sulfuric Acid	P
69	Motor Oil	Acetic Acid	P
70	Motor Oil	Acetic Acid	P

TABLE 10

PDI REAL WORLD TEST

ANALYTICAL RESULTS;RESIDUAL LADING IN DRUM SIDEWALL

LADING	TEST METHOD	AVG. RESULT	UNITS	RANGE
Methanol	G.C.-Headspace	2.4	ppm	1.5-4.6
Acetic Acid	Extraction/G.C.	56.5	ppm	5.0-97
Sulfuric Acid	X-Ray Fluoresc.	26.2	mg S/sq. in.	16-44
Mineral Spirits	Loss on Heating	9000	ppm	1600-12100
Motor Oil	Extraction/G.C.	5300	ppm	2600-11500

TABLE 11

PDI REAL WORLD TEST

RESIDUAL SECOND LADING AS A FUNCTION OF FIRST LADING

SECOND LADING: SULFURIC ACID

FIRST LADING	RESIDUAL SECOND LADING
ACRYLIC ACID	36 mg S/sq. in.
ACETIC ACID	20 mg S/sq. in.
MOTOR OIL	27 mg S/sq. in.
MINERAL SPIRITS	36 mg S/sq. in.
SULFURIC ACID	17 mg S/sq. in.
METHANOL	22 mg S/sq. in.

TABLE 12

PDI REAL WORLD TEST

RESIDUAL SECOND LADING AS A FUNCTION OF FIRST LADING

SECOND LADING: METHANOL

FIRST LADING	RESIDUAL SECOND LADING
ACRYLIC ACID	4.6 ppm
ACETIC ACID	2.8 ppm
MOTOR OIL	1.5 ppm
MINERAL SPIRITS	1.9 ppm
SULFURIC ACID	1.6 ppm

TABLE 13

PDI REAL WORLD TEST

RESIDUAL SECOND LADING AS A FUNCTION OF FIRST LADING

SECOND LADING: MINERAL SPIRITS

FIRST LADING	RESIDUAL SECOND LADING
ACRYLIC ACID	6500 ppm
ACETIC ACID	8700 ppm
MOTOR OIL	12100 ppm
MINERAL SPIRITS	10000 ppm

TABLE 14

PDI REAL WORLD TEST

RESIDUAL SECOND LADING AS A FUNCTION OF FIRST LADING

SECOND LADING: ACETIC ACID

FIRST LADING	RESIDUAL SECOND LADING
ACRYLIC ACID	50 ppm
ACETIC ACID	97 ppm
MOTOR OIL	32 ppm
MINERAL SPIRITS	37 ppm
SULFURIC ACID	78 ppm
METHANOL	40 ppm

TABLE 15

PDI REAL WORLD TEST

RESIDUAL SECOND LADING AS A FUNCTION OF FIRST LADING

SECOND LADING: MOTOR OIL

FIRST LADING	RESIDUAL SECOND LADING
ACRYLIC ACID	7800 ppm
ACETIC ACID	3400 ppm
MOTOR OIL	3800 ppm
MINERAL SPIRITS	9100 ppm
SULFURIC ACID	2600 ppm
METHANOL	5200 ppm

TABLE 16

PDI REAL WORLD TEST

ANALYTICAL RESULTS;RESIDUAL LADING, PARTITIONED SIDEWALL

DUPLICATE LADINGS

LADING	UNITS	INNER	MIDDLE	OUTER
Methanol	ppm	3.79	0.95	0.67
Acetic Acid	ppm	147	231	95
Mineral Spirits	ppm	18600	14700	8800
Motor Oil	ppm	9900	300	1100

APPENDIX A

PART I
LADING QUALITY:
Shippers' Results

Report on Real-World Test

Hunter Drums Ltd.

We provided C.I.L. in Cornwall, Ontario with 12 drums permanently branded S.A. 1 - 12 for Sulfuric Acid filling and 12 drums permanently branded A.A. 1 - 12 for Acetic Acid filling.

These drums were filled on July 31, 1986 and stored for three months.

The following chart illustrates the C.I.L. criterea for commercial grade Acetic Acid and commercial grade Sulfuric Acid.

ACETIC ACID		SULFURIC ACID	
TEST	COMMERCIAL SPEC.	TEST	COMMERCIAL SPEC.
ASSAY	99.5% Min.	ASSAY	93.19% Min.
COLOR	5 APHA Units	COLOR	40 HAZEN Units
IRON	2.00 ppm Max.	IRON	50 ppm Max.
LEAD	3.00 ppm Max.	SO2	50 ppm Max.
RESIDUE AFTER EVAPORATION	100 ppm Max.	TRANSMITTANCE	60% Min.
CHLORIDES	15 ppm Max.		
ACETIC ANHYDRIDE	0.03% Max.		
SULPHATES	15.0 ppm Max.		

On Oct. 30, 1986 C.I.L. compared the samples taken from the 24 drums with their commerical specifications for acetic and sulfuric acid.

All samples met their criterea for commercial grade acids.

The drums were then emptied and forwarded to Bakerstown Containers for reconditioning and subsequently distributed for refilling with the other ladings outlined in the test.

On May the 25th Hunter Drums received from Bakerstown 24 drums which had been reconditioned and numbered as follows:

Metacrylic Acid	- 7, 8, 9, 10
Methanol	- 19, 20, 21, 22
Mineral Spirits	- 31, 32, 33, 34
Sulfuric Acid	- 43, 44, 45, 46
Acetic Acid	- 55, 56, 57, 58
S.A.E. 30 Motor Oil	- 67, 68, 69, 70

These 24 drums were forwarded to C.I.L. and two drums from each previous lading were filled with acetic acid and two drums from each previous lading were filled with sulfuric acid.

The following chart illustrates the specifications of each lading when filled on July 10, 1987 and the results of the tests on the acids removed from the drums Nov. 8, 1987.

Commercial Grade Sulfuric Acid

Drum previously filled with:	Metacrylic Acid	S.A.E. 30 Motor Oil	Acetic Acid	Sulfuric Acid	Mineral Spirits	Methan
Sample #	613	614	615	616	617	618
Drum #	7, 8	67, 68	55, 56	43, 44	31, 32	19, 20
Specific Gravity	1.835	1.835	1.835	1.835	1.835	1.835
Assay % Sulfuric 93.19% Min.	93.54	93.45	93.50	93.46	93.56	93.55
Color 40 Hazen Units Max.	20	20	20	25	25	25
Iron 50 ppm. Max.	8.174	8.174	10.899	8.164	10.899	10.899
Sulfur Dioxide 50 ppm. Max.	76.80	20.25	18.85	23.04	32.12	21.64
Transmittance 60% Min.	92	85.5	91	90	73	90

Commercial Acetic Acid

Drum previously filled with:	Metacrylic Acid	S.A.E. 30 Motor Oil	Acetic Acid	Sulfuric Acid	Mineral Spirits	Methan
Sample #	619	620	621	622	623	624
Drum #	9, 10	69, 70	57, 58	45, 46	33, 34	21, 22
Assay % Acetic 99.5% Min.	99.98	99.93	99.98	99.98	99.98	99.98
Color 5 APHA Units	Water White	W.W.	W.W.	W.W.	W.W.	W.W.
Iron 2.0 ppm Max.	0.25	0.25	0.25	0.25	0.20	0.20
Lead 3.0 ppm Max.	0.80	0.50	0.40	0.50	1.00	1.20
Residue After Evaporation 100 ppm Max.	8	7	5	4	10	5
Chlorides 15 ppm Max.	3.75	1.00	0.75	1.00	3.75	2.00
Acetic Anhydride 0.03% Max.	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
Sulphates 15.0 ppm Max.	8.0	4.0	3.5	4.0	8.0	4.5

Ashland Chemicals

Industrial Chemicals and Solvents Division
Customer Service Laboratory

LABORATORY REPORT

TO: Dan Brown

LOCATION: Dublin

DATE: August 21, 1987

FROM: Rick Layman

SUBJECT: Service

CUSTOMER: Internal

SAMPLE: Methanol/Mineral Spirits

LAB NO.: 87-1687-1710

Analysis:

Drum #	Product	% by Vol. Contamination
3	Methanol	0.4
4	Methanol	0.1
5	Mineral Spirits	0.01
6	Mineral Spirits	0.01
39	Methanol	0.1
15	Methanol	0.1
16	Methanol	0.1
17	Mineral Spirits	0.01
27	Methanol	0.2
28	Methanol	0.1
29	Mineral Spirits	0.001
30	Mineral Spirits	0.02
40	Methanol	0.1
41	Mineral Spirits	0.01
42	Mineral Spirits	0.01
51	Methanol	0.1
52	Methanol	0.1
53	Mineral Spirits	0.001
54	Mineral Spirits	0.01
63	Methanol	0.04
64	Methanol	0.2
65	Mineral Spirits	0.01
	Mineral Spirits	0.01
66	Mineral Spirits	0.01

Comment:
The contamination in the Methanol drums was Mineral Spirits.

The contamination in the Mineral Spirits could not be identified, but is not Methanol.

The contamination in the Mineral Spirits might have been introduced before the product was put into the drums.

The plastic drums appear to be useable with alcohol solvents, but may cause contamination problems with other solvents.

RL/pjn Rick Layman

COPIES TO: B. Whitlock

M E M O R A N D U M

ESSO PETROLEUM CANADA
- RESEARCH DEPARTMENT

To: R.A. Miller

89 01 09

From: B.A. Osborne

R 40051

PLASTIC DRUM INSTITUTE TEST PROGRAM

Last summer, Esso Petroleum Canada agreed to participate in the Plastic Drum Institute Real World Test Program, a study to measure the practicality of reusing plastic drums in non-dedicated service. EPC's role in the program was to test the drums in a petroleum oil environment.

In all, eleven drums were submitted for tests. They had previously been filled with one of the following products - methacrylic acid (Drum #11), acetic acid (#59 and #60), sulphuric acid (#47 and #48), methanol (#23 and #24), mineral spirits (#35 and #36), and SAE 30 oil (#71 and #72) - stored for 3 months, reconditioned, and then shipped to EPC for testing with oil fills. One drum from each of the 5 sets of reconditioned drums was filled with Voltesso 35 transformer oil, which is very sensitive to low levels of contamination. The remaining drum from each of the 5 sets was filled with Essolube XD-3 10W engine oil, a low SAE viscosity grade product. The final drum, formerly containing methacrylic acid, was filled with Voltesso 35. The 11 drums were stored on pallets outside in a covered area from August to November, then sampled and returned to Bakerstown for reconditioning a second time and further testing.

Oil samples from each drum were analyzed after the 3 month storage period to determine any contamination present in the oils from the initial fills. Base-case samples of both Esso products were taken from the filling line before the drums were filled for comparison to the drum samples. These two oil samples were stored in amber glass bottles and kept in the laboratory away from any source of light.

Fourier Transform Infrared (FTIR) difference spectra were obtained for base-case and drum-stored oils to detect any contamination. In addition, Power Factors (ASTM D924), Interfacial Tensions (ASTM D971), and Karl Fischer water contents were run on all Voltesso 35 samples. The samples from the drums with an initial acid fill were tested for Total Acid Numbers (ASTM D974) and the samples from the drums with an initial SAE 30 oil fill were tested for additive metal contents. Also, sulphur contents were obtained on the samples with an initial sulphuric acid fill.

The FTIR spectrum, high Power Factor, low Interfacial Tension and high Total Acid Number on the oil from Drum 11 indicates contamination with methacrylic acid. The total acid number value points to a contamination level of approximately 192 ppm methacrylic acid in the oil or 34 g methacrylic acid in 178 kg oil. The remaining Voltesso 35 samples and the Essolube XD-3 10W samples showed no indication of the initial fill products in the oil.

In conclusion, 10 of the 11 reconditioned plastic drums submitted for EPC tests were satisfactory for at least 3 months storage with EPC lubricants. Drum No. 11 appeared to contain a small amount of the initial methacrylic acid fill. The EPC test results and technical information for this test program will be on file at the Esso Research Center in Sarnia.

cc: H.V. Tilley
R.S. Kartzmark
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D.A. Slack
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Attachment
1104m1w.bao

APPENDIX B

PART II
STRUCTURAL INTEGRITY:
Analytical Procedures

CONTAINER CORPORATION OF AMERICA

AN AFFILIATE OF JEFFERSON SMURFIT CORPORATION

Plastics Division
1204 EAST 12TH STREET
WILMINGTON, DE 19802

SUBJECT: Determination of volatile organic compounds in high molecular weight, high density polyethylene by thermogravimetry.

SCOPE: This procedure will measure the total volatile components present in high molecular weight, high density polyethylene (HMWHDPE) that are retrained by the material after exposure to chemicals and any cleaning procedures.

INTRODUCTION:

HMWHDPE is used in the manufacture of all-plastic drums intended for the storage and transport of hazardous chemicals. These drums may be cleaned for reuse in chemical service or ground up in 1/2"-1/4" particles for re-manufacture into other articles.

In order to safely reuse drums or reprocess ground drum material, it is necessary to determine the amount and/or identification of any residual material retained by the HMWHDPE.

Several procedures may be used to make this determination, including extraction and analysis of extractable materials and devolatilization with collection of volatile components for analysis.

The current procedure is based on devolatilization of the HMWHDPE by the technique of thermogravimetry (TG). "Thermogravimetry if a technique in which the mass of a substance is measured as a function of temperature while the substance is subjected to a controlled temperature program". (ASTM E473)

Since the ladings of the drums from which the test samples were taken for this data are known, no attempt will be made to identify volatile components and results will be given as percent based on weight loss.

This procedure can also be extended to determine the decomposition point of the HMWHDPE to evaluate any changes which may have occurred due to exposure to the chemicals contained or cleaning procedures used.

EXPERIMENTAL:

A. Samples. HMWHDPE samples are taken from sidewall sections of drums which have contained various chemicals as defined in the Plastic Drum Institute's Reuse Study: The Real World Test.

1. Black pigmented HMWHDPE - 11 samples.
2. Blue pigmented HMWHDPE - 11 samples.

B. Sample Size.

1. A 25 mg sample representative of the entire cross section is cut from the sidewall sample. Three separate samples are to be tested.

2. 25 mg samples taken from inner 1/3, middle 1/3 and outer 1/3 of drum thickness are cut from sidewall section to determine concentration gradient. Three samples each are to be analyzed.

C. Apparatus.

1. Thermogravimetric Analyzer. Consisting of an autobalance to record weight change while being tested at a controlled rate in an inert (nitrogen) atmosphere. The balance sensitivity should be $\pm$ 0.05 mg.

2. Nitrogen gas supply.

D. Procedure:

1. Place 25 mg sample (accurately weighed) in balance pan. Set range to 2 mg/full scale, each charge division equals 0.02 mg, approximately 0.1% of sample weight.

2. Place furnace into position around sample pan.

3. Start nitrogen purge gas at 50-100 ml/min., wait 5 minutes.

4. Set tempeterature program for range 50°C to 600°C.

5. Set program rate at 20°C/min.

6. Start temperature program and chart recorder.

7. Repeat for each sample.

E. Data Recorded.

1. Weight loss at 250°C, mg.

2. Decomposition Temperature of HMWHDPE. Taken as intersection of lines tangent to curve of loss vs. temperature before and after onset of rapid weight loss.

3. Weight loss at 250°C for inner 1/3, middle 1/3 and outer 1/3 (if done).

8022C-2

RESULTS:

The results are given as follows:

A. W250 (%) - Weight loss up to 250°C. Average of 3 determinations. W250 (%) = $\frac{\text{Loss (avg.)}}{\text{Sample Wt.}} \times 100$

B. T_D (°C) - Decomposition temperature for sample. Average of 3 determinations.

C. W250 (%) fraction - Percent weight loss for inner 1/3, middle 1/3 and outer 1/3 of thickness.

D. Grams/Drum - Calculate total volatile material possible in standard weight drum (20-24 lbs.).

DISCUSSION:

A discussion of the results is presented to explain results and describe any other observations made during the test such as presence of steps in the weight-temperature curve indicating possibility of multiple products being volatized.

CONCLUSIONS:

Assessment of level of contamination and suitability of drum for reuse or recycle programs.

Earl V. Lind Jr

Earl V. Lind
Manager
Materials & Systems Engineering
Container Corporation of America
Plastics Division

EVL:jr
8022C-3

TECHNICAL INFORMATION

The following is the procedure which was used in the determination of the degree to which the drum samples, supplied by the PDI, had absorbed any amounts of the sulphuric acid lading which it contained.

The procedure may involve hazardous materials, operations, and equipment. This procedure is not implying that it addresses all of the safety issues associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to its use.

MEASUREMENT OF SURFACE-SULFONATION USING X-RAY FLUORESCENCE

The analyst is referred to ASTM B 568-85, vol. 02.05, "Standard Test Method for Measurement of Coating Thickness by X-ray Spectrometry" and/or ASTM A 754-79, vol. 01.06, "Standard Test Method for Coating Thickness by X-ray Fluorescence" as a guide to testing using x-ray fluorescence.

Much of the surface-sulfonation testing can be accomplished in a manner similar to that described in ASTM C 810-75, vol. 02.05, "Standard Test Method for Nickel on Steel for Porcelain Enameling by X-ray Emission Spectrometry". Following the form of ASTM C 810-75, the necessary additions and corrections are given below for measuring surface-sulfonation using ASTM C 810-75:

1. Scope

   1.1 This test method covers the measurement of the amount of total sulfur deposited upon or reacted with the surface of high-density polyethylene.

2. Applicable Document

   2.1 Omit

3. Summary of Method

   3.1 Surface-sulfonated high-density polyethylene samples are inserted into a sample holder and irradiated with either an x-ray tube or a radioisotope source. The resulting emission characteristic of sulfur (S K-alpha) is recorded for intensity using a period sufficient to give the desired counting statistics (see ASTM A 754-79, section 3.5). The intensity count is converted by a calibration curve to micrograms of sulfur per square inch.

See reverse side

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MEASUREMENT OF SURFACE-SULFONATION USING X-RAY FLUORESCENCE (cont'd)

4. Significance

4.1 This test method is a highly accurate and rapid means for measuring surface-sulfur levels on high-density polyethylene.

5. Interferences

5.1 No interferences are expected for the S K-alpha emission. Suspicions of interferences should be checked by scanning about the S K-alpha emission line. However, low values can be obtained if overlaying material, for example, moisture or grease, is present. It is also possible to obtain incorrect values for sulfur concentration if the sulfonation depth is much different from that of the calibration materials. This is not usually a problem for the concentration range at or below that indicated in 8.1.

6. Apparatus

6.1 Suitable x-ray emission spectrometer complete with x-ray tube or radioisotope source (see manufacturer's instructions in the proper use of the spectrometer). Sulfur K-alpha emission may be measured using either energy- or wave-dispersive x-ray fluorescence (see ASTM B 568-85, sections 4.3.1 and 4.3.2) systems.

6.2 A sample holder will be required in moving the sample to the x-ray source area. This holder is usually designed for samples in the range of 1-2 inches in diameter.

6.3 High-density polyethylene discs with various levels of sulfur-coating are required for calibration and standardization.

7. Safety Precautions

7.1 Equipment should be periodically checked for radiation leaks to ensure against exposure to X-radiation.

8. Calibration and Standardization

8.1 Preparation of Standard Calibration Curve:

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8.1.1 Prepare sufficient samples to obtain an average of at least six (6) determinations (the number of determinations will be judged by the variability found for the samples and the area measured). Prepare standard samples by surface-sulfinating high-density polyethylene for varying treatment times to provide a range of surface-sulfonation values (this range is typically 50-500 micrograms of sulfur per square inch). Prepare a minimum of three (3) samples at each sulfonation level.

8.1.2 After determining the S K-alpha emission intensity versus specimen number, determine, for at least one specimen for each sulfonation level, the sulfur per square inch using an appropriate "wet-chemical" analysis.

8.1.3 Plot the sulfur emission intensity versus sulfur concentration as determined in 8.1.1 and 8.1.2, above.

9. Procedure

9.1 Standardization of Equipment

The surface-sulfonated discs, provided in 8.1, may be used for peaking response for S K-alpha emission and for calibrating the response of the x-ray emission spectrometer.

9.2 Sulfur Determination

9.2.1 Insert a sample disc into the sample holder, place the sample holder in the x-ray beam and measure the S K-alpha emission intensity. Repeat the sulfur-emission measurement. Average the counts so measured and read the sulfur concentration from the standard curve.

9.2.2 Specific details of operation of the x-ray apparatus are not included herein due to the various types of systems which may be applicable to this procedure. These details may be provided by the manufacturer.

10. Report

10.1 Convert x-ray counts to micrograms sulfur per square inch by using the calibration curve.

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B. Preparation of Sample Extract

1. Weigh 3 g of pellet-sized pieces of the HDPE drum sample to the nearest 0.1 mg into a 6 ml Hypo vial.

2. Using a volumetric pipet, add 4 ml of methylene chloride to the vial. Seal it tightly with a cap and septum, teflon side toward the sample, using the crimping tool.

3. Agitate the mixture several minutes. Allow the sample to extract overnight.

C. Preparation of Standards

1. Weigh 0.38 g of acetic acid to the nearest 0.1 mg into a 50 ml volumetric flask.

2. Dilute to volume with methylene chloride.

3. This standard contains 7600 mg/l acetic acid in methylene chloride, equivalent to the concentration found in the extract of a HDPE drum sample containing approximately 1% by weight acetic acid.

4. Further dilutions should be made, if necessary, to obtain a standard that closely matches the acetic acid concentration in the sample extract.

D. Calibration of the Gas Chromatograph

1. Inject 1 µl of the appropriate standard into the gas chromatograph.

2. Record the chromatogram and measure the area of the acetic acid peak which elutes at approximately 9.5 mins.

3. Figure 1 is a chromatogram and area report of a standard containing 4360 mg/l acetic acid in methylene chloride.

E. Analysis of a Sample Extract

1. Inject 1 µl of the sample extract into the gas chromatograph.

2. Record the chromatogram and measure the area of the acetic acid peak which elutes at approximately 9.5 mins.

IX. REPORT

Report the residual acetic acid content of the HDPE drum sample in either weight% or ppm by weight to three significant figures.

X. PRECISION AND ACCURACY

Precision data was obtained by analyzing five replicate extracts of a HDPE plaque sample containing approximately 0.6% by weight acetic acid. Five manual injections were made for each of the extracts and the standard. This analysis was repeated injecting the extracts with an autosampler. Table I is a summary of the precision data obtained in this study. The RSD was calculated for the five replicate injections of each extract and injection technique. The RSD average and range for each injection technique characterize its precision. The weight% acetic acid in each sample was calculated using the average of the five replicate injections. The method RSD for each injection technique is the variability of the resulting acetic acid concentrations.

Accuracy data for the method is not currently available. However, due to the moderately high volatility of acetic acid, the accuracy of this analysis will largely depend upon the sample history and timeliness of sample preparation and analysis.

XI. NOTES

1. As stated, the column temperature program requires a run time of 20 mins. However, presence of late-eluting compounds such as catalyst carrier hydrocarbons in the extract may require extending the final column temperature hold time and thus the overall run time.

XII. REFERENCES

Research Notebook No.: 2454
Pages: 163, 185

Author: William R. Behymer
Location: CRL

Table I

Injection Technique	Inj. Technique Avg. RSD and Range	Avg. Wt.% Acetic Acid	Method Precision(RSD)
Manual	.85%(.32-1.2%)	.605%	1.79%
Auto	.57%(.40-.79%)	.618%	1.19%

Figure 1

****** EXTERNAL STANDARD TABLE ******

********** 07-28-1988 16:19:28 Version 4.1 **********

* Sample Name: 5-12 #1 4360 mg/l Data File: W:HOAC2 *
* Date: 07-15-1988 15:46:37 Method: W:ACETIC 07-15-1988 15:56:42 # 4 *
* Interface: 4 Cycle#: 2 Operator WRB Channel#: 0 Vial#: N.A. *
* Starting Peak Width: 10 Threshold: .02 Area Threshold: 10 *

* Instrument Type: HP 5880A GC Column Type: 10ft 4% CRBWX 20M DA *
* Solvent Description: 30 cc/min Helium *
* Conditions: Column Prog. 80C for 2 mins + 10C/min to 200C *
* Detector 0: FID 230C Detector 1: N/A *
* Misc. Information: Inj. 210C, 1ul(#1701), %OFF=1, 2^0 *

Starting Delay: 0.00 Ending retention time: 20.00
Area reject: 0 One sample per 0.500 sec.
Amount injected: 1.00 Dilution factor: 1.00
Sample Weight: 1.000000

PEAK NUM	RET TIME	PEAK NAME	CONCENTRATION in ug/l	NORMALIZED CONC	AREA	HEIGHT	AREA/ HEIGHT	BL	REF PEAK	% DELTA RET TIME	CONC/AREA
1	6.288		0.0000	0.0000%	17986	2346	7.6	1			0.0000E+00
2	6.933		0.0000	0.0000%	129615	18157	7.1	1			0.0000E+00
3	9.567	Acetic Acid	4360.0000	100.0000%	2826899	401483	7.0	1	0	-.3472	1.5423E-03
4	11.580		0.0000	0.0000%	7444	1277	5.8	1			0.0000E+00

TOTAL AMOUNT = 4360.0000

Data File = W:HOAC2.PTS Printed on 07-28-1988 at 16:20:00
Start time: 0.00 min. Stop time: 20.01 min. Offset: -80 mv.
Full Range: 100 millivolts

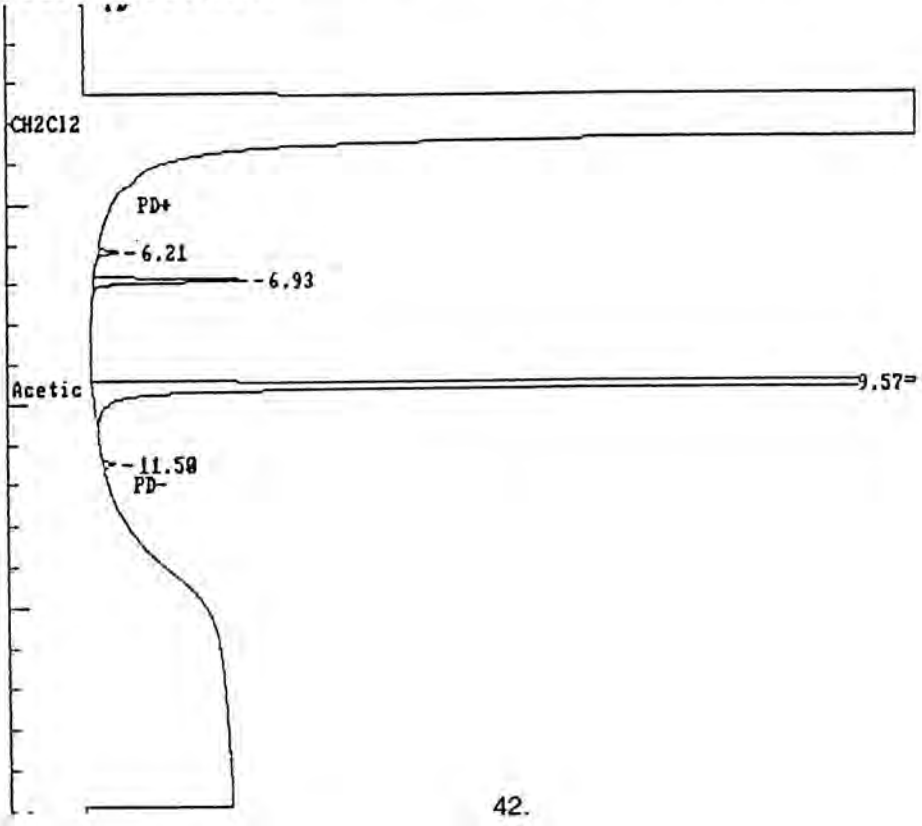


Figure 2

****** EXTERNAL STANDARD TABLE ******

********** 07-28-1988 16:21:59 Version 4.1 **********

* Sample Name: 2454-163-5 3.0283g Data File: W:HOAC3 *

* Date: 07-15-1988 16:19:34 Method: W:ACETIC 07-15-1988 15:56:42 # 4 *

* Interface: 4 Cycle#: 3 Operator WRB Channel#: 0 Vial#: N.A. *

* Starting Peak Width: 10 Threshold: .02 Area Threshold: 10 *

* Instrument Type: HP 5880A GC Column Type: 10ft 4% CRBWX 20M DA *

* Solvent Description: 30 cc/min Helium *

* Conditions: Column Prog. 80C for 2 mins + 10C/min to 200C *

* Detector 0: FID 230C Detector 1: N/A *

* Misc. Information: Inj. 210C, 1ul(#1701), %OFF=1, 2^0 *

Starting Delay: 0.00 Ending retention time: 20.00

Area reject: 0 One sample per 0.500 sec.

Amount injected: 1.00 Dilution factor: 1.00

Sample Weight: 1.000000

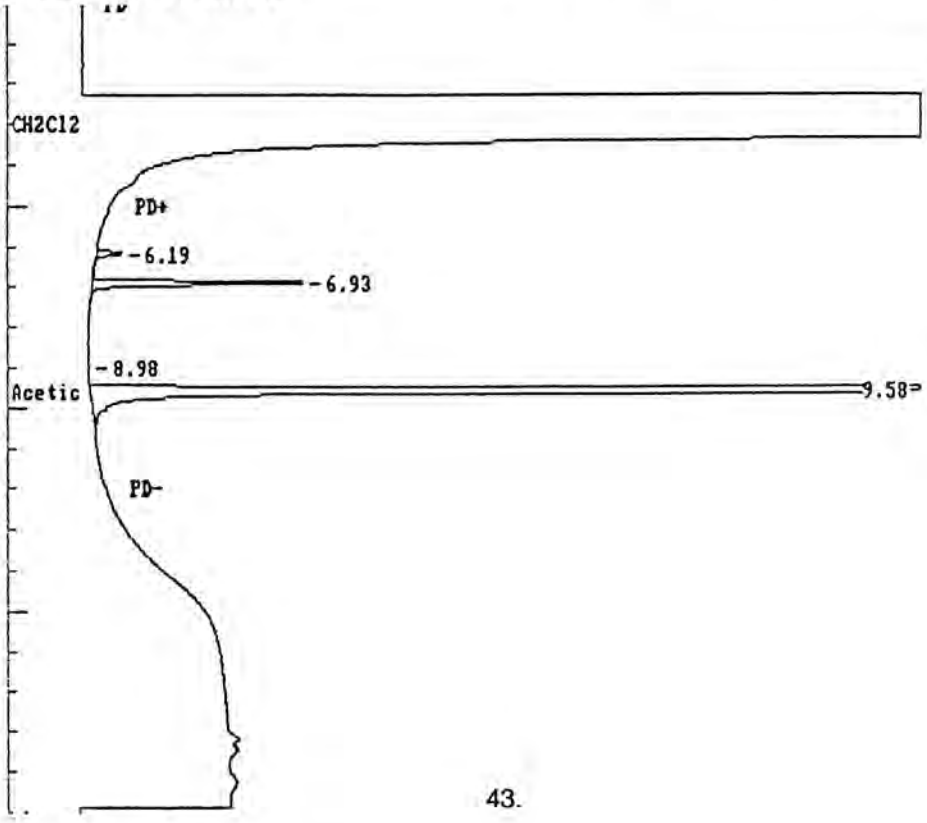
PEAK NUM	RET TIME	PEAK NAME	CONCENTRATION in ug/l	NORMALIZED CONC	AREA	HEIGHT	AREA/ HEIGHT	BL	REF PEAK	% DELTA RET TIME	CONC/AREA
1	6.192		0.0000	0.0000%	22826	3021	7.6	1			0.0000E+00
2	6.925		0.0000	0.0000%	182270	25090	7.3	1			0.0000E+00
3	8.975		0.0000	0.0000%	875	55	15.9	1			0.0000E+00
4	9.575	Acetic Acid	3986.4026	100.0000%	2532800	357943	7.1	1	0	-.2604	1.5423E-03

TOTAL AMOUNT = 3986.4026

Data File = W:HOAC3.PTS Printed on 07-28-1988 at 16:22:22

Start time: 0.00 min. Stop time: 20.01 min. Offset: -80 mv.

Full Range: 100 millivolts



HOECHST CELANESE CORPORATION
Technical Center
Corpus Christi, Texas

To: J. P. Parr - Bayport Works

November 29, 1988

From: N. M. Lamon
K. A. Fritch

ASR-77-88/NML
KAF-215-88

Methanol Analysis Scheme for High Density Polyethylene Samples from Bayport Works

A Gas Chromatography-Headspace method has been developed for the analysis of trace level concentrations of methanol in high density polyethylene samples. The method can detect methanol down to a 1 ppm concentration level. The Appendix contains the analysis procedure for this method.

The linearity and the repeatability evaluation studies of this method were performed on a Hewlett-Packard 5890A Gas Chromatography instrument equipped with a flame ionization detector and a Hewlett-Packard 19395A Headspace sampler unit. The data integration is performed on a Hewlett-Packard 3357 Laboratory Automation System computer.

Norma M. Lamon
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gl
Attachment

Keywords
Bayport Works
Methanol
Analysis
PPM
High
Density
Polyethylene
Gas Chromatography
Hewlett-Packard
Laboratory Automation System
S-1492
Instrument
Headspace
Method
Sampler
Linearity
Repeatability
Data

ANALYSIS SCHEME

OBJECTIVE: Determine trace levels of methanol (MEOH) in high density polyethylene (HDPE) samples derived from drum containers used to store methanol. Detection limits down to 5 ppm are desired.

INSTRUMENT: Hewlett-Packard 5890A Gas Chromatograph (GC) equipped with a flame ionization detector, and a Hewlett-Packard 19395A Headspace sampler unit.

I. Hewlett-Packard 5890A Gas Chromatograph Parameters:

Column: 30 meter x 0.25 mm I.D. DX-4 capillary column, 0.25 micron film thickness.

Temperature Program: Initial temperature: 40°C for 3 minutes.
Ramp rate: 10°C/minute.
Final temperature: 200°C for 10 minutes.

Equilibrium time: 0.5 minutes.

Injector temperature: 210°C.

Detector temperature: 280°C.

Carrier Gas: Helium, 0.74 ml/minute.

Split vent flow: 60 ml/minute.

Split ratio: 82:1

Auxillary gas: Nitrogen, 30 ml/minute.

Septum purge: Helium, 6 ml/minute.

Detector gases: Air: 300 ml/minute.
Hydrogen: 30 ml/minute.

Range: 2

Attenuation: 2

II. Hewlett-Packard 19395A Headspace Sampler Unit Parameters:

Equilibrium time: 30 minutes or more. The samples are equilibrated for 30 minutes and then the first sample is injected. The subsequent samples are equilibrated for 30 minutes plus the analysis time of the previous samples.

Bath temperature: 120°C.

Valve/loop temperature: 120°C.

Sample size: 1 ml sample loop.

Vial size: 10 ml.

Valve timing:

Probe: 0:01 seconds.

Press: 0:03 seconds.

Press: 0:33 seconds.

Vent: 0:38 seconds.

Vent: 0:43 seconds.

Inj: 0:44 seconds.

Inj: 37:00 minutes.

Probe: 37:01 minutes.

Carrier gas: Helium, 0.9 bar.

Auxillary gas: Helium, 1.5 bar.

Servo air: 3.5 bar.

III. Sample Preparation:

The HDPE samples are cut into small sample blocks (~5 mm x 5 mm x 7 mm).

1. Place ~0.5 gm HDPE sample and ~0.5 gm of decalin solvent (0.5 ml as measured from a 20 ml glass syringe) into a glass headspace vial.

2. Seal the headspace vial with a silicone septum and an aluminum crimp-style cap.

3. Place the sample vial into the headspace sampler tray unit. (The sampler tray is immersed into a silicone oil bath which is maintained at a temperature of 120°C. The sample will melt in the presence of decalin solvent at a temperature of 100°C.)

4. Allow the samples to sit for 30 minutes to allow for the equilibration of the methanol between the molten solid, the liquid, and the gas phases.

5. After equilibration, the headspace sampler unit pierces the septum of the sample vial and fills a 1.0 ml sample loop with the vapor phase which is transferred into the gas chromatography (GC) instrument via a heated transfer line (120°C).

IV. Method Evaluation:

The calibration standards will be conditioned in the same fashion as the test samples.

1. Prepare a control sample by combining decalin solvent with a blank HDPE sample which has not been exposed to methanol (~0.5 gm of blank HDPE sx and ~0.5 gm of decalin).

2. Make headspace injections of the control sample to determine if there are any interferences present that may elute at the same retention time as methanol. This area will be subtracted from the methanol area of all test samples and calibration standards.

3. Prepare a solution containing 11.6 ppm methanol in decalin solvent.

4. Make up five separate vials of a 50:50 mixture of the 11.6 ppm methanol solution and the blank HDPE sample to evaluate the repeatability of the method (~0.5 gm of the 11.6 ppm MEOH solution and ~0.5 gm of the blank HDPE sample in each vial). Table I shows the repeatability of these results. Figure 1 is the GC chromatogram of a 50:50 mixture sample.

5. Prepare methanol solutions at concentration levels of 1.2, 7.0, 11.6, and 15.4 ppm in decalin solvent.

6. Combine the solutions from Step IV.5. with blank HDPE sample to generate the calibration curve. Approximately 0.5 gm of each solution and ~0.5 gm of blank HDPE sample is placed in each vial. Prepare a total of 5 vials for each of the different methanol calibration standards. Table II contains the raw data obtained from this study. Figure 2 is the methanol linearity curve.

V. Statistical Evaluation of the Method:

1. The repeatability for five individual preparations of four different concentrations of blend is contained in Figure 2. The data of concentration versus area for nineteen points has been plotted and regressed as shown in Figure 2.

2. In Table III, the ratio of the concentrations versus areas have been determined for nineteen points. The standard deviation for the response factors or ratio is multiplied by the appropriate T factor (for df = n - 1 = 19 - 1 = 18) to arrive at the best approximation of the error in a sample result at the 95% confidence limits.

Calculations:

The sample result will be determined 5 times and each corrected area for methanol will be related to its concentration via the regression equation:

$$\text{conc ppm} = 0.544 + 0.00246 \text{ Area}$$

TABLE I

METHANOL REPEATABILITY STUDY
(11.6 PPM)

COMPONENT METHANOL
11
9.8
10.8
10.2
10

68% CONFIDENCE LEVEL.
AVERAGE 10.36

STD. DEV. .517687
COEFF.VAR. 4.99698

95% CONFIDENCE LEVEL
AVERAGE 10.36

STD.DEV. 1.4371
COEFF.VAR. 13.8716

STANDARD DEVIATION (STDV) AT 68% CONFIDENCE LEVEL:

$$s = \sqrt{\frac{\sum_{i=1}^{n}(x_i - \bar{x})^2}{n-1}}$$

Where:

Σ = the summation of all the values 1 thru n.

n = the number of data points evaluated.

$\bar{x}$ = average (mean) of the # values.

x_i = represents each value.

%RSDV = % relative standard deviation of the mean. (Coefficient of variation)

$$\text{COEFFICIENT OF VARIATION (\%RSDV)} = \frac{\text{Standard Deviation}}{\text{Mean}} \times 100$$

STDV (95% Conf. level) = STDV (68% Conf. level) x T value (n-1) from the table below

COEFF.VAR. (95% Conf.level) = COEFF.VAR.(68% Conf.level) x T value (n-1) from the table below

T Table for calculating the probability of data occurring at the 95% Confidence level

N degrees of freedom	T
1	12.71
2	4.30
3	3.18
4	2.78
5	2.57
6	2.45
7	2.37
8	2.31
9	2.26
10	2.23

TABLE II

LINEARITY STUDY OF MEOH IN DECALIN SOLVENT IN THE PRESENCE OF HDPE CONTROL SAMPLE

MEOH CONC.	MEOH SOLN.	HDPE WT.	MEOH AREA*	CORRECTED MEOH AREA**
1.2 ppm - 1	0.5722 gm	0.5264 gm	408	355
- 2	0.5424 gm	0.5186 gm	405	352
- 3	0.5458 gm	0.5192 gm	410	357
- 4	0.5293 gm	0.5120 gm	396	343
- 5	0.5121 gm	0.5188 gm	389	336
AVERAGE	0.5404 gm	0.5190 gm	402	349
7.0 ppm - 1	0.5877 gm	0.5106 gm	3120	3067
- 2	0.5142 gm	0.5214 gm	2657	2604
- 3	0.5601 gm	0.5275 gm	2972	2919
- 4	0.5205 gm	0.5237 gm	2705	2652
- 5	0.5856 gm	0.5129 gm	2998	2945
AVERAGE	0.5536 gm	0.5192 gm	2890	2837
11.6 ppm - 1	0.5551 gm	0.5220 gm	4292	4239
- 2	0.5191 gm	0.5096 gm	3822	3769
- 3	0.5590 gm	0.5225 gm	4226	4173
- 4	0.5146 gm	0.5063 gm	3986	3933
- 5	0.5071 gm	0.5096 gm	3899	3846
AVERAGE	0.5310 gm	0.5140 gm	4065	3992
15.4 ppm - 1	0.5332 gm	0.5115 gm	6616	6563
- 2	0.5063 gm	0.5336 gm	6235	6182
- 3	0.5097 gm	0.5149 gm	6302	6249
- 4	0.6113 gm	0.5025 gm	7490	7437
- 5	0.5173 gm	0.5006 gm	6197	6144
AVERAGE	0.5356 gm	0.5126 gm	6568	6515

* The control sample impurity has not been subtracted from the methanol area.

** The methanol area is corrected by subtracting out the control sample impurity (average of four injections = 53).

TABLE III

RATIO OF THE CONCENTRATIONS VERSUS AREAS

ROW	Conc ppm	Area	Ratio	Conc ppm	Area	Ratio	Conc ppm	Area	Ratio	Conc ppm	Area	Ratio
1	1.2	355	0.0033803	7.0	3067	0.0022824	11.6	4239	0.0027365	15.4	6563	0.0023465
2	1.2	352	0.0034091	7.0	2604	0.0026882	11.6	3769	0.0030777	15.4	6182	0.0024911
3	1.2	357	0.0033613	7.0	2919	0.0023981	11.6	4173	0.0027798	15.4	6249	0.0024644
4	1.2	343	0.0034985	7.0	2652	0.0026395	11.6	3933	0.0029494	15.4	6144	0.0025065
5	1.2	336	0.0035714	7.0	2945	0.0023769	11.6	3846	0.0030161	---	---	---

N	19
MEAN	0.00284
STDEV *	0.00090
%RSDEV *	31.7

* At the 95% confidence level.

FIGURE 1

GC CHROMATOGRAM OF A 11.6 PPM MEOH STANDARD

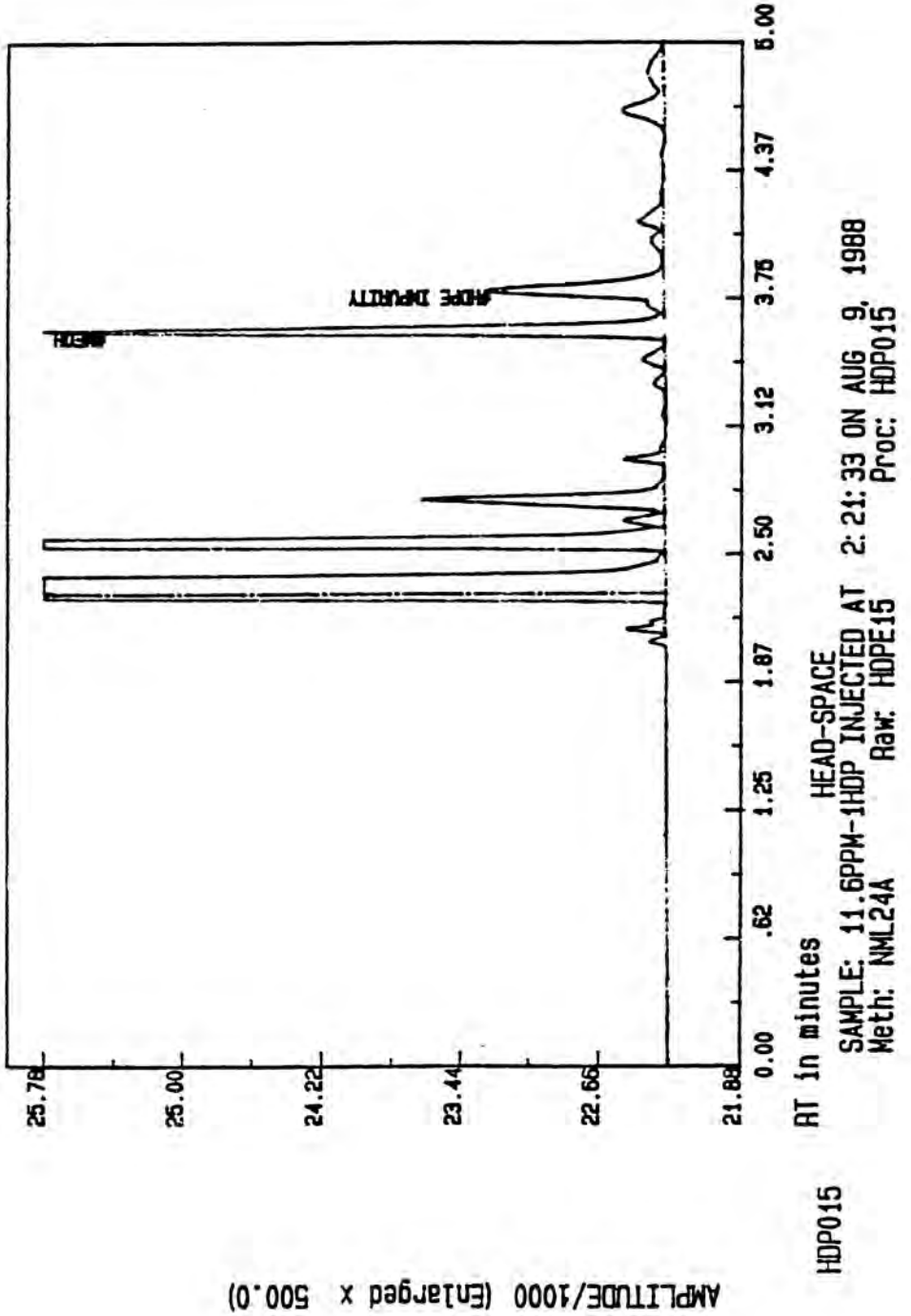

FIGURE 2

METHANOL LINEARITY STUDY
METHANOL STATISTICAL EVALUATION AT THE 95% CONFIDENCE LEVEL

ROW	1.2 ppm	area 1.2	7.0 ppm	area 7.0	11.6 ppm	area 11.6	15.4 ppm	area 15.4
1	1.2	355	7.0	3067	11.6	4239	15.4	6563
2	1.2	352	7.0	2604	11.6	3769	15.4	6182
3	1.2	357	7.0	2919	11.6	4173	15.4	6249
4	1.2	343	7.0	2652	11.6	3933	15.4	6144
5	1.2	336	7.0	2945	11.6	3846	---	---
N	5	5	5	5	5	5	4	4
MEAN	1.2	348.6	7.0	2837.4	11.6	3992.0	15.4	6284.5
STDEV	0.0	24.6	0.0	555.7	0.0	570.2	0.0	606.4
%RSDV		7.0		19.6		14.3		9.6

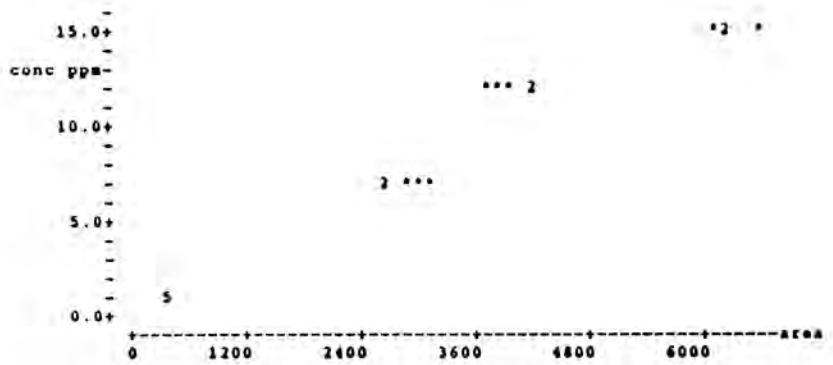

Fig. 2 Methanol linearity study

The regression equation is conc ppm = 0.544 + 0.00246 area

Predictor	Coef	Stdev	t-ratio	p
Constant	0.5438	0.3724	1.46	0.162
area	0.00246227	0.00009718	25.34	0.000

s = 0.8850 R-sq = 97.4% R-sq(adj) = 97.3%

Analysis of Variance

SOURCE	DF	SS	MS	F	p
Regression	1	502.83	502.83	641.98	0.000
Error	17	13.32	0.78		
Total	18	516.15			

Unusual Observations

Obs.	area	conc ppm	Fit	Stdev.Fit	Residual	St.Resid
12	3769	11.600	9.824	0.210	1.776	2.07R

R denotes an obs. with a large st. resid.

PROCEDURE FOR ANALYZING LADINGS ABSORBED IN POLYETHYLENE DRUMS

1.) Place 1 gram of ground polymer in a suitable container for solvent extraction. Place 20 ml of the extraction solvent, in this case methylene chloride, in the container and place on a shaker for one hour.

2.) Analyze the extraction solvent by gas chromatography. In this study a gas chromatograph with a 25 meter capillary column with methyl silicone as the stationary phase was used. The GC conditions were:

Initial temperature:	10 deg. C
Final temperature:	325 deg. C
Program rate:	15 deg./min.
Injector temperature:	325 deg. C
Detector Temperature:	350 deg. C

3.) The extraction runs are compared to standards. The standards used in this study were dilutions in methylene chloride of the engine oil and transformer oil type that was used in the second lading.

APPENDIX C

PART II
STRUCTRUAL INTEGRITY:
Analytical Results

HOECHST CELANESE CORPORATION
Technical Center
Corpus Christi, Texas

To: L. E. Wade

April 18, 1989

From: N. M. Lamon
K. A. Fritch

ASR-83-89/NML
KAF-219-89

Methanol Analysis Scheme and Sample Data Results for High Density Polyethylene Samples from Bayport Works

A round-robin testing program was initiated to determine the compatibility of high density polyethylene drum liners with various solvents. The program was supported with analytical labs from multiple companies. The CCTC provided the analytical support for Bayport Works during this program.

A Gas Chromatographic-Headspace method was used for the determination of trace level concentrations of methanol in high density polyethylene samples. The methanol detection limit for this method is ~0.8 ppm. The appendix contains the analysis procedure and the sample analysis results obtained with this method.

The linearity and the repeatability evaluation studies of this method were performed on a Hewlett-Packard 5890A Gas Chromatography instrument equipped with a flame ionization detector and a Hewlett-Packard 19395A Headspace sampler unit. The data integration is performed on a Hewlett-Packard 3357 Laboratory Automation System computer.

The six samples received from Bayport Works each contained less than 5 ppm methanol as shown in Table V.

Norma M. Lamon
N. M. Lamon

K. A. Fritch
K. A. Fritch

gl
Attachment

Keywords
Bayport Works
Methanol
Headspace
Gas Chromatography
Hewlett-Packard
Laboratory Automation System
S-1492
Density
Polyethylene
Analysis
Sampler
PPM
High
Instrument
Data
Method
Linearity
Repeatability

"QUALITY AND TEAMWORK–A WINNING COMBINATION"

APPENDIX

METHANOL ANALYSIS SCHEME AND SAMPLE DATA RESULTS

ANALYSIS SCHEME

Objective: Determine trace levels of methanol (MEOH) in high density polyethylene (HDPE) samples derived from drum containers used to store methanol. Detection limits down to 5 ppm are desired.

Instrument: Hewlett-Packard 5890A Gas Chromatograph (GC) equipped with a flame ionization detector, and a Hewlett-Packard 19395A Headspace sampler unit.

I. Hewlett-Packard 5890A Gas Chromatograph parameters:

Column: 30 meter x 0.25 mm I.D. DX-4 capillary column, 0.25 micron film thickness.

Temperature program:

Initial temperature: 40°C for 3 minutes.

Ramp rate: 10°C/minute.

Final temperature: 200°C for 10 minutes.

Equilibrium time: 0.5 minutes.

Injector temperature: 210°C.

Detector temperature: 280°C.

Carrier Gas: Helium, 0.74 ml/minute.

Split vent flow: 51 ml/minute.

Split ratio: 69:1

Auxillary gas: Nitrogen, 30 ml/minute.

Septum purge: Helium, 6 ml/minute.

Detector gases:

Air: 300 ml/minute.
Hydrogen: 30 ml/minute.

Range: 2

Attenuation: 2

II. Hewlett-Packard 19395A Headspace Sampler Unit Parameters:

Equilibrium time: 30 minutes or more. The samples are equilibrated for 30 minutes and then the first sample is injected. The subsequent samples are equilibrated for 30 minutes plus the analysis time of the previous sample.

Bath temperature: 120°C.

Valve/loop temperature: 120°C.

Sample size: 1 ml sample loop.

Vial size: 10 ml.

Valve timing:

Probe: 0:01 seconds.

Press: 0:03 seconds.

Press: 0:33 seconds.

Vent: 0:38 seconds.

Vent: 0:48 seconds.

Inj: 0:49 seconds.

Inj: 1:49 seconds.

Probe: 1:50 seconds.

Carrier gas: Helium, 0.9 bar.

Auxillary gas: Helium, 1.5 bar.

Servo air: 3.5 bar.

III. Sample preparation: The HDPE samples are cut into small sample blocks (~5 mm x 5 mm x 7 mm)

1. Place ~0.5 gm HDPE sample and ~0.5 gm of decalin solvent (0.5 ml as measured from a 20 ml glass syringe) into a glass headspace vial.

2. Seal the headspace vial with a silicone septum and an aluminum crimp-style cap.

3. Place the sample vial into the headspace sampler tray unit. (The sampler tray is immersed into a silicone oil bath which is maintained at a temperature of 120°C. The sample will melt in the presence of decalin solvent at a temperature of 100°C.)

4. Allow the samples to sit for 30 minutes to allow for the equilibration of the methanol between the molten solid, the liquid, and the gas phases.

5. After equilibration, the headspace sampler unit pierces the septum of the sample vial and fills a 1.0 ml sample loop with the vapor phase which is transferred into the gas chromatography (GC) instrument via a heated transfer line (120°C).

IV. Method evaluation:

The calibration standards will be conditioned in the same fashion as the test samples.

1. Prepare a control sample by combining decalin solvent with a blank HDPE sample which has not been exposed to methanol. (~0.5 gm of blank HDPE sample and ~0.5 gm of decalin)

2. Make headspace injections of the control sample to determine if there are any interferences present that may elute at the same retention time as methanol. This area will be subtracted from the methanol area of all test samples and calibration standards.

3. Prepare a solution containing 9.8 ppm methanol in decalin solvent.

4. Make up five separate vials of a 50:50 mixture of the 9.8 ppm methanol solution and the blank HDPE sample to evaluate the repeatability of the method (~0.5 gm of the 9.8 ppm MEOH solution and ~0.5 gm of the blank HDPE sample in each vial). Table I shows the repeatability of these results. Figure 1 is the GC chromatogram of a 50:50 mixture sample.

5. Prepare methanol solutions at concentration levels of 0.768, 6.3, 9.8, and 17.6 ppm in decalin solvent.

6. Combine the solutions from Step IV.5 with blank HDPE sample to generate the calibration curve. Approximately 0.5 gm of each solution and ~0.5 gm of blank HDPE sample is placed in each vial. Prepare a total of five vials for each of the different methanol calibration standards. Table II contains the raw data obtained from this study. Figure 2 is the methanol linearity curve.

V. Statistical evaluation of the method:

1. The repeatability for five individual preparations of four different concentrations of blend is contained in Table II. The data of concentration versus area for 20 points has been plotted and regressed as shown in Figure 2.

2. In Table III, the ratio of the concentrations versus areas have been determined for 20 points. The standard deviation for the response factors or ratio is multiplied by the appropriate T factor (for df = n - 1 = 20 - 1 = 19) to arrive at the best approximation of the error in a sample result at the 95% confidence limits.

VI. Calculations:

The sample result will be determined five times and each corrected area for methanol will be related to its concentration via the regression equation:

$$\text{conc ppm} = 1.08 + 0.00187\ \text{AREA}$$

VII. Sample analysis results:

Table IV contains the HDPE sample analysis raw data. The analysis results are statistically evaluated at the 95% confidence level in Table V.

TABLE I

METHANOL REPEATABILITY STUDY
(9.8 PPM)

COMPONENT METHANOL

9.9
9.9
10.2
10.4
9.6

68% CONFIDENCE LEVEL.
AVERAGE 10

STD. DEV.	.308221
COEFF.VAR.	3.08221

95% CONFIDENCE LEVEL
AVERAGE 10

STD.DEV.	.855621
COEFF.VAR.	8.55621

FIGURE 1

GC CHROMATOGRAM OF A 9.8 PPM MEOH STANDARD

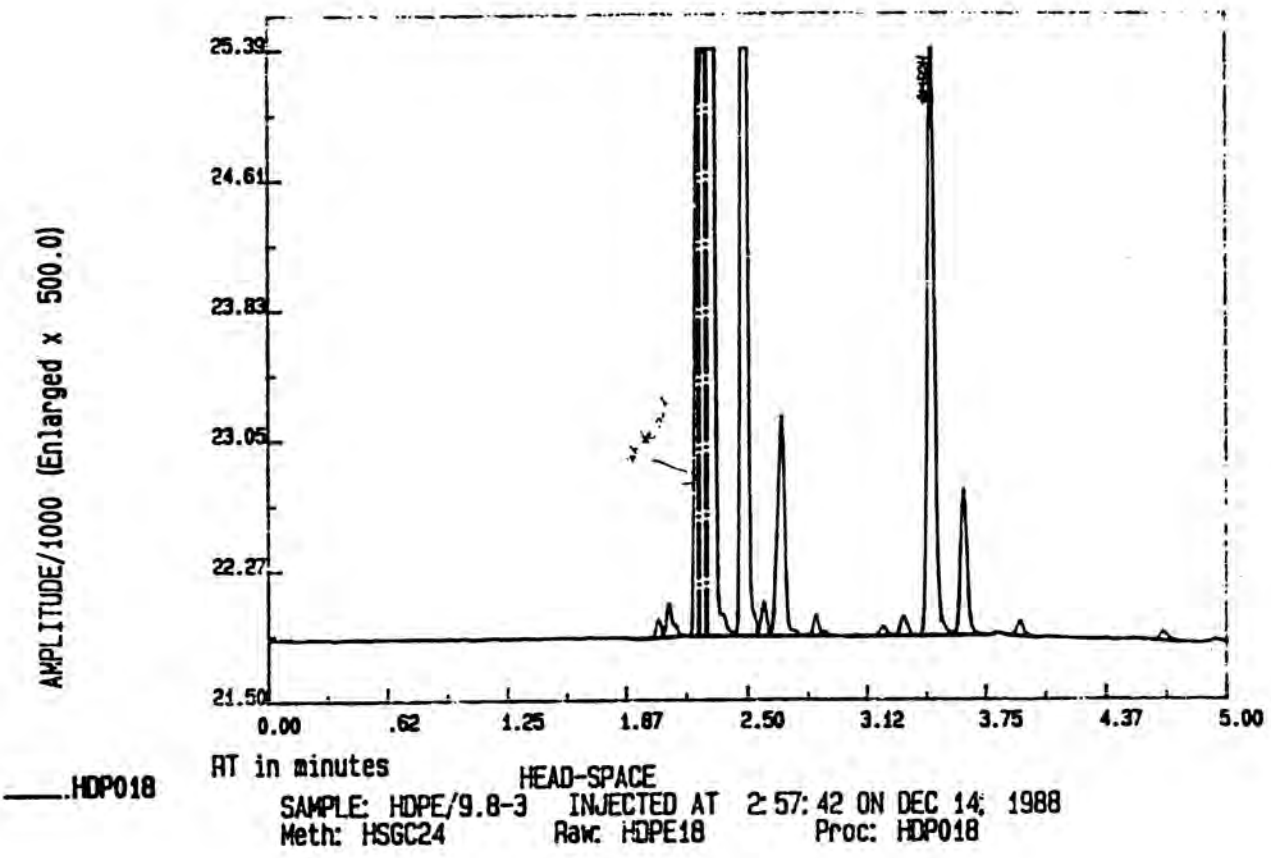

FIGURE 2

METHANOL LINEARITY STUDY

METHANOL STATISTICAL EVALUATION AT THE 95% CONFIDENCE LEVEL

ROW	0.768 ppm	area 0.768	6.3 ppm	area 6.3	9.8 ppm	area 9.8	17.6 ppm	area 17.6
1	0.768	326	6.3	2183	9.8	4784	17.6	9689
2	0.768	290	6.3	2146	9.8	4698	17.6	8475
3	0.768	335	6.3	1996	9.8	4887	17.6	9016
4	0.768	356	6.3	2272	9.8	4996	17.6	8995
5	0.768	315	6.3	2086	9.8	4559	17.6	4246
N	5	5	5	5	5	5	5	5
MEAN	0.768	324.4	6.3	2136.6	9.8	4768.6	17.6	8868.2
STDEV	0.0	67.8	0.0	267.5	0.0	478.4	0.0	1474.6
%RSDEV		20.9		13.4		10.0		16.6

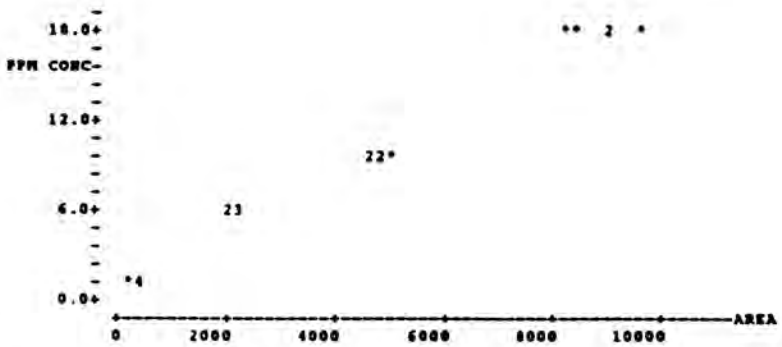

Fig. 2 Methanol linearity study

The regression equation is PPM CONC = 1.08 + 0.00187 AREA

Predictor	Coef	Stdev	t-ratio	p
Constant	1.0768	0.3420	3.15	0.006
AREA	0.00187359	0.00006634	28.24	0.000

s = 0.9559 R-sq = 97.8% R-sq(adj) = 97.7%

Analysis of Variance

SOURCE	DF	SS	MS	F	p
Regression	1	728.90	728.90	797.71	0.000
Error	18	16.45	0.91		
Total	19	745.35			

QUANTUM CHEMICAL CORPORATION
USI DIVISION

INTEROFFICE CORRESPONDENCE

To: John Bergerhouse (RML) Date: December 15, 1988

cc: William Behymer (CRL) File: BERG1215

Subject: Analysis of PDI Drum Samples for Residual Acetic Acid

Copies: P. Hanik (RML)
R. Henkel (RML)
I. Peat (CRL)
R. Vordenberg (CRL)

The analysis of the eleven drum samples received in November for residual acetic acid is complete. Approximately 1/8 to 1/4 in. cubes cut from each of the drum wall samples were extracted overnight in methylene chloride. The acetic acid concentration in the extracts, determined by Gas Chromatography, was then related by weight to the original drum samples.

The first of the eleven samples was labelled #6 or #9 whose second lading was acetic acid or mineral spirits. The chromatogram of this sample contained a large group of peaks whose overall pattern was nearly identical to those of samples #33 and #34, both of which had been laden with mineral spirits. From this information, it was concluded that the sample sent to us was actually #6, which had been laden with mineral spirits, and not #9.

The following is a summary of the results obtained from the analysis of the eleven drum wall samples:

Sample	First Lading	Second Lading	ppm Acetic Acid
# 6	Methacrylic Acid	Mineral Spirits	3 ppm
#10	Methacrylic Acid	Acetic Acid	96 ppm
#21	Methanol	Acetic Acid	5 ppm
#22	Methanol	Acetic Acid	74 ppm
#33	Mineral Spirits	Acetic Acid	70 ppm
#34	Mineral Spirits	Acetic Acid	5 ppm
#45	Sulfuric Acid	Acetic Acid	83 ppm
#46	Sulfuric Acid	Acetic Acid	72 ppm
#58	Acetic Acid	Acetic Acid	97 ppm
#69	SAE 30 Motor Oil	Acetic Acid	49 ppm
#70	SAE 30 Motor Oil	Acetic Acid	14 ppm

As requested, a migration or concentration gradient study was also conducted using sample #58 whose first and second ladings were acetic acid. The sample was milled in such a way as to obtain sections of the outer, middle and inner thirds of the approximately 1/4 in. thick drum wall. Due

DOW CHEMICAL U.S.A.

Building B-1607
TEXAS OPERATIONS
FREEPORT, TEXAS 77541

January 10, 1989

J. E. Bergerhouse
Quantum
USI Division
3100 Golf Road
Rolling Meadows, IL 60008

Dear Mr. Bergerhouse:

Upon request by the Reuse Committee of The Plastic Drum Institute, twelve (12) 55-gallon drums were analyzed for residual traces of sulphuric acid. When high density polyethylene is exposed to sulphuric acid, a chemical reaction takes place in which sulphur atoms are incorporated in the chemical structure of the polyethylene. The reaction is commonly called sulfonation.

Three coupons were cut from the side walls of each drum. Each coupon was analyzed for the amount of surface sulfonation using x-ray fluorescence. The unit of measure is micrograms of sulphur per square inch of sample and the accuracy of the test is ±10% of the measured value. For reference purposes, an average automotive gas tank that has been properly sulphonated will contain a barrier layer that contains approximately 250 to 300 micrograms of sulphur per square inch.

Regards,

Joseph M. Tanner

Joseph M. Tanner
Polyethylene TS&D
Plastics Department

chg/Attachment

TECHNICAL INFORMATION

CONCENTRATION OF SULPHUR IN THE WALLS OF 55-GALLON DRUMS

DRUM NUMBER	SULPHUR LEVEL (Micrograms/sq. in.) SAMPLE #1	#2	#3	AVERAGE SULPHUR LEVEL (Micrograms/sq. in.)
7	18	26	41	28
8	41	34	58	44
19	21	18	20	20
20	24	25	24	24
31	42	33	45	40
32	31	32	33	32
43	16	17	16	16
44	17	18	19	18
55	16	16	16	16
56	21	21	26	23
67	29	27	35	30
68	22	24	24	23

JMT:chg
1/89

See reverse side

OLEFIN AND STYRENE PLASTICS DEPARTMENT
THE DOW CHEMICAL COMPANY • MIDLAND, MICHIGAN 48674

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Form No. _____

CONTAINER CORPORATION OF AMERICA

AN AFFILIATE OF JEFFERSON SMURFIT CORPORATION

Plastics Division
1204 EAST 12TH STREET
WILMINGTON, DE 19802

February 3, 1989

Mr. John Bergerhouse
USI Division
Quantum Chemical Corp.
3100 Golf Rd.
Rolling Meadows, IL 60008

Dear John:

I have attached the results of our analysis of the drums returned to us after the reconditioning and drop testing that was carried out at Bakerstown Container.

We did not use the test procedure using the TGA that I sent to you in March, 1988. The reason is that we first ran a quick loss-on-heating test in a standard lab hot air oven and found a significant level of volatile materials which indicated we did not need the added sensitivity available by using TGA.

We used a two stage procedure as follows:

1. A sample approximately 3" x 3" was taken from each drum and weighed. The weights ranged from 40-60 grams.

2. The sample was placed in a 100°C hot air oven for one hour.

3. After one hour, the sample was removed, cooled and re-weighed.

4. Percent loss at 100°C was calculated.

5. The oven temperature was raised to 130°C and the samples heated for an additional hour at this temperature.

6. The samples were again cooled and re-weighed with the additional percent loss calculated.

7. Total hours was obtained by adding the loss at 100°C and 130°C.

The results we obtained are consistent with previous values we have found for hydrocarbons stored in polyethylene containers.

If you have any questions regarding the attached results, please give me a call.

Very truly yours,

CONTAINER CORPORATION OF AMERICA/
PLASTICS DIVISION

Earl V. Lind
Manager
Materials & Systems Engineering

EVL:jr
8889C

cc: J. A. Geyer

Attachment

LOSS-ON-HEATING TEST RESULTS
PDI REUSE PROGRAM

DRUM #	MANUFACTURER	COLOR	LADING 1	LADING 2	% - 100°C	% - 130°C	TOTAL
5	Sonoco Plstics	Blue	Acrylic	Mineral	0.45	0.68	1.13
6	Drum	Blue	Acid	Spirits	0.06	0.10	0.16
29	CCA	Black	Mineral	Mineral	0.25	0.43	0.68
30	CCA	Blue	Spirits	Spirits	0.51	0.82	1.33
54	Hunter	Blue	Acetic Acid	Mineral Spirits	0.29	0.58	0.87
66	Eastern	Black	SAE 40	Mineral Spirits	0.48	0.73	1.21

These are the only drums that we received. The results appear to be consistent with previous data we have obtained using hydrocarbons with HMWHDPE, except for Drum #6.

We have not tried to determine the reason for the lower value found for this drum.

EVL:jr
8889C

SAMPLE DESCRIPTION	INNER OIL IN POLYMER WT%	MIDDLE OIL IN POLYMER WT%	OUTER OIL IN POLYMER WT%
#11 Blue PE	transformer oil 1.86%	transformer oil 0.31%	transformer oil 0.18%
#23 Black PE	transformer oil 1.39%	transformer oil 0.29%	transformer oi 0.16%
#24 Black PE	engine oil 0.86%	engine oil 0.22%	transformer oil 0.48%
#35 Black PE	transformer oil 2.06% (mineral spirits)	transformer oil 1.21% (has bigger area mineral spirits)	transformer oil 0.18% (has mineral spirits)
#36 Blue PE	engine oil 1.86% (mineral spirits)	engine oil 0.37%	transformer oil 0.51% (has mineral spirits)
#47 Dk Blue PE	engine oil 0.66%	engine oil 0.01%	transformer oil 0.11%
#48 Dk Blue PE	transformer oil 1.39%	transformer oil 0.40%	transformer oil 0.04%
#59 Dk Blue PE	transformer oil 0.80%	transformer oil 0.14%	transformer oil 0.05%
#60 Dk Blue PE	engine oil 0.93%	engine oil 0.02%	transformer oil 0.06%
#71 Black PE	engine oil 0.99%	engine oil 0.03%	transformer oil 0.11%
#72 Blue PE	transformer oil 1.32%	transformer oil 0.19%	transformer oil 0.03%

Data from Phillips Petroleum

The PDI Real World Test Part II

The Resin Reconditioning Study

A Special Purpose Group of
The Society of the Plastics Industry, Inc.
1275 K Street, NW, Ste. 400
Washington, DC 20005
(202) 371-5200

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.

The Plastic Drum Institute (PDI)
The Society of the Plastics Industry, Inc.
1275 K Street, NW, Suite 400
Washington, DC 20005
(202)371-5200

Copies can be ordered through SPI Literature Sales Department at (202) 371-5256, or (800) 541-0736 (Visa/Mastercard orders only). Order # AV-111; $10.00 for members, $20.00 for non-members.

ACKNOWLEDGEMENTS

The Plastic Drum Institute wishes to acknowledge and thank the following companies for their assistance in this study:

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.

Plastic Drum Institute
Technical Committee

John Bergerhouse, Chairman	Quantum Chemical
Don Belcher	Dow Chemical
D'Jaris Richardson	Fina Corporation
Ken Ford	Mobil Chemical
Nancy Conley	Novacor Chemical
Don Loti	Paxon Polymer
John Rathman	Phillips 66
Stan Sandler	Rieke Canada
Earl Lind	Russell-Stanley
John Malik	Sonoco Plastic Drum

PDI REAL WORLD TEST PART 2

"RESIN RECONDITIONING STUDY"

TABLE OF CONTENTS

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 ladings 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 ladings determined through quality control analysis upon the ladings 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 ladings packaged remained in the reconditioned drums. Most shippers concluded that the reconditioned plastic drums were acceptable for the ladings used. Also reconfirmed was that the ladings 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 ladings 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 ladings 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:

Lading	Test Method	Laboratory
Hydraulic Oil	Extraction GC	Mobil Chemical
Acrylic Acid	GC Chromotograpy	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 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 decrepancies 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® text. 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 Regrind				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

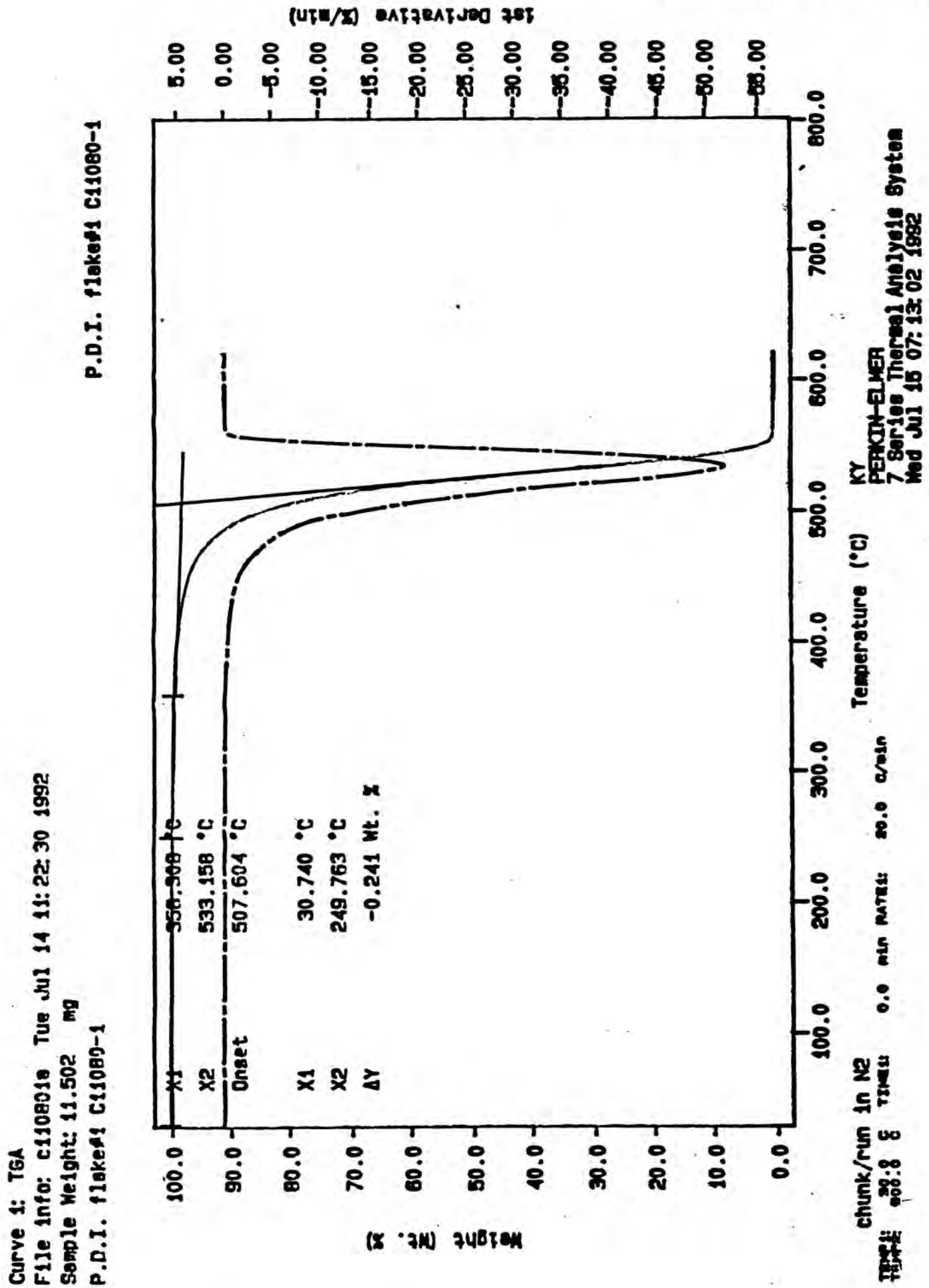
FLAKE = UNWASHED PELLET = WASHED

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

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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File Info: C11080ia Tue Jul 14 11:22:30 1992
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P.D.I. flake#1 C11080-1
X1 350.308 °C
X2 533.158 °C
Onset 507.604 °C
X1 30.740 °C
X2 249.763 °C
ΔY -0.241 Wt. %
Weight (Wt. %)
1st Derivative (%/min)
Temperature (°C)
PERKIN-ELMER
7 Series Thermal Analysis System
Wed Jul 15 07:13:02 1992
chunk/run in N2

Curve 1: TGA
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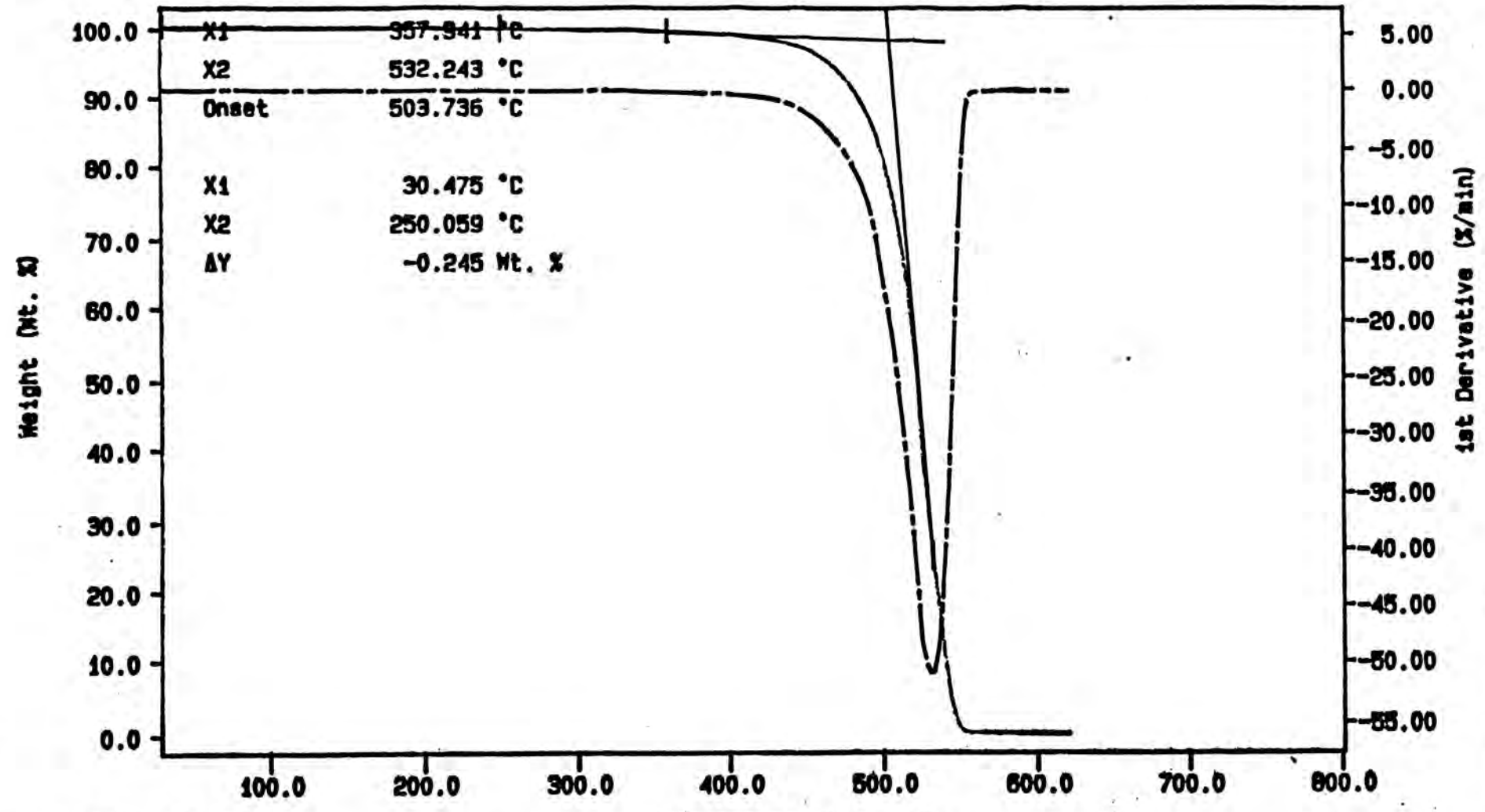
P.D.I. flake#1 C11080-1



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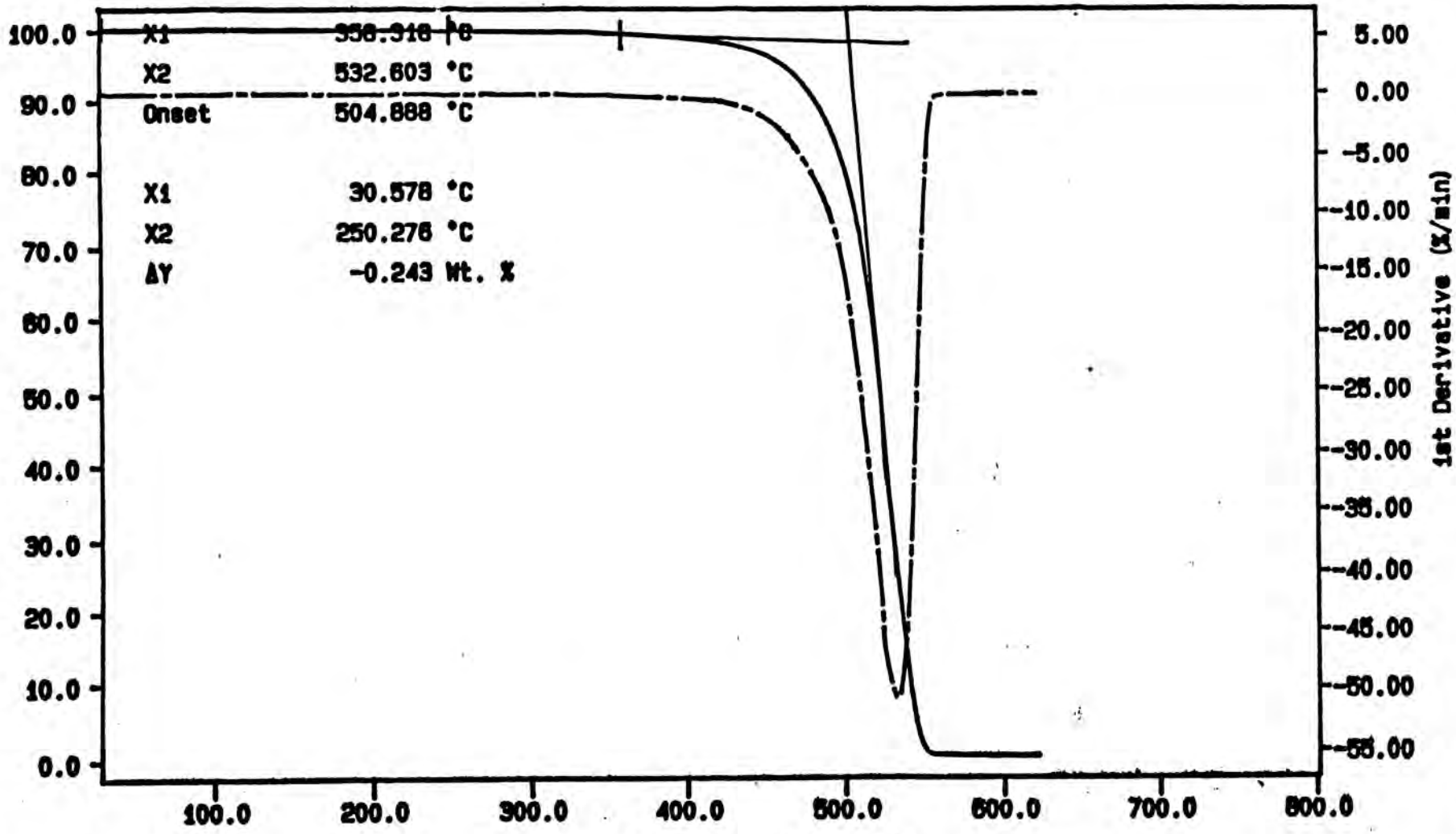
TEMP2: 600.0 C

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. flake#1 C11080-1

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



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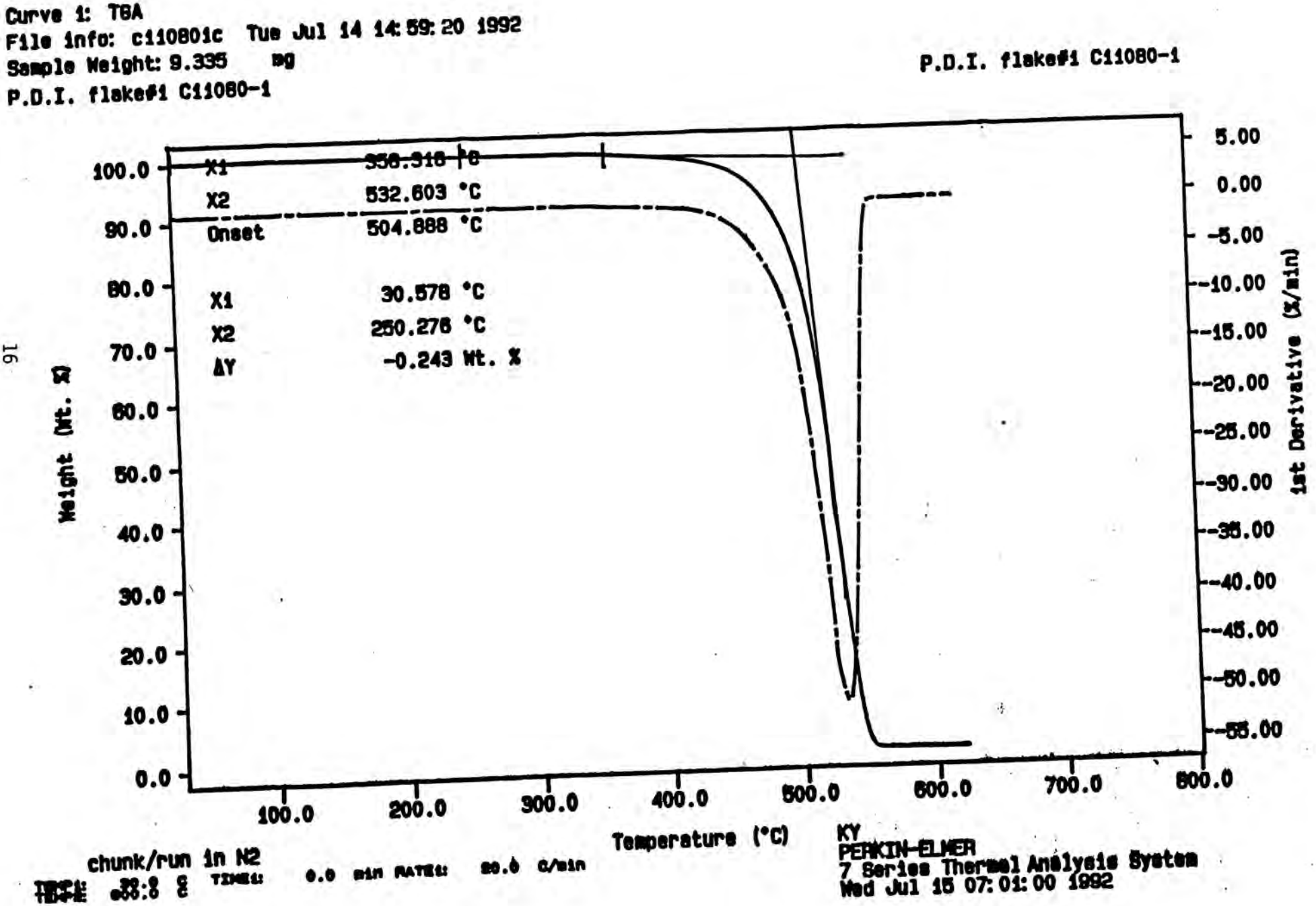
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P.D.I. flake#1 C11080-1
X1 358.318 °C
X2 532.603 °C
Onset 504.888 °C
X1 30.578 °C
X2 250.276 °C
ΔY -0.243 Wt. %
Weight (Wt. %)
1st Derivative (%/min)
Temperature (°C)
chunk/run in N2
TIME: 0.0 min RATE: 20.0 C/min
KY
PERKIN-ELMER
7 Series Thermal Analysis System
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Sample Weight: 10.650 mg
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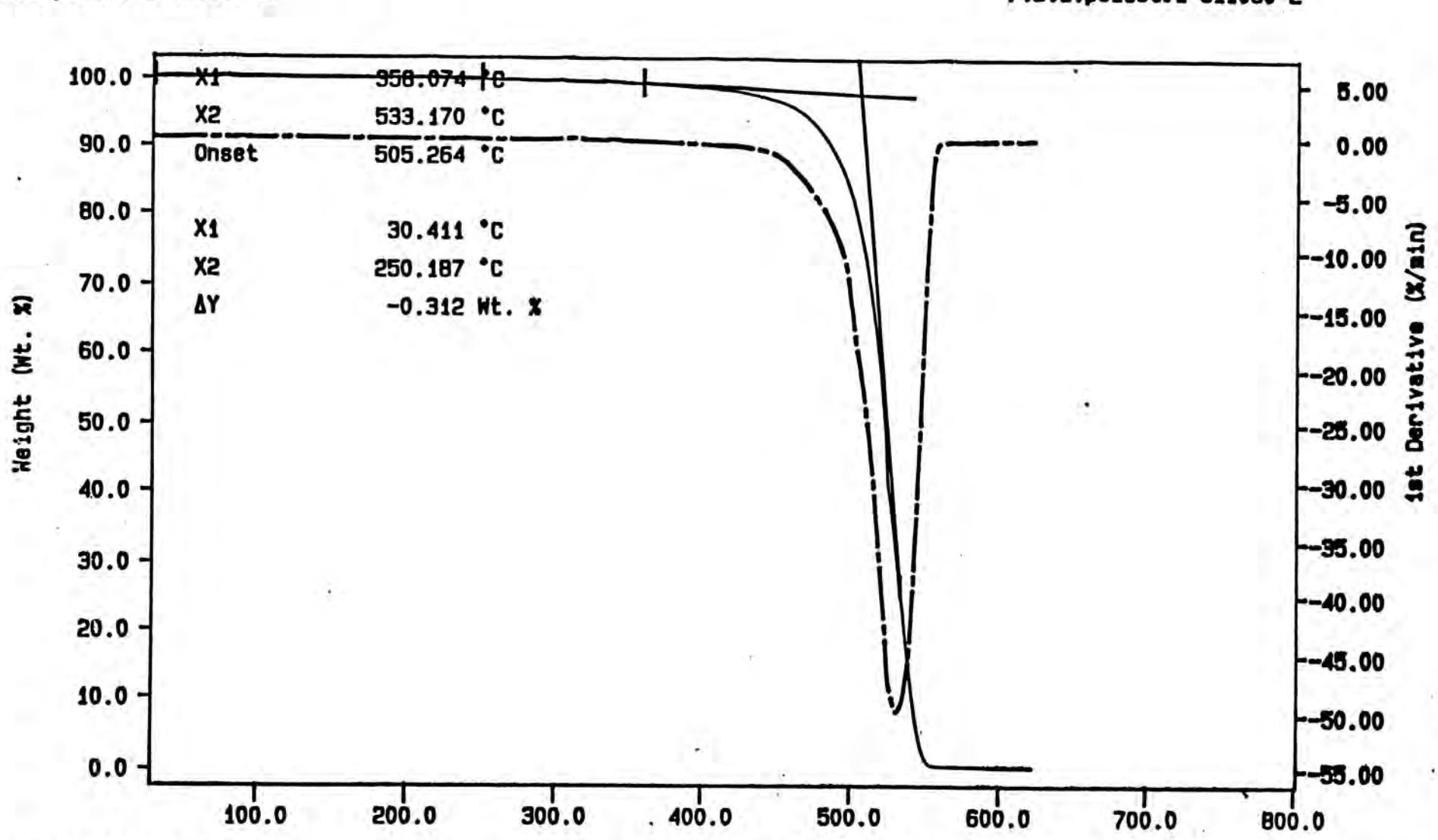

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TEMP2: 600.0 C

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PERKIN-ELMER
7 Series Thermal Analysis System
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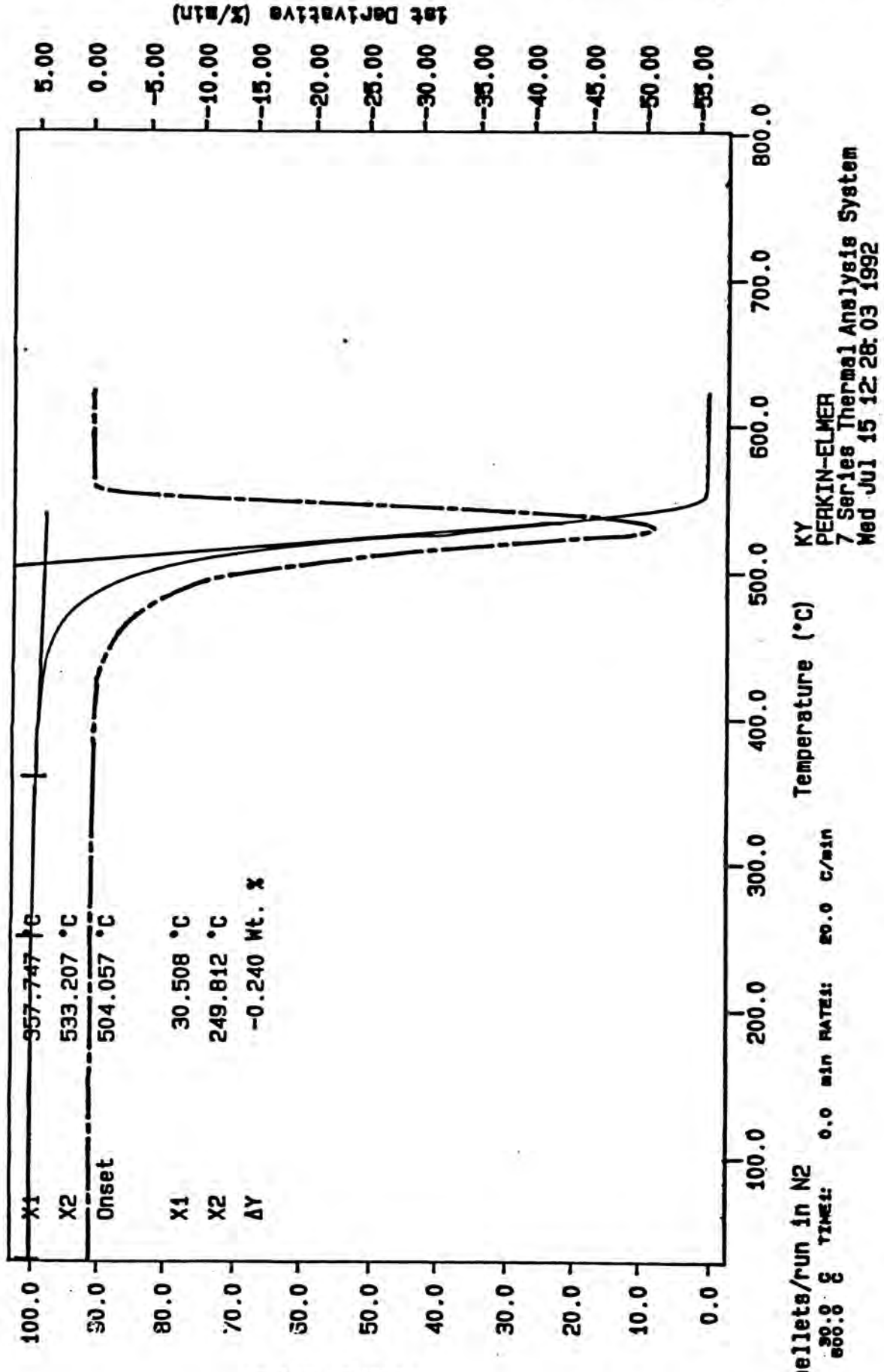
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P.D.I.pellet#1 C11080-2
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X2 533.207 °C
Onset 504.057 °C
X1 30.508 °C
X2 249.812 °C
ΔY -0.240 Wt. %
Weight (%)
1st Derivative (%/min)
Temperature (°C)
pellets/run in N2
KY
PERKIN-ELMER
7 Series Thermal Analysis System
Wed Jul 15 12:28:03 1992

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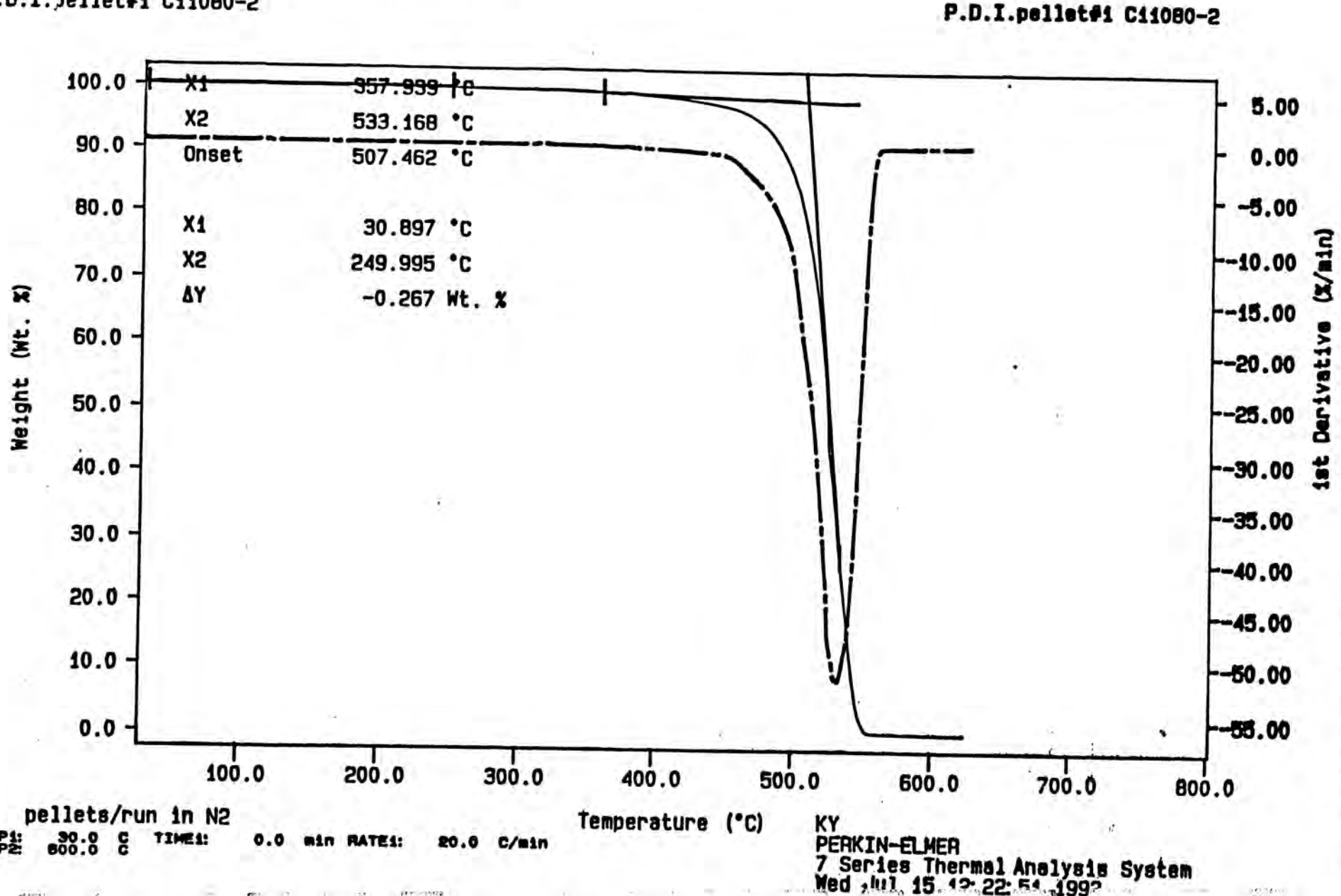

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TEMP2: 600.0 C

KY
PERKIN-ELMER
7 Series Thermal Analysis System
Wed Jul 15 12:22:54 1992

19.

Curve 1: TGA
File info: c110803a Wed Jul 15 13:14:18 1992
Sample Weight: 11.656 mg
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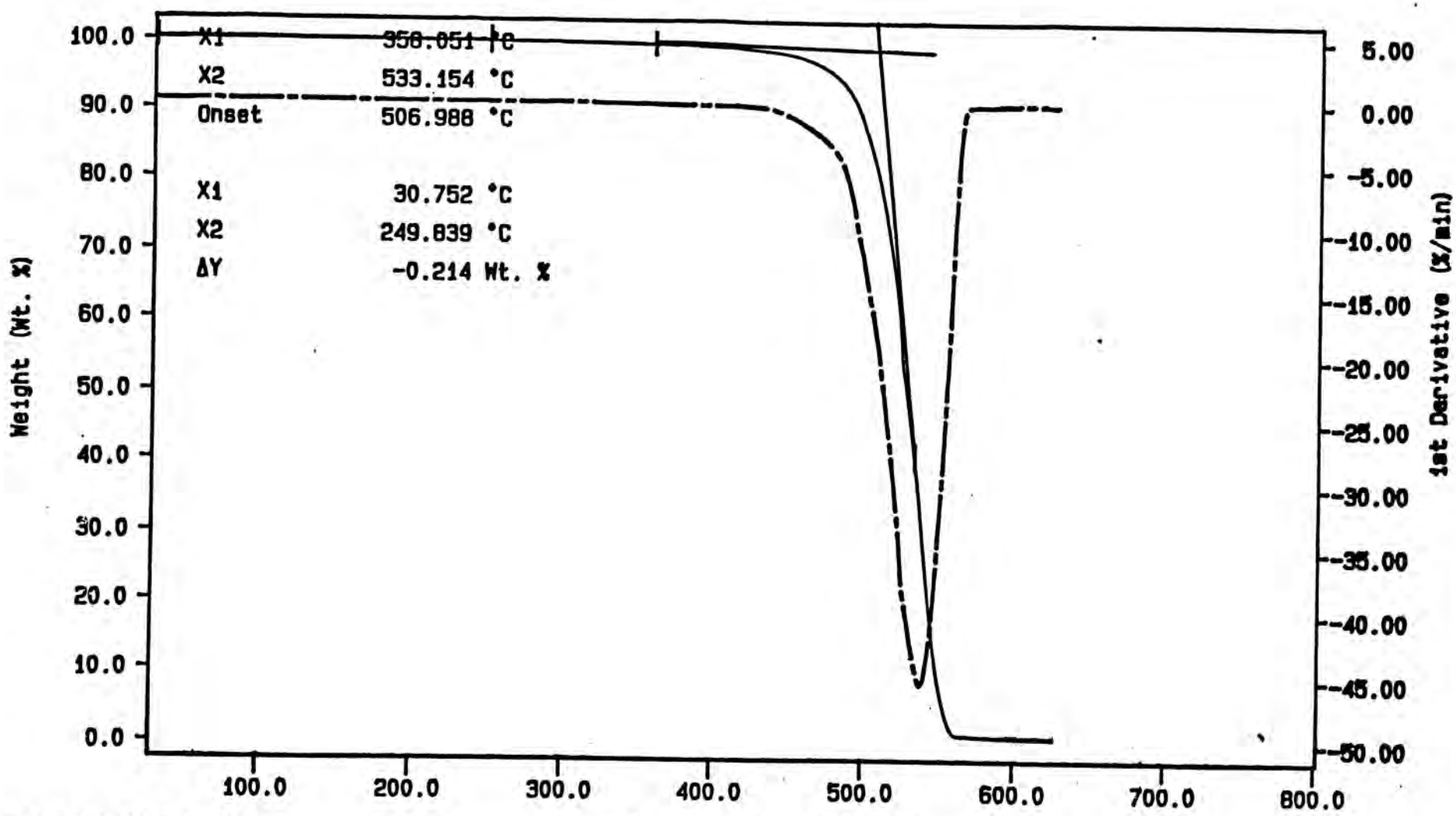
P.D.I. flake #4 C11080-3



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PERKIN-ELMER
7 Series Thermal Analysis System
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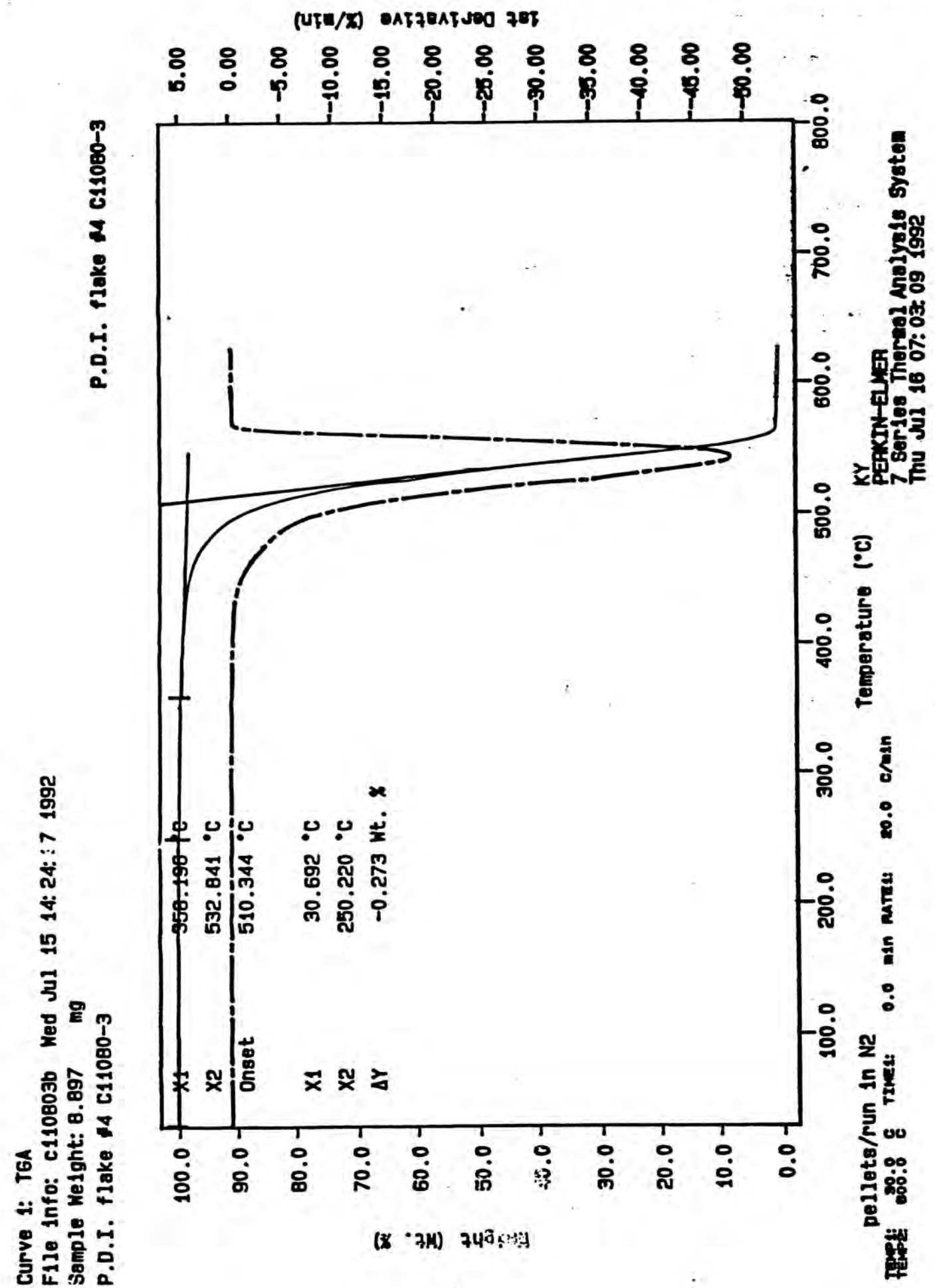
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P.D.I. flake #4 C11080-3
X1 358.190 °C
X2 532.841 °C
Onset 510.344 °C
X1 30.692 °C
X2 250.220 °C
ΔY -0.273 Wt. %
Weight (Wt. %)
1st Derivative (%/min)
Temperature (°C)
pellets/run in N2
TEMP: 30.0 C TIME: 0.0 min RATE: 20.0 C/min
TEMP: 600.0 C
KY
PERKIN-ELMER
7 Series Thermal Analysis System
Thu Jul 16 07:03:09 1992

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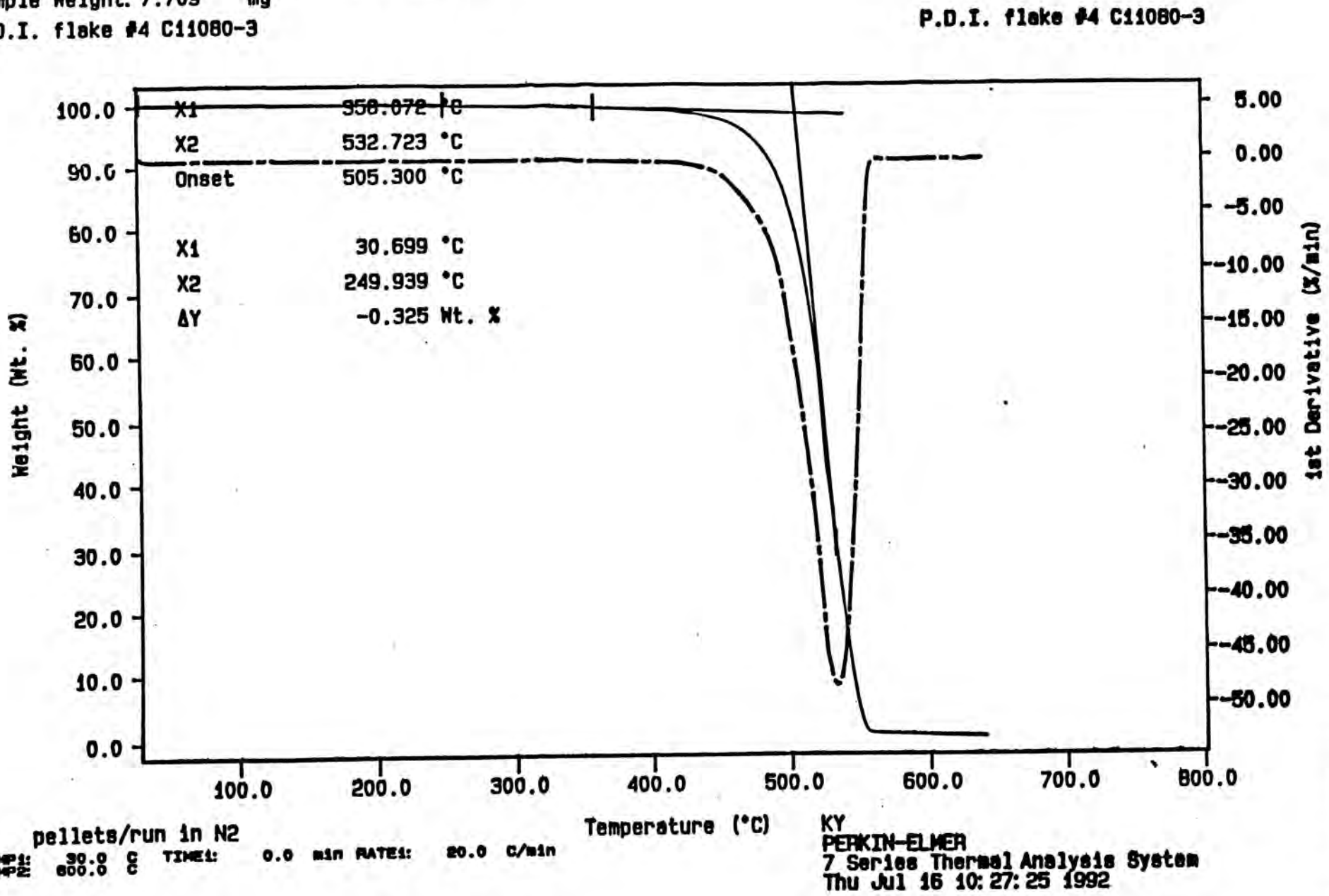
P.D.I. flake #4 C11080-3

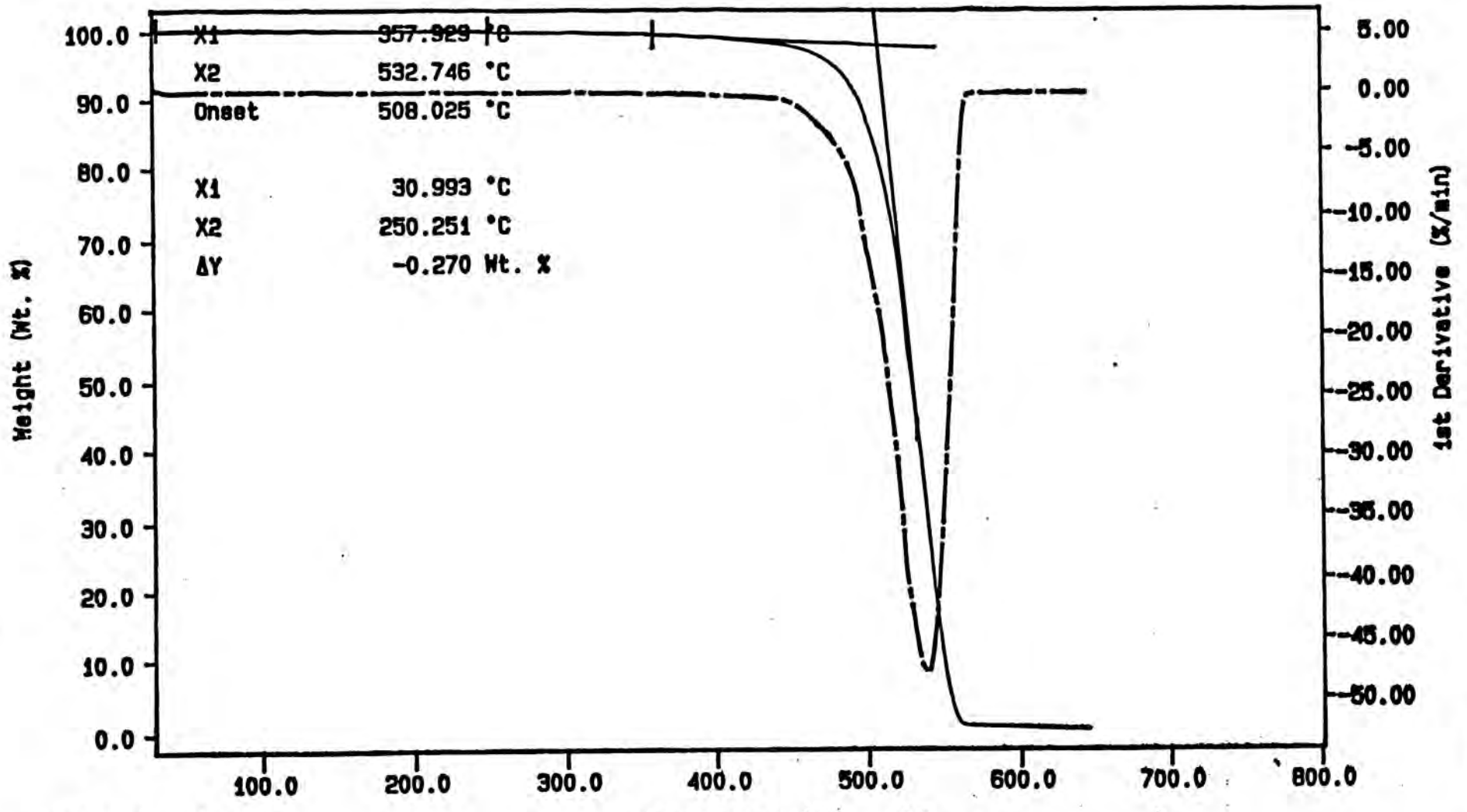


pellets/run in N2
TEMP1: 30.0 C TIME1: 0.0 min RATE1: 20.0 C/min
TEMP2: 600.0 C

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

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

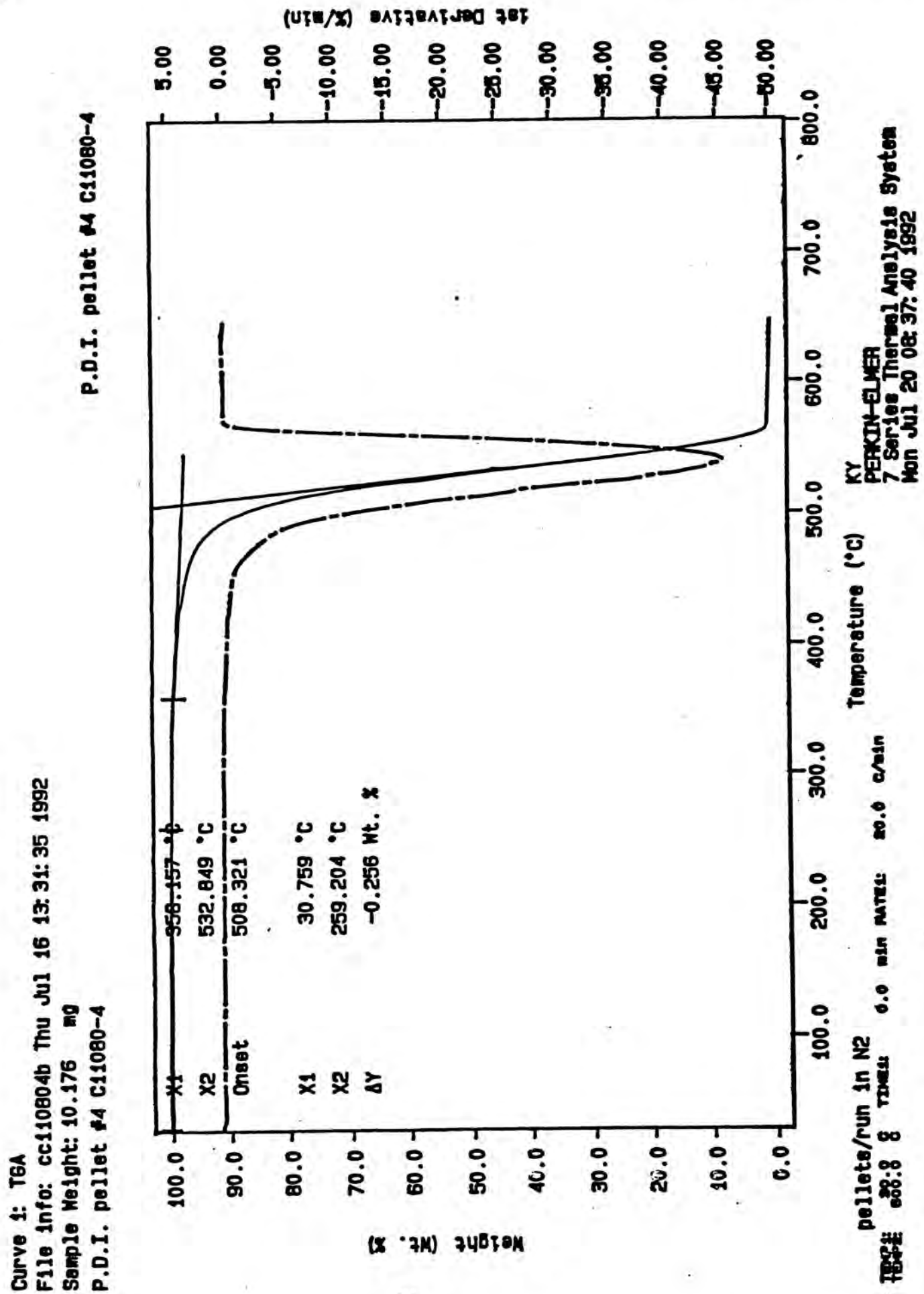
P.D.I. pellet #4 C11080-4



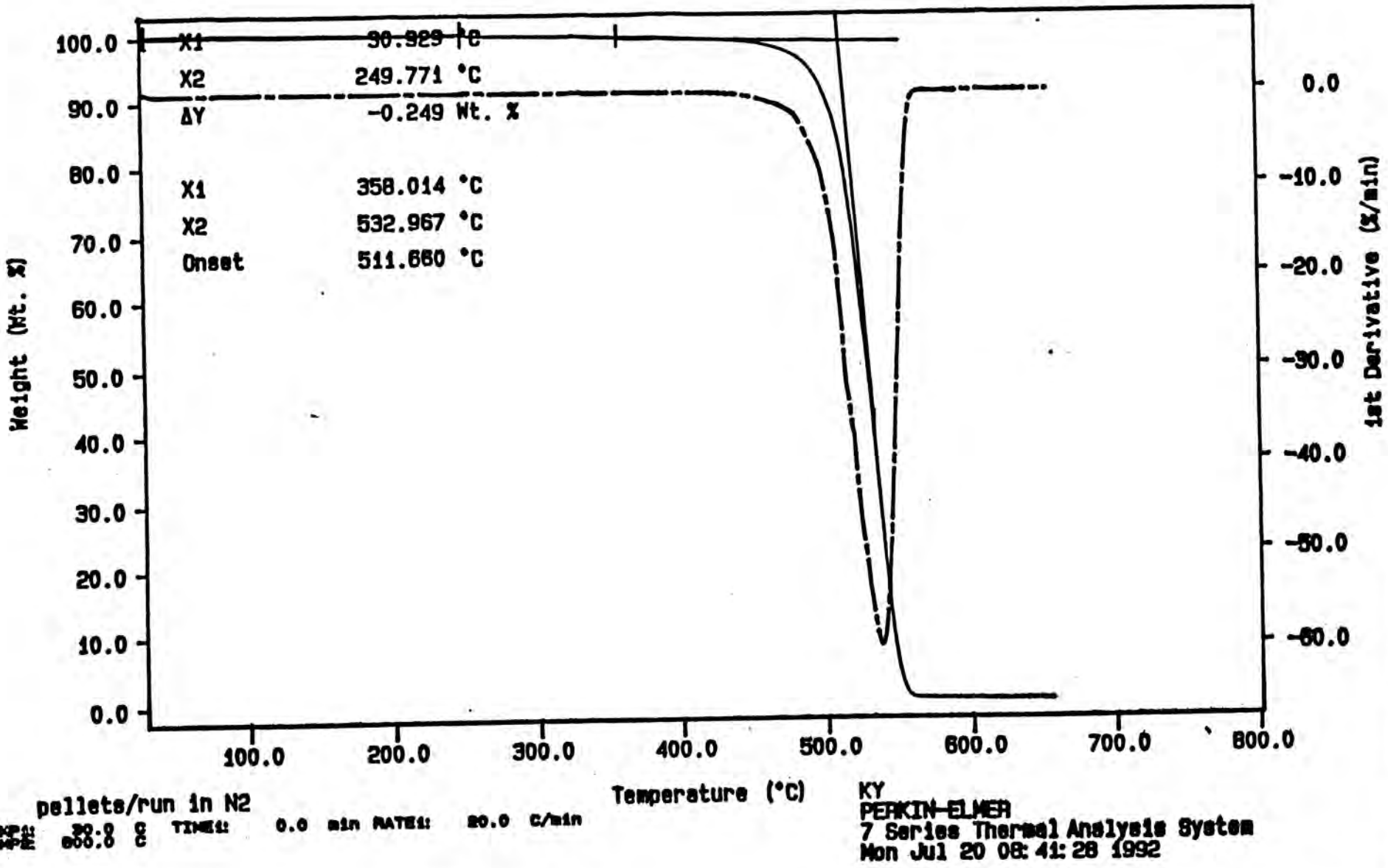
pellets/run in N2

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

KY
PERKIN-ELMER
7 Series Thermal Analysis System
Mon Jul 20 08:19:33 1992

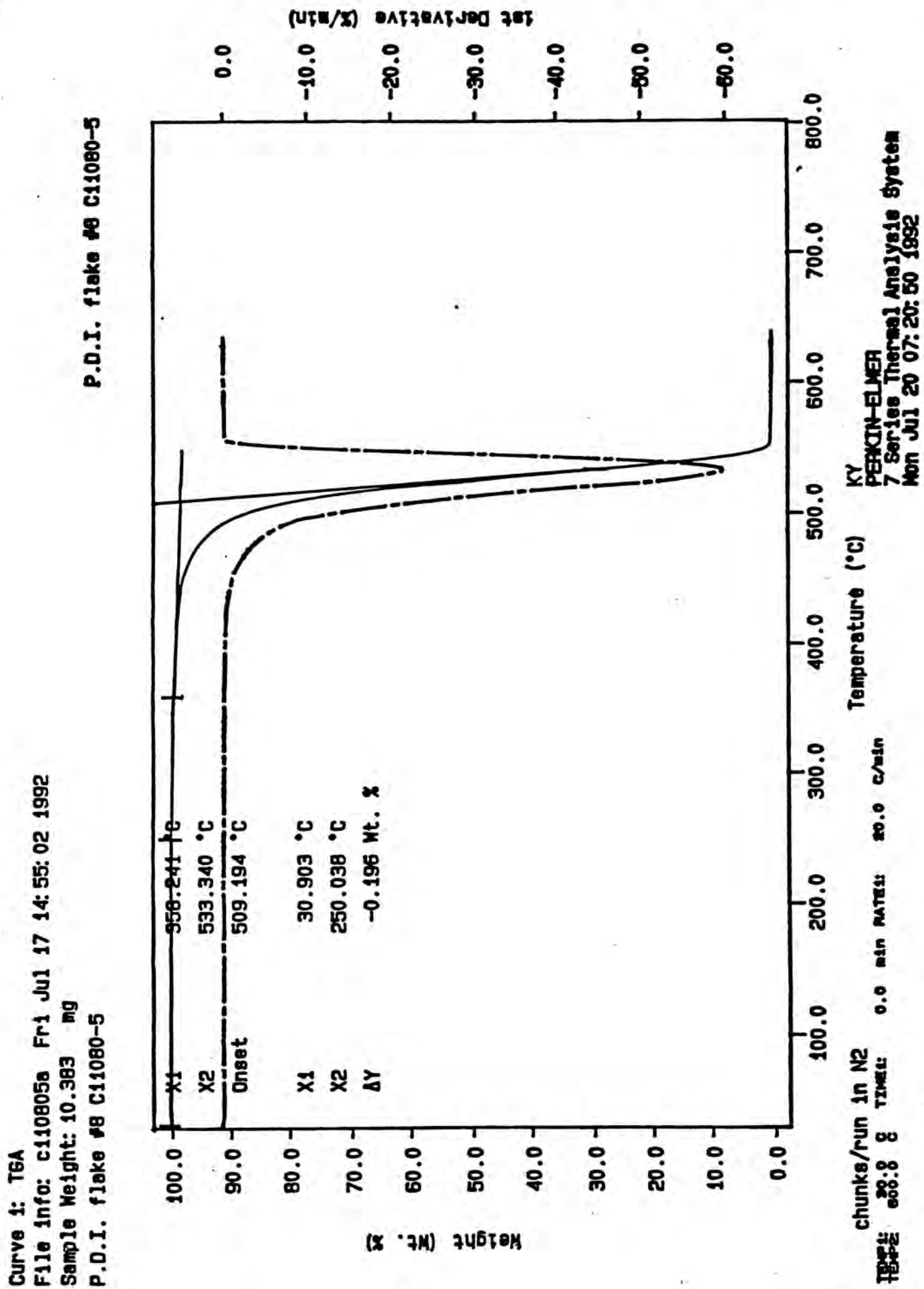

Curve 1: TGA
File Info: cc110804b Thu Jul 16 13:31:35 1992
Sample Weight: 10.176 mg
P.D.I. pellet #4 C11080-4
P.D.I. pellet #4 C11080-4
X1 958.157 °C
X2 532.849 °C
Onset 508.321 °C
X1 30.759 °C
X2 259.204 °C
ΔY -0.256 Wt. %
Weight (Wt. %)
1st Derivative (%/min)
Temperature (°C)
pellets/run in N2
KY
PERKIN-ELMER
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



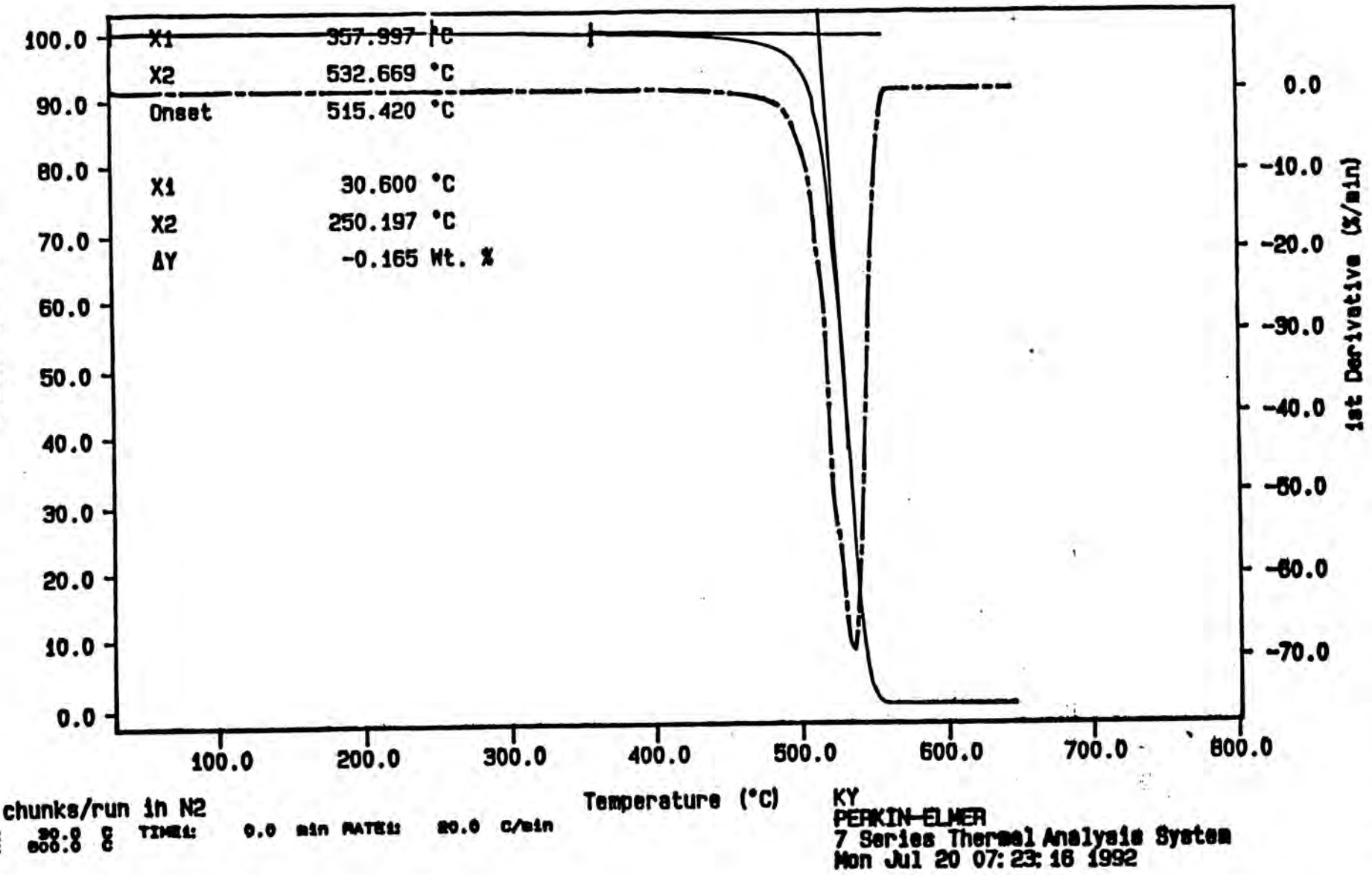
pellets/run in N2
TEMP: 30.0 C TIME: 0.0 min RATE: 20.0 C/min
TEMP: 800.0 C

KY
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
X1 356.241 °C
X2 533.340 °C
Onset 509.194 °C
X1 30.903 °C
X2 250.038 °C
ΔY -0.196 Wt. %
Weight (Wt. %)
1st Derivative (%/min)
Temperature (°C)
chunks/run in N2
TEMP1: 800.0 C TIME1: 0.0 min RATE1: 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

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



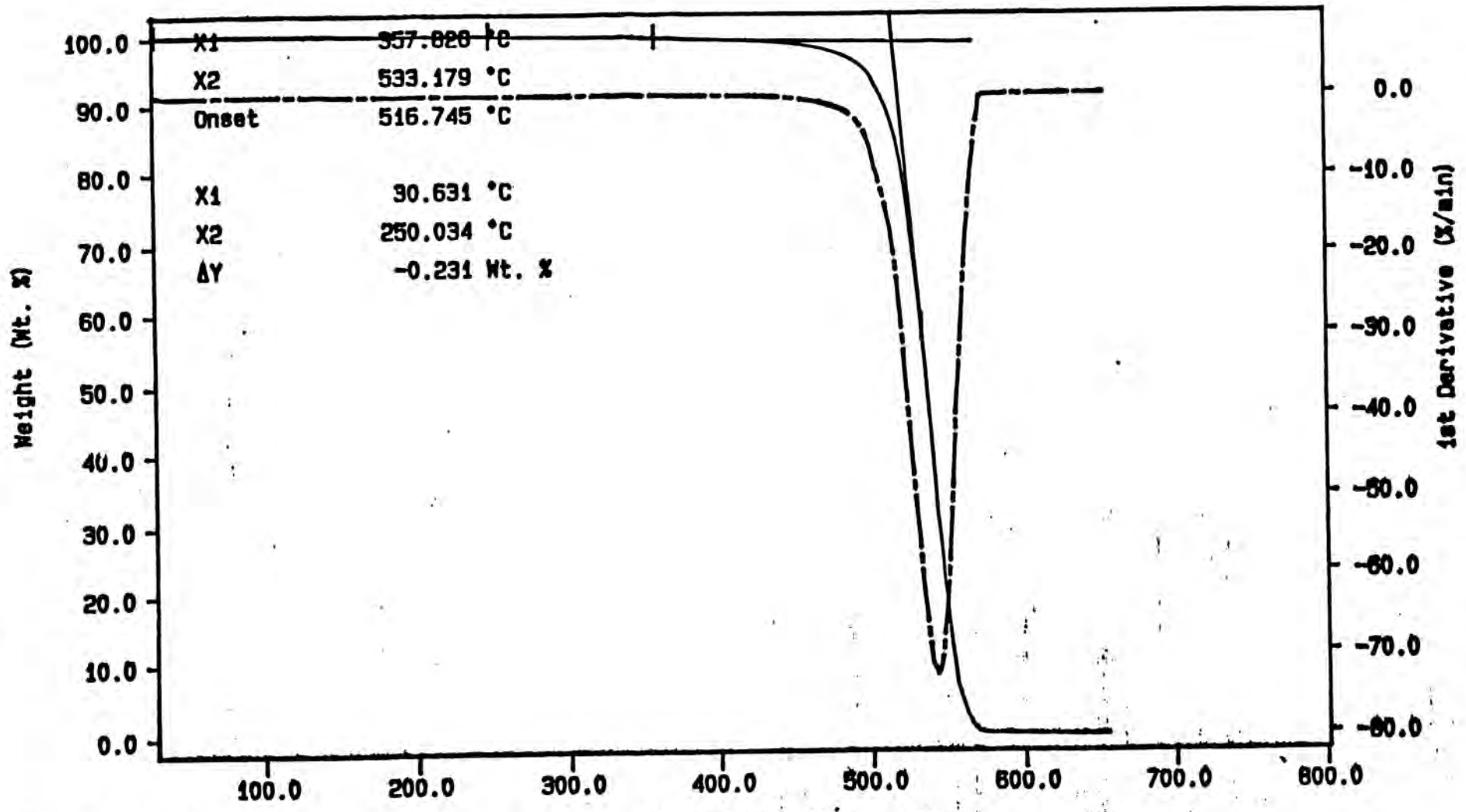
chunks/run in N2
TEMP1: 30.0 C TIME1: 0.0 min RATE1: 20.0 C/min
TEMP2: 800.0 C

KY
PERKIN-ELMER
7 Series Thermal Analysis System
Mon Jul 20 07:23:16 1992

27.

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

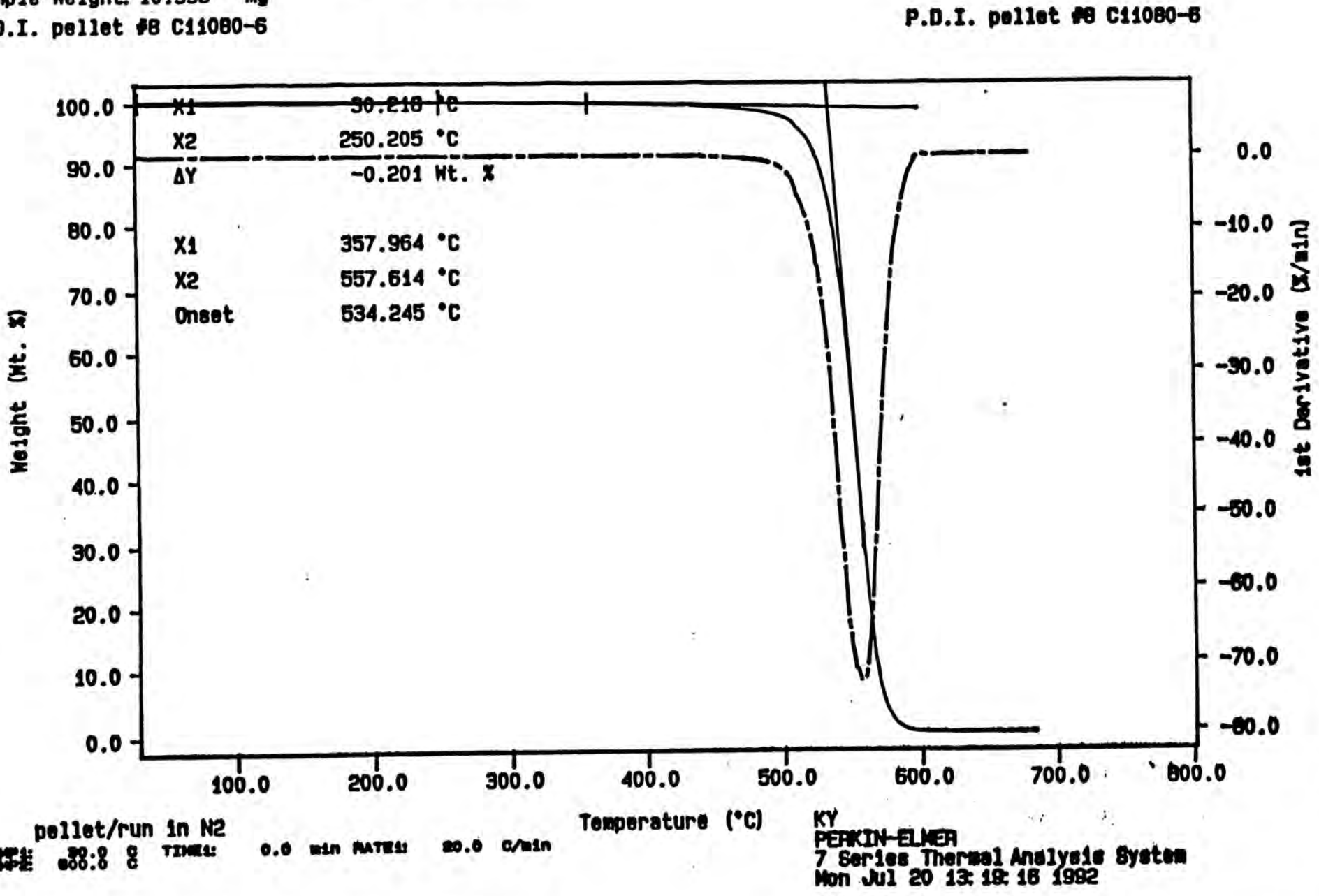
P.D.I. flake #8 C11080-5



chunks/run in N2
TEMP1: 30.0 C TIME1: 0.0 min RATE1: 20.0 C/min
TEMP2: 600.0 C

KY
PERKIN-ELMER
7 Series Thermal Analysis System
Mon Jul 20 08:13:13 1992

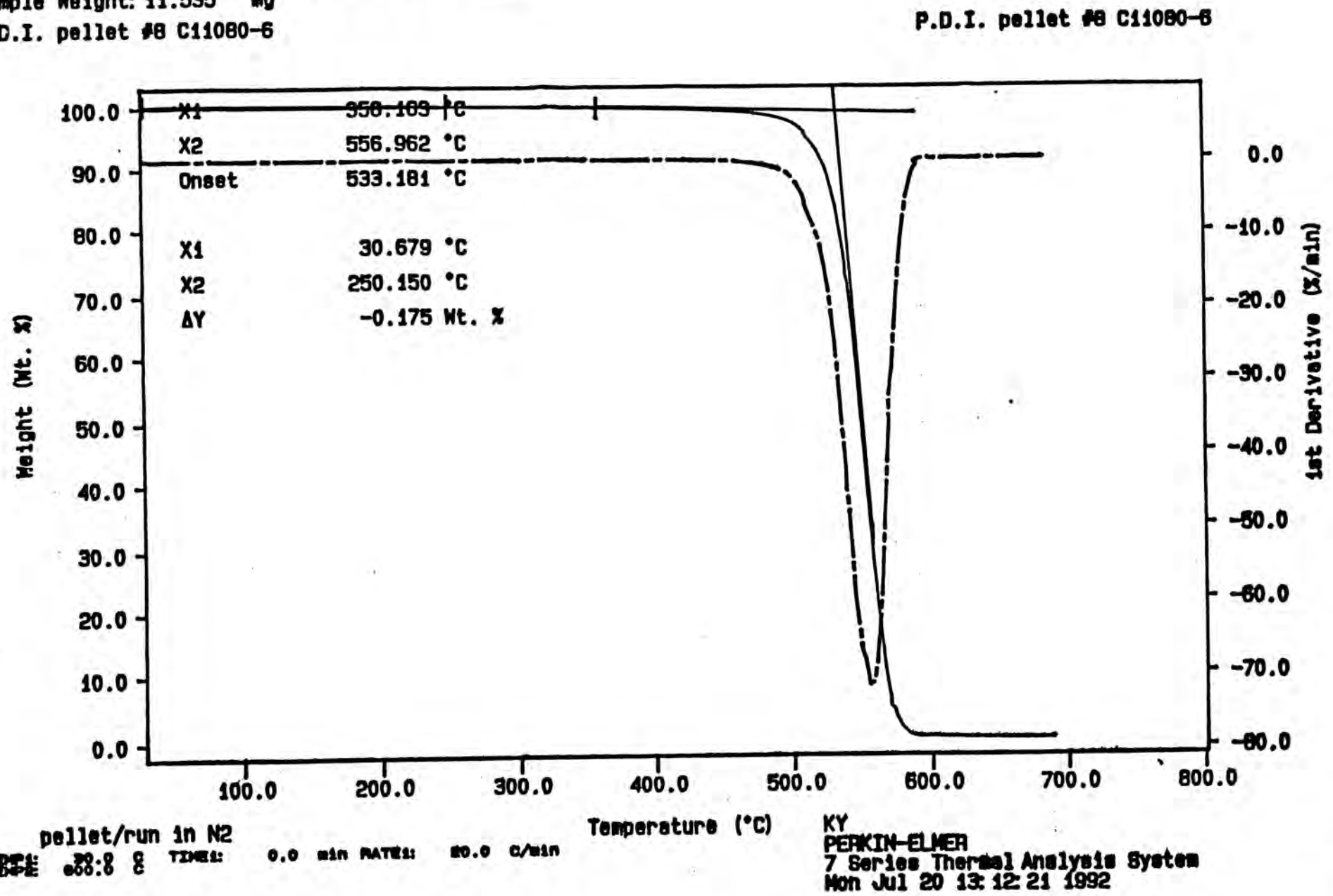
Curve 1: TGA
File info: c110806a Mon Jul 20 09:20:46 1992
Sample Weight: 10.993 mg
P.D.I. pellet #8 C11080-6



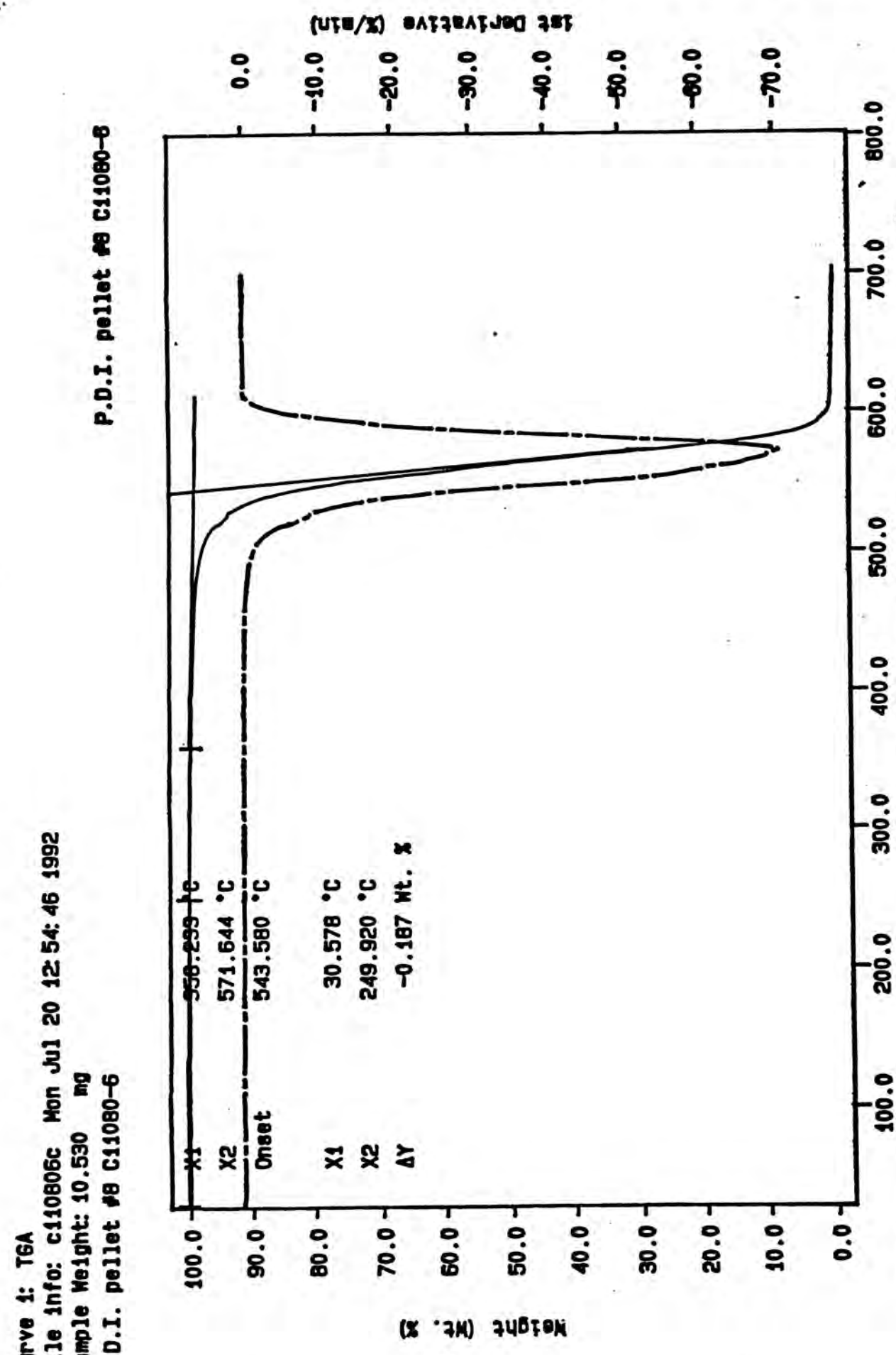
pellet/run in N2
TEMP: 30.0 C TIME: 0.0 min RATE: 20.0 C/min
TEMP: 800.0 C

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



pellet/run in N2
TIME: 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
X1 356.233 °C
X2 571.644 °C
Onset 543.580 °C
X1 30.578 °C
X2 249.920 °C
ΔY -0.187 Wt. %
Weight (Wt. %)
1st Derivative (%/min)
Temperature (°C)
pellet/run in N2
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.

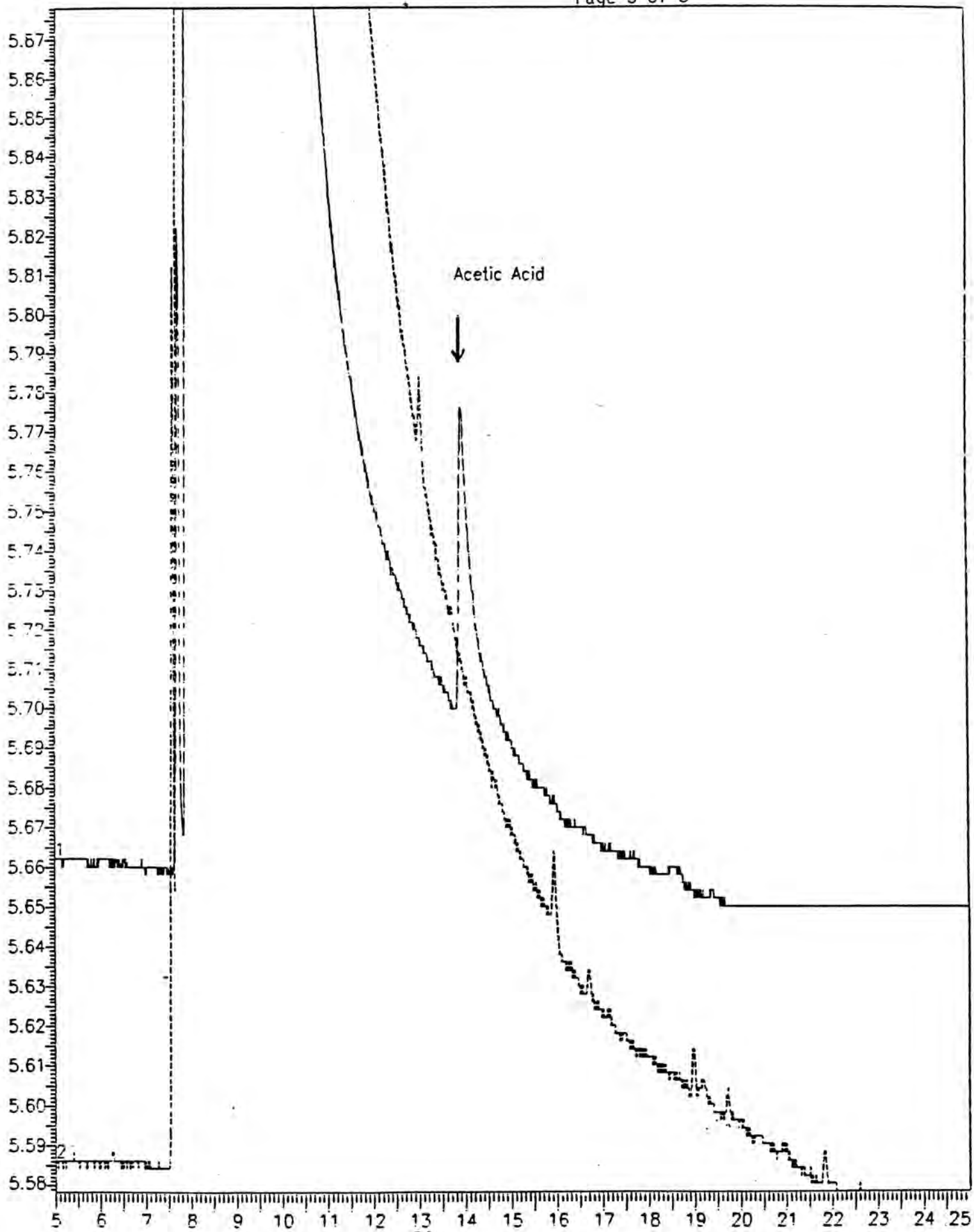
II. Hydraulic Oil Lading

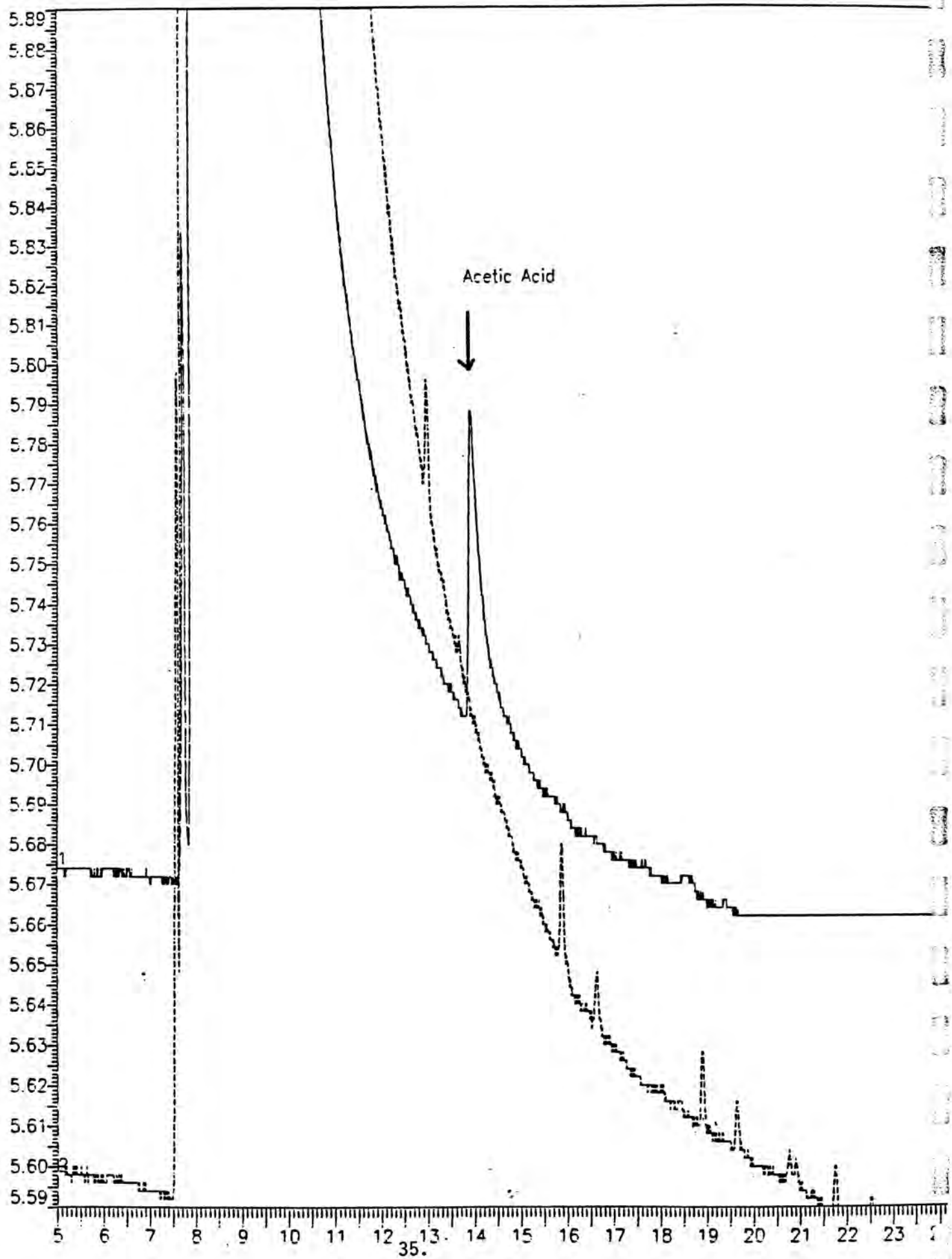
A. Procedure

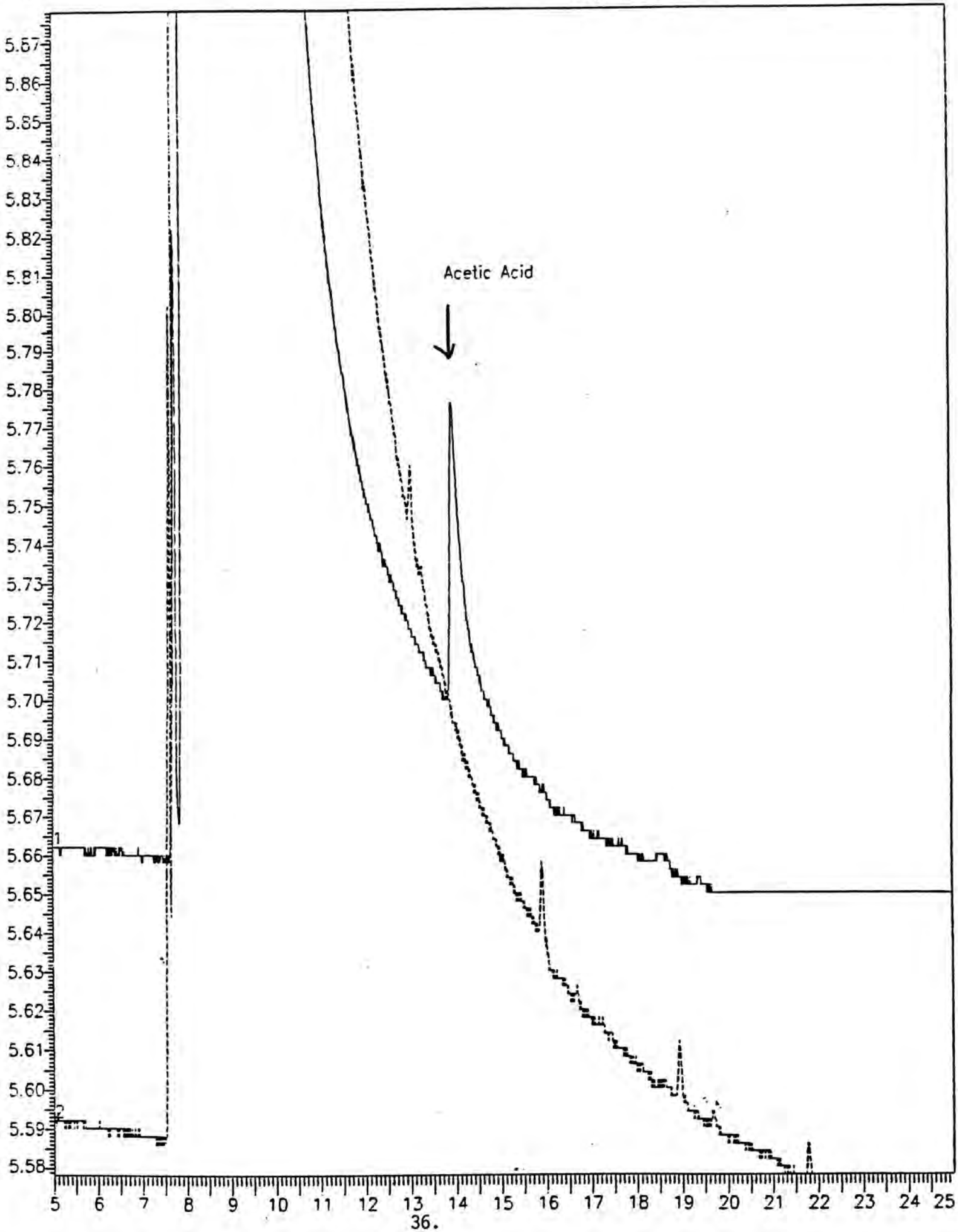
1. Grind sample to 30 mesh.
2. Place 2 g of ground sample and 10 of dichloromethane in a 25 ml screw-top bottle with a polyethylene-lined cap. Shake the mixture to disperse the sample in the dichloromethane, and allow it to stand at ambient temperature for 72 hours. (The oil extraction was done at greater 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 um 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 uL.
   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.

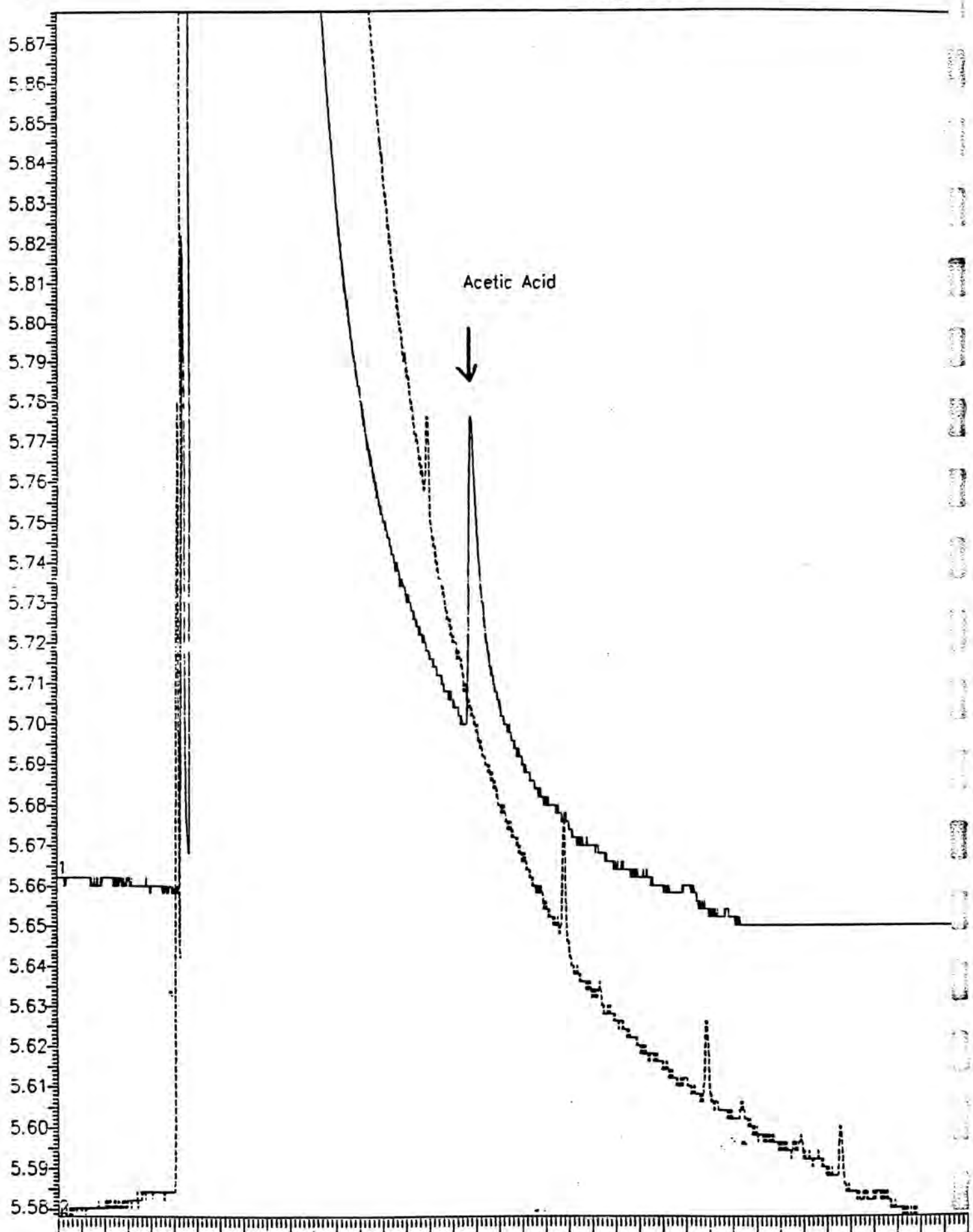
B. Results and Discussion

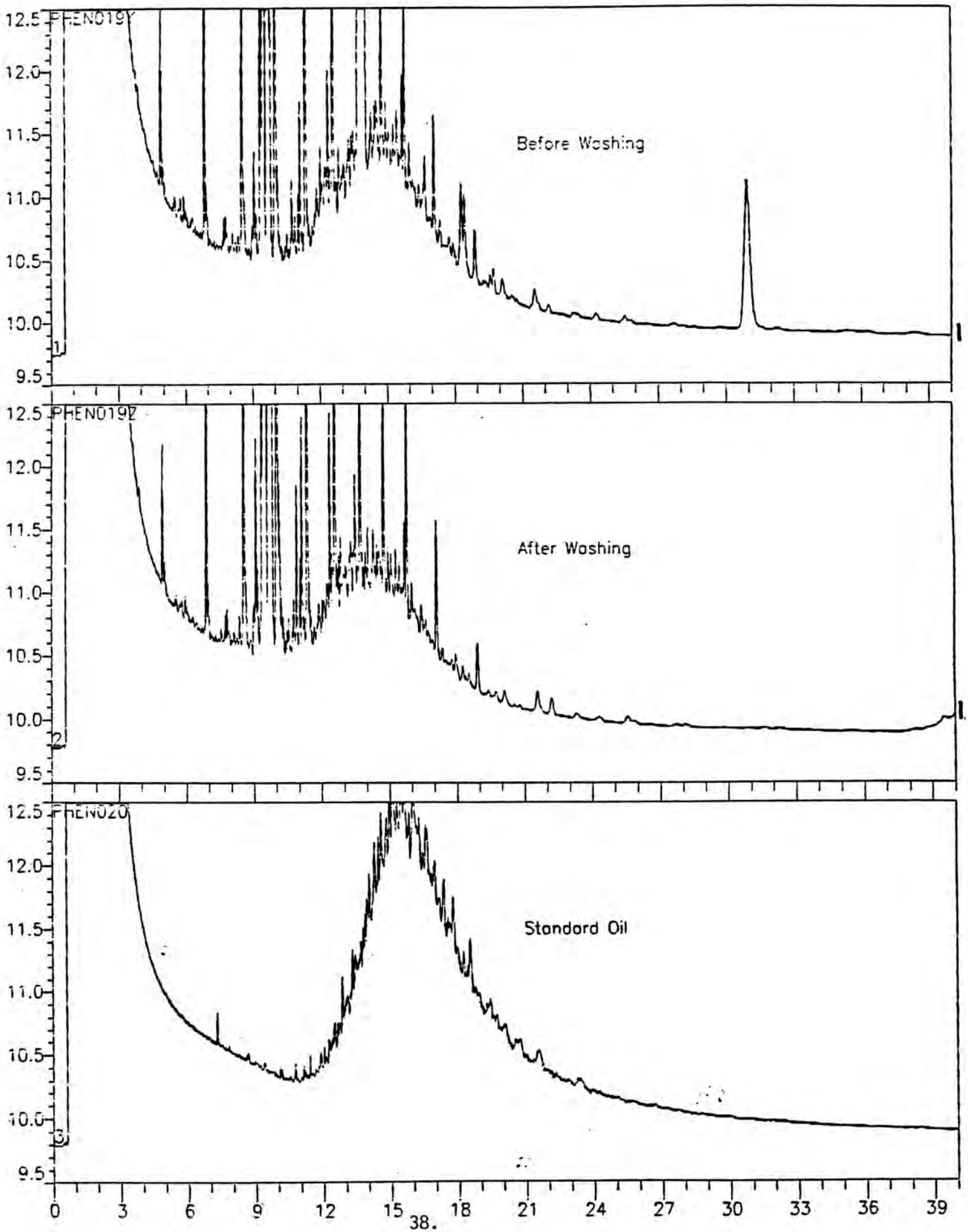
1. There was significant oil remaining in the polyethylene. The oil concentrations in the dichloromethane extract solutions (external standard calibration):
   a. Extract of unwashed sample: 0.25 g/L.
   b. Extract of washed sample: 0.2 g/L.
2. Thus approximately 20% of the oil in/on the polyethylene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

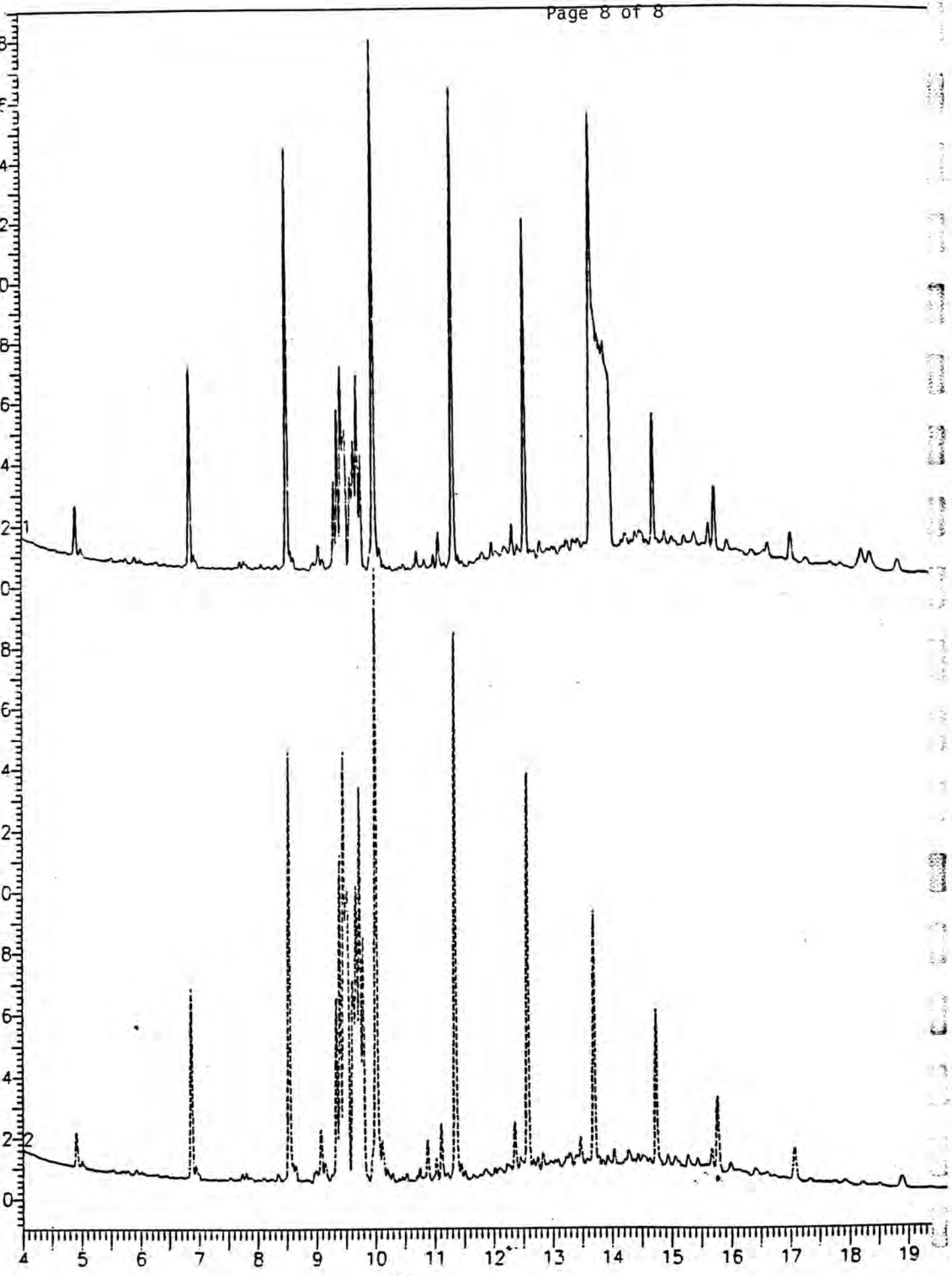

Acetic Acid
5.67
5.66
5.65
5.64
5.63
5.62
5.61
5.60
5.59
5.58
1
2
5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25


Acetic Acid
5.89
5.88
5.87
5.86
5.85
5.84
5.83
5.82
5.81
5.80
5.79
5.78
5.77
5.76
5.75
5.74
5.73
5.72
5.71
5.70
5.69
5.68
5.67
5.66
5.65
5.64
5.63
5.62
5.61
5.60
5.59
5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23


Acetic Acid
5.87
5.86
5.85
5.84
5.83
5.82
5.81
5.80
5.79
5.78
5.77
5.76
5.75
5.74
5.73
5.72
5.71
5.70
5.69
5.68
5.67
5.66
5.65
5.64
5.63
5.62
5.61
5.60
5.59
5.58
1
2
5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25


Acetic Acid
5.87
5.86
5.85
5.84
5.83
5.82
5.81
5.80
5.79
5.78
5.77
5.76
5.75
5.74
5.73
5.72
5.71
5.70
5.69
5.68
5.67
5.66
5.65
5.64
5.63
5.62
5.61
5.60
5.59
5.58
5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24


PHEN019Y
Before Washing
PHEN019Z
After Washing
PHEN020
Standard Oil
12.5
12.0
11.5
11.0
10.5
10.0
9.5
0
3
6
9
12
15
18
21
24
27
30
33
36
39


48
44
42
40
38
36
34
32
30
28
26
24
22
20
18
16
14
12
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1
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19

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 Instituite

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

SUBJECT: Test Results for the Drum Recycling Project

	Flow Rate ASTM 1238 HLMI 21.6 kg	Flow Rate ASTM 1238 MI 5.0 kg	Density ASTM D 792 g/cm
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 chromotograpy 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.

Sample	acrylic acid	sulfuric acid
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

FINA OIL AND CHEMICAL COMPANY LA PORTE PLANT QUALITY ASSURANCE METHODS		QA-C-015-4
		Page 1 of 4
		Issue Date: 10/28/80
		Revision Date: 10/01/92
		Approval:

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 ± 20° 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 8½" x 12" x 1/8" aluminum backup plates
   b. Two 8½" x 12" chrome ferrotype plates
   c. Two sheets of mylar film, 1.5 mil thick
   d. One mold, 8½" x 12" x 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

f. Chrome ferrotype plate
g. Aluminum backup plate

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.

Table I

Program	Elements
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

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.

JJM 1/5/93

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

Novacor
PLASTICS DIVISION

Novacor Chemicals Ltd.
Technical Centre
3620 - 32 Street N.E.
Calgary, Alberta
Canada T1Y 6G7
(403) 291-8444
FAX (403) 291-0493

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

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

Nancy Conley

Nancy Conley
Technical Service Specialist

cc: Don Parks
Chris Gick

NC/sm

NOVACOR CHIMIE (CANADA) LTÉE
3100, Côte Vertu, Bureau 460
Saint-Laurent, Québec
H4R 2J8
Téléphone: (514) 745-1414
Fax: (514) 745-0874

Novacor

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

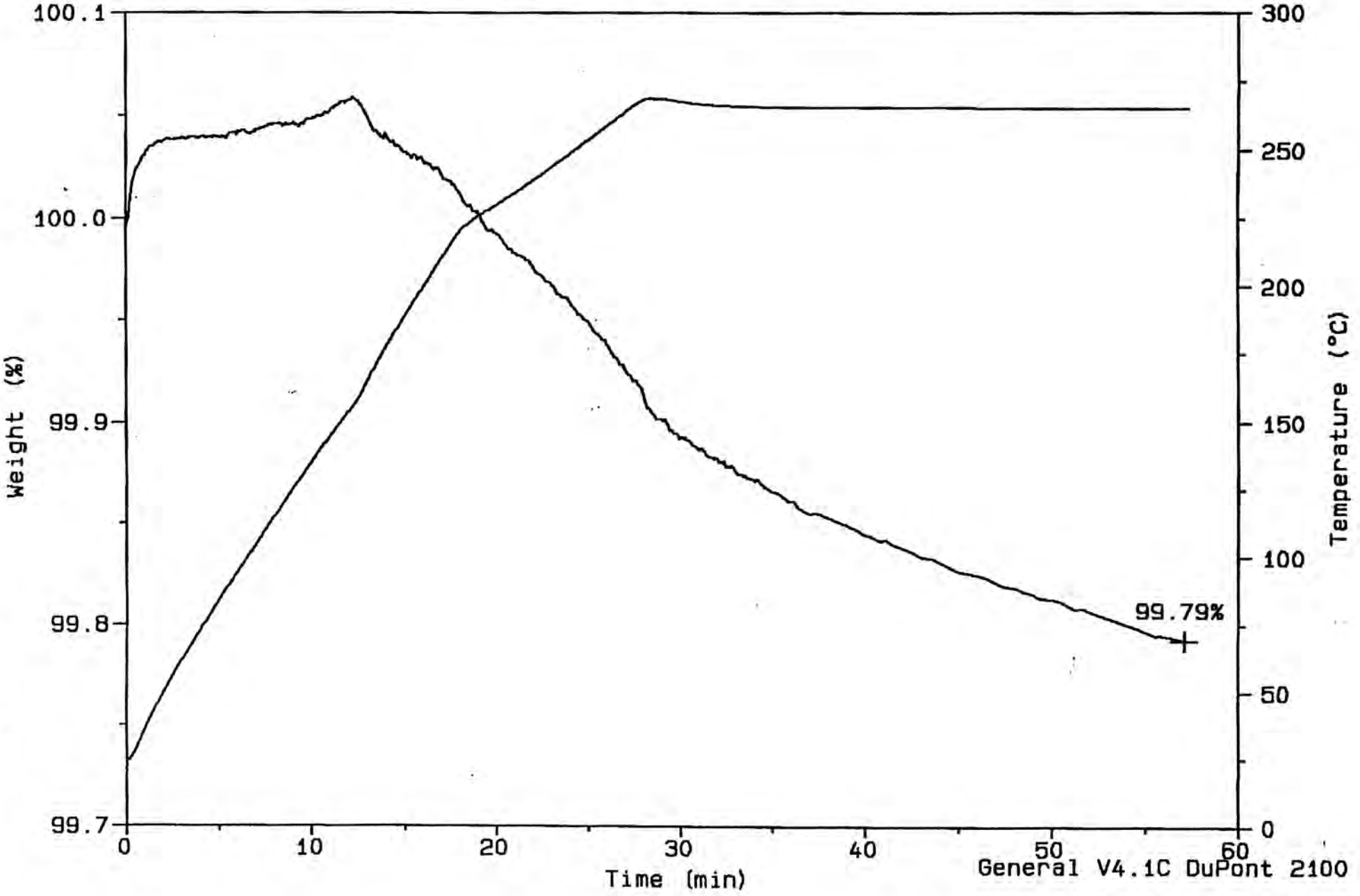
Si vous éprouvez des difficultés de transmission, veuillez appeler le (514) 745-1414.

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

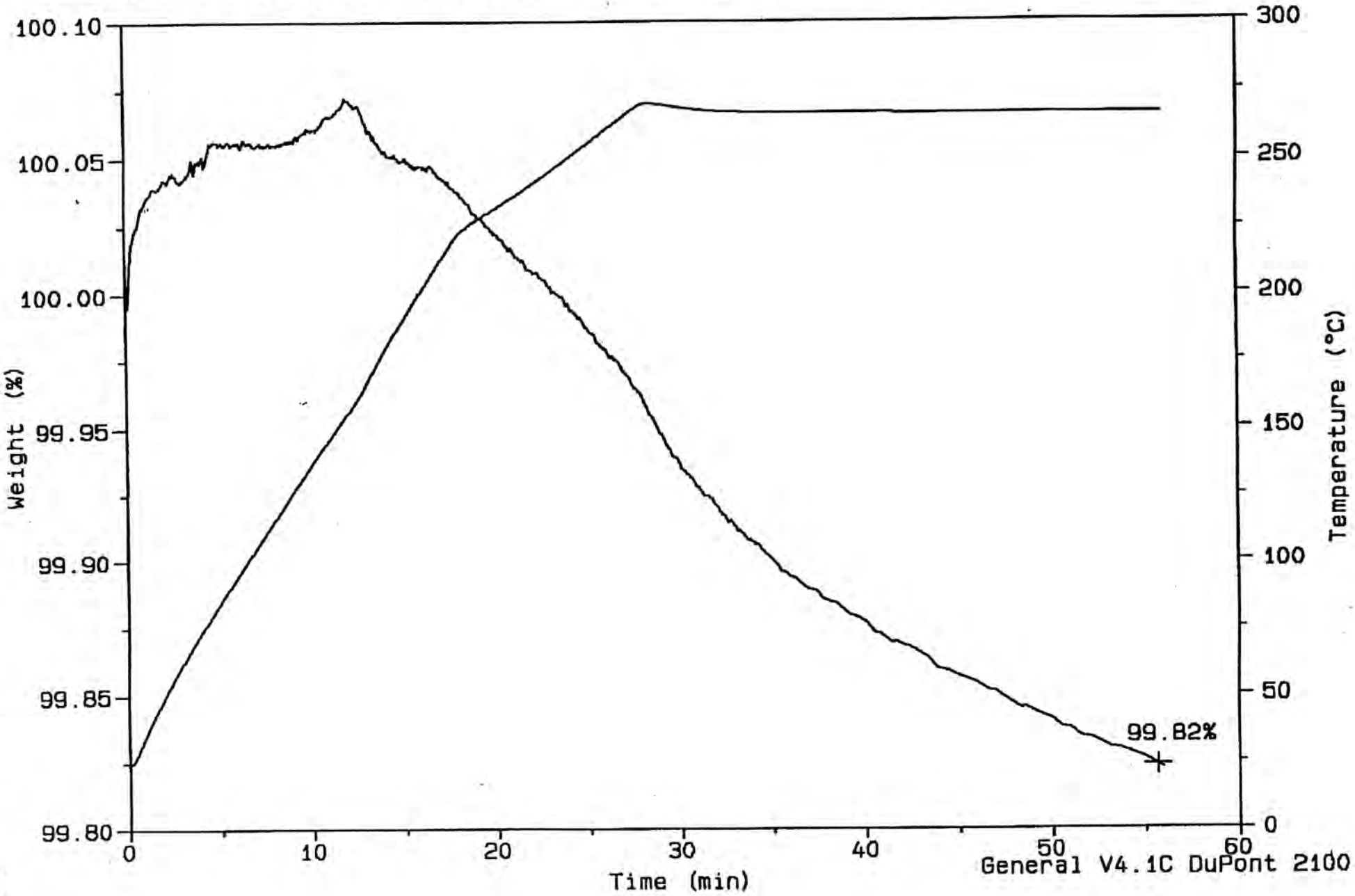


General V4.1C DuPont 2100

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

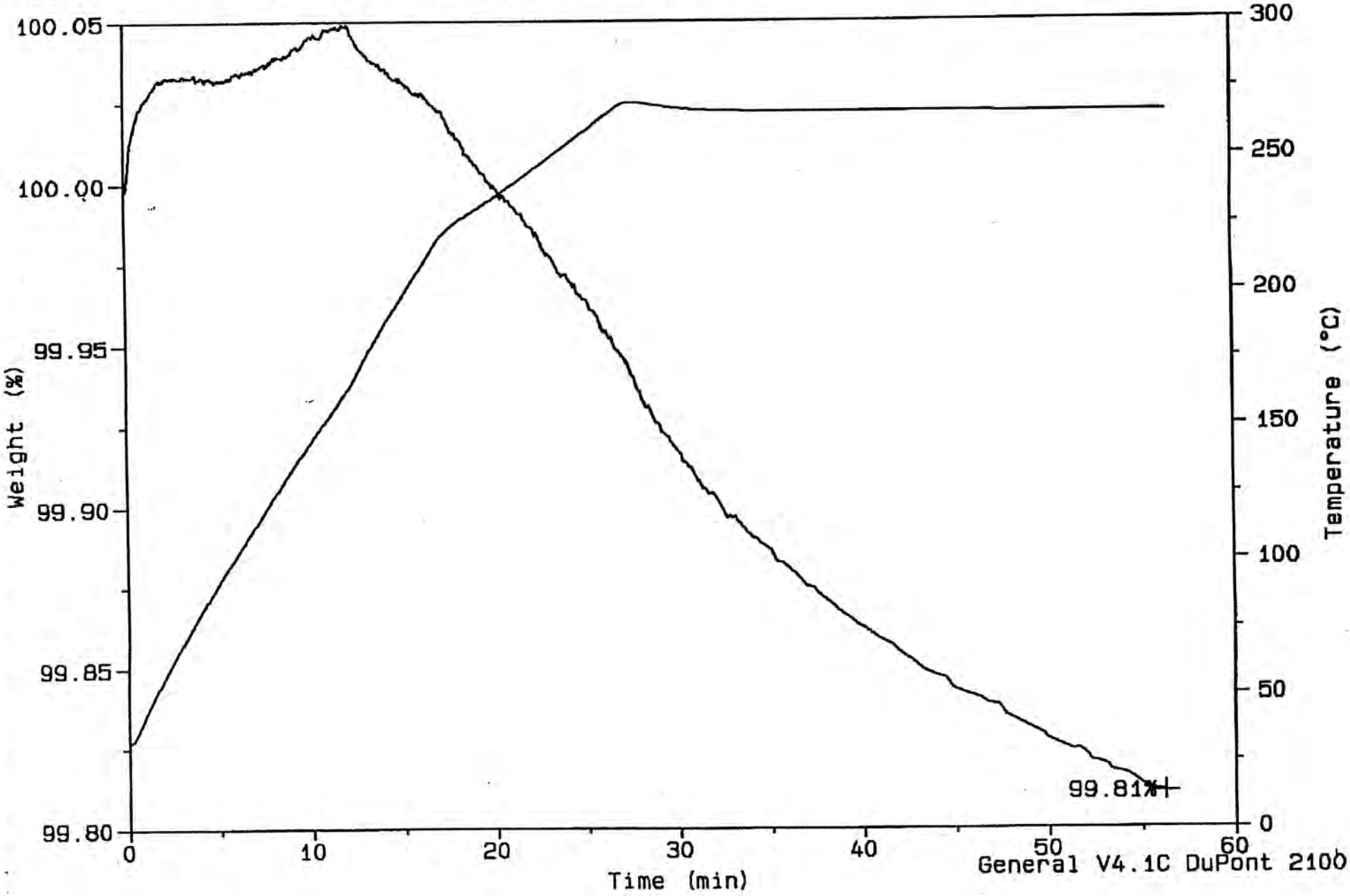
File: A:93-564.02
Operator: VD
Run Date: 10-Mar-93 13:41



Sample: 93-564 HBW355A 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



General V4.1C DuPont 2100

Novacor MEMORANDUM

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

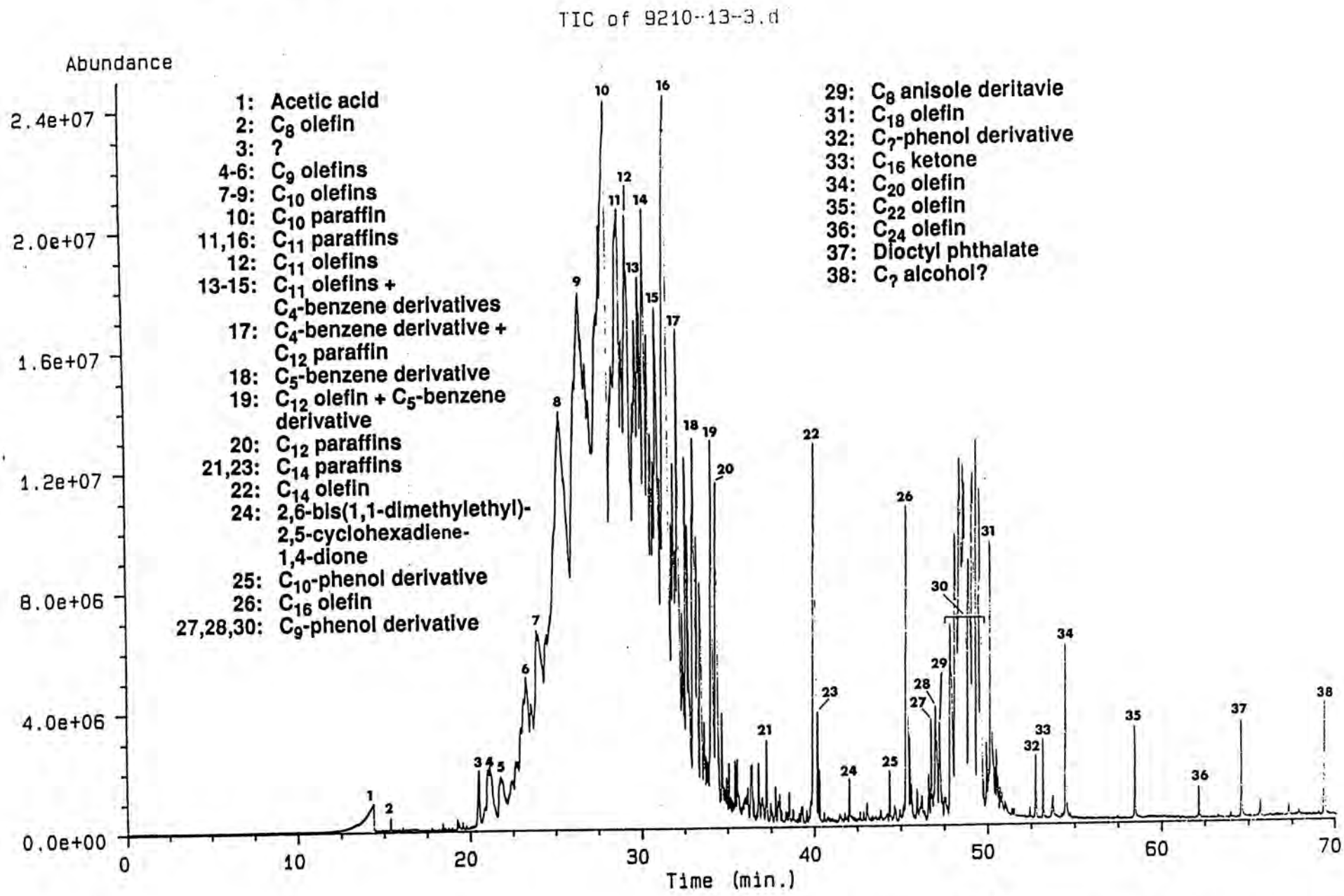
Chromatographic Conditions for the GC-MS Analysis of PDI Drum Samples

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

TT: 144.mem

FIGURE 7: TOTAL ION CHROMATOGRAM OF THE UNKNOWN SAMPLE

Sample weight: 169 mg



TT: 144.mem

FIGURE 2: TOTAL ION CHROMATOGRAM OF THE WASHED SAMPLE

Sample weight: 163 mg

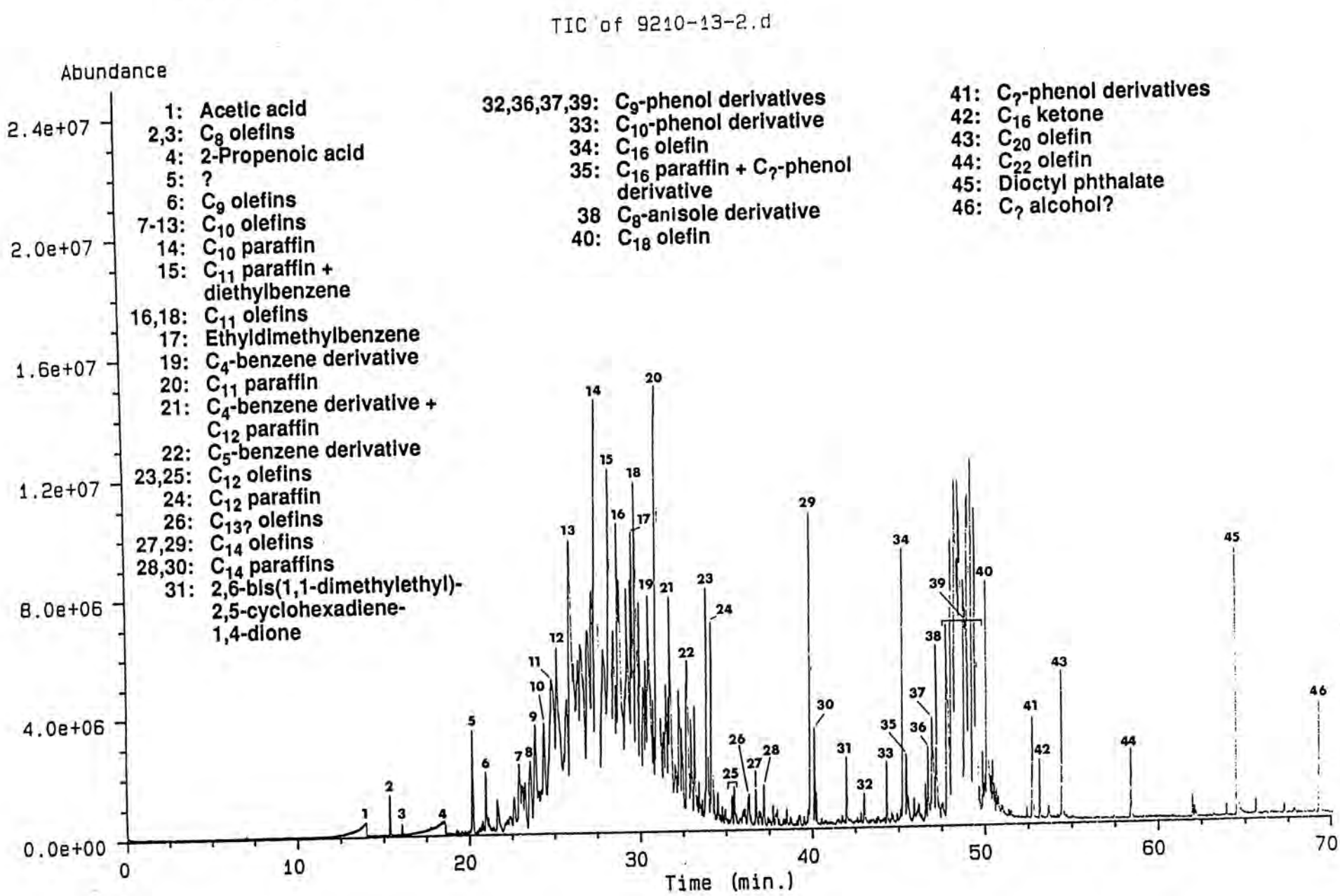


FIGURE 3: TOTAL ION CHROMATOGRAM OF THE [illegible] W55[illegible] RE[illegible] SA[illegible]LE

Sample weight: 192 mg

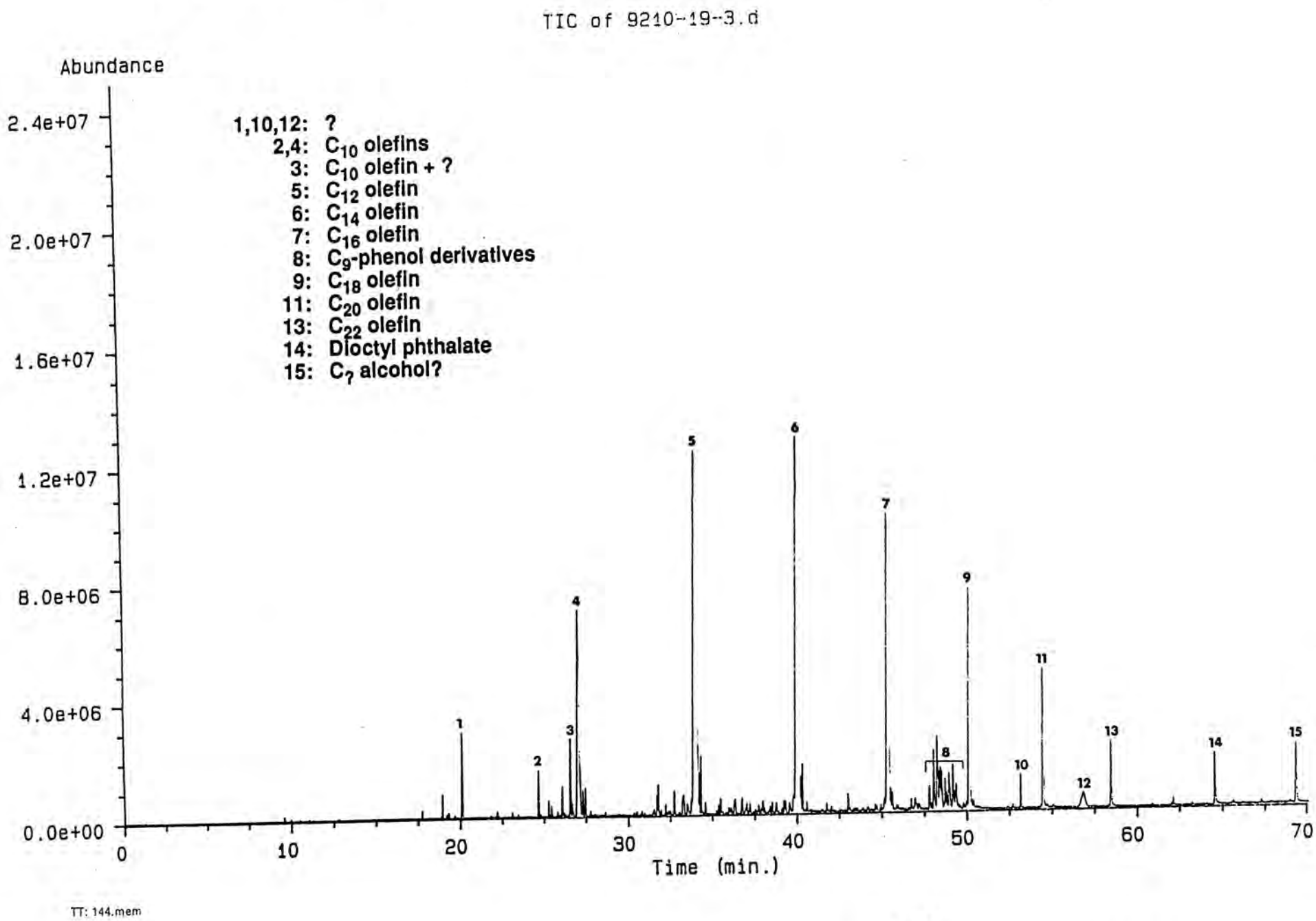
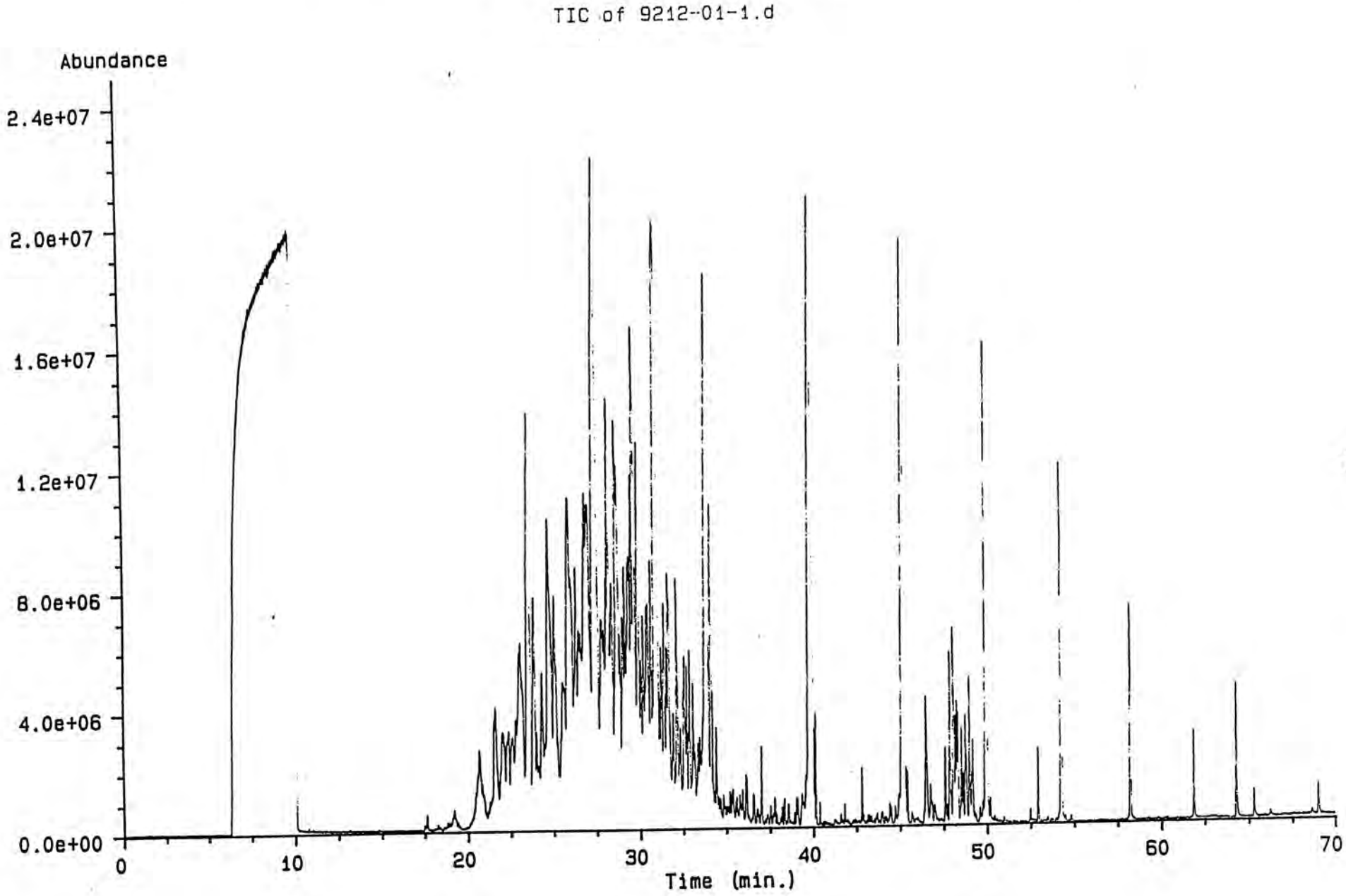
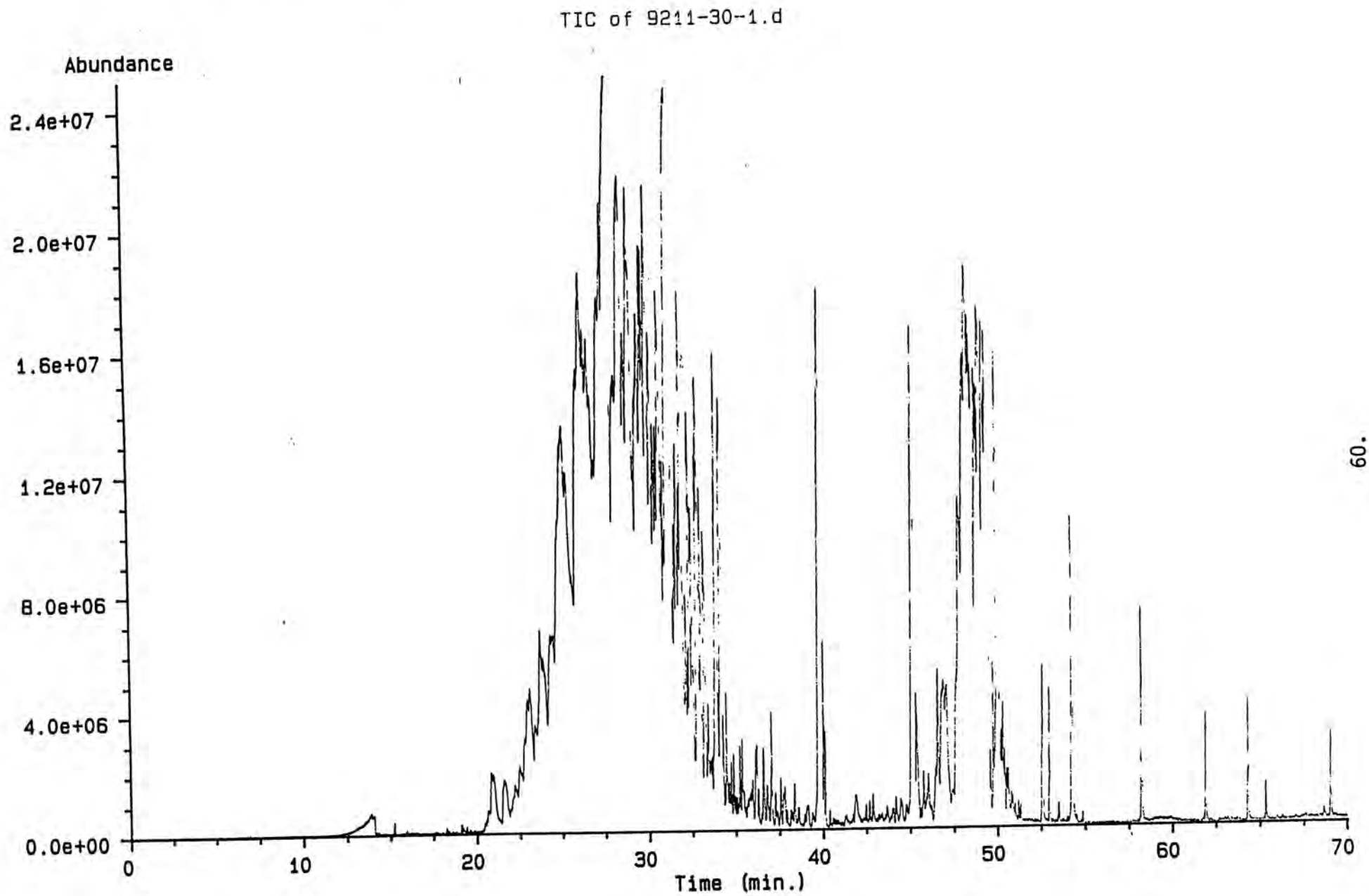


FIGURE 4: TOTAL ION CHROMATOGRAM OF HB-W555-A RESIN SPIKED WITH 72 uG OF STODDARD'S REAGENT (FIRST DESORPTION)



TT: 144.mem

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



TT: 144.mem

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

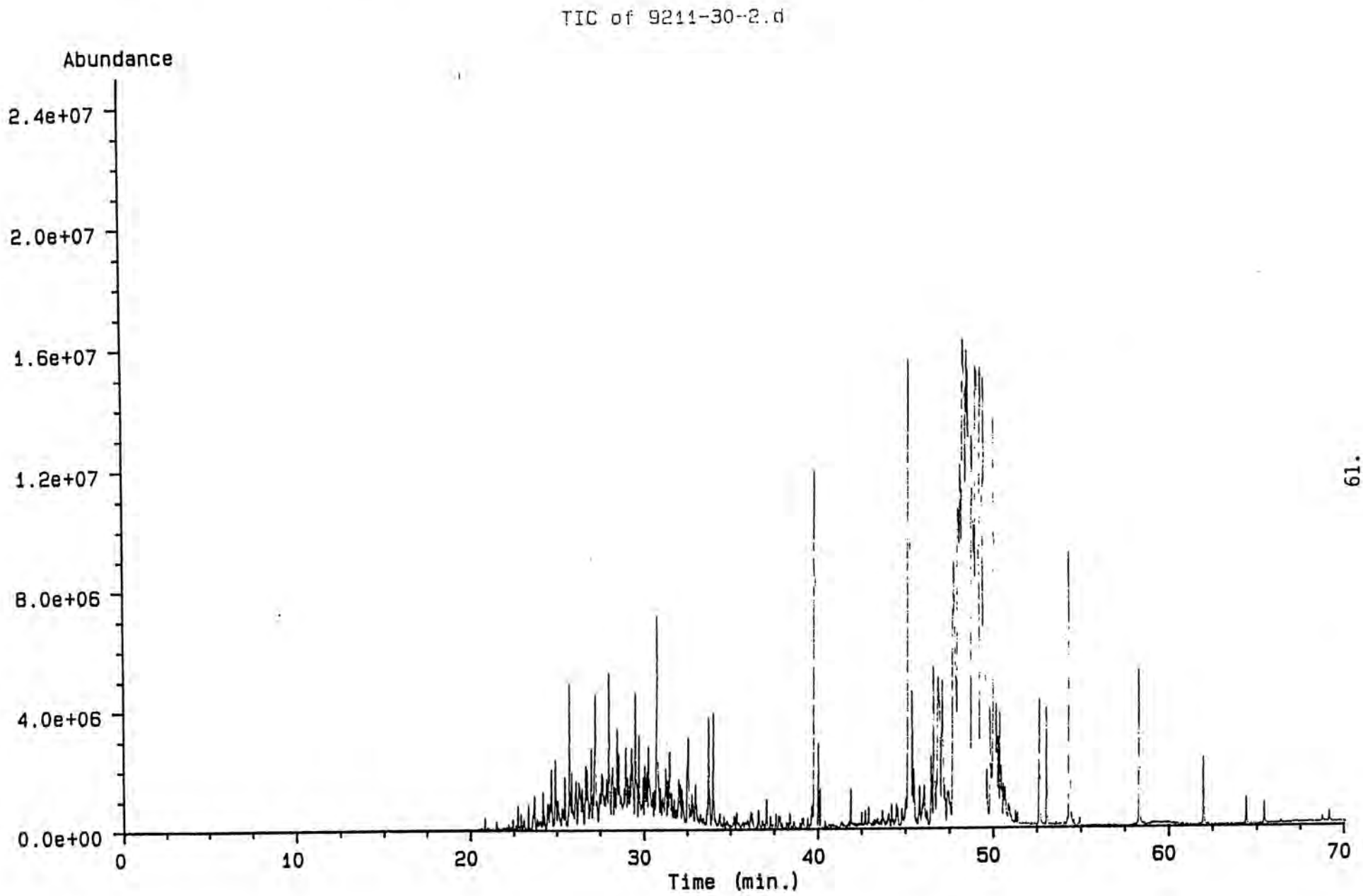
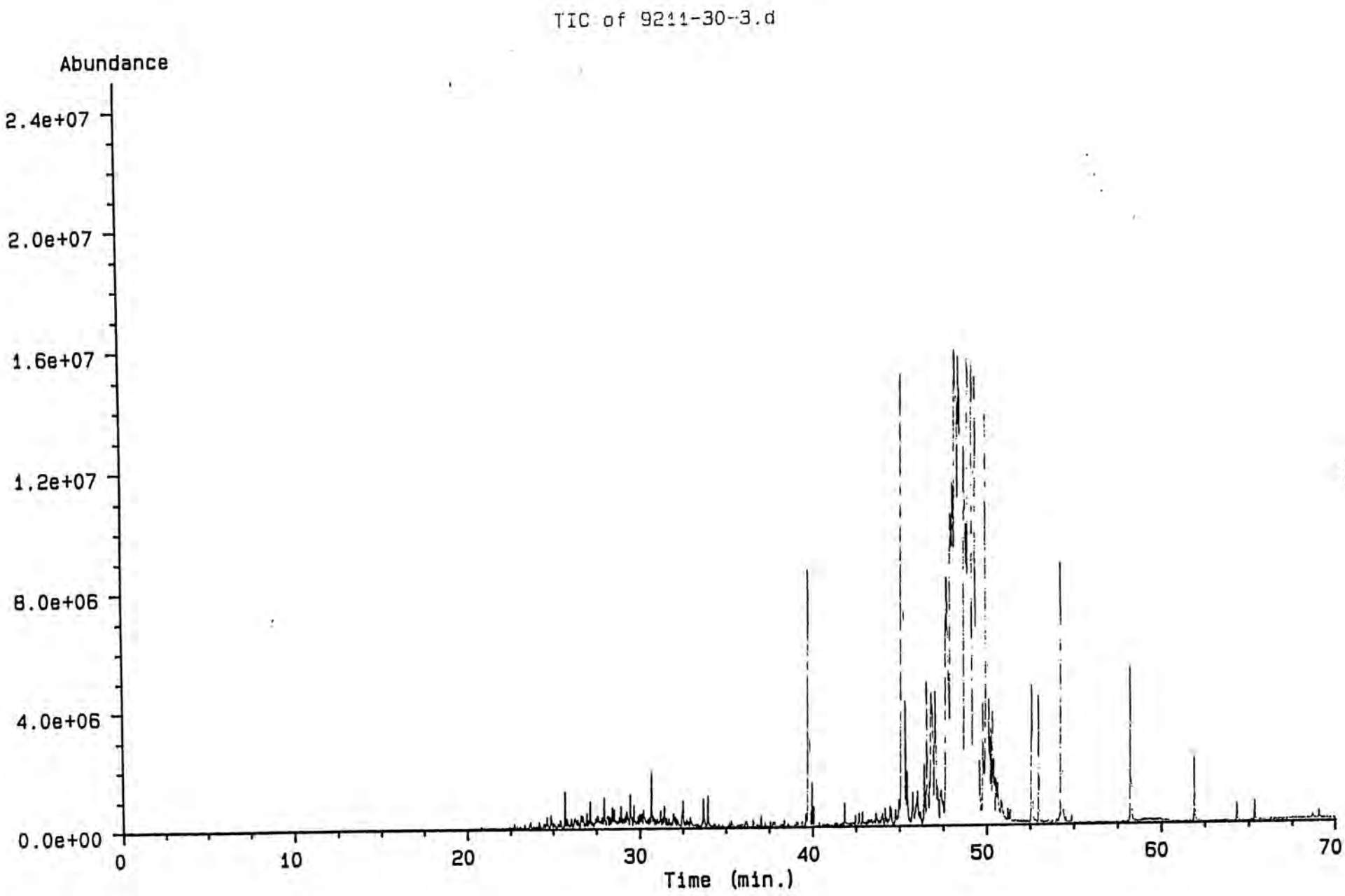
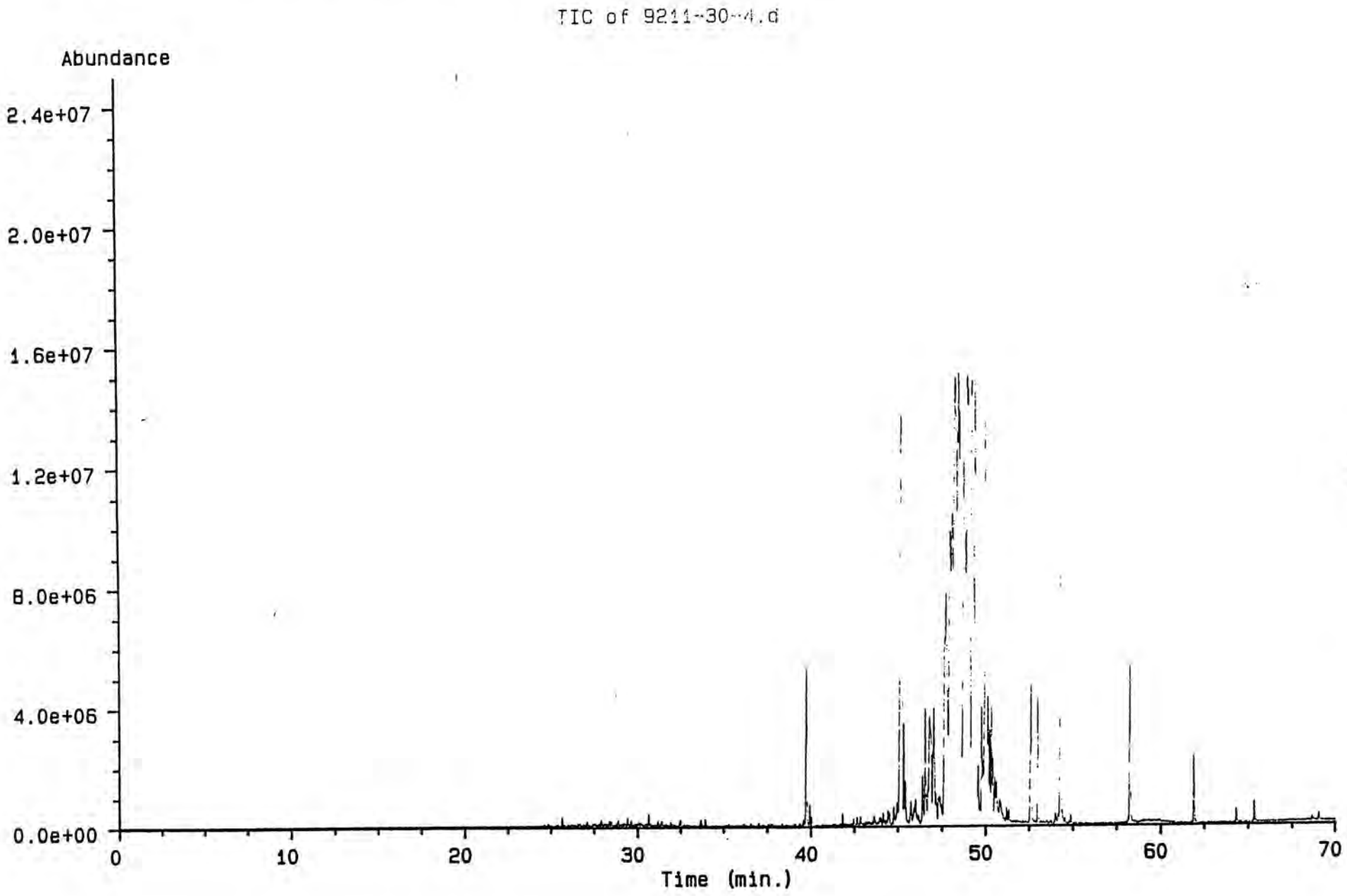


FIGURE 7: TOTAL ION CHROMATOGRAM OF THE UNWASHED SAMPLE (THIRD DESORPTION)



TT: 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, HIMI, density and TGA. Attached are the results of the X-ray fluorescence and TGA. The results of the HIMI and density testing are as follows:

Sample #	Form	Color	HIMI	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

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

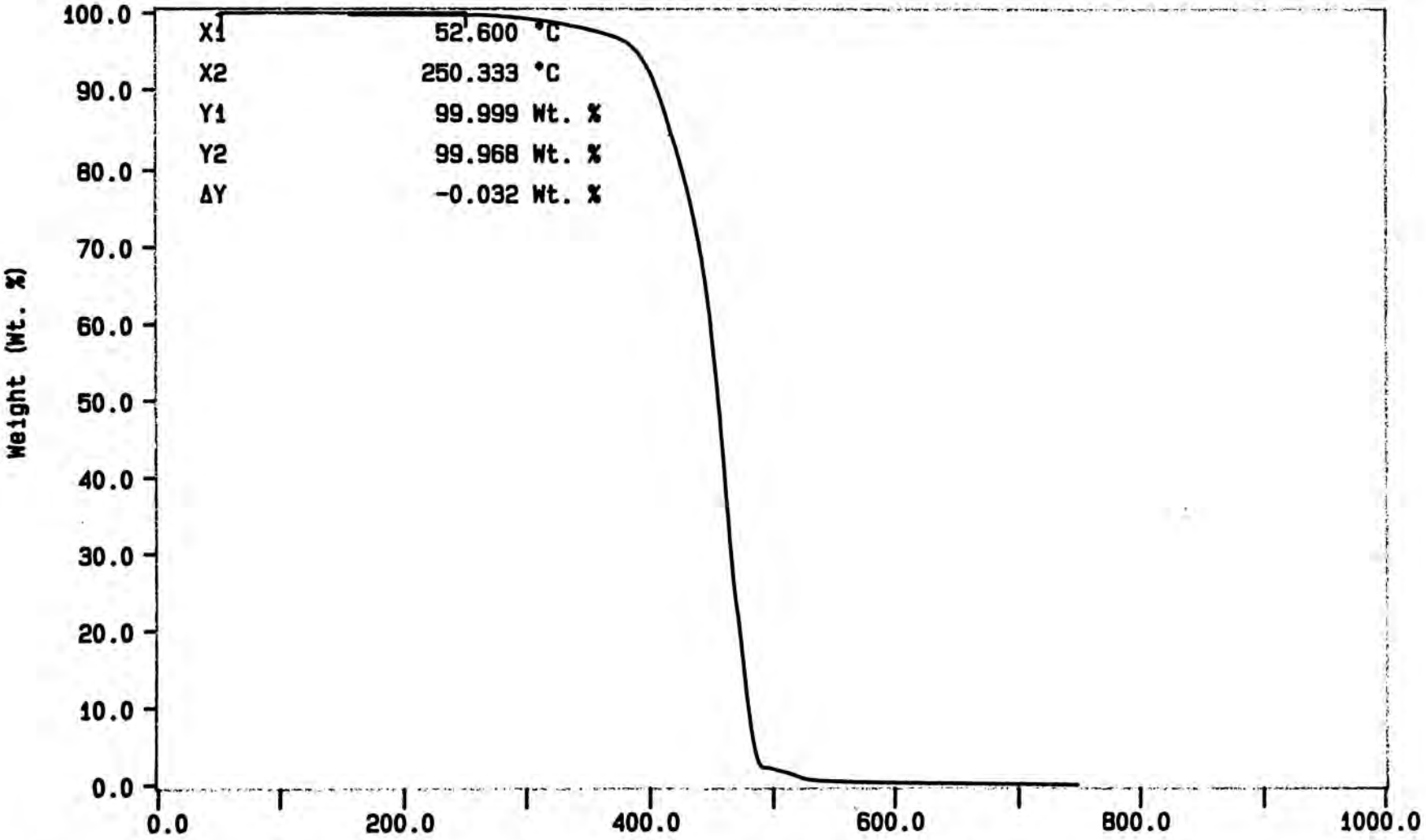
Attached are TGA data on the recycled PE flake and pellet. The experiments were performed using a dry nitrogen purge at a heating rate of 20 C/min for a temperature range of 50 to 900 C. The temperature was calibrated using an Alumel and a Nicoseal metal standard, according to the instructions in the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

I leave interpretation of the results 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)

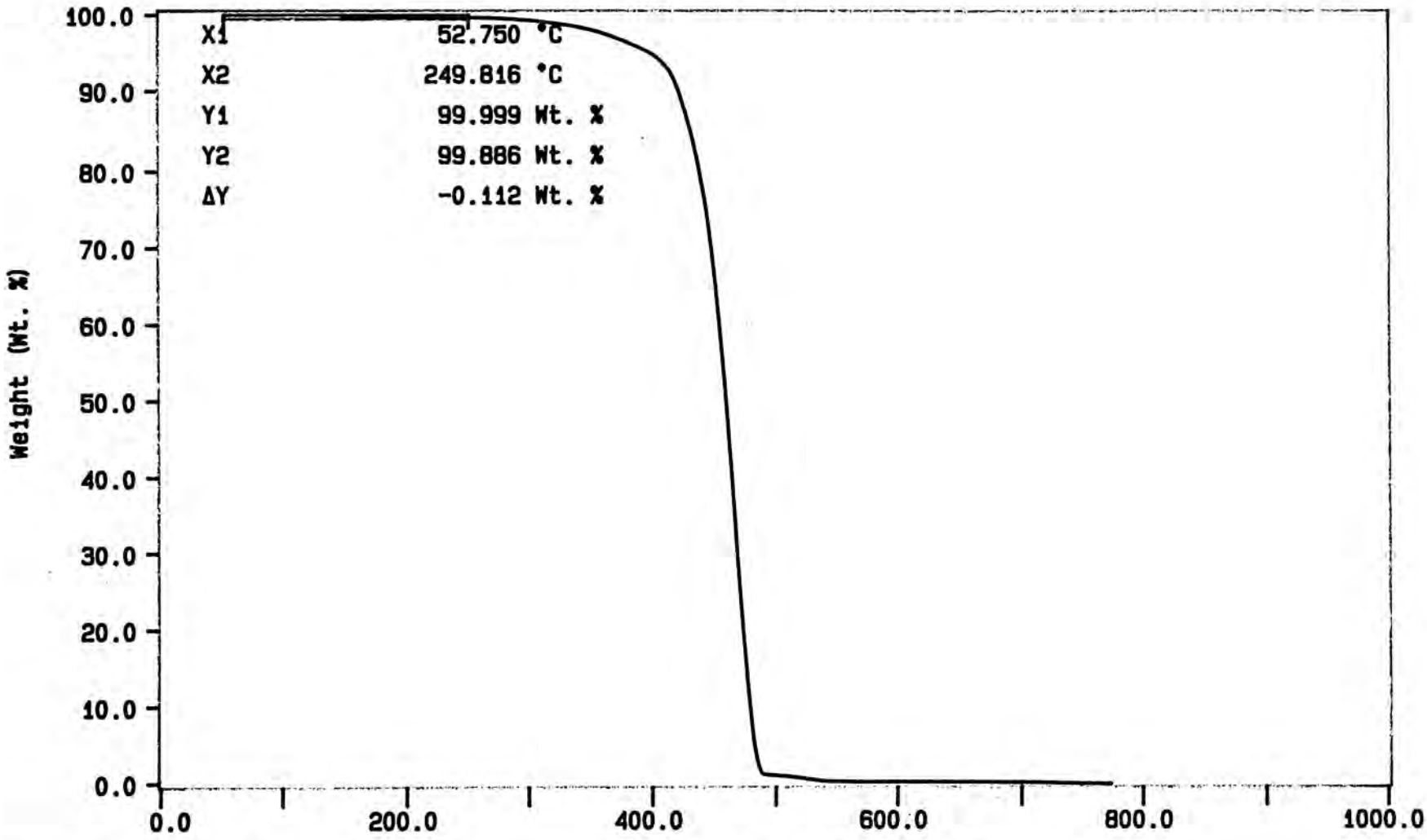


Requested by John Rathman

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

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)



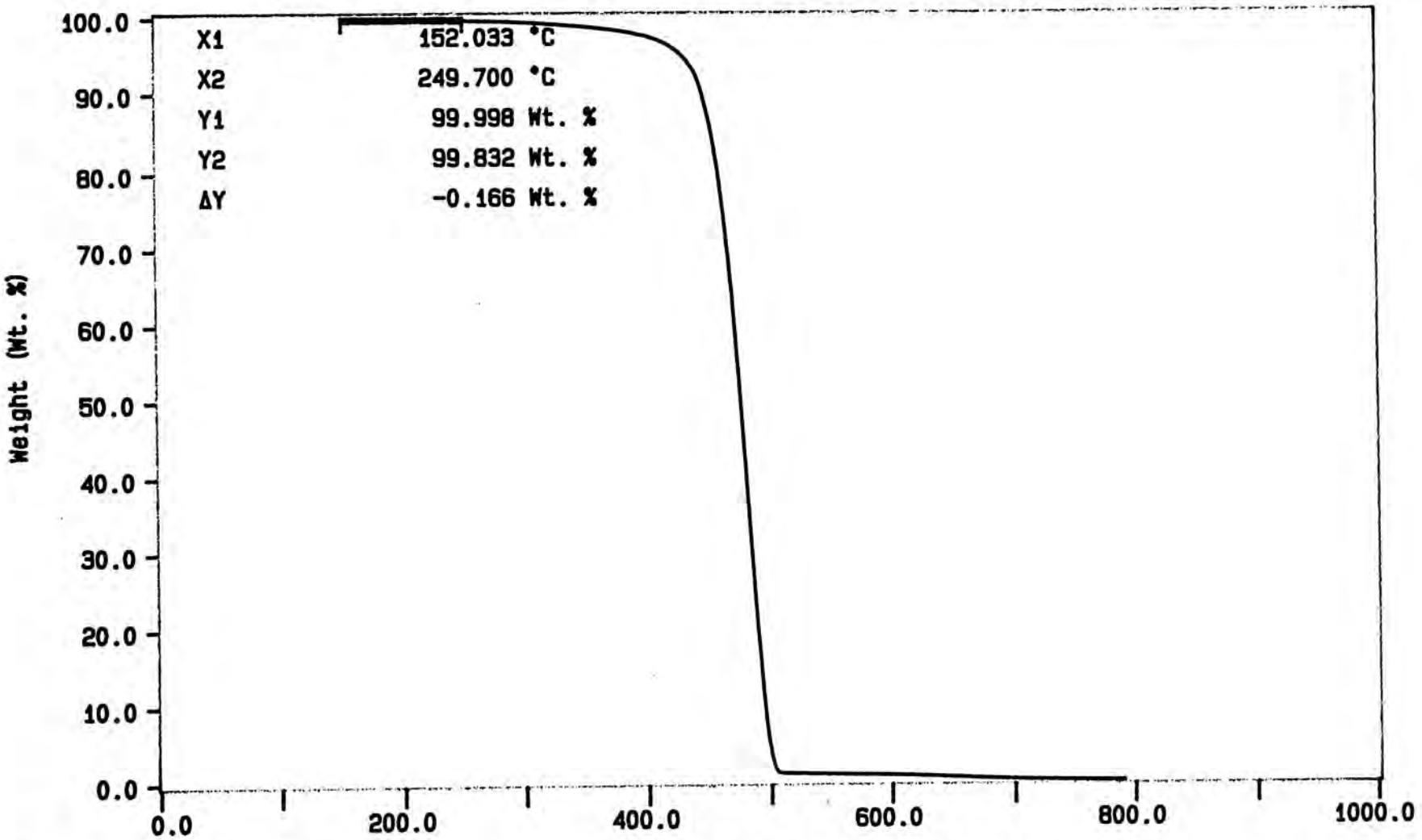
Requested by John Rathman

TEMP1: 50.0 C TIME1: 0.0 min RATE1: 20.0 C/min
TEMP2: 800.0 C

WWT
PERKIN-ELMER
7 Series Thermal Analysis System
Thu Feb 25 08:01:44 1993

67

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



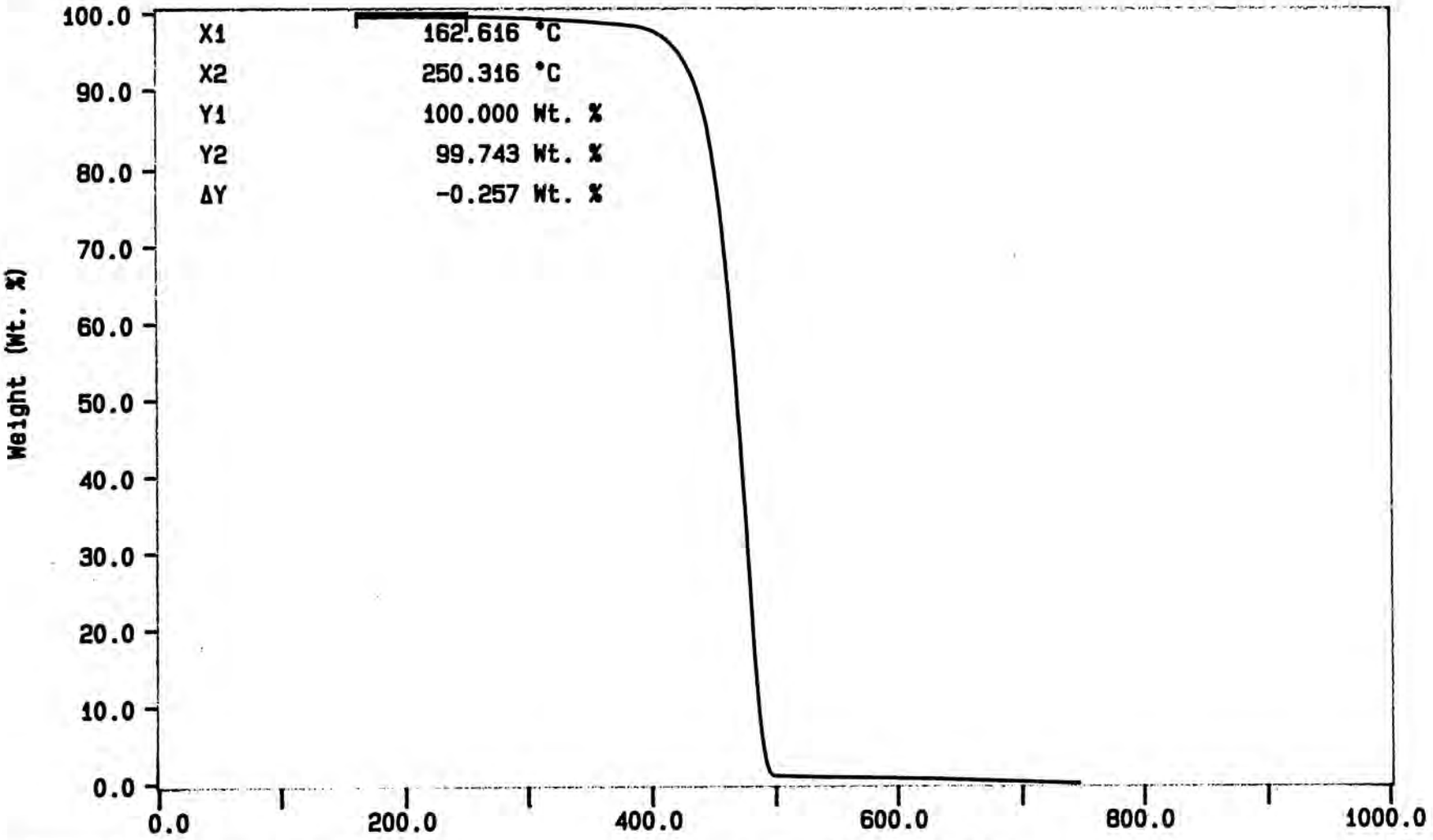
Requested by John Rathman

TEMP1: 50.0 C TIME1: 0.0 min RATE1: 20.0 C/min
TEMP2: 900.0 C

WWT
PERKIN-ELMER
7 Series Thermal Analysis System
Thu Feb 25 08:00:35 1993

'89

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



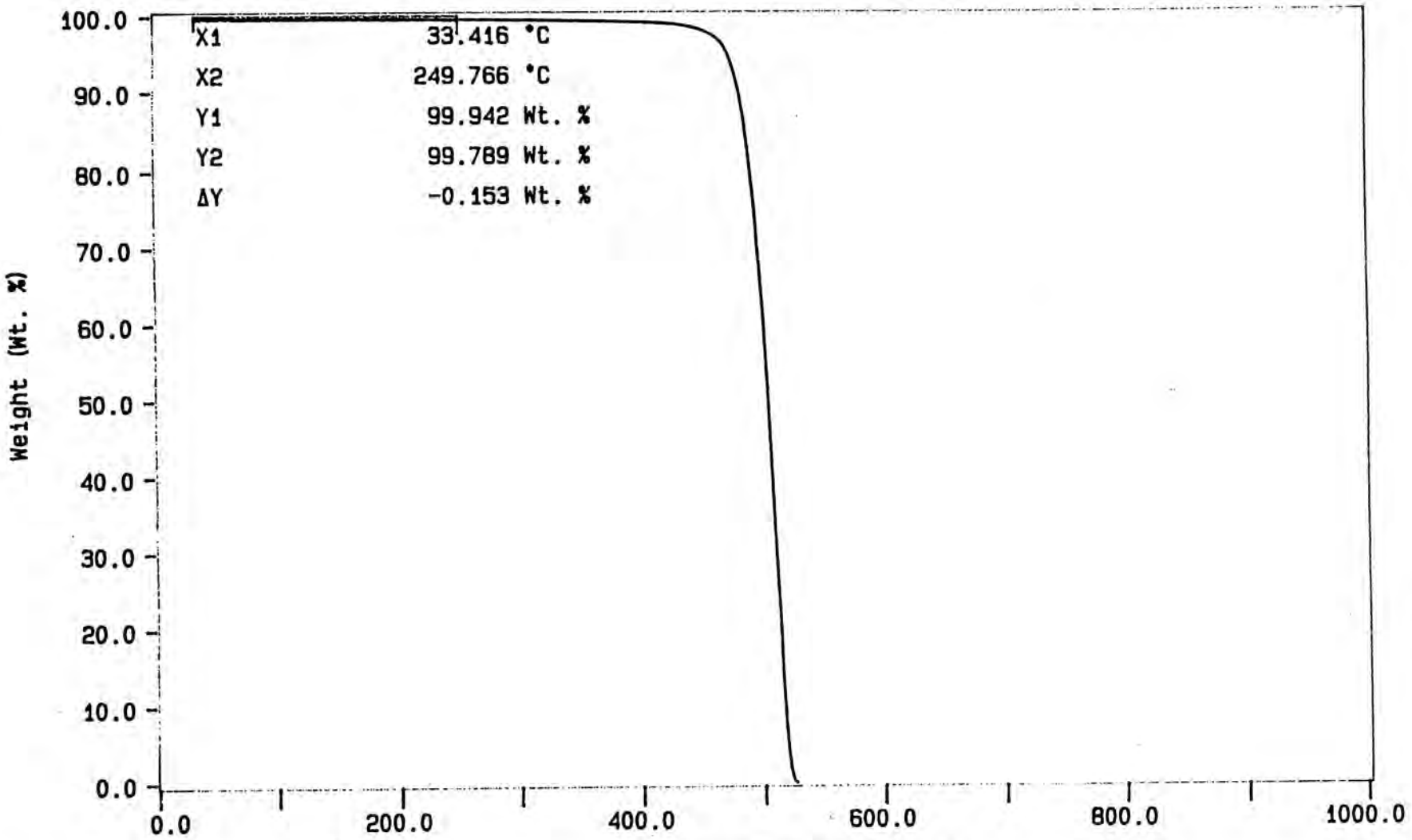
Requested by John Rathman

TEMP1: 50.0 C TIME1: 0.0 min RATE1: 20.0 C/min
TEMP2: 900.0 C

WWT
PERKIN-ELMER
7 Series Thermal Analysis System
Thu Feb 25 08:00:09 1993

69

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

Phillips HDPE Control TGA Results

PHILLIPS 66

Notegram

September 4, 1992

To: John Rathman
PTC

From: Kathy Swallows
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 pursing 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^{o}C

4. Decant extract into 500 mL volumetric flask. Wash polymer residue 2-3 times with 50 mL portions of destilled, 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 /METHOD	Reported to nearest 5 ppm S
BLUE FLAKE UNWASHED /5 BLUE FLAKE UNWASHED	8/14/92	9215657	SULFUR * /X-RAY	130 ppm S
COMMENT FOR S BY X-RAY ON 9215657: DIRECT COMPARISON TO S IN MINERAL OIL STDS.. X-RAY FLUPRESCENCE. APPROX. ~~WT.%~~ ppm SULFUR ONLY!!!!!!! LCP				
A BLUE PELLET WASHED /5A BLUE PELLET WASHED	8/14/92	9215658	SULFUR * /X-RAY	65
COMMENT FOR S BY X-RAY ON 9215658: DIRECT COMPARISON TO S IN MINERAL OIL STDS.. X-RAY FLUPRESCENCE. APPROX. ~~WT.%~~ ppm SULFUR ONLY!!!!!!! LCP				
BLACK FLAKE UNWASHED /6 BLACK FLAKE UNWASHED	8/14/92	9215659	SULFUR * /X-RAY	335
COMMENT FOR S BY X-RAY ON 9215659: DIRECT COMPARISON TO S IN MINERAL OIL STDS.. X-RAY FLUPRESCENCE. APPROX. ~~WT.%~~ ppm SULFUR ONLY!!!!!!! LCP				
A BLACK PELLET WASHED /6A BLACK PELLET WASHED	8/14/92	9215660	SULFUR * /X-RAY	155
COMMENT FOR S BY X-RAY ON 9215660: DIRECT COMPARISON TO S IN MINERAL OIL STDS.. X-RAY FLUPRESCENCE. APPROX. ~~WT.%~~ ppm SULFUR ONLY!!!!!!! LCP				

INDICATES PRIORITY REQUEST FOR TESTS.

*** END OF REPORT ***

SULFUR 4

P.P. FILM

	Avg. mm S	RMAC correction	Corrected Avg mm S	Report as Tenthest S
MOLDED SPEC. 3 each. WATERS				
STDE "A"	130.22	1.0170	132.43	130 mm
	62.32	1.0173	63.40	65 mm
STDE "B"				
STDE "A"	336.2	1.0024	337.01	335 mm
	156.73	1.0021	157.06	155 mm
STDE "B"				

PHILLIPS 66

Notegram

November 23, 1992

To: John Rathman
PTC

From: Kathy Swallows KJS
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 pellitized 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 inhomogenity 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 inhomogenity 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, contaminates 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

Attachment 1

Summary of Microtox Data for Plastic Drum Resins

Sample No.	Label	Color	Microtox EC50 5 min.	Microtox EC50 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™

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.

WHAT DO PEOPLE USE MICROTOX FOR?

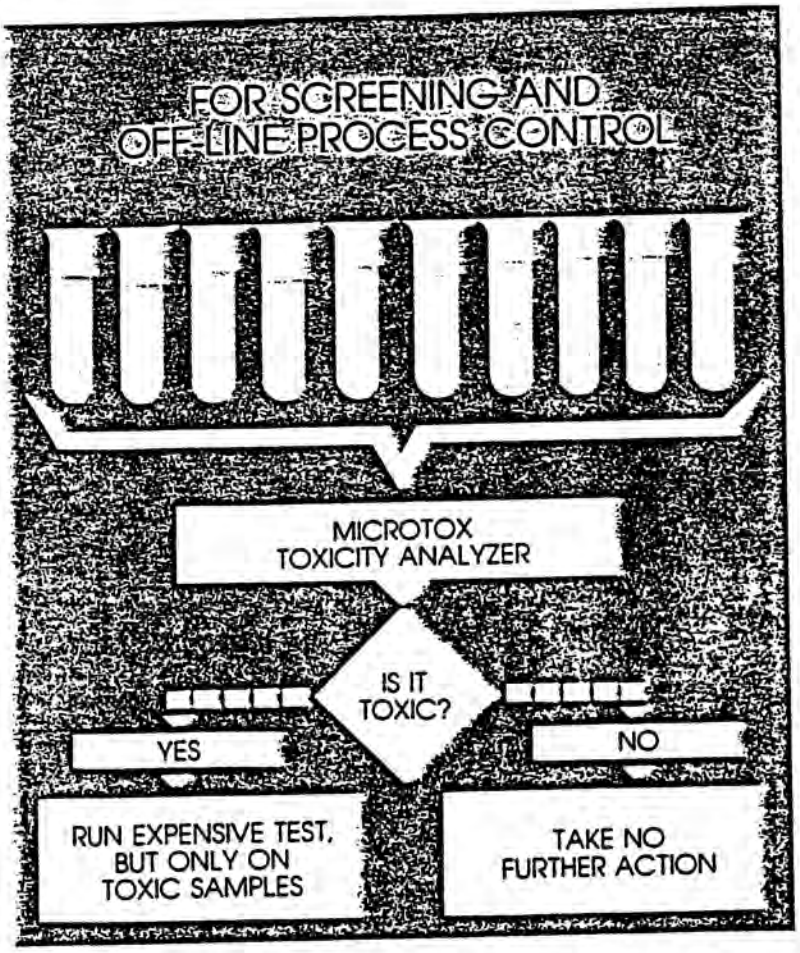
Microtox is used chiefly in two ways:

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.

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.



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.

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 conta with artificial ice by expedient routes to insure that arrives in good condition. It has a long shelf-life whe stored at refrigerator or colder temperatures.

MICROBI
CORPORAT
2233 Faraday
Carlsbad, CA 92008
(619) 438
Telex: 5106

The PDI Real World Test Part III

Drum Performance with Recycled Content

A Special Purpose Group of
The Society of the Plastics Industry, Inc.
1275 K Street, NW, Ste.400
Washington, DC 20005
(202) 371-5200

PDI REAL WORLD TEST, PART III

'DRUM PERFORMANCE WITH RECYCLED CONTENT"

TABLE OF CONTENTS

DRUM PERFORMANCE WITH RECYCLED CONTENT

I. INTRODUCTION

Three prior Plastic Drum Institute (PDI) reports have been issued on studies of High Density Polyethylene drums to determine their ability to be reused, the effect of lading contamination and ability to clean residual lading from used drum recycle resin. 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 ladings 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 ladings determined through quality control analysis upon the ladings 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 ladings packaged remained in the reconditioned drums. Most shippers concluded that the reconditioned plastic drums were acceptable for the ladings used. Also reconfirmed was that the ladings and reconditioning did not affect the structural integrity of the plastic drum, as seen in the 1983 study.

In 1993 the PDI issued a third report entitled "PDI Real World Test Part 2 - Resin Reconditioning Study". The PDI was interested in investigating the ability to recondition the High Density Polyethylene for a recycle material from plastic drums taken out of service. The drums were cleaned under standard drum reconditioning, ground into flake, the flake was washed in a secondary washing operation that is typical of post-consumer polyethylene recycle plants and then repelletized. The primary objective of this study was to then quantify the residual ladings remaining absorbed into the High Density Polyethylene at various step during the cleaning and repelleting process.

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 during various stages of the study.

II. DISCUSSION

The work conducted by PDI to date has shown that plastic drums can be successfully used in packaging various products without detriment to performance properties of the drum due to effects of the lading, primary or secondary, or cleaning processes. Also after the plastic drums have come to the end of their service life that the drums can then be ground up and the resin cleaned so that it can be recycled into a secondary application.

At the present time HM 181 standards state the following: "No used material other than production residues or regrind from the same manufacturing process may be used." This prohibits the use of drum recycle resin back into the production of new drums for service. Therefore, the PDI determined that an investigation into the feasibility of incorporating drum recycle resin back into new production 55 gallon plastic drums would be done. A program was developed with the following project steps:

- Material Procurement
- Material Preparation
- Drum Production
- Drum Testing

MATERIAL PROCUREMENT

An equal number of drums which had contained the following seven ladings were selected for use in this study:

- Apple Juice
- Sulfuric Acid
- Sodium Hydroxide
- Alcohol based flavor compound
- Hydrogen Peroxide
- Surfactant
- Biocide

The above ladings were chosen to represent a cross-section of the majority of drum types available. MSDS sheets for all regulated products are included in Appendix A. In order to ensure a consistent flow rate for the recycled resin, drums were also selected based on the knowledge of the flow properties of the original resins used by each drum manufacturer. All drums were produced with high density, high molecular weight polyethylene ASTM D1248, Type III, Category 5. Drums were selected to ensure a recycled product with a high load melt index of 3 - 5 dg/min.

MATERIAL PREPARATION

All drums were washed by Recycle, Inc. using a wash procedure which conforms to the EPA guidelines for triple rinsing. Drums were flaked and blended into 5 boxes containing 1000 lb. each. A composite sample was taken from each box for flow rate testing to ensure that the target flow rate range was met. The data are provided in Table 1. Oxygen induction time measurements were also taken to determine whether additional stabilization would be required during the pelletization of the flakes. It was determined that the resin was adequately stabilized. Corrosivity testing was also conducted to ensure that there would be no machine damage in processing this material. The tests showed the material to be non-corrosive. A headspace GC-MS analysis was conducted on the drum recycle regrind and drum recycle repelletized resin. Test results for both the corrosivity testing and the headspace GC-MS analysis are provided in Appendix B.

The boxes containing the flaked material were sent to Custom-Pak Extrusion to be pelletized. The material was pelletized for improved processing during the drum production trials. Material was then sent to Hunter Drums Ltd. for drum production trials.

DRUM PRODUCTION / TESTING

It has been shown in previous studies by the PDI that the physical properties of the plastic drum are not significantly altered through multiple reconditionings. It has also been established that only minimal residual lading remains in the polyethylene. The primary objective of this study was to demonstrate that recycled polyethylene taken from used plastic drums could be used in the manufacture of new drums. Also that these drums would be capable of passing performance testing as set out in HM 181.

The drum test program was conducted in two parts. In test I, drums were produced with a wide range of recycled content. The primary purpose for this test was to determine the feasibility of producing drums with various levels of regrind and in addition the effect on primary drum properties such as ESCR and stack performance. Following the success of test I, it was decided to conduct a second test in order to produce a set of drums for passing all HM 181 performance testing. Current legislation includes mandates for certain

consumer packaging of up to 25% recycled content. It was decided to conduct a full scale test to produce drums with 30% recycled content for complete testing.

TEST I

Drums with five percentages of drum recycle regrind (0, 10, 25, 50, 100) were produced during a one-day production trial at Hunter Drums Ltd. The virgin resin used to blend with the drum recycle regrind was a nominal 3.5 HLMI, high molecular weight, high density polyethylene from Novacor. The drum style produced for this trial was the Polycon 210 Liter Tight Head Drum. Observations were made of all critical processing conditions. Slight process changes due to the addition of the recycled material were adjusted for using routine production procedures. There was no change in the temperature settings, cycle time or throughput. A record of the weights and dimensions for every drum set was also recorded. A summary of bung dimensions and average drum weight are included in Tables 2 and 3, respectively. Drums with each of the five regrind levels were sent to TEN-E Packaging Services, Inc. for ESCR and 28-day stack testing. Drum ESCR testing was performed according to ASTM D5571 and 28-day stack testing was performed in accordance with the Department of Transportation's Title 49 CFR: Section 178.606. A summary of the test results are shown in the Table 4. A complete test report is included in Appendix C, which also includes flow rate and density values as well as wall thickness profiles and weights measured on each drum set. All drums, including those produced with 100% of the recycled material, passed the ESCR and 28-day stack testing.

TEST II

Drums with 0% and 30% recycled content were produced during a one-day production trial at Hunter Drums Ltd. The virgin resin used to blend with the drum recycle regrind was a nominal 3.5 HLMI, high molecular weight, high density polyethylene from Novacor. The drum style produced for this trial was the Polycon 210 Liter Tight Head Drum. Drums produced with 0 and 30% recycled content were sent to TEN-E Packaging Services, Inc. for complete performance testing. A summary of the test results are shown in the Table 5. A complete test report is included in Appendix 4. Drum testing was conducted in conformance with the Department of Transportation's Title 49 CFR for packing group II. Drums produced with 30% recycled drum resin passed all performance tests.

III. CONCLUSIONS

» Recycled material from various drum ladings can be successfully handled through grinding and repelletization.

» Recycled material could be manufactured into new drums at up to 100%.

» Drum ESCR and 28 day stack performance was maintained with up to 100% recycled content.

» Drums produced at a chosen recycled content of 30% passed all UN performance testing for packing group II.

Table 1

FLAKED MATERIAL FLOW PROPERTIES

BOX	FLOW RATE (dg/min), (190/21.6)
1	5.0
2	4.7
3	4.8
4	4.5
5	4.9

Table 2

BUNG DIMENSIONS

DRUM	COARSE THREAD DIAMETER VERTICAL (inch)	COARSE THREAD DIAMETER HORIZ. (inch)	OUT OF ROUND	FINE THREAD DIAMETER VERTICAL (inch)	FINE THREAD DIAMETER HORIZ. (inch)	OUT OF ROUND
0% RECYCLE	2.38	2.40	0.02	2.22	2.24	0.02
10% RECYCLE	2.38	2.40	0.02	2.23	2.23	0.00
25% RECYCLE	2.39	2.40	0.01	2.22	2.24	0.02
50% RECYCLE	2.37	2.40	0.03	2.21	2.22	0.01
100% RECYCLE	2.38	2.37	0.01	2.22	2.24	0.02

Table 3

DRUM WEIGHT DATA

DRUM		DRUM WEIGHT (KG)	FLASH WEIGHT (KG)	TOTAL WEIGHT (KG)
0% RECYCLE	AVERAGE	10.22	1.96	12.19
	STAND. DEV.	0.02	0.03	0.03
10% RECYCLE	AVERAGE	10.21	1.93	12.13
	STAND. DEV.	0.01	0.02	0.02
25% RECYCLE	AVERAGE	10.24	1.98	12.22
	STAND. DEV.	0.03	0.05	0.05
50% RECYCLE	AVERAGE	10.26	1.90	12.16
	STAND. DEV.	0.02	0.02	0.03
100% RECYCLE	AVERAGE	10.16	1.90	12.06
	STAND. DEV.	0.03	0.05	0.06

Table 4

TEN-E TEST RESULTS FROM SEPTEMBER 30, 1994

	100 % Virgin 0% PCR	90 % Virgin 10% PCR	75 % Virgin 25% PCR	50 % Virgin 50% PCR	0 % Virgin 100% PCR
28 DAY STACK TEST Specific Gravity: 1.8	3/3 Passed	3/3 Passed	3/3 Passed	3/3 Passed	3/3 Passed
ESCR (days) 10% Igepal, 10% rated capacity, 2 psi internal pressure, 50°C					
Drum 1	21	12	18	16	21
Drum 2	21	18	21	23	21
Drum 3	18	20	14	No Failure	25

Table 5

TEN-E TEST RESULTS
FROM
MARCH 24, 1995

	100 % Virgin 0% PCR	70% Virgin 30% PCR
DROPS 1.5M, -18°C		
Diagonal Top Chime	3/3 Passed	3/3 Passed
Flat On Side	3/3 Passed	3/3 Passed
LEAK PROOFNESS Test Pressure 20 Kpa	3/3 Passed	3/3 Passed
HYDROSTATIC PRESSURE Test Pressure 100 Kpa	3/3 Passed	3/3 Passed
28 DAY STACK TEST Specific Gravity: 1.8	3/3 Passed	3/3 Passed
VIBRATION TEST	3/3 Passed	3/3 Passed
DYNAMIC COMPRESSION	3/3 Passed	3/3 Passed
ESCR (days) 10% Igepal, 10% rated capacity, 2 psi internal pressure, 50°C		
Drum 1	13	21
Drum 2	16	13
Drum 3	15	15

APPENDIX A

MSDS SHEETS

RECYCLE INC.

450 METUCHEN ROAD • SOUTH PLAINFIELD • NEW JERSEY • 07080 • (908) 756-2200 • FAX (908) 756-4524

March 15, 1994

Ms. Nancy Conley
NOVACOR CHEMICALS LTD.
Plastics Division
3100 Cote Vertu, Suite 460
Saint Laurent, Quebec, Canada H4R 2J8

Dear Nancy:

Enclosed please find the Material Safety Data Sheets you requested. Please feel free to call if you have any questions.

Sincerely,

Jeffrey L. Bey
V.P. Operation

JLB/jmm

Enclosures

DU PONT

MATERIAL SAFETY DATA SHEET

IDENTIFICATION

NAME
Sulfuric Acid, 77 to 100%

GRADE
60° Tech., 66° Tech.,
1.835 Electrolyte, 98% Tech.,
99% Tech., 100% Tech.

CAS REGISTRY NO.
7664-93-9

CAS NAME
Sulfuric Acid

I.D. NOS./CODES
NIOSH Registry No.: WS5600000

MANUFACTURER/DISTRIBUTOR
E. I. du Pont de Nemours & Co. (Inc.)

ADDRESS
Wilmington, DE 19898

CHEMICAL FAMILY
Mineral Acid

FORMULA
H_2SO_4

TSCA INVENTORY STATUS
Reported/Included

SARA/TITLE III STATUS
See ADDITIONAL INFORMATION and HAZARDOUS COMPONENTS sections

PRODUCT INFORMATION PHONE
(800) 441-9442

MEDICAL EMERGENCY PHONE
(800) 441-3637

TRANSPORTATION EMERGENCY PHONE
CHEMTREC (800) 424-9300

PHYSICAL DATA

BOILING POINT, 760 mmHg
193°C to 327°C (380°F to 621°F)
See page 2 for specific grades

SPECIFIC GRAVITY
1.70 to 1.85
See page 2

VAPOR DENSITY
3.4

pH INFORMATION
<1

FORM
Liquid

COLOR
Colorless to light gray

MELTING POINT
-35°C to 11°C (-31°F to 51°F)
See page 2

VAPOR PRESSURE
<0.3 mmHg at 25°C (77°F)
<0.6 mmHg at 38°C (100°F)

SOLUBILITY IN WATER
100%

EVAPORATION RATE (Butyl Acetate = 1)
<1

APPEARANCE
Oily; clear to turbid

ODOR
Odorless

PHYSICAL PROPERTIES (FROM PAGE 1)

Grade	Boiling Point °C	Boiling Point °F	Melting Point °C	Melting Point °F	Specific Gravity
60° Technical	193	380	-12	10	1.706
66° Technical	279	535	-35	-31	1.835
1.835 Electrolyte	279	535	-35	-31	1.835
98% Technical	327	621	-2	29	1.844
99% Technical	310	590	4	40	1.842
100% Technical	274	526	11	51	1.839

HAZARDOUS COMPONENTS

MATERIAL(S)	CAS NO.			APPROXIMATE %
Sulfuric Acid*	7664-93-9	60°	Technical	77.7
		66°	Technical	93.2
		1.835	Electrolyte	93.2
		98%	Technical	98.0
		99%	Technical	99.0
		100%	Technical	100

*Regulated as a Toxic Chemical under Section 313 of Title III/SARA, and 40 CFR Part 372.

NONHAZARDOUS COMPONENT

Water	7732-18-5	0-22

HAZARDOUS REACTIVITY

INSTABILITY
Stable, but reacts violently with water and organic materials with evolution of heat.

INCOMPATIBILITY
Vigorous reactions occur with water; alkaline solutions; metals; metal powders; carbides; chlorates; fulminates; nitrates; picrates; strong oxidizing, reducing, or combustible organic materials. Hazardous gases are evolved on contact with chemicals such as cyanides, sulfides, and carbides.

DECOMPOSITION
Releases sulfur dioxide at extremely high temperatures.

POLYMERIZATION
Will not occur.

FIRE AND EXPLOSION DATA

FLASH POINT
Will not burn.

FLAMMABLE LIMITS IN AIR, % BY VOL.
LOWER Not applicable.
UPPER Not applicable.

AUTOIGNITION TEMPERATURE
Not applicable.

AUTODECOMPOSITION TEMPERATURE
Not available.

FIRE AND EXPLOSION HAZARDS
Reacts with most metals, especially when dilute, to give flammable, potentially explosive hydrogen gas.

EXTINGUISHING MEDIA
Use media as appropriate for combustibles in area. Use water spray to cool containers exposed to fire; do not get water inside containers.

SPECIAL FIREFIGHTING INSTRUCTIONS
Evacuate personnel to a safe area. Keep personnel removed and upwind of fire. Generates heat upon addition of water, with possible spattering. Wear full protective clothing. Runoff from fire control may cause pollution. Neutralize runoff with lime, soda ash, etc., to prevent corrosion of metals and formation of hydrogen gas. Wear self-contained breathing apparatus (SCBA) if fumes or mists are present.

HEALTH HAZARD INFORMATION

PRINCIPAL HEALTH HAZARDS (Including Significant Routes, Effects, Symptoms of Overexposure, and Medical Conditions Aggravated by Exposure)

Causes severe burns to eyes, skin and all body tissues. Inhalation of mist may cause lung damage.

Inhalation 1-hour LC_{50}: 347 ppm in rats
Oral LD_{50}: 2140 mg/kg in rats

The concentrated compound is corrosive to the skin and the eye of animals. By ingestion it is moderately toxic in animals causing corrosion of mucosal surfaces. Toxic effects described in animals from single exposures by inhalation include respiratory irritation. Animal testing indicates that this compound does not have the potential to produce carcinogenic, mutagenic, embryotoxic, or reproductive effects.

Human health effects of overexposure to the liquid by skin or eye contact may cause eye corrosion with corneal or conjunctival ulceration; or skin burns or ulceration. Ingestion of the liquid may cause severe burns to the mucous membranes of the mouth and esophagus. Repeated or prolonged contact with mists may cause eye irritation with discomfort, tearing, or blurring of vision; or skin irritation with discomfort or rash. Human health effects of overexposure by inhalation may include irritation of the upper respiratory passages or erosion of dental surfaces. Higher exposures by inhalation may lead to temporary lung irritation effects with cough, discomfort, difficulty breathing, or shortness of breath; or possibly modest initial symptoms, followed in hours by severe shortness of breath, requiring prompt medical attention.

HEALTH HAZARD INFORMATION (cont'd)

Although two epidemiology studies did suggest a possible association between sulfuric acid exposure and respiratory tract tumors, conclusions from these studies are very limited because of significant deficiencies. In one study for example, only a small number of workers was sampled, and there was exposure to other materials including diethyl sulfate, an IARC and NTP carcinogen. In the other study there was exposure to other acids, plus the smoking histories of the workers were ignored in the development of the study conclusion. Based on the overall weight of evidence from all human and chronic animal studies, we conclude that no causal relationship between sulfuric acid exposure and respiratory tract tumors has been shown.

Individuals with preexisting diseases of the lungs may have increased susceptibility to the toxicity of excessive exposures.

CARCINOGENICITY

Not listed as a carcinogen by IARC, NTP, OSHA, or ACGIH.

EXPOSURE LIMITS [PEL (OSHA), TLV (ACGIH), AEL (DU PONT), ETC.]

OSHA 8-hour Time Weighted Average (TWA) and ACGIH TLV(R)-TWA are 1 mg/m^3 (ACGIH STEL = 3 mg/m^3). The Du Pont AEL 8 and 12-hour TWA is 1 mg/m^3.

SAFETY PRECAUTIONS

Do not get in eyes, on skin, on clothing.
Avoid breathing vapor or mists.
Keep containers closed.
Do not add water to contents while in container because of violent reaction.
Wash thoroughly after handling.

FIRST AID

In case of contact: immediately (within seconds) flush the skin or eyes with plenty of water (preferably cold water) for at least 15 minutes while removing contaminated clothing and shoes. Call a physician. Wash clothing before reuse.

While the patient is being transported to a medical facility, apply compresses of ice water. If medical treatment must be delayed, immerse the affected area in iced water. If immersion is not practical, compresses of iced water can be applied. Avoid freezing tissues.

Note to Physician: Continued washing of the affected area with cold or iced water will be helpful in removing the last traces of sulfuric acid. Creams or ointments should not be applied before or during the washing phase of the treatment.

If inhaled: Remove to fresh air immediately and have patient lie down and remain quiet. Apply artificial respiration if breathing has stopped. Give oxygen if breathing is difficult. Call a physician.

If swallowed: Do not induce vomiting. Give large quantities of water. Call a physician. Do not give carbonates. Never give anything by mouth to an unconscious person.

ROTECTION INFORMATION

GENERALLY APPLICABLE CONTROL MEASURES

Good general ventilation should be provided to keep vapor and mist concentrations below the exposure limits.

PERSONAL PROTECTIVE EQUIPMENT

Have available and wear as appropriate for exposure conditions when handling containers or operating equipment containing sulfuric acid: chemical splash goggles; full-length face shield/chemical splash goggle combination; acid-proof gauntlet gloves, apron, and boots; long sleeve wool, acrylic, or polyester clothing; acid-proof suit and hood; and OSHA permissible respiratory protection. In case of emergency or where there is a strong possibility of considerable exposure, wear a complete acid suit with hood, boots, and gloves. If acid vapor or mist are present and exposure limits may be exceeded, wear OSHA permissible respiratory protection.

ISPOSAL INFORMATION

AQUATIC TOXICITY

The 48-hour TLm in flounder is 100-300 ppm.

SPILL, LEAK OR RELEASE

Stop flow if possible. Review "Fire and Explosion Hazards" and "Safety Precautions" before proceeding with clean up. Use appropriate protective equipment during clean up. Soak up small spills with dry sand, clay, or diatomaceous earth. Dike large spills and cautiously dilute, neutralize with lime or soda ash, and transfer to waste water treatment system. Prevent liquid from entering sewers, waterways, or low areas. Comply with Federal, State, and local regulations on reporting releases. Superfund reportable discharge is 1000 lbs.

WASTE DISPOSAL

Cleaned-up material is a RCRA Hazardous Waste. Do not flush to surface water or sanitary sewer system. Comply with Federal, State, and local regulations. If approved, neutralize and transfer to waste treatment system.

HIPPING INFORMATION

DOT (172.101)

PROPER SHIPPING NAME
Sulfuric Acid

HAZARD CLASS
Corrosive Material

UN NO.
1830

DOT LABEL(S)
Corrosive

DOT PLACARD (TT/TC)
Corrosive

DOT/IMO (172.102)

PROPER SHIPPING NAME
Sulphuric Acid

HAZARD CLASS
Corrosive Material, 8

UN NO.
1830

PACKAGING GROUP II

SHIPPING INFORMATION (cont'd)

REPORTABLE QUANTITY
1000 lb/454 kg

SHIPPING CONTAINERS
Tank cars, tank trucks, 55 gallon stainless steel drums.

ADDITIONAL INFORMATION AND REFERENCES

STORAGE CONDITIONS
Keep out of sun and away from heat, sparks, and flame. Keep container tightly closed and (drum) closure up to prevent leakage. Loosen closure carefully. Relieve internal pressure when received and at least weekly thereafter. Do not use pressure to empty. Be sure closure is securely fastened before moving container. Do not wash out container or use it for other purposes; replace closure after each withdrawal and return it with empty container.

NPCA - HMIS RATINGS

Health (Acute)	3
Flammability	0
Reactivity	2
Personal Protection	-

Personal Protection rating to be supplied by user depending on use conditions.

NFPA RATINGS

Health	3
Flammability	0
Reactivity	2
Unusual Hazards	W

SARA/TITLE III HAZARD CATEGORIES AND LISTS

Product Hazard Categories:

Chronic Health	- Yes
Acute Health	- Yes
Fire Hazard	- No
Pressure Hazard	- No
Reactivity Hazard	- Yes

Lists:

Extremely Hazardous Substance	- Yes
CERCLA Hazardous Substance	- Yes
Toxic Chemical	- Yes

For further information, see Du Pont Sulfuric Acid "Storage and Handling Bulletin."

DATE OF LATEST REVISION/REVIEW: 10/88

PERSON RESPONSIBLE FOR MSDS:
J. C. WATTS
Du Pont Co.
Chemicals & Pigments Department
Chestnut Run Plaza
P.O. Box 80709
Wilmington, DE 19880-0709
(302) 999-4946

118547

MATERIAL SAFETY DATA SHEET

Diamond Shamrock
Chemicals Company

MSDS NUMBER: M4806

MSDS DATE: 05-15-86

PRODUCT NAME: **50% CAUSTIC SODA-DB**

24 HOUR EMERGENCY PHONE: (214) 922-2700 Scott / Brown Chem

I. PRODUCT IDENTIFICATION

3 HEALTH HAZARD, 0 FIRE HAZARD, & 1 REACTIVITY rating based on NIOSH "Identification System for Occupationally Hazardous Materials" (1974)

MANUFACTURER'S NAME AND ADDRESS: Diamond Shamrock Chemicals Company, Chlor-Alkali Division, 351 Phelps Court, P.O. Box 152300, Irving, Texas 75015-2300

CHEMICAL NAME: Sodium Hydroxide CAS NUMBER: 1310-73-2

SYNONYMS/COMMON NAMES: Sodium Hydroxide; NaOH

CHEMICAL FORMULA: NaOH

DOT PROPER SHIPPING NAME: Caustic Soda, Liquid

DOT HAZARD CLASS: Corrosive Material

DOT I.D. NUMBER: UN1824 HAZARDOUS SUBSTANCE: RQ1000

II. HAZARDOUS INGREDIENTS

MATERIAL OR COMPONENT	HAZARD DATA	CAS NUMBER	%
Sodium Hydroxide	PEL = 2 mg/m^3 TLV = 2 mg/m^3 Ceiling	1310-73-2	50
Water		7732-18-5	50

See Section V

This material is listed in the TSCA Inventory.
Not listed as carcinogenic by IARC, NTP, OSHA, ACGIH.

III. PHYSICAL DATA

BOILING POINT @ 760 mm Hg: 143°C VAPOR DENSITY (Air=1): N/A

FREEZING POINT: 12.1°C (54°F) % VOLATILES BY VOL.: <50%

VAPOR PRESSURE: 13 mm Hg @ 60°C

SPECIFIC GRAVITY (H_2O=1): 1.54 @ 15.6°C

SOLUBILITY IN H_2O % BY WT: Completely Soluble

APPEARANCE AND ODOR: Clear with no odor

pH: 7.5% solution has pH 14.0

CAS = Chemical Abstract Service Number
PEL = OSHA Permissible Exposure Limit
TLV = TLV®, ACGIH Threshold Limit Value, Current

N/A = No relevant information found or not available
NA = Not applicable

IV. FIRE AND EXPLOSION DATA

FLASH POINT: NA AUTOIGNITION TEMPERATURE: Nonflammable

FLAMMABLE LIMITS IN AIR, % BY VOLUME- UPPER: Nonflammable
LOWER: Nonflammable

EXTINGUISHING MEDIA:
This product is not combustible. Water spray, foam, carbon dioxide or dry chemicals may be used where this product is stored.

SPECIAL FIRE FIGHTING PROCEDURES:
Protective clothing and pressure-demand, self-contained breathing apparatus should be worn by firefighters in areas where product is stored.

UNUSUAL FIRE AND EXPLOSION HAZARD:
None. See Reactivity (Section VI).

V. HEALTH HAZARD INFORMATION

HEALTH HAZARD DATA:
Caustic Soda is a corrosive material.
Sodium Hydroxide: Acute Oral LD_{50} = 140-340 mg/kg (rat)
Acute Dermal LD_{50} = 1.35 gm/kg (rabbit)

ROUTES OF EXPOSURE

INHALATION:
Airborne concentrations of dust, mist, or spray of caustic soda may cause damage to the upper respiratory tract and even to the lung tissue proper which could produce chemical pneumonia, depending upon severity of exposure.

SKIN CONTACT:
This product is destructive to tissues contacted and produces severe burns.

SKIN ABSORPTION:
See Skin Contact above.

EYE CONTACT:
This product is destructive to eye tissues on contact. Will cause severe burns that result in damage to the eyes and even blindness.

INGESTION:
This product, if swallowed, can cause severe burns and complete tissue perforation of mucous membranes of the mouth, throat, esophagus and stomach.

EFFECTS OF OVEREXPOSURE

ACUTE:
Corrosive to all body tissues with which it comes in contact.

CHRONIC:
The chronic local effect may consist of multiple areas of superficial destruction of the skin or of primary irritant dermatitis. Similarly, inhalation of dust, spray, or mist may result in varying degrees of irritation or damage to the respiratory tract tissues and an increased susceptibility to respiratory illness.

EMERGENCY AND FIRST AID PROCEDURES

EYES:
OBJECT IS TO FLUSH MATERIAL OUT IMMEDIATELY THEN SEEK MEDICAL ATTENTION. IMMEDIATELY flush eyes with large amounts of water for at least 15 minutes, holding lids apart to ensure flushing of the entire surface. Washing eyes within several seconds is essential to achieve maximum effectiveness. Seek medical attention immediately.

SKIN:
Wash contaminated areas with plenty of water for 15 minutes. Remove contaminated clothing and footwear and wash clothing before reuse. Discard footwear which cannot be decontaminated. Seek medical attention immediately.

V. HEALTH HAZARD INFORMATION ....continued

INHALATION:
Get person out of contaminated area to fresh air. If breathing has stopped, resuscitate and administer oxygen if readily available. Seek medical attention immediately.

INGESTION:
NEVER give anything by mouth to an unconscious person. If swallowed, DO NOT INDUCE VOMITING. Give large quantities of water. If available, give several glasses of milk. If vomiting occurs spontaneously, keep airway clear. Seek medical attention immediately.

VI. REACTIVITY DATA

CONDITIONS CONTRIBUTING TO INSTABILITY:
Under normal conditions, this material is stable.

INCOMPATIBILITY:
See Mixing and Handling (Section IX). Avoid direct contact with water. This product may be added slowly to water or acids with dilution and agitation to avoid a violent reaction. When handling this product, avoid contact with aluminum, tin, zinc, and alloys containing these metals. Do not mix with strong acids without dilution and agitation to prevent violent or explosive reaction. Avoid contact with leather or wool.

HAZARDOUS DECOMPOSITION PRODUCTS:
None.

CONDITIONS CONTRIBUTING TO HAZARDOUS POLYMERIZATION:
Material is not known to polymerize.

VII. ENVIRONMENTAL PROCEDURES

STEPS TO BE TAKEN IF MATERIAL IS RELEASED OR SPILLED:
Leaks should be stopped. Spills should be contained and cleaned up immediately. Spills should be removed by using a vacuum truck. Neutralize remaining traces of material with any dilute inorganic acid such as hydrochloric, sulfuric, nitric, phosphoric, and acetic acid. The spill area should then be flushed with water followed by liberal covering of sodium bicarbonate. All clean-up material should be removed and placed in approved containers, labeled and stored in a safe place to await proper treatment or disposal. Spills on areas other than pavement, e.g., dirt or sand, may be handled by removing the affected soils and placing in approved containers. Persons performing clean-up work should wear adequate personal protective equipment and clothing.

Caution: Caustic Soda may react violently with acids and water.

WASTE DISPOSAL METHOD:
The materials resulting from clean-up operations may be hazardous wastes and, therefore, subject to specific regulations. Package, store, transport, and dispose of all clean-up materials and any contaminated equipment in accordance with all applicable federal, state, and local health and environmental regulations. Shipments of waste materials may be subject to manifesting requirements per applicable regulations. Appropriate disposal will depend on the nature of each waste material and should be performed by competent and properly permitted contractors. Ensure that all responsible federal, state, and local agencies receive proper notification of spill and disposal methods.

VIII. INDUSTRIAL HYGIENE CONTROL MEASURES

VENTILATION REQUIREMENTS:
Use adequate local exhaust ventilation where mist, sprays or resuspended dust may be generated. NOTE: Where carbon monoxide or other reaction products may be generated, special ventilation may be required.

SPECIFIC PERSONAL PROTECTIVE EQUIPMENT

RESPIRATORY:
Respiration protection is not required under normal use. Use NIOSH/MSHA approved respirator where mists, sprays or resuspended dust may be generated. Follow manufacturer's recommendations.

EYE:
Face shield and goggles or chemical goggles should be worn.

GLOVES:
Gloves should be worn. Gloves may be decontaminated by washing with mild soap and water. Natural and butyl rubber have been suggested.

OTHER CLOTHING AND EQUIPMENT:
Protective clothing to minimize skin contact. Chemically resistant safety shoes. Wash contaminated clothing with soap and water and dry before reuse. Showers and eyewash facilities should be accessible.

MONITORING EXPOSURE

BIOLOGICAL:
N/A

PERSONAL/AREA:
Use NIOSH Analytical Method No. 7401.

IX. SPECIAL PRECAUTIONS

SIGNAL WORD: DANGER!

STATEMENT OF HAZARDS:
CAUSES SEVERE BURNS TO SKIN AND EYES
CONTACT WITH EYES CAN CAUSE PERMANENT EYE DAMAGE
INHALATION OF DUST, MIST OR SPRAY CAN CAUSE SEVERE LUNG DAMAGE
CAN REACT VIOLENTLY WITH WATER, ACIDS AND OTHER SUBSTANCES

PRECAUTIONARY STATEMENTS:
Do not get into eyes, on skin, on clothing.
Avoid breathing dust, mists, or spray.
Do not take internally.
Use with adequate ventilation and employ respiratory protection when exposure to dust, mist or spray is possible.
When handling, wear chemical splash goggles, face shield, rubber gloves and protective clothing.
Wash thoroughly after handling or contact - exposure can cause burns which are not immediately painful or visible.
Keep container closed.
Product can react violently with water, acids, and other substances - read Special Mixing and Handling Instructions below carefully before using.
Product is corrosive to tin, aluminum, zinc and alloys containing these metals, and will react violently with these metals in powder form.
Hazardous carbon monoxide gas can form upon contact with food and beverage products in enclosed spaces and can cause death. Follow appropriate tank entry procedures (ANSI Z117.1-1977).

IX. SPECIAL PRECAUTIONS ...continued

FIRST AID:

IN CASE OF CONTACT:

For eyes:

Immediately flush with plenty of water for at least 15 minutes, holding eyelids apart to ensure flushing of the entire eye surface. Washing eyes within several seconds is essential to achieve maximum effectiveness. Seek medical attention immediately.

For skin:

Immediately wash with plenty of water for 15 minutes. Remove contaminated clothing and footwear. Wash clothing before reuse and discard footwear which cannot be decontaminated. Seek medical attention immediately.

IF INHALED:

Remove person out of contaminated area to fresh air. If breathing has stopped, artificial respiration should be started. Oxygen may be administered, if available. Seek medical attention immediately.

IF SWALLOWED:

Do not induce vomiting. Give large quantities of water. If available, give several glasses of milk. Never give anything by mouth to an unconscious person. Seek medical attention immediately.

IN CASE OF SPILL OR LEAK:

Leaks should be stopped. Spills, after containment, should be shoveled up or removed by vacuum truck (if liquid) to chemical waste area. Neutralize residue with dilute acid, flush spill area with water followed by liberal covering of sodium bicarbonate. Dispose of wash water and spill by-products according to federal, state, and local regulations.

SPECIAL MIXING AND HANDLING INSTRUCTIONS:

Considerable heat is generated when product is mixed with water. Therefore, when making solutions always carefully follow these steps:

ALWAYS wear ALL protective clothing described above. Never add water to product. ALWAYS add product - with constant stirring - slowly to surface of lukewarm (80-100°F) water, to assure product is being completely dissolved as it is added.

If product is added too rapidly, or without stirring, and becomes concentrated at bottom of mixing vessel, excessive heat may be generated, resulting in DANGEROUS boiling and spattering, and a possible IMMEDIATE AND VIOLENT ERUPTION of highly caustic solution.

Note: 50 pounds of product dissolved in 30 gallons of 90°F water will raise temperature of resulting solution to approximately 180°F. Never add more product than can be absorbed by solution while maintaining temperature below 200°F (@ sea level) to prevent boiling and spattering.

Product can react EXPLOSIVELY with acids, aldehydes, and many other organic chemicals - when mixing product with solutions containing such chemicals, follow all of above mixing instructions, and add product very gradually, while stirring constantly.

ALWAYS empty and clean containers of all residues before adding product, to avoid possible EXPLOSIVE reaction between product and unknown residue.

Returnable containers should be shipped in accordance with supplier's recommendations. Return shipments should comply with all federal, state, and DOT regulations. All residual caustic soda should be removed from containers prior to disposal.

More information on the hazards and handling of caustic soda appear in Diamond Shamrock Corporation's Material Safety Data Sheet and Caustic Soda Handbook EC-LDC-1d.

DISPOSAL:

The materials resulting from clean-up operations may be hazardous wastes and, therefore, subject to specific regulations. Package, store, transport, and dispose of all clean-up materials and any contaminated equipment in accordance with all applicable federal, state, and local health environmental regulations. Shipments of waste materials may be subject to manifesting requirements per applicable regulations. Appropriate disposal will depend on the nature of each waste material and should be performed by competent and properly permitted contractors. Ensure that all responsible federal, state, and local agencies receive proper notification of disposal.

MATERIAL SAFETY DATA SHEET

3V INC.- P.O. DRAWER Y GEORGETOWN, S.C. 29442 - (803) 546-8556

PRODUCT NAME: OXIDAN TCA/P Effective date: 12/01/92

SECTION 1. IDENTIFICATION

Chemical characterization:

Form : Solid granular or powder material
Color : White
Odor : Pungent as of chlorine

SECTION 2. INGREDIENTS/HAZARD STATEMENTS

Chemical identity	CAS NO.	PEL	TLV	PERCENT
Trichloro-s-Triazinetrione	87-90-1	NA	NA*	Ca. 100%

*The TLV for chlorine is 0.5 ppm (1.5 mg/m_3) TWA and 1ppm STEL.

WARNING STATEMENTS: DANGER! Highly corrosive. Causes irreversible eye damage and burns to skin. May be fatal if swallowed. Will burn with the evolution of chlorine and equally toxic gases. Do not get in eyes, on skin or clothing. Avoid breathing dust and fumes.

SECTION 3. PHYSICAL DATA

Melting point (°C)	decomposes above 225°C	Boiling point (°C)	NA
Density (g/ml)	NA	Bulk density (g/ml)	1.00 - 1.05
Vapor pressure (mm Hg at 20 °C)	NA	Viscosity (cp at 20 °C)	NA
pH (1% in H_2O)	2.5 - 3.5	Solubility in water	ca. 12 g/l

SECTION 4. FIRE AND EXPLOSION HAZARDS

Flash point (method): NA °C ( ) LEL: % UEL: %

Extinguishing media: ( ) CO_2 ( ) Dry Chemical ( ) Foam
(x) Water spray

Special fire fighting procedures: Use large amounts of water. Small quantities of water with large quantities of hot product may lead to fire/explosion. Do not use CO_2, dry chemical, foam or halon.

Eye exposure:	Flush with water for at least 15 minutes. Get medical attention.
Inhalation:	Remove to fresh air. Seek medical attention if irritation develops.

SECTION 7. PRECAUTIONS FOR SAFE HANDLING AND USE

Steps to take in case of spill or release: Contain material and do not allow material to sit on ground or floor and become moist. Do not contaminate with water, organics or nitrogen containing compounds.

Waste disposal methods: If material cannot be disposed of by use contact state or local environmental control agent or the nearest EPA offices for guidance.

Handling and storage: Keep containers tightly closed in a dry well ventilated location. Protect from heat. Keep away from combustible and incompatible materials. Avoid dust formation. Recommended storage temperature < 25°C.

Other precautions: Store in accordance with local building codes and or NFPA codes. Refer to "Guidelines for Safe Handling and Storage of Calcium Hypochlorite and Chlorinated Isocyanurate Pool Chemicals" for further detail.

SECTION 8. CONTROL MEASURES

Respiratory protection: Protective apparatus for chlorine and dust if exposure limits exceeded.

Dermal protection: Rubber gloves. Full length work clothing.

Eye protection: Dust proof goggles.

Ventilation: Recommended to control airborne levels below limits.

Protective equipment: Eye wash & safety shower within 25 ft. of potential exposure area.

SECTION 9. ENVIRONMENTAL AND REGULATORY INFORMATION

Ecological effects: This product is toxic to fish and aquatic life. Do not empty into drains or water courses.

CERCLA RQ (lbs): Not listed per se. For chlorine, RQ is 10 lb.
TSCA status: On Inventory.
SARA 313 list: Not listed.
SARA 304 list: Not listed.
DOT status: UN2468. Label required: OXIDIZER.

SECTION 4. FIRE AND EXPLOSION HAZARDS (cont.)

Protective measures to avoid fire/explosion: Flood with copious amounts of water. Use great care in approaching wet containers of hot product as these conditions lead to NCl_3 formation, an explosive substance.

NFPA Hazard Rating Index: (3) Health (0) Flammability
(2) Reactivity (Ox) Special

SECTION 5. REACTIVITY DATA

Stability: (x) Stable ( ) Unstable

Conditions to avoid: Decomposes if heated above 225°C with liberation of toxic gases.

Incompatibility: ( ) water (x) strong acids ( ) strong bases
( ) oxidizers (x) reducers (x) combustibles
(x) other: Product is a strong oxidizer. Avoid contact with any oxidizable organic and inorganic material. Contact with organic matter may cause fire; decomposition with chlorine by contact with acids. Especially, avoid contact with nitrogen containing compounds like ammonia, urea, amines and similar. Mixing with small quantities of water may cause violent reaction.

Hazardous decomposition products: Chlorine and traces of phosgene.

SECTION 6. HEALTH HAZARD INFORMATION

Acute effects: Corrosive to eyes and irritating to skin of rabbits. Acute dermal (rabbit) LD_{50} estimated to be > 5000 mg/kg. Acute oral (rat) LD_{50} about 800 mg/kg. Irritating to nose and throat.

Chronic effects: Not established.

First Aid Procedures:

Swallowing:	Promptly drink a large quantity of milk, egg whites, gelatin solution, or if these are not available, drink large quantities of water. Do not induce vomiting. Contact a physician immediately.
Dermal exposure:	Immediately brush off excess chemical and flush skin with cold water for at least 15 minutes. If irritation develops, get medical attention.

"FOR CHEMICAL EMERGENCY"
Spill, Leak, Fire, Exposure or Accident
Call CHEMTREC - Day or Night
1-800-424-9300

The statements made herein are intended to provide product safety information. This material safety data sheet has been prepared with information currently available. Although care has been taken to provide accurate information, no warranties either expressed or implied are being made as a result of the presentation of this information.

UNION CARBIDE

UNION CARBIDE CHEMICALS AND PLASTICS COMPANY INC.
Industrial Chemicals Division

MATERIAL SAFETY DATA SHEET

NH-01

EFFECTIVE DATE: 01/24/91

Union Carbide urges each customer or recipient of this MSDS to study it carefully to become aware of and understand the hazards associated with the product. The reader should consider consulting reference works or individuals who are experts in ventilation, toxicology, and fire prevention, as necessary or appropriate to use and understand the data contained in this MSDS.

To promote safe handling, each customer or recipient should: (1) notify its employees, agents, contractors and others whom it knows or believes will use this material of the information in this MSDS and any other information regarding hazards or safety; (2) furnish this same information to each of its customers for the product; and (3) request its customers to notify their employees, customers, and other users of the product of this information.

I. IDENTIFICATION

PRODUCT NAME: ETHYLENEDIAMINE

CHEMICAL NAME: 1,2-Ethanediamine

CHEMICAL FAMILY: Ethyleneamines

FORMULA: $C_2H_8N_2$ MOLECULAR WEIGHT: 60.10

SYNONYMS: EDA; EDA-HP; EDA-UHP

CAS # and CAS NAME: 107-15-3 1,2-Ethanediamine

II. PHYSICAL DATA (Determined on typical material)

BOILING POINT, 760 mm Hg: 117.3 C (243.1 F)

FREEZING POINT: 11.3 C (52.3 F)

SPECIFIC GRAVITY(H_2O = 1): 0.898 at 20/20 C

VAPOR PRESSURE AT 20°C: 10.4 mm Hg

VAPOR DENSITY (air = 1): 2.07

SOLUBILITY IN WATER by wt: 100%

EVAPORATION RATE (Butyl Acetate = 1): 0.91

APPEARANCE AND ODOR: Water-white liquid; strong amine odor.

EMERGENCY PHONE NUMBER: 1-800-UCC-HELP (Number available at all times) or 304-744-3487

UNION CARBIDE CHEMICALS AND PLASTICS COMPANY INC.
Industrial Chemicals Division
39 Old Ridgebury Road, Danbury, CT. 06817-0001

PRODUCT NAME: ETHYLENEDIAMINE

III. INGREDIENTS

MATERIAL	%	TLV (Units)	HAZARD
Ethylenediamine (CAS# 107-15-3)	99.0-99.9	10ppm TWA, ACGIH and OSHA 10ppm PEL, OSHA	See Section V

IV. FIRE AND EXPLOSION HAZARD DATA

FLASH POINT (test method(s)): 105 F, Tag closed cup ASTM D 56
108 F, Tag open cup ASTM D 1310

FLAMMABLE LIMITS IN AIR. % by volume: LOWER: 4.2 UPPER: 14.4

EXTINGUISHING MEDIA: Apply alcohol-type or all-purpose-type foams by manufacturers' recommended techniques for large fires. Use C02 or dry chemical media for small fires.

SPECIAL FIRE FIGHTING PROCEDURES: Do not direct a solid stream of water or foam into hot, burning pools; this may cause splattering and increase fire intensity. Use protective clothing, eye protection and self-contained breathing apparatus.

UNUSUAL FIRE AND EXPLOSION HAZARDS: Oxides of nitrogen will be evolved.

V. HEALTH HAZARD DATA

EXPOSURE LIMIT(S): See Section III.

EFFECTS OF SINGLE OVEREXPOSURE:

SWALLOWING: Moderately toxic. May cause burns of mouth and throat, abdominal pain, nausea vomiting, diarrhea, dizziness, weakness, thirst, collapse and possible coma. The nature and severity of these signs and symptoms will be dependent on the amount swallowed. Aspiration into the lungs may occur during ingestion or vomiting, resulting in lung injury.

SKIN ABSORPTION: Prolonged or widespread exposure may result in the absorption of potentially harmful amounts of material.

INHALATION: Vapors are irritating and may cause excessive tear formation, burning sensation of the nose and throat, coughing, wheezing, shortness of breath, nausea and vomiting. Extremely high vapor concentrations may cause lung damage. Some individuals may develop asthma. (See "Other Effects of Overexposure".)

SKIN CONTACT: Causes severe local redness, swelling and chemical burns.

EYE CONTACT: Causes severe conjunctival irritation, corneal injury and iritis. Corneal injury may be marked, extensive, and if not promptly treated, may possibly lead to permanent impairment of vision. Vapor may cause temporary disturbances of vision. (See "Notes to Physician.")

EFFECTS OF REPEATED OVEREXPOSURE: Repeated exposure to high vapor concentration may cause injury to liver, kidney, and respiratory tract.

MEDICAL CONDITIONS AGGRAVATED BY OVEREXPOSURE: Because of its irritating properties, this material may aggravate an

PRODUCT NAME: ETHYLENEDIAMINE

existing dermatitis. Breathing of vapor and/or mists may aggravate asthma and inflammatory or fibrotic pulmonary disease.

SIGNIFICANT LABORATORY DATA WITH POSSIBLE RELEVANCE TO HUMAN HEALTH HAZARD EVALUATION: None currently known.

OTHER EFFECTS OF OVEREXPOSURE:

- Inhalation of ethyleneamines may cause sensitization of the respiratory tract, and the development of an asthmatic reaction on further exposure.
- There may be some susceptible* individuals who develop long-term hyperreactive airways, asthma and other respiratory injury following exposure to extremely low concentrations of ethyleneamines, even below the irritation threshold. Other respiratory irritants may produce a reaction in individuals whose airways have become hyperreactive.
  *Since there are no definitive screening methods available to identify susceptible individuals, we suggest that people with asthma, or other long-standing respiratory conditions (for example, chronic bronchitis, emphysema, etc.) should be protected from any potential exposure to ethyleneamines.
- Skin contact may cause sensitization and an allergic skin reaction.
- Cross-sensitization may also occur by skin contact with this material and other amines.

EMERGENCY AND FIRST AID PROCEDURES:

SWALLOWING:	If patient is fully conscious, give two glasses of water or milk at once. Do not induce vomiting. Obtain medical attention without delay.
SKIN:	Immediately flush skin with plenty of water while removing contaminated clothing and shoes. Obtain medical attention. Wash clothing before wearing again. Discard shoes.
INHALATION:	Remove to fresh air. Give artificial respiration if not breathing. Oxygen may be given by qualified personnel if breathing is difficult. Obtain medical attention.
EYES:	Immediately flush eyes thoroughly with water and continue washing for at least 15 minutes. Obtain medical attention, preferably from an ophthalmologist, urgently.
NOTES TO PHYSICIAN:	There is no specific antidote. Treatment should be directed at the control of symptoms and the clinical condition of the patient. Due to the corrosive nature of the material, swallowing may lead to severe ulceration and inflammation of the upper alimentary tract with hemorrhage and fluid loss. Also, perforation of the esophagus or stomach may occur, leading to mediastinitis or peritonitis and the resultant complications. Due to the severely corrosive nature of the material, any aspiration during vomiting could result in severe lung injury. Therefore, emesis should not be induced mechanically or pharmacologically. However, the acute peroral systemic toxicity of the material indicates that evacuation of the stomach contents should be undertaken at the earliest possible time by means carrying the least likelihood for aspiration (e.g. the use of gastric lavage with endotracheal intubation). Exposure to the vapor may cause minor edema of the corneal epithelium. This condition, referred to as "glaucopsia," "blue haze," or "blue-gray haze," produces a blurring of vision against a general bluish haze and the appearance of halos around bright objects. The effect disappears spontaneously within a few hours of the end of an exposure, and leaves no sequelae. Although not detrimental to the eye per se, glaucopsia predisposes an affected individual to physical accidents, and reduces the ability to undertake skilled tasks such as driving a motorized vehicle.

VI. REACTIVITY DATA

STABILITY: Stable

CONDITIONS TO AVOID: Some decomposition can occur upon vigorous heating: e.g., application of

high-pressure steam or flame.

INCOMPATIBILITY (materials to avoid):
Avoid contamination with acids, epoxides, oxidizing agents, aldehydes, ketones, acrylates and organic halides.

HAZARDOUS COMBUSTION OR DECOMPOSITION PRODUCTS:
Burning can produce carbon monoxide, carbon dioxide and oxides of nitrogen. Vigorous heating above ambient temperatures can produce other volatile amines and ammonia.

HAZARDOUS POLYMERIZATION: Will Not Occur

CONDITIONS TO AVOID: None

VII. SPILL OR LEAK PROCEDURES

STEPS TO BE TAKEN IF MATERIAL IS RELEASED OR SPILLED:
Wear suitable protective equipment; avoid contact with liquid and vapors! Small spills could be flushed with large amounts of water. Larger spills should be collected for disposal. Avoid discharge to sewers or waterways. At very low concentration in water (about 10ppm), ethylenediamine is biodegradable in a biological wastewater treatment system. However, at about 500 ppm concentration or higher, it can be toxic to the biomass in a treatment system. Also, it is toxic to fish. Therefore, avoid discharge to sewers or to natural waters.

WASTE DISPOSAL METHOD: Incinerate in a furnace where permitted under appropriate Federal, State, and local regulations.

VIII. SPECIAL PROTECTION INFORMATION

RESPIRATORY PROTECTION (specify type):
Positive pressure supplied air respirator equipped with a full facepiece should be used during any operation where there is potential for release of this product to workplace air.

VENTILATION: This product should be confined within closed equipment, in which case general (mechanical) room ventilation should be satisfactory. Special local ventilation is needed at points where vapors can be expected to escape to workplace air.

PROTECTIVE GLOVES: Butyl or Neoprene

EYE PROTECTION: Monogoggles

OTHER PROTECTIVE EQUIPMENT:
Eye bath, safety shower and chemical apron.
When breaking open any equipment (or in the event of a large spill or release), full protective clothing is needed (i.e. rainsuit, boots, cuffed gloves, etc.).

IX. SPECIAL PRECAUTIONS

PRECAUTIONS TO BE TAKEN IN HANDLING AND STORAGE:
DANGER! Causes eye and skin burns.
Harmful and corrosive if swallowed.
Harmful if inhaled or absorbed through skin.

PRODUCT NAME: ETHYLENEDIAMINE

May cause asthma with possible long-term lung damage.
May cause allergic skin reaction.
Cross-sensitation to other amines may occur.
Combustible.
Aspiration may cause lung damage.
May cause liver, kidney and respiratory system damage.
Do not get in eyes, on skin on clothing.
Do not swallow.
Avoid breathing vapor.
Keep away from heat and flame.
Keep container closed.
Use with adequate ventilation.
Wash thoroughly after handling.
FOR INDUSTRY USE ONLY

OTHER PRECAUTIONS: WARNING: Sudden release of hot organic chemical vapors or mists from process equipment operating at elevated temperature and pressure, or sudden ingress of air into vacuum equipment, may result in ignitions without the presence of obvious ignition sources. Published "autoignition" or "ignition" temperature values cannot be treated as safe operating temperatures in chemical processes without analysis of the actual process conditions.

Any use of this product in elevated-temperature processes should be thoroughly evaluated to establish and maintain safe operating conditions. Further information is available in a technical bulletin entitled "Ignition Hazards of Organic Chemical Vapors."

X. REGULATORY INFORMATION

STATUS ON SUBSTANCE LISTS:

The concentrations shown are maximum or ceiling levels (weight %) to be used for calculations for regulations. Trade Secrets are indicated by "TS".

FEDERAL EPA

Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (CERCLA) requires notification of the National Response Center of release of quantities of Hazardous Substances equal to or greater than the reportable quantities (RQs) in 40 CFR 302.4.

Components present in this product at a level which could require reporting under the statute are:

CHEMICAL	CAS NUMBER	UPPER BOUND CONCENTRATION %
Ethylenediamine	107-15-3	99.9

Superfund Amendments and Reauthorization Act of 1986 (SARA) Title III

requires emergency planning based on Threshold Planning Quantities (TPQs) and release reporting based on Reportable Quantities (RQs) in 40 CFR 355 (used for SARA 302, 304, 311 and 312).

Components present in this product at a level which could require reporting under the statute are:

CHEMICAL	CAS NUMBER	UPPER BOUND CONCENTRATION %
Ethylenediamine	107-15-3	99.9

Superfund Amendments and Reauthorization Act of 1986 (SARA) Title III

requires submission of annual reports of release of toxic chemicals that appear in 40 CFR 372 (for SARA 313). This information must be included in all MSDSs that are copied and distributed for this material.

Components present in this product at a level which could require reporting under the statute are:

*** None ***

STATE RIGHT-TO-KNOW

CALIFORNIA Proposition 65 This product does not contain materials which the State of California has

found to cause cancer, birth defects or other reproductive harm.

MASSACHUSETTS Right-To-Know, Substance List (MSL) Hazardous Substances and Extraordinarily Hazardous Substances on the MSL must be identified when present in products.
Components present in this product at a level which could require reporting under the statute are:
EXTRAORDINARILY HAZARDOUS SUBSTANCES (=> 0.0001%)

CHEMICAL	CAS NUMBER	UPPER BOUND CONCENTRATION %
Ethylenediamine	107-15-3	99.9

PENNSYLVANIA Right-To-Know, Hazardous Substance List Hazardous Substances and Special Hazardous Substances on the List must be identified when present in products.
Components present in this product at a level which could require reporting under the statute are:
*** NONE ***

Toxic Substances Control Act(TSCA) STATUS:
The ingredients of this product are on the TSCA inventory.

CALIFORNIA SCAQMD RULE 443.1 VOC'S:
Not presently available

OTHER REGULATORY INFORMATION:
*** None Known ***

NOTE ----
The opinions expressed are those of qualified experts within Union Carbide.. We believe that the information contained is current as of the date of the Material Safety Data Sheet. Since the use of this information and of these opinions and the conditions of the use of the product are not within the control of Union Carbide, it is the user's obligation to determine the conditions of safe use of the product.

REVISED SECTIONS: Revisions in this MSDS occurred in the following sections:
Section V: HEALTH HAZARD DATA
Section IX: PRECAUTIONS TO BE TAKEN IN HANDLING AND STORAGE

PC: 34810
F NUMBER: N0138C

Fritzsche Dodge & Olcott™
76 Ninth Avenue, New York, N. Y. 10011

A Unit of BASF K&F Corporation

BASF Group

MATERIAL SAFETY DATA SHEET

PIN#: *67160

In case of emergency call:
Fritzsche Dodge & Olcott 212-929-4100
Chemtrec 800-424-9300

HEALTH	2
FLAMMABILITY	2
REACTIVITY	0

HAZARD RATING
LEAST 0 SLIGHT 1 MODERATE 2 HIGH 3 EXTREME 4

REVISION DATE 7/09/8

SECTION I - IDENTIFICATION AND GENERAL INFORMATION

PRODUCT NUMBER: *67160
PRODUCT NAME: PHENYLETHYL ALCOHOL FCC EXTRA

CHEMICAL NAME: BENZENEETHANOL
CHEMICAL FAMILY: AROMATIC ALCOHOL
SYNONYMS: BENZYL CARBINOL

MOLECULAR WEIGHT: 122.16
MOLECULAR FORMULA: C8H10O

CAS NUMBER: 000060-12-8
TSCA REGISTERED: YES
FDA APPROVED: YES
RIFM APPROVED: YES
RIFM NUMBER: 0145
FEMA APPROVED: YES
FEMA NUMBER: 2858

SECTION II - HAZARDOUS INGREDIENT(S)

CONTAINS: PHENYLETHYL ALCOHOL FCC EXTRA

SECTION III - PHYSICAL DATA

BOILING POINT (DEG C):	217.00 - 218.00 AT 760 MM HG	FLASHPOINT:	195
MELTING POINT (DEG C):	26	VAPOR DENSITY:	N/F
SPECIFIC GRAVITY:	1.0170 - 1.0200 AT 25 C	% VOLATILE:	N/F
REFRACTIVE INDEX:	N/F		
ALCOHOL CONTENT:	N/F		
WATER CONTENT:	N/F		
VAPOR PRESSURE:	1.000 MM HG AT 58		
SOLUBILITY IN WATER:	SLIGHTLY SOLUBLE		

APPEARANCE: COLORLESS LIQUID WITH MILD, WARM, ROSE, HONEY-LIKE ODOR. CHARACTERISTIC ODOR AND FLAVOR.

SECTION IV - FIRE AND EXPLOSION HAZARD DATA

Extinguishing Media: Carbon Dioxide, Dry Chemicals or Foam.
Special Fire Fighting Procedures: Use of self contained breathing apparatus and protective clothing is recommended.
Unusual Fire and Explosion Hazards: Sealed containers may build up pressure at high temperatures. Keep containers cool whenever possible.

SECTION V - HEALTH HAZARD DATA

THRESHOLD LIMIT VALUE (ACGIH TLV): N/F
THRESHOLD LIMIT VALUE (OSHA PEL): N/F

CARCINOGENICITY: NTP MONOGRAPH: N/F IARC MONOGRAPH: N/F

ACUTE TOXICITY DATA:

ORAL LD50: 1,790.00 MG/KG RAT

INHALATION LC50: N/F

DERMAL LD50: 790.00 MG/KG RABBIT

HEALTH HAZARDS (ACUTE AND CHRONIC):

INGESTION: SLIGHT HEALTH HAZARD

INHALATION: N/F

SKIN CONTACT: TOXIC

I[illegible]TATION (SKIN): CAUSES IRRITATION

IRRITATION (EYE): CAUSES EYE IRRITATION

OTHER EFFECTS:

INGESTION:

N/F

INHALATION:

N/F

SKIN CONTACT:
REPEATED DERMAL APPLICATION OF
LARGE AMOUNTS TO PREGNANT RATS
CAUSED REPRODUCTIVE EFFECTS.

The health hazard information is based on laboratory animal test data for the individual components of this product in their pure form as provided under 29 CFR 1910.1200 (d)(5)(ii). The user should consider the relevancy of

SECTION VI - EMERGENCY FIRST AID PROCEDURES

ye contact- Flush immediately with cold clean water for at least 15 inutes. Contact a physician immediately.
in contact- The affected area should be thoroughly washed with soap and ter. Flush with large quantities of water. If irritation persists call physician.
nhalation: remove to fresh air and contact a physician if necessary.
gestion: Administer water or milk to dilute. Contact a physician or cal poison control center immediately.

SECTION VII - REACTIVITY DATA

is material presents no signifigant reactivity hazard.
zardous Polymerization will not occur and it will not react violently th water.
ntact with highly reactive chemical oxidants should be avoided.
burned this product will produce carbon monoxide and/or carbon dioxide.

SECTION VIII - PERSONAL PROTECTION

: The use of goggles or a face shield is recommended.
iratory: Respiratory protection is normally not required in well ntilated areas however, NIOSH approved respiratory protection may be quired when the material is rated toxic by inhalation or if the material to be used in a confined area.
her Protective Equipment: Chemical resistant gloves are recommended.

ntilation: Ventilation meeting ACGIH standards should be employed.
rk/Hygienic Practices: Use good, personal hygiene practices: Limit posure to product whenever possible. Wash after any contact. Thoroughly

sh any contaminated clothing or shoes before reuse.

SECTION IX - SPILL, LEAK AND WASTE DISPOSAL PROCEDURES

move any sources of flame or sparks. If in a confined area NIOSH proved respiratory protection may be required.
sorb spills on vermiculite or other suitable absorbant material and move to an approved disposal container.
spose of in accordance with current laws and regulations.

SECTION X - STORAGE AND HANDLING

Store in tight full containers in a cool dry place, away from light.

THE INFORMATION CONTAINED HEREIN IS BASED ON DATA CONSIDERED ACCURATE AND RELIABLE, NEVERTHELESS, YOU SHOULD PERFORM YOUR OWN INVESTIGATION AND INDEPENDENT VERIFICATION. NO WARRANTY IS EXPRESSED OR IMPLIED REGARDING THE ACCURACY OR CORRECTNESS OF THESE DATA. SINCE THE USE OF THIS INFORMATION AND THE CONDITIONS OF USE OF PRODUCT ARE NOT WITHIN THE CONTROL OF FRITZSCHE, DODGE & OLCOTT, IT IS THE USER'S OBLIGATION TO DETERMINE THE CONDITION OF SAFE USE OF THE PRODUCT.

********** END OF MSDS **********

APPENDIX B

CORROSIVITY TESTING

AND

HEADSPACE GC-MS ANALYSIS

Quantum Chemical Corporation

Quantum

USI Division

Allen Research Center
11530 Northlake Drive
P.O. Box 429566
Cincinnati, Ohio 45249
513-530-6500

January 11, 1994

Ms. Nancy Conley
Nova PetroChemicals
Plastics Division
3100 Cote Vertu
St. Laurent, Quebec, CN H4R 2J8

Dear Nancy,

As agreed to through the PDI, Quantum has completed the corrosion testing of the samples from the five boxes of washed drum flake that were sent to me. The results are as follows:

Box #	Corrosion Rating
1	1
2	1
3	1
4	3
5	1

The corrosion rating is a 1 to 5 scale with 5 indicating the highest level of corrosivity and 1 the lowest. A rating of 3 is generally considered acceptable for typical plastics molding operations. The test follows the directions found in the Rhorback Cosasco Corrosion Test Kit.

Nancy, I still have to perform the oxygen induction time testing on the samples. As soon as this is done, I will pass the results on to you.

Very truly yours,

John E. Bergerhouse
Project Leader, Polyolefins Development
LJ12108L

CC: Mr. John Rathman
Phillips 66 Company
Plastics Tech. Center
Bartlesville, OK 74004

Novacor

MEMORANDUM

Date: May 17, 1994

To: Tony Tikuisis, NTC

From: Ken Sonnenberg

Re: **Headspace GC-MS Analysis of Recycled Drum Samples**

Two composite samples from the PDI recycled drum project, one each of the flaked and repelletized materials, have been analyzed by headspace GC-MS to identify volatile organic compounds. Since the composite flaked material consisted of pieces of varying sizes, a subsample was first cut into small pieces of approximately the same size in order to increase sample homogeneity and thus improve repeatability. (The composite pelletized material had a relatively uniform pellet size and was therefore not cut into smaller pieces.) Subsamples of both materials were placed into 22mL headspace-autosampler vials and heated for 20 minutes at 110°C before the headspace was sampled. A 1mL volume of each headspace was injected directly onto the gas-chromatographic column through a heated transfer line. Analytical conditions for the headspace autosampler are listed in Table 1; those for the GC-MS are listed in Table 2. (The GC-MS run was started at the beginning of the headspace-autosampler inject time.) Figures 1 and 2 show the total ion chromatograms of the headspace samples.

Figure 1 shows the chromatogram obtained from the composite flaked sample. The majority of the detected components consist of C6 to C16 hydrocarbons. As shown by the chromatogram of virgin HDPE analyzed under the same conditions (HB-W555-A, Figure 3), the higher hydrocarbons (C12 to C16, and some of the C10's) arise from the polymer itself. However, numerous other compounds not observed in the HDPE were present: chlorinated compounds (chloromethylbenzene, 2-chloro-2-methylpropane, carbon tetrachloride and tetrachloroethylene), organic acids (acetic and 2-butenoic), other oxygenated compounds (acetone, 2-methyl-2-propanol and dimethoxymethane), flavour and odour compounds (ethyl butanoate, limonene and possibly 1-methoxy-2-propyl acetate), and pyrazine, a nitrogen-containing compound.

Figure 2 shows the chromatogram of the headspace from the pelletized sample. The pattern of components is similar to that seen in the flaked sample, with C6 to C16 hydrocarbons comprising the majority of the observed components. Similar non-hydrocarbon components were seen; however, 2-chloro-2-methylpropane and carbon tetrachloride were not detected, while ethyl-3-methylbutanoate, chlorooctane and a C6 ketone were present in this sample but not in the flaked material. As well, dimethyl disulfide was tentatively identified. These differences between the samples were confirmed by duplicate analysis of fresh subsamples of both materials (chromatograms not shown).

As requested, headspace samples were also obtained from the materials after heating to 40°C and 80°C. Figures 4 and 5 compare the headspace samples obtained from subsamples of the flaked and pelletized materials (respectively) heated for 20 minutes at 40, 80 and 110°C. (All other conditions were the same.) As shown by the chromatograms, heating the materials at 40°C produced very few components, and the components observed at 80°C appear to represent a subset of those seen at 110°C. The chromatograms generated at the highest temperature were therefore selected for further analysis, with the results outlined above.

Figure 6 shows the chromatogram of the headspace sample obtained from a commercial apple juice (Sun-Rype®) after heating to 40°C for 20 minutes. As shown in the chromatogram, the observed components consist largely of esters and alcohols, at least one of which (ethyl butanoate) was also detected in both composite samples.

If you have any further questions, please do not hesitate to contact me.

K.S.
encl.
2344.doc

Table 1

Headspace Autosampler Conditions for the GC-MS Analysis of Recycled Drum Samples

Instrument:	Tekmar Autosampler/Carrousel 7000/7050
Platen Temperature:	110°C
Platen Equilibration Time:	0.10 minutes
Sample Equilibration Time:	20 minutes
Mixer:	Off
Vial Pressurization Setpoint:	4 psig
Vial Pressurization Time:	0.80 minutes
Pressure Equilibration Time:	0.10 minutes
Sample Loop Size:	1 mL
Loop Fill Time:	0.60 minutes
Loop Equilibration Time:	0.10 minutes
Injection Time:	2.50 minutes
Sampling Valve Temperature:	120°C
Transfer Line Temperature:	120°C
Transfer Line Backpressure:	70 kPa @ 50°C
Injections/Vial	1
Standby Vial Needle Flowrate:	13 mL/minute

Table 2

Chromatographic Conditions for the Headspace GC-MS Analysis of Recycled Drum Samples

Instrument:	HP GC-MSD 5890/5970
Column:	HP-5ms, 30 m length x 0.25 mm i.d., d_f 0.25 µm
Carrier Gas:	UHP Helium
Column Head Pressure:	70 kPa @ 50°C
Temperature Program:	1) 3 minutes @ –20°C 2) –20°C to 140°C @ 3°C/minute 3) 140°C to 300°C @ 20°C/minute
Detector Temperature:	280°C
MSD	
Scan Range:	20–200 a.m.u.
Threshold:	1000
A/D Samples:	2^3

Figure 1: Total Ion Chromatogram of Headspace Sample from 3.8 g of NTC 94-1135, Composite Flaked Material

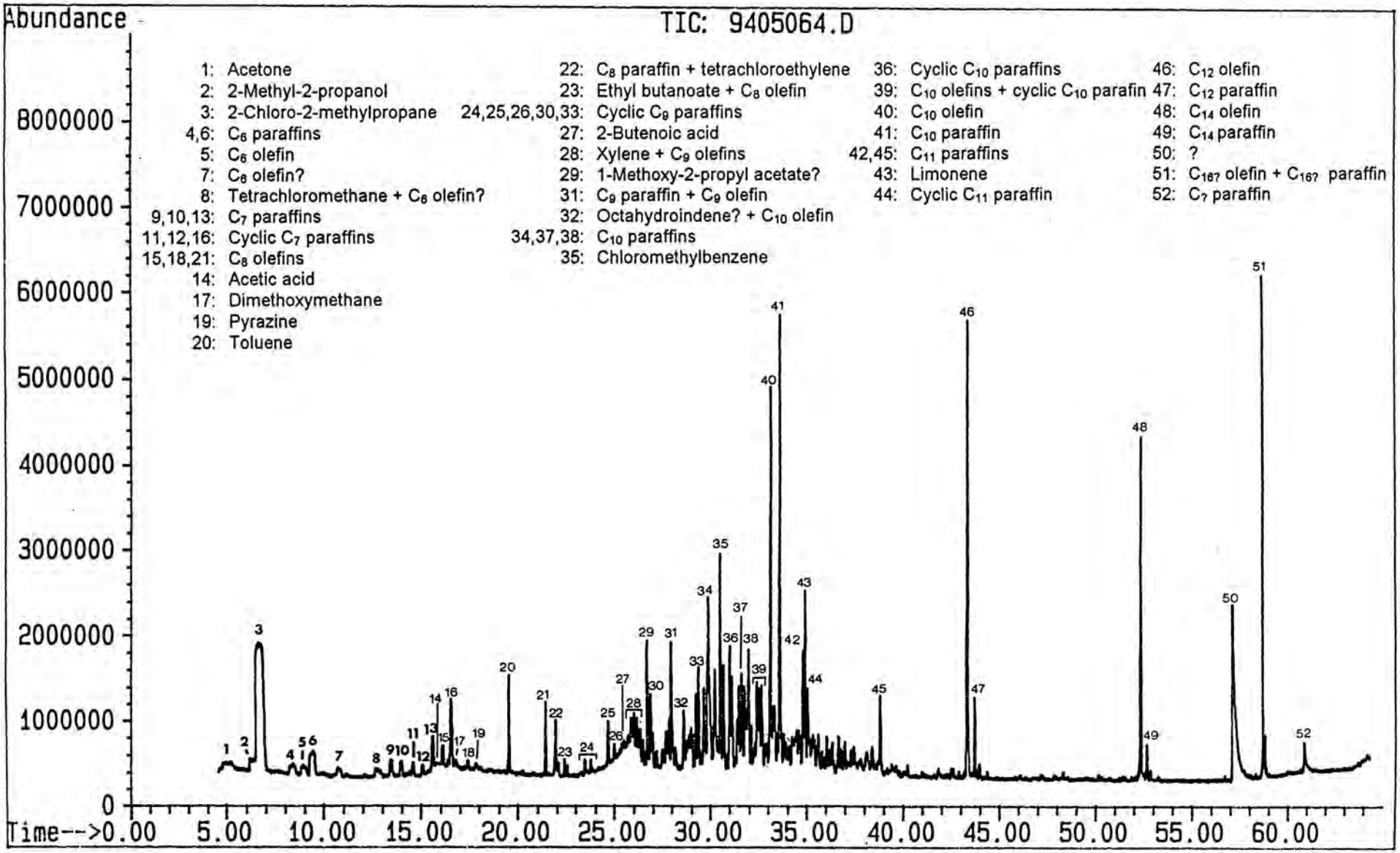


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Figure 2: Total Ion Chromatogram of Headspace Sample from 3.5 g of NTC 94-1136, Composite Pelletized Material

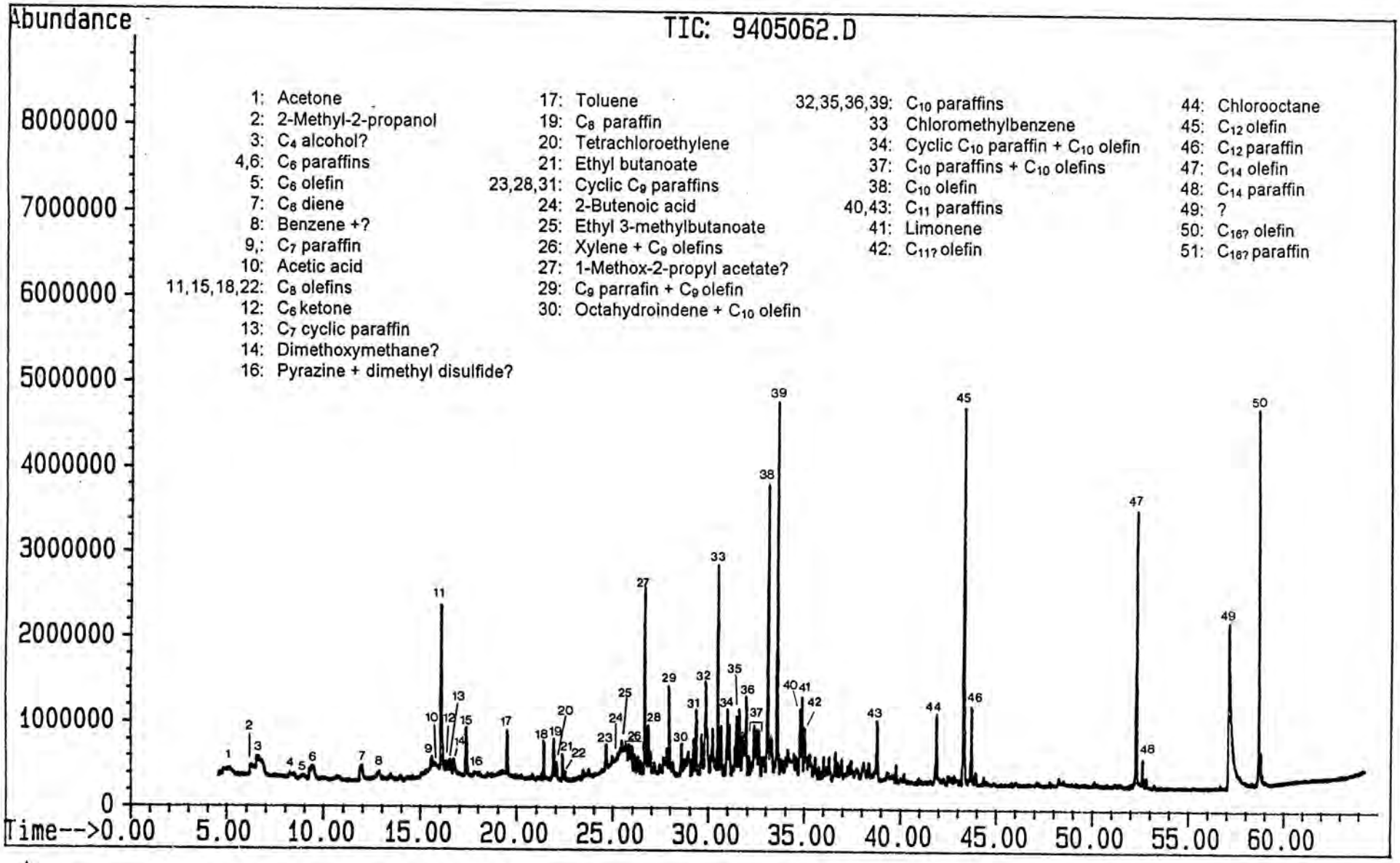

Figure 3: Total Ion Chromatogram of Headspace Sample from 3.5 g of HB-W555-A

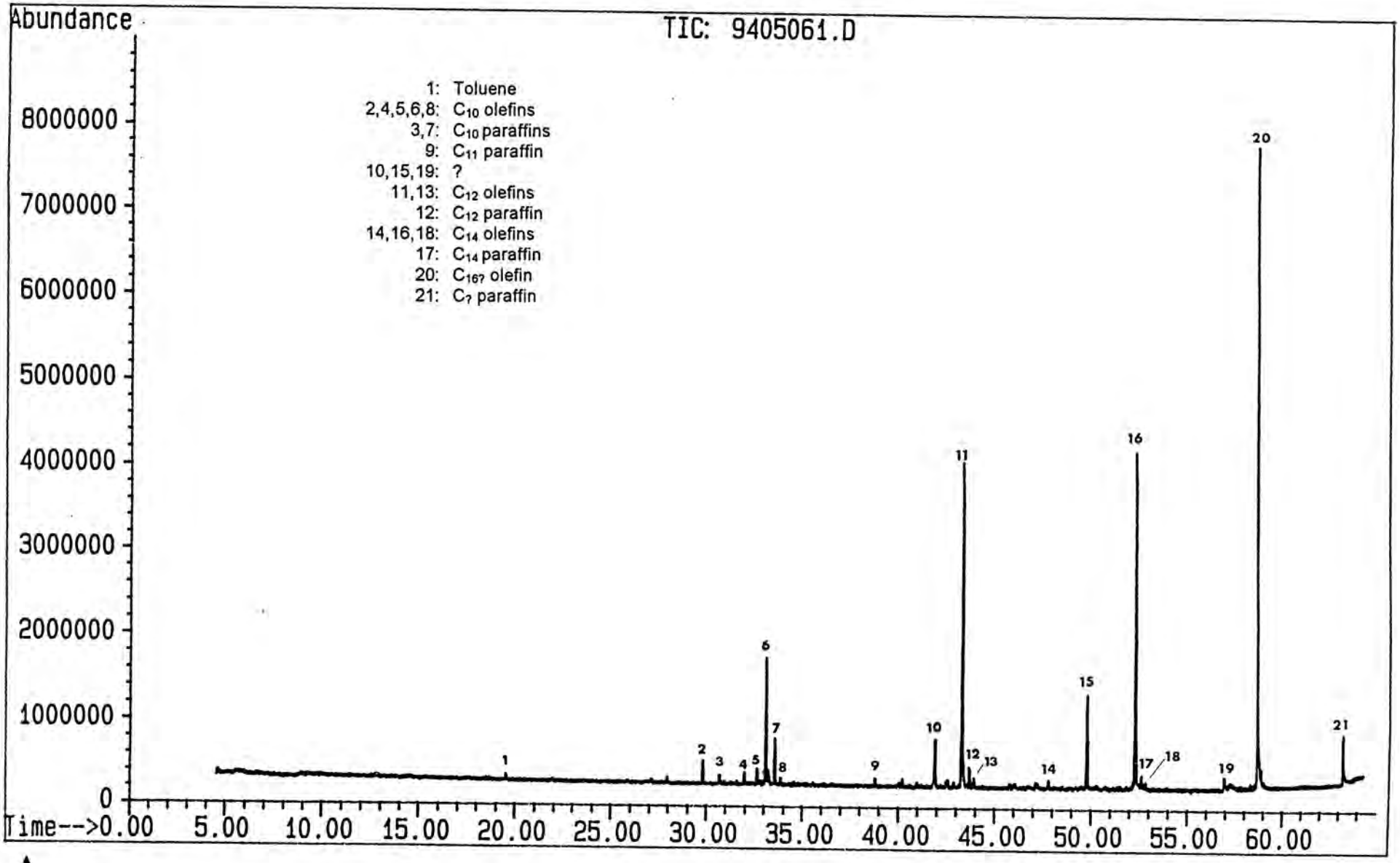

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Figure 4: Comparison of Headspace Samples obtained from NTC 94-1135 (Composite Flaked) at A) 110°C, B) 80°C, and C) 40°C

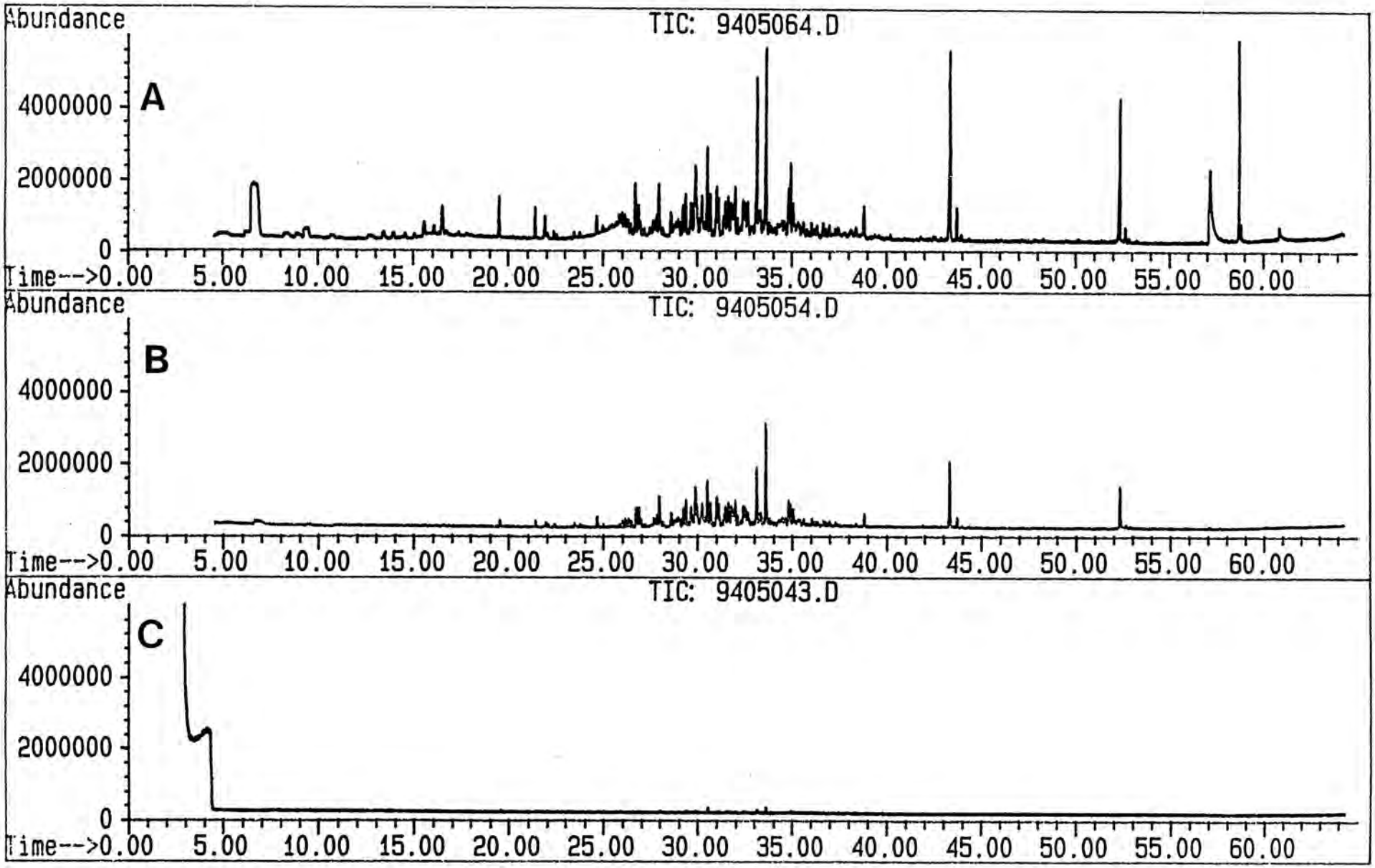
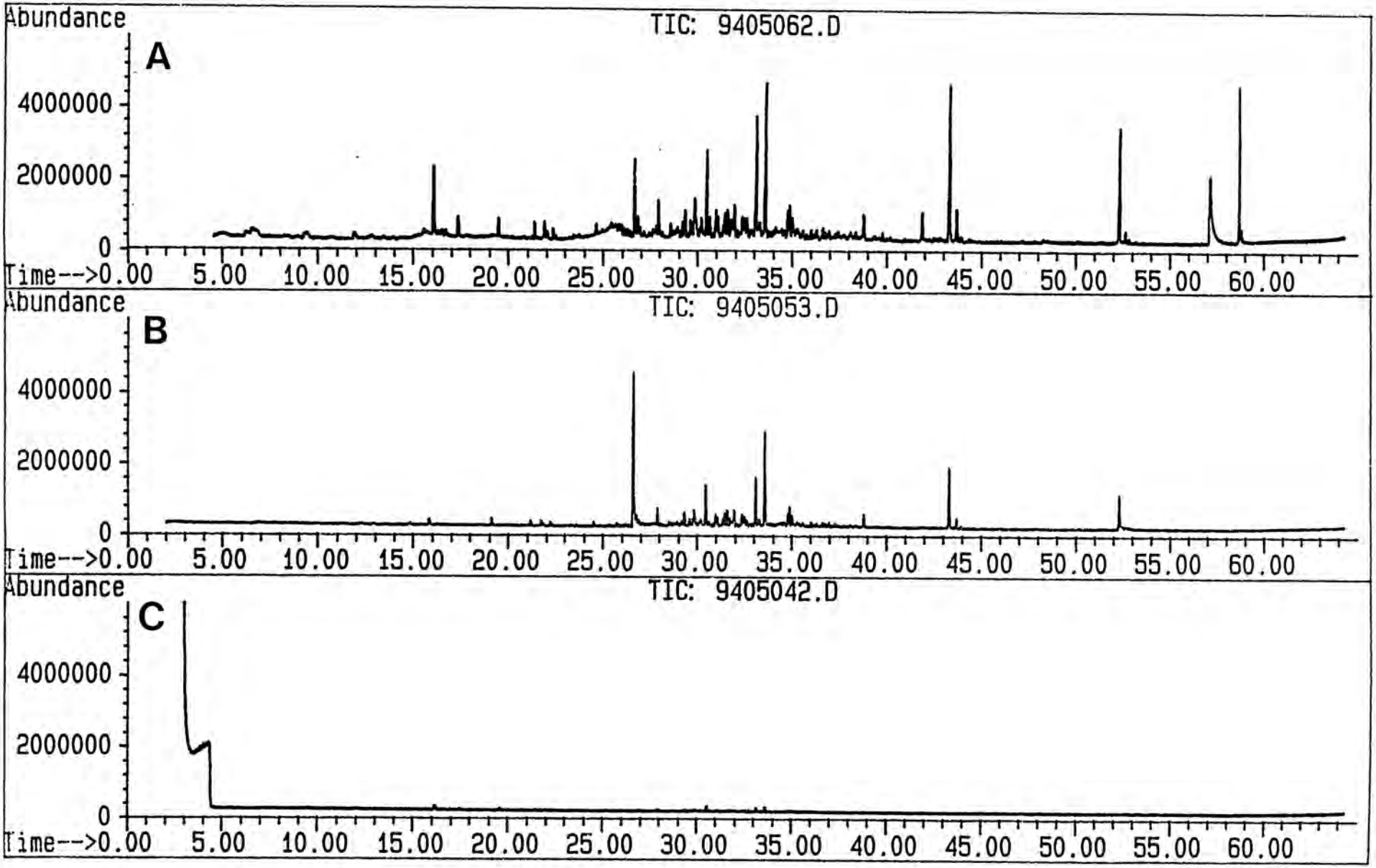


Figure 5: Comparison of Headspace Samples obtained from NTC 94-1136 (Composite Pelletized) at A) 110°C, B) 80°C, and C) 40°C



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APPENDIX C

TEST I

TEN-E REPORT

DATED SEPTEMBER 30, 1994

TENE
PACKAGING SERVICES INCORPORATED

PLASTIC DRUM INSTITUTE
210 LITER PCR DRUM EVALUATION

- 100% Virgin Resin
- 10% Recycle Resin & 90% Virgin Resin
- 25% Recycle Resin & 75% Virgin Resin
- 50% Recycle Resin & 50% Virgin Resin
- 100% Recycle Resin

REPORT #: 13145

September 30, 1994

TABLE OF CONTENTS

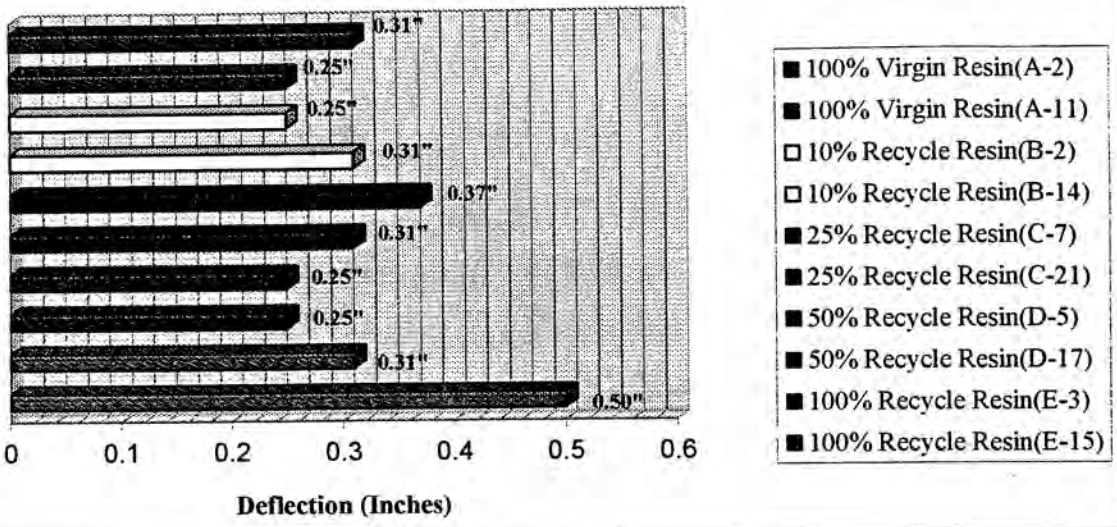
OBJECTIVE

To conduct an evaluation of 210 liter drums manufactured with varying amounts of post consumer and virgin resin. Each drum variable was evaluated by performing a UN Chapter 9 stack test (104°F), environmental stress crack resistance test(122°F), and a complete quality control audit.

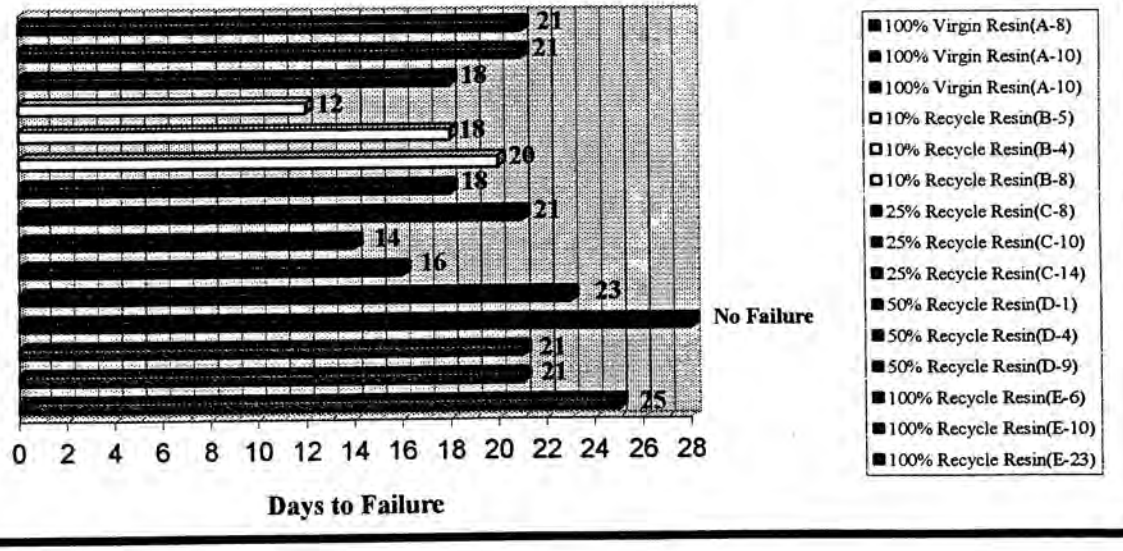
Variable	Description
A	100% Virgin Resin
B	10% Recycle Resin & 90% Virgin Resin
C	25% Recycle Resin & 75% Virgin Resin
D	50% Recycle Resin & 50% Virgin Resin
E	100% Recycle Resin

SUMMARY OF TEST RESULTS

TOTAL DEFLECTION AFTER 28DAY STACK TEST



ESCR TESTS (DAYS TO FAILURE)

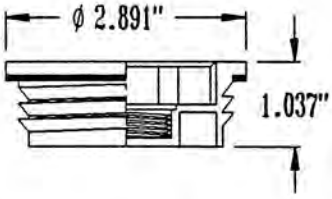


TEST PROCEDURES AND RESULTS

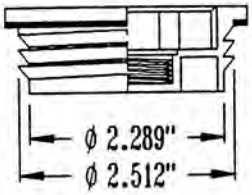
QUALITY CONTROL AUDIT RESULTS - Continued

CLOSURE	
• **Description:**	2" Threaded Buttress Plug (Rieke PPA-53-B-5)
• **Material:**	High Density Polyethylene
• **Pigment:**	Natural
• **Tare Weight:**	32.067 Grams
• **Markings:**	Rieke PPA-53-B-5

Overall Closure Dimensions



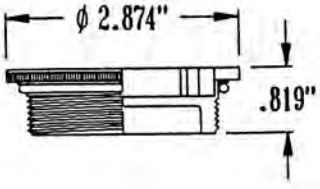
Finish Dimensions



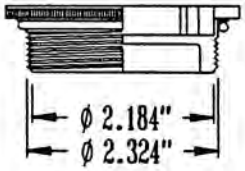
GASKET	
• **Description/Material:**	PVC Rubber Gasket (round)
• **Tare Weight:**	3.367 Grams
• **Thickness:**	0.181"

CLOSURE	
• **Description:**	2" Threaded NPT Plug (Rieke PPA-55-1)
• **Material:**	High Density Polyethylene
• **Pigment:**	Natural
• **Tare Weight:**	24.172 Grams
• **Markings:**	Rieke PPA-55-1

Overall Closure Dimensions



Finish Dimensions



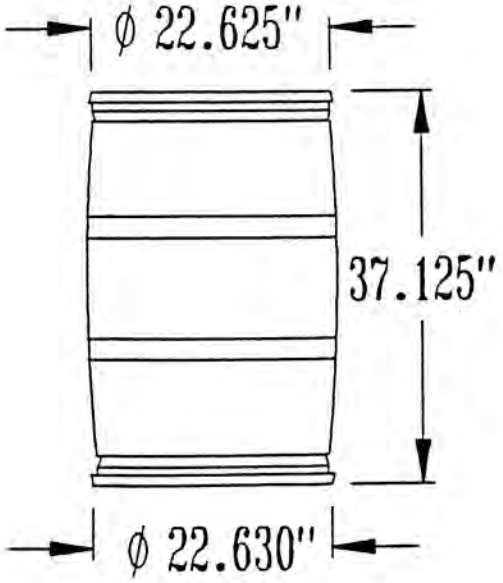
GASKET	
• **Description/Material:**	PVC Rubber Gasket (round)
• **Tare Weight:**	2.836 Grams
• **Thickness:**	0.156"

TEST PROCEDURES AND RESULTS

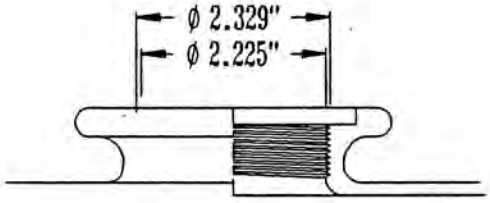
QUALITY CONTROL AUDIT RESULTS - Continued

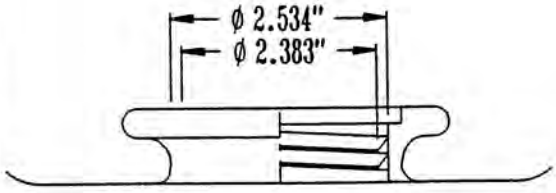
DRUM VARIABLE - A (100% VIRGIN RESIN)			
• **Description:**	210 Liter Tight Head Drum		
• **Material:**	High Density Polyethylene		
• **Pigment:**	Black		
• **Openings:** (Number & Size)	(1) 2" Buttress (1) 2" NPT		
• **Tare Weight:**	28.2 Lbs		
• **Overflow Capacity:**	59.4 Gallons	224.9 Kg (495.5 Lbs)	
• **98% Overflow Capacity:**	58.2 Gallons	220.4 Kg (485.6 Lbs)	
• **Melt Index (I^{21}):**	2.56 g/10 minutes		
• **Density:**	0.955 g/cc		
• **Markings:**	HUNTER 7-23-94	210 LITERS	"2" HDPE

Overall Drum Dimensions



Finish Dimensions





13145 - PD1 - A (100% VIRGIN RESIN)

WALL THICKNESS PROFILE (inches)

TOP HEAD

MSMT	DRUM 1	MSMT	DRUM 1
1	0.163	8	0.160
2	0.149	9	0.160
3	0.154	10	0.169
4	0.151	11	0.170
5	0.174	12	0.153
6	0.159	13	0.158
7	0.154	14	0.182

SIDEWALL

MSMT	DRUM 1	MSMT	DRUM 1
15	0.212	18	0.171
16	0.187	19	0.161
17	0.183	20	0.191

SIDEWALL

MSMT	DRUM 1	MSMT	DRUM 1
21	0.279	24	0.161
22	0.171	25	0.156
23	0.173	26	0.315

SIDEWALL

MSMT	DRUM 1	MSMT	DRUM 1
27	0.220	30	0.164
28	0.177	31	0.151
29	0.189	32	0.194

SIDEWALL

MSMT	DRUM 1	MSMT	DRUM 1
33	0.259	36	0.146
34	0.181	37	0.149
35	0.162	38	0.315

BOTTOM HEAD

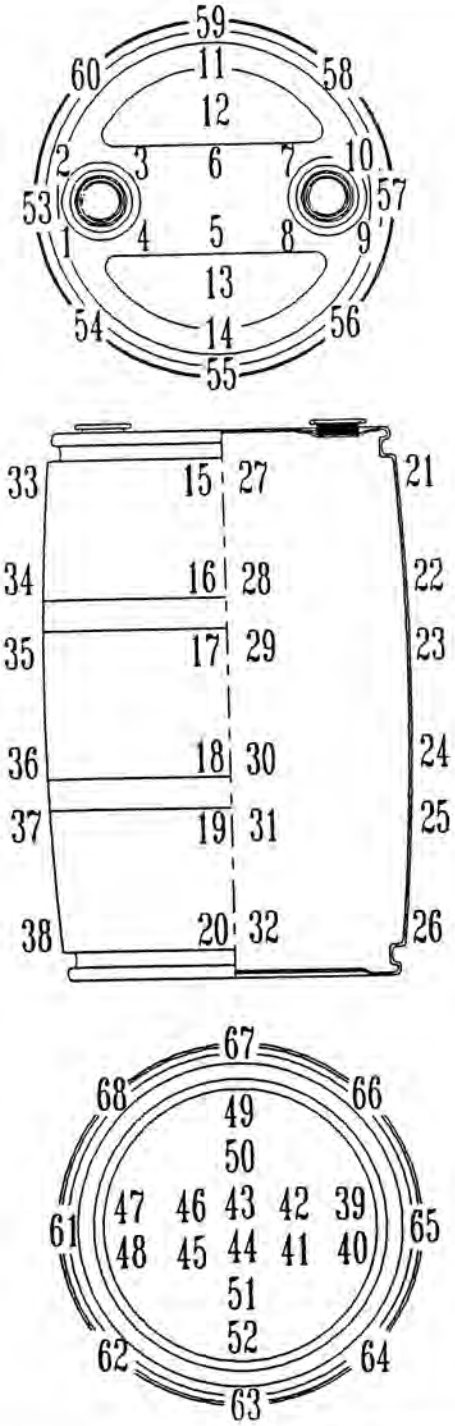
MSMT	DRUM 1	MSMT	DRUM 1
39	0.242	46	0.216
40	0.260	47	0.267
41	0.214	48	0.259
42	0.205	49	0.266
43	0.191	50	0.198
44	0.233	51	0.232
45	0.233	52	0.250

TOP RADIUS (Under Handling Ring)

MSMT	DRUM 1	MSMT	DRUM 1
53	0.179	57	0.152
54	0.153	58	0.155
55	0.180	59	0.176
56	0.126	60	0.155

BOTTOM RADIUS

MSMT	DRUM 1	MSMT	DRUM 1
61	0.258	65	0.262
62	0.228	66	0.229
63	0.220	67	0.214
64	0.248	68	0.267

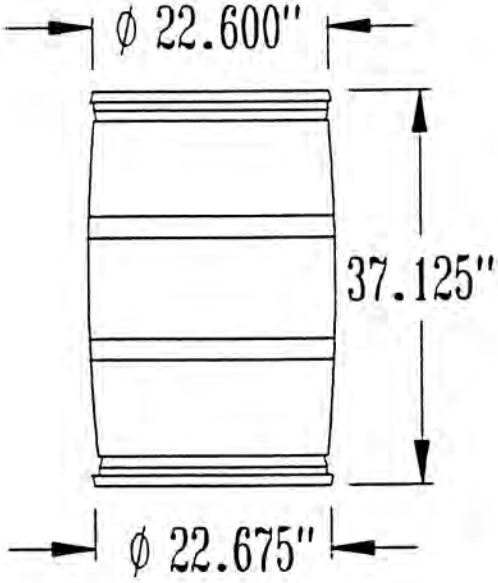


TEST PROCEDURES AND RESULTS

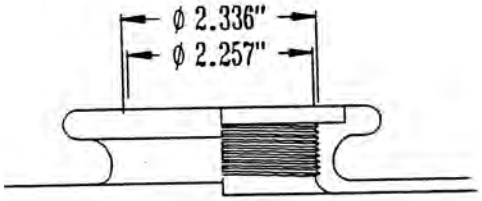
QUALITY CONTROL AUDIT RESULTS - Continued

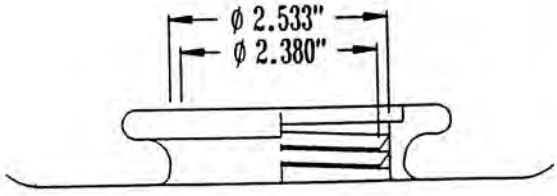
DRUM VARIABLE - B (10% RECYCLE RESIN & 90% VIRGIN RESIN)				
• **Description:**	210 Liter Tight Head Drum			
• **Material:**	High Density Polyethylene			
• **Pigment:**	Black			
• **Openings:** (Number & Size)	(1) 2" Buttress (1) 2" NPT			
• **Tare Weight:**	28.2 Lbs			
• **Overflow Capacity:**	59.4 Gallons		224.9 Kg (495.5 Lbs)	
• **98% Overflow Capacity:**	58.2 Gallons		220.4 Kg (485.6 Lbs)	
• **Melt Index (I^{21}):**	3.28 g/10 minutes			
• **Density:**	0.954 g/cc			
• **Markings:**	HUNTER	7-23-94	210 LITERS	"2" HDPE

Overall Drum Dimensions



Finish Dimensions





13145 - PDI B (10% DRUM RECYCLE & 90% VIRGIN RESIN)

WALL THICKNESS PROFILE (inches)

TOP HEAD

MSMT	DRUM 1	MSMT	DRUM 1
1	0.186	8	0.176
2	0.186	9	0.128
3	0.170	10	0.180
4	0.168	11	0.193
5	0.166	12	0.175
6	0.165	13	0.178
7	0.170	14	0.199

SIDEWALL

MSMT	DRUM 1	MSMT	DRUM 1
15	0.210	18	0.160
16	0.171	19	0.148
17	0.176	20	0.231

SIDEWALL

MSMT	DRUM 1	MSMT	DRUM 1
21	0.258	24	0.149
22	0.171	25	0.154
23	0.167	26	0.299

SIDEWALL

MSMT	DRUM 1	MSMT	DRUM 1
27	0.201	30	0.165
28	0.182	31	0.163
29	0.181	32	0.197

SIDEWALL

MSMT	DRUM 1	MSMT	DRUM 1
33	0.281	36	0.159
34	0.184	37	0.152
35	0.168	38	0.316

BOTTOM HEAD

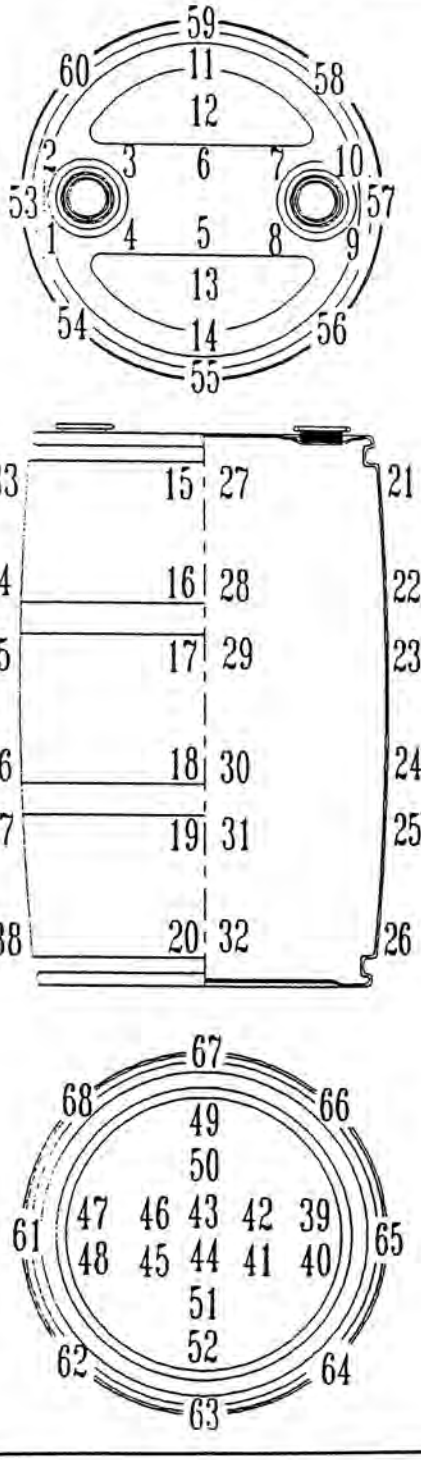
MSMT	DRUM 1	MSMT	DRUM 1
39	0.252	46	0.203
40	0.268	47	0.284
41	0.222	48	0.274
42	0.204	49	0.231
43	0.160	50	0.194
44	0.233	51	0.193
45	0.229	52	0.246

TOP RADIUS (Under Handling Ring)

MSMT	DRUM 1	MSMT	DRUM 1
53	0.233	57	0.187
54	0.173	58	0.148
55	0.176	59	0.183
56	0.159	60	0.174

BOTTOM RADIUS

MSMT	DRUM 1	MSMT	DRUM 1
61	0.233	65	0.234
62	0.208	66	0.258
63	0.230	67	0.258
64	0.258	68	0.249

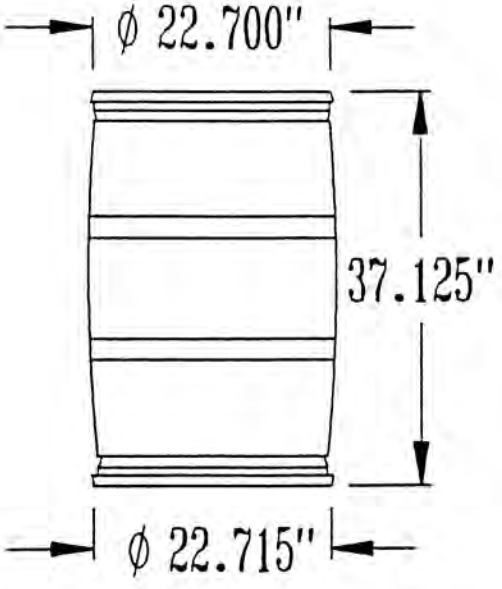


TEST PROCEDURES AND RESULTS

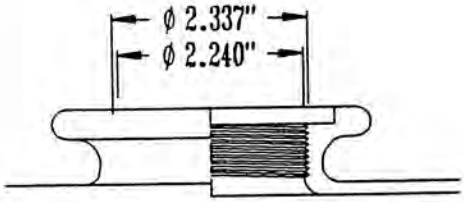
QUALITY CONTROL AUDIT RESULTS - Continued

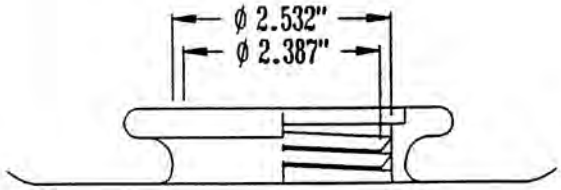
DRUM VARIABLE - C (25% RECYCLE RESIN & 75% VIRGIN RESIN)			
• **Description:**	210 Liter Tight Head Drum		
• **Material:**	High Density Polyethylene		
• **Pigment:**	Black		
• **Openings:** (Number & Size)	(1) 2" Buttress (1) 2" NPT		
• **Tare Weight:**	28.2 Lbs		
• **Overflow Capacity:**	59.4 Gallons	224.9 Kg (495.5 Lbs)	
• **98% Overflow Capacity:**	58.2 Gallons	220.4 Kg (485.6 Lbs)	
• **Melt Index (I^{21}):**	3.62 g/10 minutes		
• **Density:**	0.958 g/cc		
• **Markings:**	HUNTER 7-23-94	210 LITERS	"2" HDPE

Overall Drum Dimensions



Finish Dimensions





13145 - PDI C (25% DRUM RECYCLE & 75% VIRGIN RESIN)

WALL THICKNESS PROFILE (inches)

TOP HEAD

MSMT	DRUM 1	MSMT	DRUM 1
1	0.194	8	0.198
2	0.185	9	0.184
3	0.197	10	0.188
4	0.191	11	0.204
5	0.185	12	0.190
6	0.188	13	0.192
7	0.195	14	0.198

SIDEWALL

MSMT	DRUM 1	MSMT	DRUM 1
15	0.213	18	0.170
16	0.181	19	0.160
17	0.181	20	0.234

SIDEWALL

MSMT	DRUM 1	MSMT	DRUM 1
21	0.290	24	0.153
22	0.180	25	0.160
23	0.183	26	0.293

SIDEWALL

MSMT	DRUM 1	MSMT	DRUM 1
27	0.193	30	0.164
28	0.184	31	0.153
29	0.182	32	0.194

SIDEWALL

MSMT	DRUM 1	MSMT	DRUM 1
33	0.280	36	0.145
34	0.179	37	0.161
35	0.164	38	0.296

BOTTOM HEAD

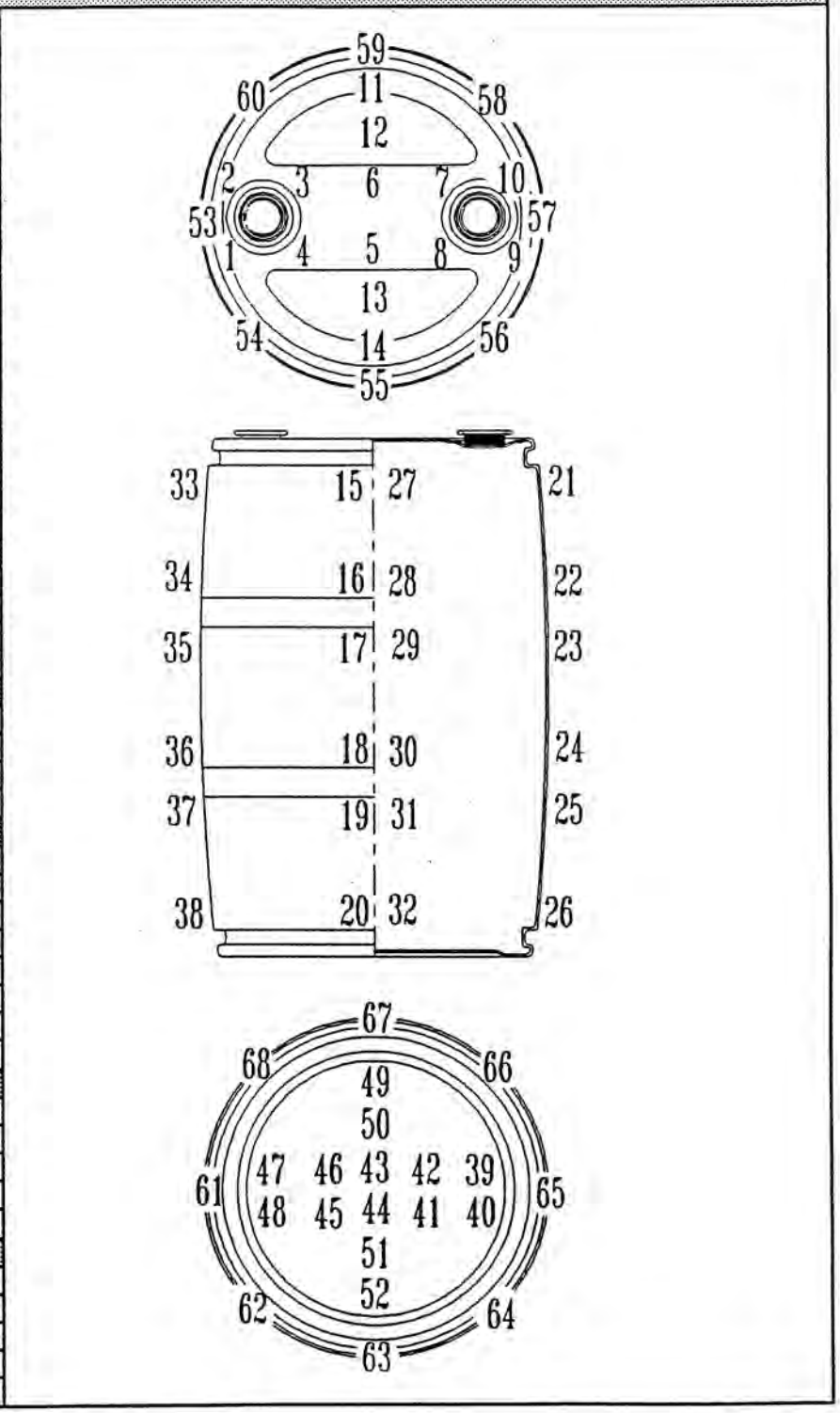
MSMT	DRUM 1	MSMT	DRUM 1
39	0.211	46	0.190
40	0.231	47	0.242
41	0.204	48	0.240
42	0.175	49	0.166
43	0.186	50	0.148
44	0.205	51	0.216
45	0.213	52	0.174

TOP RADIUS (Under Handling Ring)

MSMT	DRUM 1	MSMT	DRUM 1
53	0.197	57	0.166
54	0.169	58	0.169
55	0.181	59	0.166
56	0.188	60	0.167

BOTTOM RADIUS

MSMT	DRUM 1	MSMT	DRUM 1
61	0.251	65	0.167
62	0.186	66	0.202
63	0.191	67	0.162
64	0.218	68	0.195

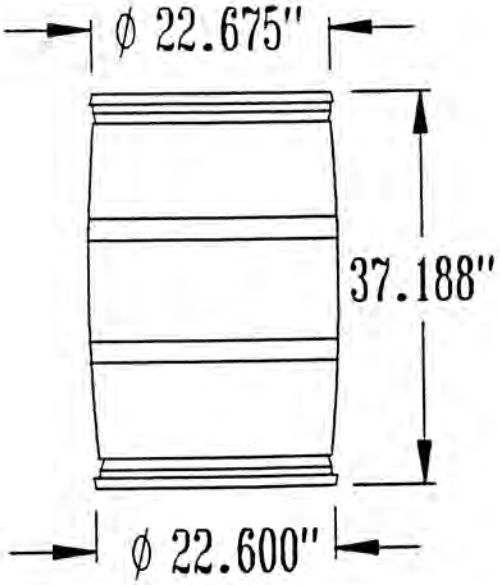


TEST PROCEDURES AND RESULTS

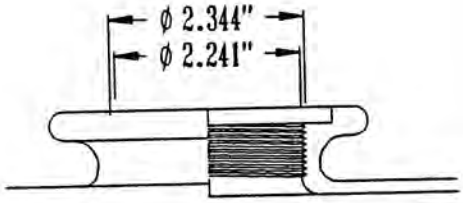
QUALITY CONTROL AUDIT RESULTS - Continued

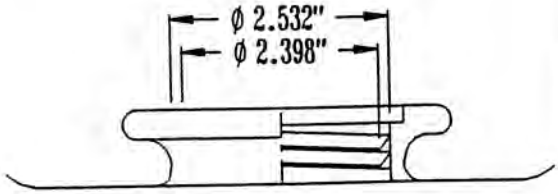
DRUM VARIABLE - D (50% RECYCLE RESIN & 50% VIRGIN RESIN)				
• **Description:**	210 Liter Tight Head Drum			
• **Material:**	High Density Polyethylene			
• **Pigment:**	Black			
• **Openings:** (Number & Size)	(1) 2" Buttress (1) 2" NPT			
• **Tare Weight:**	28.2 Lbs			
• **Overflow Capacity:**	59.4 Gallons		224.9 Kg (495.5 Lbs)	
• **98% Overflow Capacity:**	58.2 Gallons		220.4 Kg (485.6 Lbs)	
• **Melt Index (I^{21}):**	4.02 g/10 minutes			
• **Density:**	0.954 g/cc			
• **Markings:**	HUNTER	7-23-94	210 LITERS	"2" HDPE

Overall Drum Dimensions



Finish Dimensions





13145 - PD1 D (50% DRUM RECYCLE & 50% VIRGIN RESIN)

WALL THICKNESS PROFILE (inches)

TOP HEAD

MSMT	DRUM 1	MSMT	DRUM 1
1	0.208	8	0.211
2	0.194	9	0.209
3	0.199	10	0.207
4	0.203	11	0.203
5	0.211	12	0.203
6	0.207	13	0.206
7	0.206	14	0.202

SIDEWALL

MSMT	DRUM 1	MSMT	DRUM 1
15	0.220	18	0.171
16	0.202	19	0.156
17	0.197	20	0.226

SIDEWALL

MSMT	DRUM 1	MSMT	DRUM 1
21	0.299	24	0.157
22	0.185	25	0.161
23	0.187	26	0.292

SIDEWALL

MSMT	DRUM 1	MSMT	DRUM 1
27	0.198	30	0.156
28	0.184	31	0.149
29	0.178	32	0.201

SIDEWALL

MSMT	DRUM 1	MSMT	DRUM 1
33	0.281	36	0.130
34	0.182	37	0.151
35	0.160	38	0.296

BOTTOM HEAD

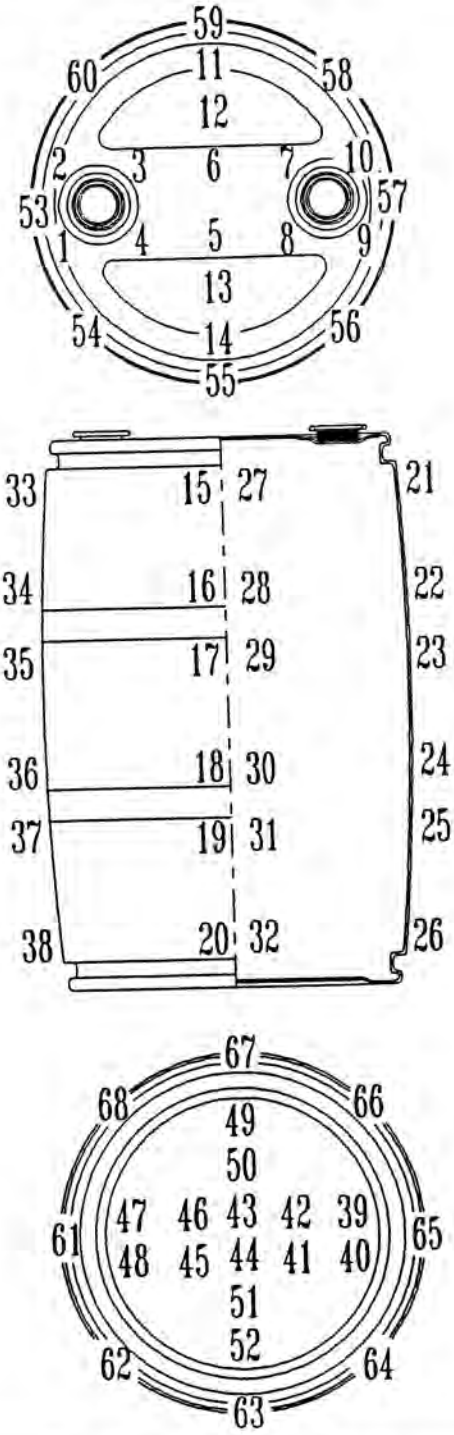
MSMT	DRUM 1	MSMT	DRUM 1
39	0.235	46	0.201
40	0.213	47	0.258
41	0.201	48	0.239
42	0.199	49	0.167
43	0.192	50	0.164
44	0.209	51	0.170
45	0.215	52	0.176

TOP RADIUS (Under Handling Ring)

MSMT	DRUM 1	MSMT	DRUM 1
53	0.220	57	0.265
54	0.155	58	0.162
55	0.171	59	0.165
56	0.155	60	0.152

BOTTOM RADIUS

MSMT	DRUM 1	MSMT	DRUM 1
61	0.216	65	0.211
62	0.173	66	0.137
63	0.173	67	0.177
64	0.174	68	0.151

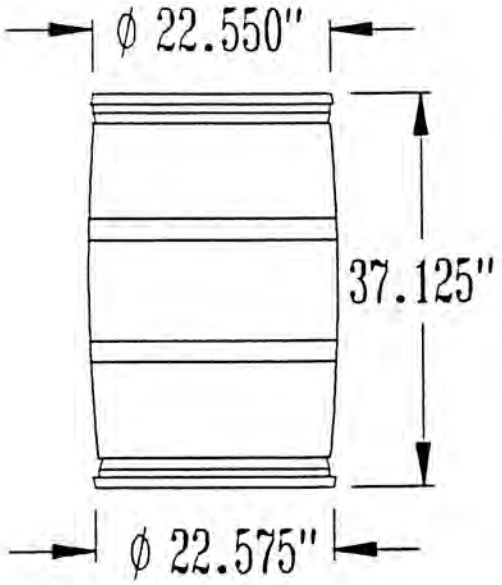


TEST PROCEDURES AND RESULTS

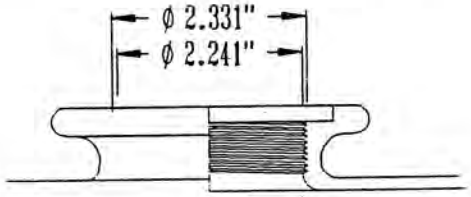
QUALITY CONTROL AUDIT RESULTS - Continued

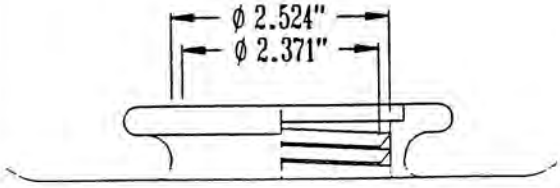
DRUM VARIABLE - E (100% RECYCLE RESIN)			
• **Description:**	210 Liter Tight Head Drum		
• **Material:**	High Density Polyethylene		
• **Pigment:**	Black		
• **Openings:** (Number & Size)	(1) 2" Buttress (1) 2" NPT		
• **Tare Weight:**	28.2 Lbs		
• **Overflow Capacity:**	59.4 Gallons	224.9 Kg (495.5 Lbs)	
• **98% Overflow Capacity:**	58.2 Gallons	220.4 Kg (485.6 Lbs)	
• **Melt Index (I^{21}):**	5.26 g/10 minutes		
• **Density:**	0.954 g/cc		
• **Markings:**	HUNTER 7-23-94	210 LITERS	"2" HDPE

Overall Drum Dimensions



Finish Dimensions





13145 - PDI E (100% DRUM RECYCLE)

WALL THICKNESS PROFILE (inches)

TOP HEAD

MSMT	DRUM 1	MSMT	DRUM 1
1	0.218	8	0.218
2	0.211	9	0.231
3	0.204	10	0.204
4	0.213	11	0.225
5	0.218	12	0.212
6	0.213	13	0.215
7	0.214	14	0.234

SIDEWALL

MSMT	DRUM 1	MSMT	DRUM 1
15	0.200	18	0.145
16	0.182	19	0.152
17	0.179	20	0.195

SIDEWALL

MSMT	DRUM 1	MSMT	DRUM 1
21	0.280	24	0.126
22	0.178	25	0.151
23	0.156	26	0.285

SIDEWALL

MSMT	DRUM 1	MSMT	DRUM 1
27	0.219	30	0.161
28	0.188	31	0.166
29	0.184	32	0.234

SIDEWALL

MSMT	DRUM 1	MSMT	DRUM 1
33	0.329	36	0.146
34	0.180	37	0.161
35	0.179	38	0.277

BOTTOM HEAD

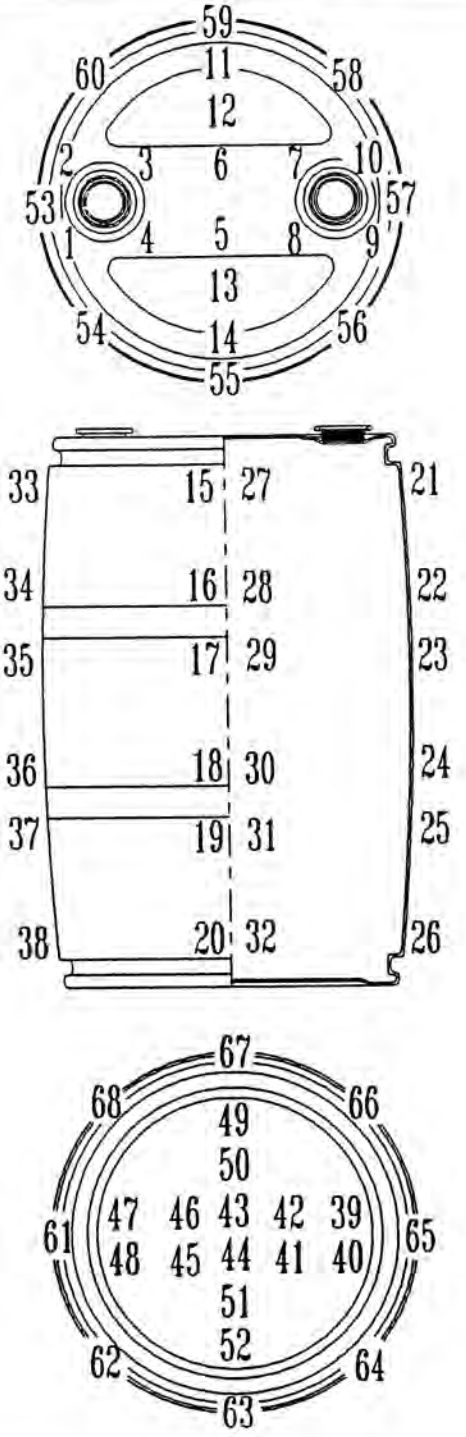
MSMT	DRUM 1	MSMT	DRUM 1
39	0.227	46	0.216
40	0.219	47	0.264
41	0.202	48	0.218
42	0.186	49	0.179
43	0.184	50	0.184
44	0.177	51	0.181
45	0.203	52	0.188

TOP RADIUS (Under Handling Ring)

MSMT	DRUM 1	MSMT	DRUM 1
53	0.193	57	0.185
54	0.163	58	0.160
55	0.148	59	0.148
56	0.151	60	0.147

BOTTOM RADIUS

MSMT	DRUM 1	MSMT	DRUM 1
61	0.191	65	0.176
62	0.160	66	0.153
63	0.163	67	0.169
64	0.158	68	0.159



TEST PROCEDURES AND RESULTS - 28 DAY STACK TESTS

DRUM VARIABLE - A (100% VIRGIN RESIN)

SAMPLE SIZE:
- 2 Samples / Variable

FILL CAPACITY:
- 98% of Maximum Capacity
- 220.46 Kg (485.60 Lbs) Net / Drum

CLOSURE APPLICATION :
- 20 Ft-Lbs (Buttress & NPT)

STACK TEST DURATION:
- 28 Days

TEST LOAD APPLIED:
- 908 Kg (2000 Lbs)

FILLING SUBSTANCE:
- Water

DRUM TEST WEIGHT:
- 233.27 Kg (513.80 Lbs)

CONDITIONING:
- 40°C (104°F)

STACK TEST EQUIPMENT:
- Dead Load Steel Weights
  Guided Load Fixtures
- Vollrath Walk-In Environmental Chamber
- Rieke W-168 PT (20/9) Torque Wrench

TEST STANDARD:
- Department of Transportation's Title 49 CFR; Section 178.606 (c)
- ASTM D4577-Standard Test Method for Compression Resistance of a Container Under Constant Load

STACK TEST LOAD CALCULATION

• Number of Drums in a 3 meter (118") high stack (-1):	2.18 Drums
• Package gross weight (Based On 1.8 Specific Gravity):	409.63 Kg (902.28 Lbs)

2.18 Drums x 409.63 Kg (902.28 Lbs) = 892.99 Kg (1966.95 Lbs) Min. Required Load Per Drum

STACK TEST RESULTS

Sample #	Maximum Deflection Following 28 Day Stack Test (Inches)	Results
1 (A-2)	0.31"	Pass
2 (A-11)	0.25"	Pass

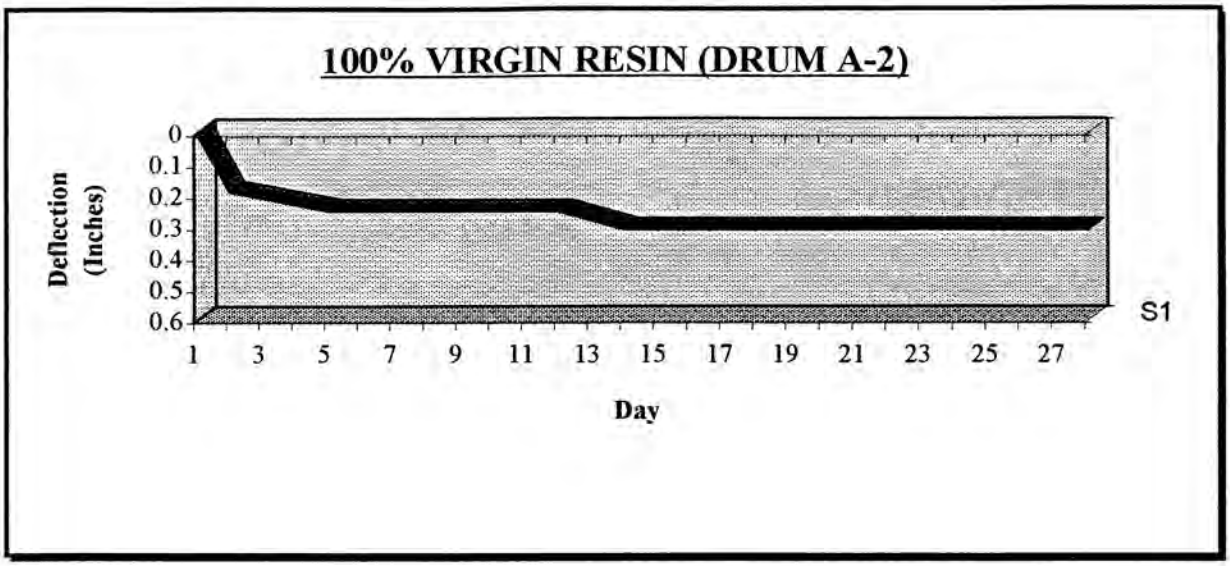
Refer to Photo 1 in Appendix I for 28 Day Stack Test Set-Up
Refer to next page for Time vs. Deflection Graphs

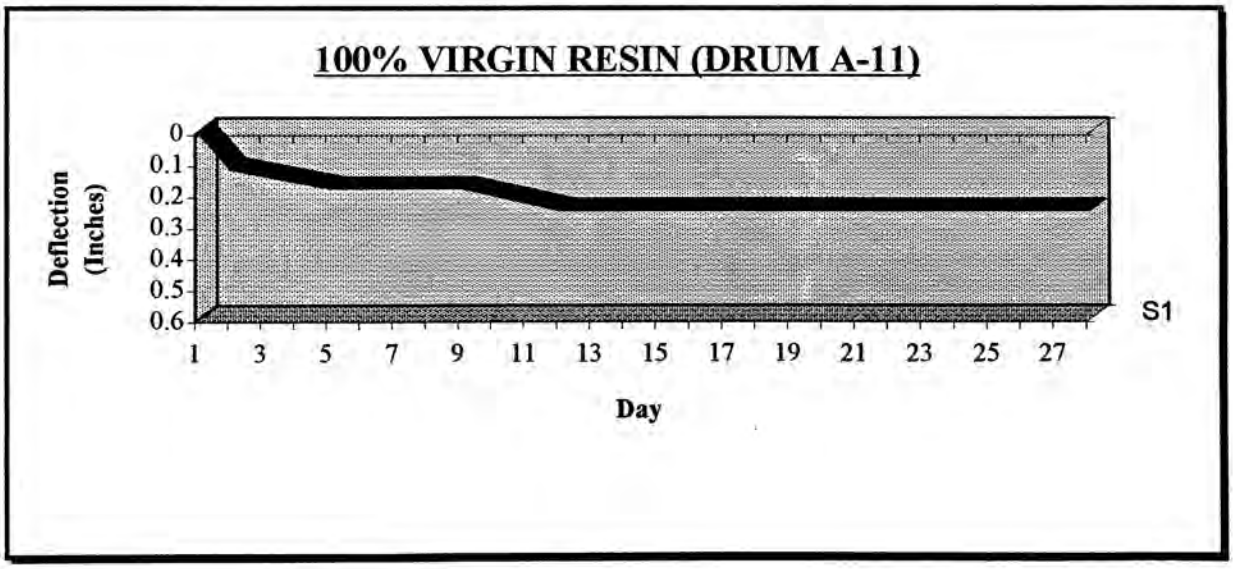
CRITERIA FOR PASSING THE TEST

- There is no deterioration that could adversely affect transport safety or any distortion liable to reduce its strength or cause instability in stacks of packages.
- No test sample may leak from the inner receptacle or inner packagings.

TEST PROCEDURES AND RESULTS - 28 DAY STACK TESTS

DRUM VARIABLE - A (100% VIRGIN RESIN)





TEST PROCEDURES AND RESULTS - 28 DAY STACK TESTS

DRUM VARIABLE - B (10% RECYCLE RESIN & 90% VIRGIN RESIN)

SAMPLE SIZE:
- 2 Samples / Variable

FILLING SUBSTANCE:
- Water

FILL CAPACITY:
- 98% of Maximum Capacity
- 220.46 Kg (485.60 Lbs) Net / Drum

DRUM TEST WEIGHT:
- 233.27 Kg (513.80 Lbs)

CLOSURE APPLICATION :
- 20 Ft-Lbs (Buttress & NPT)

CONDITIONING:
- 40°C (104°F)

STACK TEST DURATION:
- 28 Days

STACK TEST EQUIPMENT:
- Dead Load Steel Weights Guided Load Fixtures
- Vollrath Walk-In Environmental Chamber
- Rieke W-168 PT (20/9) Torque Wrench

TEST LOAD APPLIED:
- 908 Kg (2000 Lbs)

TEST STANDARD:
- Department of Transportation's Title 49 CFR; Section 178.606 (c)
- ASTM D4577-Standard Test Method for Compression Resistance of a Container Under Constant Load

STACK TEST LOAD CALCULATION

• Number of Drums in a 3 meter (118") high stack (-1):	2.18 Drums
• Package gross weight (Based On 1.8 Specific Gravity):	409.63 Kg (902.28 Lbs)

2.18 Drums x 409.63 Kg (902.28 Lbs) = 892.99 Kg (1966.95 Lbs) Min. Required Load Per Drum

STACK TEST RESULTS

Sample #	Maximum Deflection Following 28 Day Stack Test (Inches)	Results
1 (B-2)	0.25"	Pass
2 (B-14)	0.31"	Pass

Refer to Photo 1 in Appendix I for 28 Day Stack Test Set-Up
Refer to next page for Time vs. Deflection Graphs

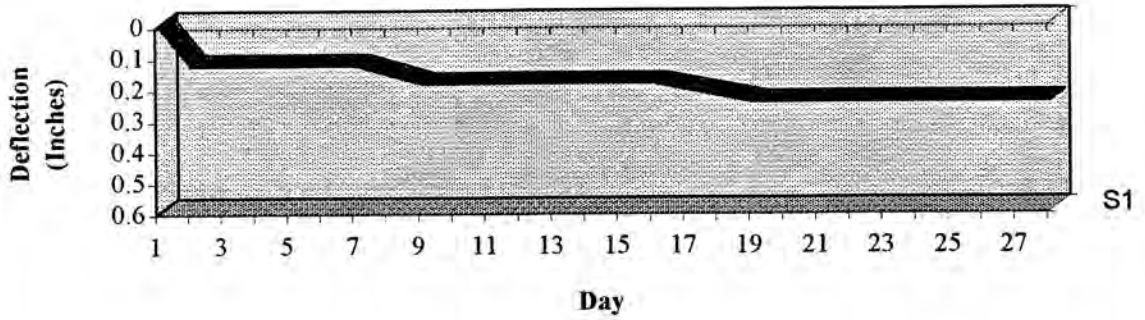
CRITERIA FOR PASSING THE TEST

- There is no deterioration that could adversely affect transport safety or any distortion liable to reduce its strength or cause instability in stacks of packages.
- No test sample may leak from the inner receptacle or inner packagings.

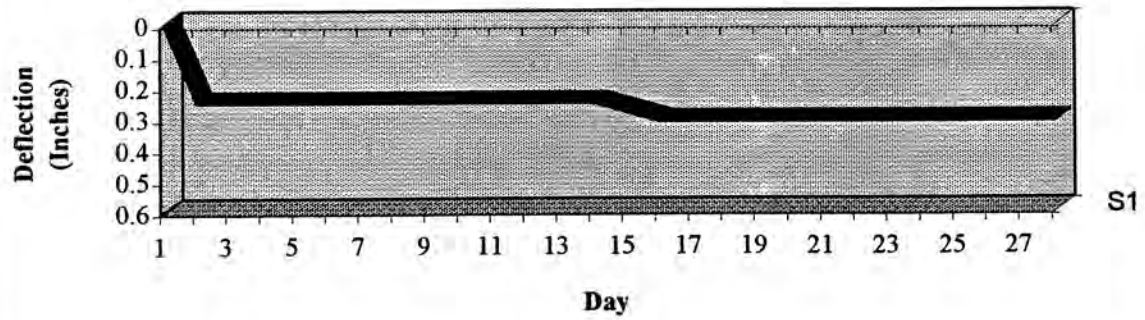
TEST PROCEDURES AND RESULTS - 28 DAY STACK TESTS

DRUM VARIABLE - B (10% RECYCLE RESIN & 90% VIRGIN RESIN)

10% RECYCLE RESIN (DRUM B-2)



10% RECYCLE RESIN (DRUM B-14)



TEST PROCEDURES AND RESULTS - 28 DAY STACK TESTS

DRUM VARIABLE - C (25% RECYCLE RESIN & 75% VIRGIN RESIN)

SAMPLE SIZE:
- 2 Samples / Variable

FILLING SUBSTANCE:
- Water

FILL CAPACITY:
- 98% of Maximum Capacity
- 220.46 Kg (485.60 Lbs) Net / Drum

DRUM TEST WEIGHT:
- 233.27 Kg (513.80 Lbs)

CLOSURE APPLICATION :
- 20 Ft-Lbs (Buttress & NPT)

CONDITIONING:
- 40°C (104°F)

STACK TEST DURATION:
- 28 Days

STACK TEST EQUIPMENT:
- Dead Load Steel Weights
  Guided Load Fixtures
- Vollrath Walk-In Environmental Chamber
- Rieke W-168 PT (20/9) Torque Wrench

TEST LOAD APPLIED:
- 908 Kg (2000 Lbs)

TEST STANDARD:
- Department of Transportation's Title 49 CFR; Section 178.606 (c)
- ASTM D4577-Standard Test Method for Compression Resistance of a Container Under Constant Load

STACK TEST LOAD CALCULATION	
• Number of Drums in a 3 meter (118") high stack (-1):	2.18 Drums
• Package gross weight (Based On 1.8 Specific Gravity):	409.63 Kg (902.28 Lbs)

2.18 Drums x 409.63 Kg (902.28 Lbs) = 892.99 Kg (1966.95 Lbs) Min. Required Load Per Drum

STACK TEST RESULTS

Sample #	Maximum Deflection Following 28 Day Stack Test (Inches)	Results
1 (C-7)	0.37"	Pass
2 (C-21)	0.31"	Pass

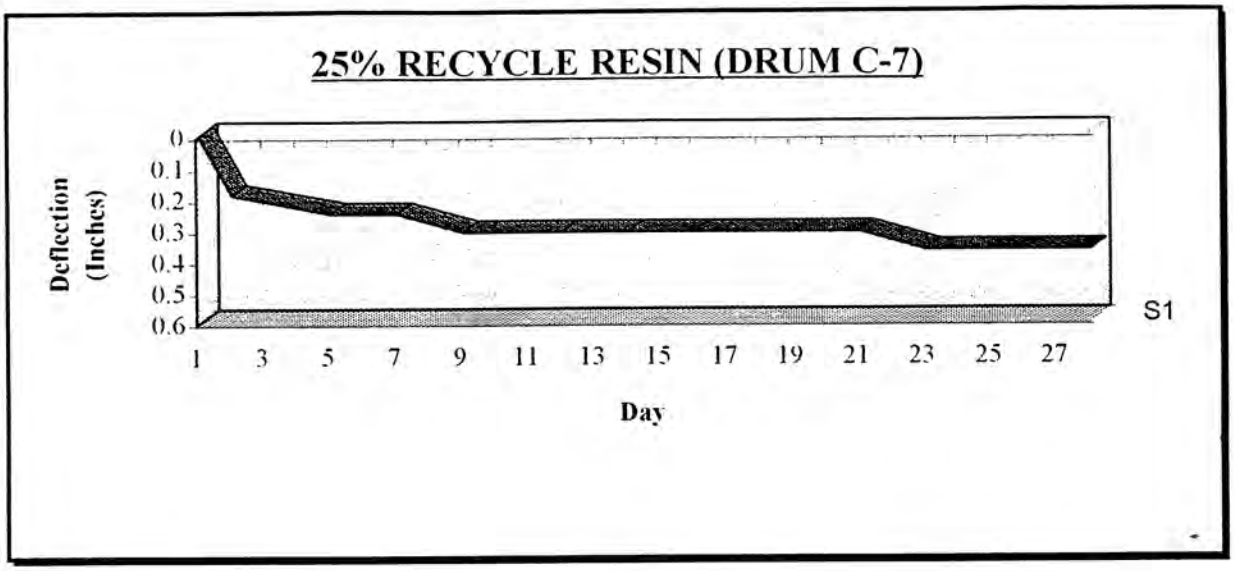
Refer to Photo 1 in Appendix I for 28 Day Stack Test Set-Up
Refer to next page for Time vs. Deflection Graphs

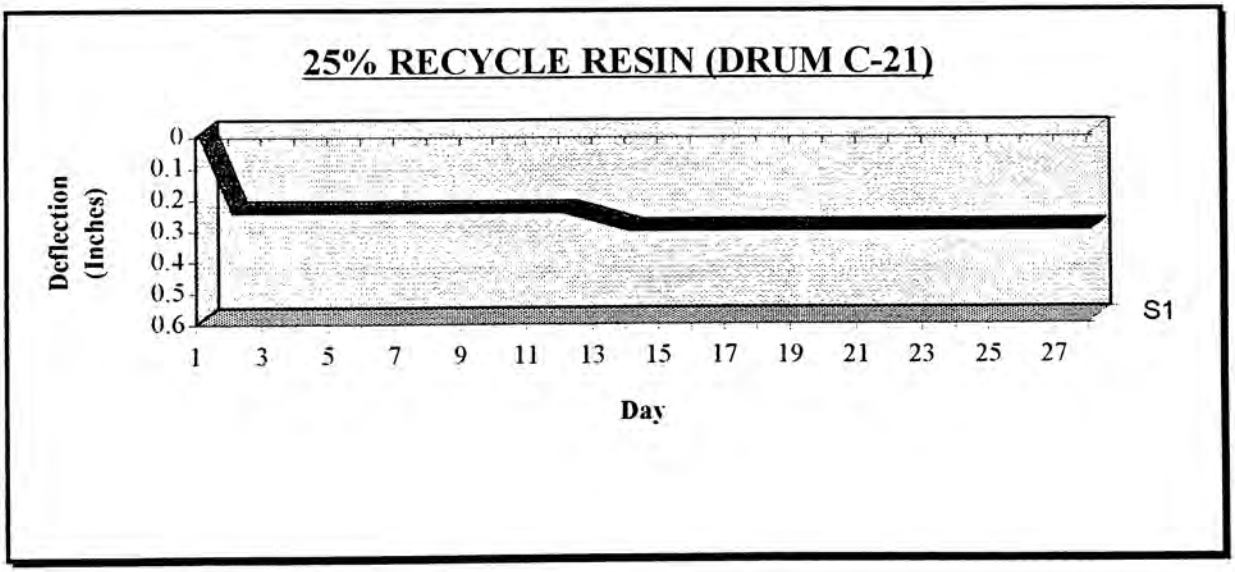
CRITERIA FOR PASSING THE TEST

- There is no deterioration that could adversely affect transport safety or any distortion liable to reduce its strength or cause instability in stacks of packages.
- No test sample may leak from the inner receptacle or inner packagings.

TEST PROCEDURES AND RESULTS - 28 DAY STACK TESTS

DRUM VARIABLE - C (25% RECYCLE RESIN & 75% VIRGIN RESIN)





TEST PROCEDURES AND RESULTS - 28 DAY STACK TESTS

DRUM VARIABLE - D (50% RECYCLE RESIN & 50% VIRGIN RESIN)

SAMPLE SIZE:
- 2 Samples / Variable

FILL CAPACITY:
- 98% of Maximum Capacity
- 220.46 Kg (485.60 Lbs) Net / Drum

CLOSURE APPLICATION :
- 20 Ft-Lbs (Buttress & NPT)

STACK TEST DURATION:
- 28 Days

TEST LOAD APPLIED:
- 908 Kg (2000 Lbs)

FILLING SUBSTANCE:
- Water

DRUM TEST WEIGHT:
- 233.27 Kg (513.80 Lbs)

CONDITIONING:
- 40°C (104°F)

STACK TEST EQUIPMENT:
- Dead Load Steel Weights
  Guided Load Fixtures
- Vollrath Walk-In Environmental Chamber
- Rieke W-168 PT (20/9) Torque Wrench

TEST STANDARD:
- Department of Transportation's Title 49 CFR; Section 178.606 (c)
- ASTM D4577-Standard Test Method for Compression Resistance of a Container Under Constant Load

STACK TEST LOAD CALCULATION	
• Number of Drums in a 3 meter (118") high stack (-1):	2.18 Drums
• Package gross weight (Based On 1.8 Specific Gravity):	409.63 Kg (902.28 Lbs)

2.18 Drums x 409.63 Kg (902.28 Lbs) = 892.99 Kg (1966.95 Lbs) Min. Required Load Per Drum

STACK TEST RESULTS		
Sample #	**Maximum Deflection Following 28 Day Stack Test (Inches)**	**Results**
1 (D-5)	0.25"	Pass
2 (D-17)	0.25"	Pass

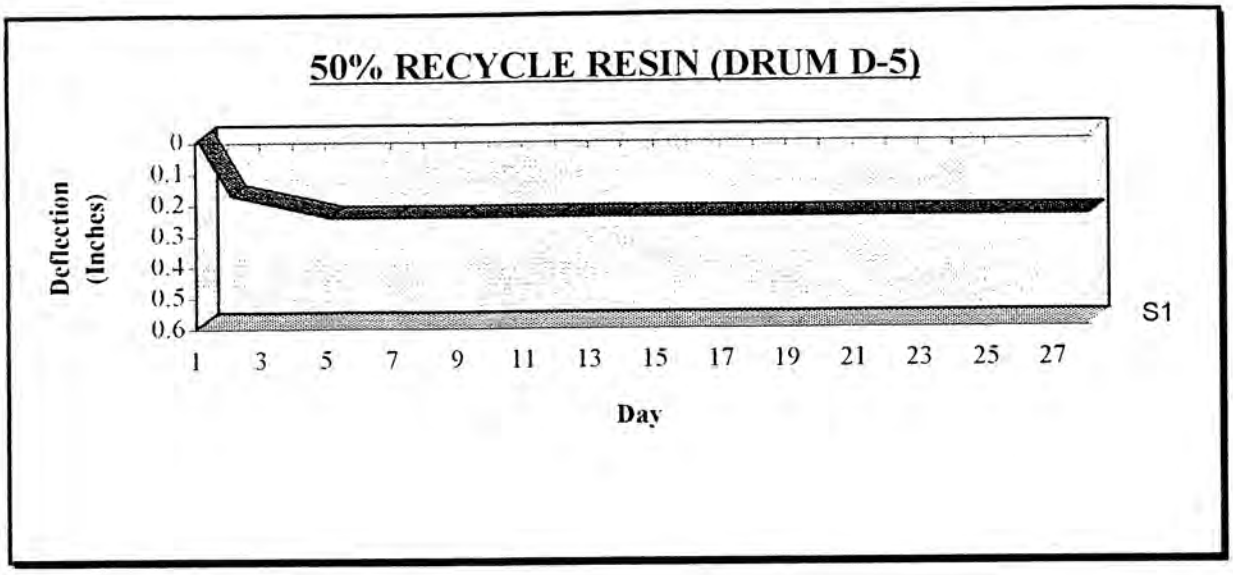
Refer to Photo 1 in Appendix I for 28 Day Stack Test Set-Up
Refer to next page for Time vs. Deflection Graphs

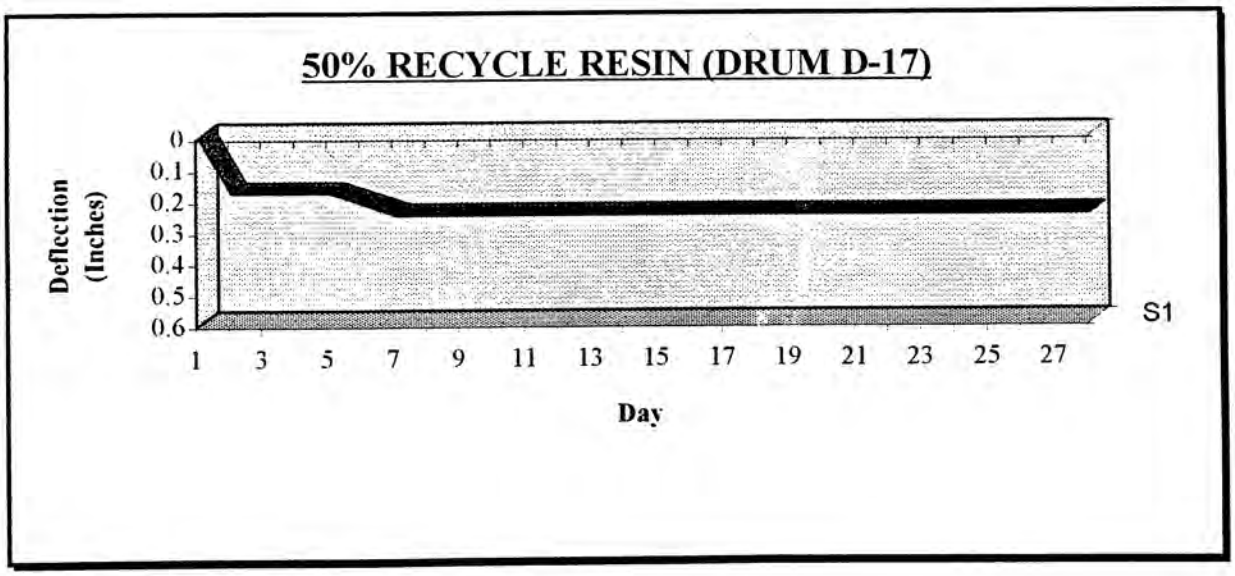
CRITERIA FOR PASSING THE TEST

- There is no deterioration that could adversely affect transport safety or any distortion liable to reduce its strength or cause instability in stacks of packages.
- No test sample may leak from the inner receptacle or inner packagings.

TEST PROCEDURES AND RESULTS - 28 DAY STACK TESTS

DRUM VARIABLE - D (50% RECYCLE RESIN & 50% VIRGIN RESIN)





TEST PROCEDURES AND RESULTS - 28 DAY STACK TESTS

DRUM VARIABLE - E (100% RECYCLE RESIN)

SAMPLE SIZE:
- 2 Samples / Variable

FILLING SUBSTANCE:
- Water

FILL CAPACITY:
- 98% of Maximum Capacity
- 220.46 Kg (485.60 Lbs) Net / Drum

DRUM TEST WEIGHT:
- 233.27 Kg (513.80 Lbs)

CLOSURE APPLICATION :
- 20 Ft-Lbs (Buttress & NPT)

CONDITIONING:
- 40°C (104°F)

STACK TEST DURATION:
- 28 Days

STACK TEST EQUIPMENT:
- Dead Load Steel Weights
  Guided Load Fixtures
- Vollrath Walk-In Environmental Chamber
- Rieke W-168 PT (20/9) Torque Wrench

TEST LOAD APPLIED:
- 908 Kg (2000 Lbs)

TEST STANDARD:
- Department of Transportation's Title 49 CFR; Section 178.606 (c)
- ASTM D4577-Standard Test Method for Compression Resistance of a Container Under Constant Load

STACK TEST LOAD CALCULATION

• Number of Drums in a 3 meter (118") high stack (-1):	2.18 Drums
• Package gross weight (Based On 1.8 Specific Gravity):	409.63 Kg (902.28 Lbs)

2.18 Drums x 409.63 Kg (902.28 Lbs) = 892.99 Kg (1966.95 Lbs) Min. Required Load Per Drum

STACK TEST RESULTS

Sample #	Maximum Deflection Following 28 Day Stack Test (Inches)	Results
1 (E-3)	0.31"	Pass
2 (E-15)	0.50"	Pass

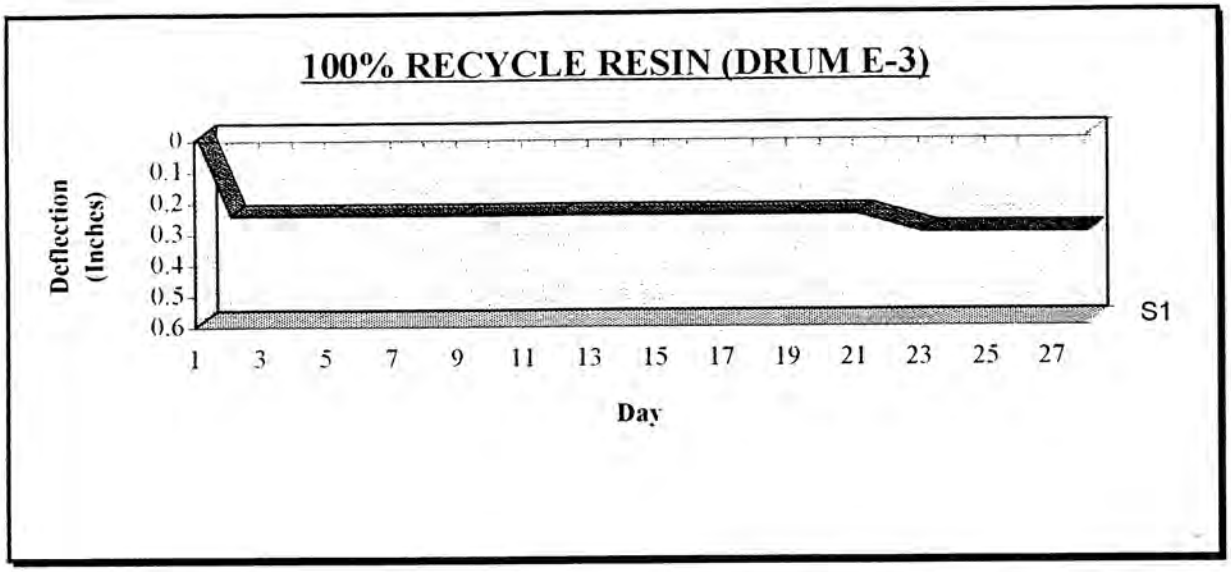
Refer to Photo 1 in Appendix I for 28 Day Stack Test Set-Up
Refer to next page for Time vs. Deflection Graphs

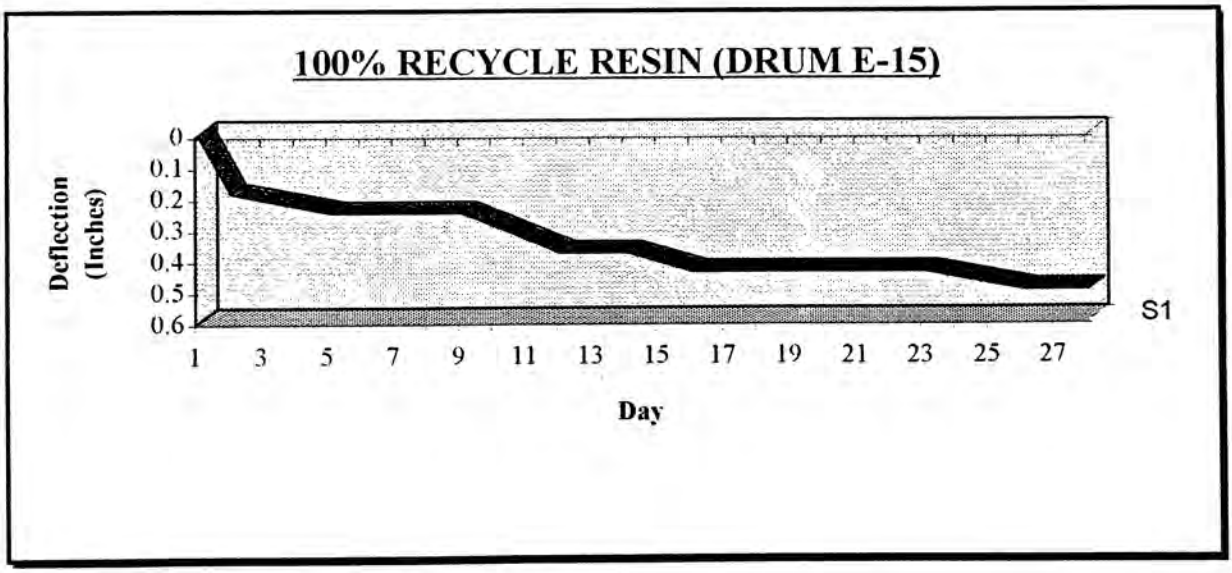
CRITERIA FOR PASSING THE TEST

- There is no deterioration that could adversely affect transport safety or any distortion liable to reduce its strength or cause instability in stacks of packages.
- No test sample may leak from the inner receptacle or inner packagings.

TEST PROCEDURES AND RESULTS - 28 DAY STACK TESTS

DRUM VARIABLE - E (100% RECYCLE RESIN)





TEST PROCEDURES AND RESULTS - ESCR TESTING (PROCEDURE - A)

DRUM VARIABLE - A (100% VIRGIN RESIN)

SAMPLE SIZE:
- 3 Samples / Variable

FILL CAPACITY:
- 10% Rated Capacity
- 20.84 Kg (45.90 Lbs) Net / Drum

CLOSURE APPLICATION :
- 20 Ft-Lbs (Buttress & NPT)

ESCR TEST DURATION:
- 28 Days

INTERNAL SURFACES RECOATED:
- Every 48 Hours

SAMPLES VISUALLY INSPECTED:
- Every 24 Hours

FILLING SUBSTANCE:
- 10% Nonyl Phenoxypoly Ethanol Solution

DRUM TEST WEIGHT:
- 233.27 Kg (513.80 Lbs) (10% of Rated Capacity)

CONDITIONING:
- 50.0°C (122°F)

INTERNAL TEST PRESSURE:
- 2.0 psi +/- 0.1 psi (13.8 kPa +/- 1.4 kPa)

ESCR TEST EQUIPMENT:
- Marshalltown G23606 3½" Gauge
- Vollrath Walk-In Environmental Chamber
- Rieke W-168 PT (20/9) Torque Wrench

TEST STANDARD:
- ASTM Draft Standard - Standard Practice For Environmental Stress Crack Resistance (ESCR) of Plastic Tighthead Drums Not Exceeding 60 Gallons (227 L) In Rated Capacity

ESCR TEST RESULTS

Sample #	Days to Failure	Comments \ Observations
1 (A-4)	21 Days	A 1" stress crack occurred along the bottom radius of drum A-4 (underneath the lower ring) on the NPT opening side *Refer to photo # 3 in appendix I*
2 (A-8)	21 Days	A 5" stress crack occurred along the bottom radius of drum A-8 (underneath the lower ring) on the NPT opening side *Refer to photo # 4 in appendix I*
3 (A-10)	18 Days	A 3" stress crack occurred along the bottom radius of drum A-10 (underneath the lower ring) on the NPT opening side *Refer to photo # 5 in appendix I*

Refer to Photo 2 in Appendix I for Environmental Stress Crack Resistance Test Set-Up

CRITERIA FOR PASSING THE TEST

- If there are no stress cracks evident (which would be indicated by bubbling or leaking of the regent through the points of failure) following the predetermined test duration.

TEST PROCEDURES AND RESULTS - ESCR TESTING (PROCEDURE - A)

DRUM VARIABLE - B (10% RECYCLE RESIN & 90% VIRGIN RESIN)

SAMPLE SIZE:
- 3 Samples / Variable

FILL CAPACITY:
- 10% Rated Capacity
- 20.84 Kg (45.90 Lbs) Net / Drum

CLOSURE APPLICATION :
- 20 Ft-Lbs (Buttress & NPT)

ESCR TEST DURATION:
- 28 Days

INTERNAL SURFACES RECOATED:
- Every 48 Hours

SAMPLES VISUALLY INSPECTED:
- Every 24 Hours

FILLING SUBSTANCE:
- 10% Nonyl Phenoxypoly Ethanol Solution

DRUM TEST WEIGHT:
- 233.27 Kg (513.80 Lbs) (10% of Rated Capacity)

CONDITIONING:
- 50.0°C (122°F)

INTERNAL TEST PRESSURE:
- 2.0 psi +/- 0.1 psi (13.8 kPa +/- 1.4 kPa)

ESCR TEST EQUIPMENT:
- Marshalltown G23606 3½" Gauge
- Vollrath Walk-In Environmental Chamber
- Rieke W-168 PT (20/9) Torque Wrench

TEST STANDARD:
- ASTM Draft Standard - Standard Practice For Environmental Stress Crack Resistance (ESCR) of Plastic Tighthead Drums Not Exceeding 60 Gallons (227 L) In Rated Capacity

ESCR TEST RESULTS

Sample #	Days to Failure	Comments \ Observations
1 (B-4)	18 Days	A 2" stress crack occurred along the bottom radius of drum B-4 (underneath the lower ring) on the NPT opening side **Refer to photo # 6 in appendix I*
2 (B-5)	12 Days	A 1" stress crack occurred along the bottom radius of drum B-5 (underneath the lower ring) on the NPT opening side **Refer to photo # 7 in appendix I*
3 (B-8)	20 Days	A 3" stress crack occurred along the bottom radius of drum B-8 (underneath the lower ring) on the NPT opening side **Refer to photo # 8 in appendix I*

Refer to Photo 2 in Appendix I for Environmental Stress Crack Resistance Test Set-Up

CRITERIA FOR PASSING THE TEST

- If there are no stress cracks evident (which would be indicated by bubbling or leaking of the regent through the points of failure) following the predetermined test duration.

TEST PROCEDURES AND RESULTS - ESCR TESTING (PROCEDURE - A)

DRUM VARIABLE - C (25% RECYCLE RESIN & 75% VIRGIN RESIN)

SAMPLE SIZE:
- 3 Samples / Variable

FILL CAPACITY:
- 10% Rated Capacity
- 20.84 Kg (45.90 Lbs) Net / Drum

CLOSURE APPLICATION :
- 20 Ft-Lbs (Buttress & NPT)

ESCR TEST DURATION:
- 28 Days

INTERNAL SURFACES RECOATED:
- Every 48 Hours

SAMPLES VISUALLY INSPECTED:
- Every 24 Hours

FILLING SUBSTANCE:
- 10% Nonyl Phenoxypoly Ethanol Solution

DRUM TEST WEIGHT:
- 233.27 Kg (513.80 Lbs) (10% of Rated Capacity)

CONDITIONING:
- 50.0°C (122°F)

INTERNAL TEST PRESSURE:
- 2.0 psi +/- 0.1 psi (13.8 kPa +/- 1.4 kPa)

ESCR TEST EQUIPMENT:
- Marshalltown G23606 3½" Gauge
- Vollrath Walk-In Environmental Chamber
- Rieke W-168 PT (20/9) Torque Wrench

TEST STANDARD:
- ASTM Draft Standard - Standard Practice For Environmental Stress Crack Resistance (ESCR) of Plastic Tighthead Drums Not Exceeding 60 Gallons (227 L) In Rated Capacity

ESCR TEST RESULTS

Sample #	Days to Failure	Comments \ Observations
1 (C-8)	18 Days	A 2" stress crack occurred along the bottom radius of drum C-8 (underneath the lower ring) on the buttress opening side **Refer to photo # 9 in appendix I*
2 (C-10)	21 Days	A 1" stress crack occurred along the bottom radius of drum C-10 (underneath the lower ring) on the buttress opening side **Refer to photo # 10 in appendix I*
3 (C-15)	14 Days	A 1" stress crack occurred along the bottom radius of drum C-15 (underneath the lower ring) on the NPT opening side **Refer to photo # 11 in appendix I*

Refer to Photo 2 in Appendix I for Environmental Stress Crack Resistance Test Set-Up

CRITERIA FOR PASSING THE TEST

- If there are no stress cracks evident (which would be indicated by bubbling or leaking of the regent through the points of failure) following the predetermined test duration.

TEST PROCEDURES AND RESULTS - ESCR TESTING (PROCEDURE - A)

DRUM VARIABLE - D (50% RECYCLE RESIN & 50% VIRGIN RESIN)

SAMPLE SIZE:
- 3 Samples / Variable

FILL CAPACITY:
- 10% Rated Capacity
- 20.84 Kg (45.90 Lbs) Net / Drum

CLOSURE APPLICATION :
- 20 Ft-Lbs (Buttress & NPT)

ESCR TEST DURATION:
- 28 Days

INTERNAL SURFACES RECOATED:
- Every 48 Hours

SAMPLES VISUALLY INSPECTED:
- Every 24 Hours

FILLING SUBSTANCE:
- 10% Nonyl Phenoxypoly Ethanol Solution

DRUM TEST WEIGHT:
- 233.27 Kg (513.80 Lbs) (10% of Rated Capacity)

CONDITIONING:
- 50.0°C (122°F)

INTERNAL TEST PRESSURE:
- 2.0 psi +/- 0.1 psi (13.8 kPa +/- 1.4 kPa)

ESCR TEST EQUIPMENT:
- Marshalltown G23606 3½" Gauge
- Vollrath Walk-In Environmental Chamber
- Rieke W-168 PT (20/9) Torque Wrench

TEST STANDARD:
- ASTM Draft Standard - Standard Practice For Environmental Stress Crack Resistance (ESCR) of Plastic Tighthead Drums Not Exceeding 60 Gallons (227 L) In Rated Capacity

ESCR TEST RESULTS

Sample #	Days to Failure	Comments \ Observations
1 (D-1)	16 Days	A 1" stress crack occurred along the bottom radius of drum D-1 (underneath the lower ring) on the NPT opening side **Refer to photo # 12 in appendix I*
2 (D-4)	23 Days	A 3" stress crack occurred along the top radius of drum D-4 (underneath the top ring) on the NPT opening side **Refer to photo # 13 in appendix I*
3 (D-9)	No Failure After 28 Days	

Refer to Photo 2 in Appendix I for Environmental Stress Crack Resistance Test Set-Up

CRITERIA FOR PASSING THE TEST

- If there are no stress cracks evident (which would be indicated by bubbling or leaking of the regent through the points of failure) following the predetermined test duration.

TEST PROCEDURES AND RESULTS - ESCR TESTING (PROCEDURE - A)

DRUM VARIABLE - E (100% RECYCLE RESIN)

SAMPLE SIZE:
- 3 Samples / Variable

FILL CAPACITY:
- 10% Rated Capacity
- 20.84 Kg (45.90 Lbs) Net / Drum

CLOSURE APPLICATION :
- 20 Ft-Lbs (Buttress & NPT)

ESCR TEST DURATION:
- 28 Days

INTERNAL SURFACES RECOATED:
- Every 48 Hours

SAMPLES VISUALLY INSPECTED:
- Every 24 Hours

FILLING SUBSTANCE:
- 10% Nonyl Phenoxypoly Ethanol Solution

DRUM TEST WEIGHT:
- 233.27 Kg (513.80 Lbs) (10% of Rated Capacity)

CONDITIONING:
- 50.0°C (122°F)

INTERNAL TEST PRESSURE:
- 2.0 psi +/- 0.1 psi (13.8 kPa +/- 1.4 kPa)

ESCR TEST EQUIPMENT:
- Marshalltown G23606 3½" Gauge
- Vollrath Walk-In Environmental Chamber
- Rieke W-168 PT (20/9) Torque Wrench

TEST STANDARD:
- ASTM Draft Standard - Standard Practice For Environmental Stress Crack Resistance (ESCR) of Plastic Tighthead Drums Not Exceeding 60 Gallons (227 L) In Rated Capacity

ESCR TEST RESULTS

Sample #	Days to Failure	Comments \ Observations
1 (E-6)	21 Days	A 1" stress crack occurred along the bottom radius of drum E-6 (underneath the lower ring) on the buttress opening side **Refer to photo # 14 in appendix I*
2 (E-10)	21 Days	A 4" stress crack occurred along the bottom radius of drum E-10 (underneath the lower ring) on the NPT opening side **refer to photo # 15 in appendix I*
3 (E-23)	25 Days	A 1" stress crack occurred along the top radius of drum E-23 (underneath the top ring) on the NPT opening side **Refer to photo # 16 in appendix I*

Refer to Photo 2 in Appendix I for Environmental Stress Crack Resistance Test Set-Up

CRITERIA FOR PASSING THE TEST

- If there are no stress cracks evident (which would be indicated by bubbling or leaking of the regent through the points of failure) following the predetermined test duration.

APPENDIX I - PHOTOGRAPHS

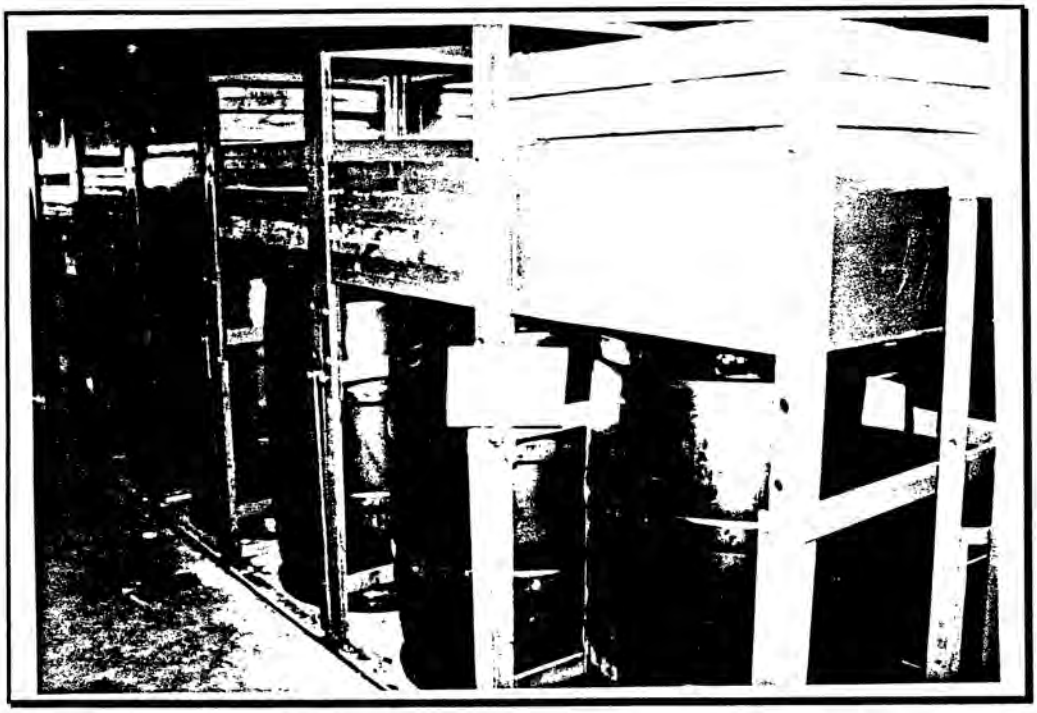


Photo # 1 - 28 Day Stack
⇦ Test Set-up

Photo # 2 - ESCR Test
Set-up ⇨

Photo # 3 - ESCR Test Failure

⇦ • Drum Variable = 100% Virgin Resin
• Days to Failure = 21 Days

Photo # 4 - ESCR Test Failure

⇨ • Drum Variable = 100% Virgin Resin
• Days to Failure = 21 Days

Photo # 5 - ESCR Test Failure

⇦ • Drum Variable = 100% Virgin Resin
• Days to Failure = 18 Days

Photo # 6 - ESCR Test Failure

⇨ • Drum Variable = 10% Recycle Resin
• Days to Failure = 18 Days

Photo # 7 - ESCR Test Failure

⇦ • Drum Variable = 10% Recycle Resin
• Days to Failure = 12 Days

Photo # 8 - ESCR Test Failure

⇨ • Drum Variable = 10% Recycle Resin
• Days to Failure = 20 Days

Photo # 9 - ESCR Test Failure

⇦ • Drum Variable = 25% Recycle Resin
• Days to Failure = 18 Days

Photo # 10 - ESCR Test Failure

⇨ • Drum Variable = 25% Recycle Resin
• Days to Failure = 21 Days

Photo # 11 - ESCR Test Failure

⇦ • Drum Variable = 25% Recycle Resin
• Days to Failure = 14 Days

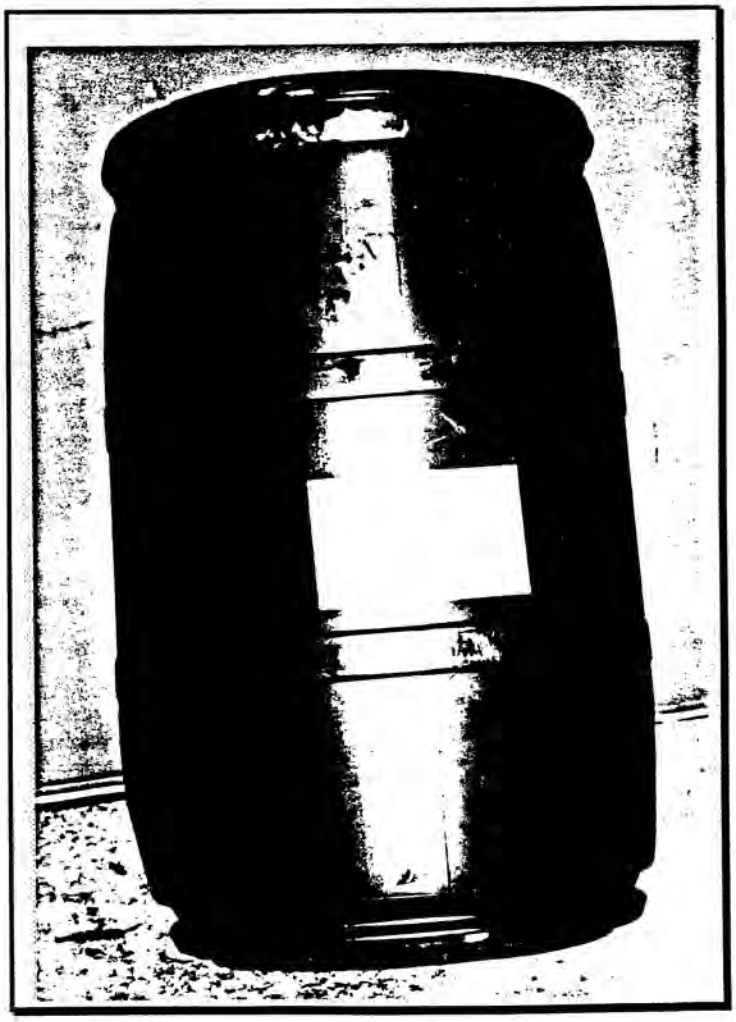


Photo # 12 - ESCR Test Failure

⇨ • Drum Variable = 50% Recycle Resin
• Days to Failure = 16 Days

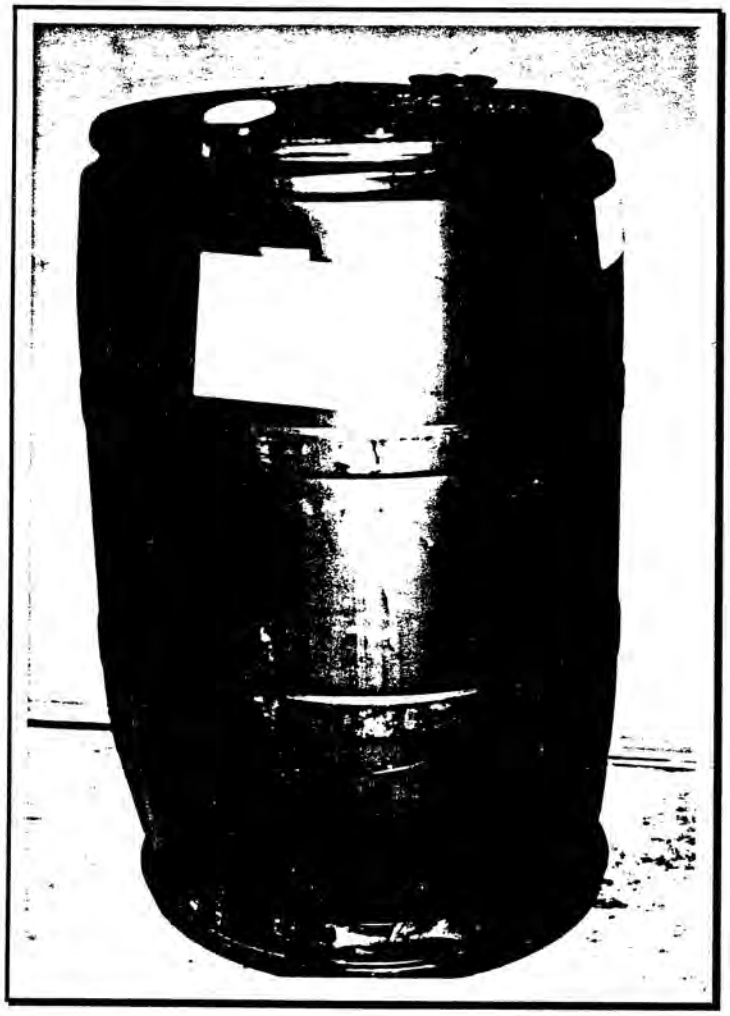


Photo # 13 - ESCR Test Failure

⇦ • Drum Variable = 50% Recycle Resin
• Days to Failure = 23 Days

Photo # 14 - ESCR Test Failure

⇨ • Drum Variable = 100% Recycle Resin
• Days to Failure = 21 Days

Photo # 15 - ESCR Test Failure

⇦ • Drum Variable = 100% Recycle Resin
• Days to Failure = 21 Days

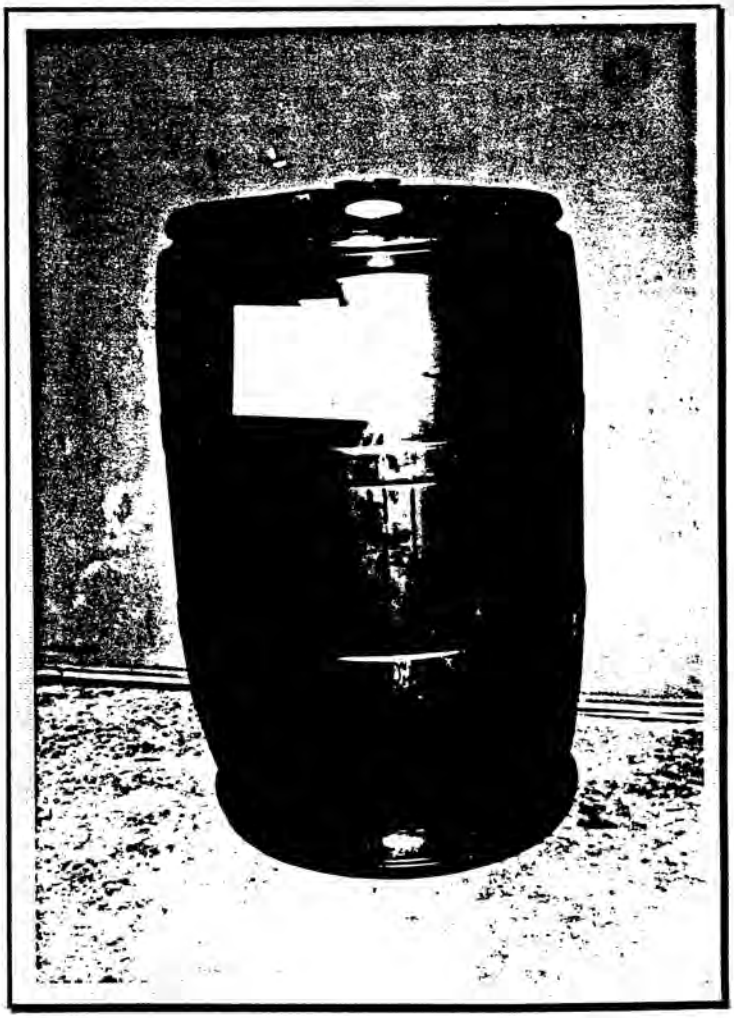


Photo # 16 - ESCR Test Failure

⇨• Drum Variable = 100% Recycle Resin
• Days to Failure = 25 Days

DISCLAIMER OF WARRANTIES

TEN-E PACKAGING SERVICES, INC. certifies that the previously described testing services have been performed in accordance with standard good laboratory practices. **ALL OTHER WARRANTIES, EXPRESSED OR IMPLIED, INCLUDING ANY WARRANTY THAT THE PACKAGING TESTED IS MERCHANTABLE, FIT FOR A PARTICULAR PURPOSE OR IN COMPLIANCE WITH ANY FEDERAL OR STATE REGULATIONS, ARE DISCLAIMED.**

Lewis G. Anderson
Packaging Engineer

APPENDIX D

TEST II

TEN-E REPORT

DATED MARCH 24, 1995

TENE
PACKAGING SERVICES INCORPORATED

PERFORMANCE EVALUATION
210 LITRE DRUM VARIABLES

- 30% Recycled / 70% Virgin Resin
- 100% Virgin Resin

REPORT #: 13465

PLASTIC DRUM INSTITUTE
1275 K Street N.W.
Suite 400
Washington, D.C. 20005

March 24, 1995

TABLE OF CONTENTS

OBJECTIVE

To conduct a performance evaluation on 210 litre tight head drums manufactured with varying amounts of recycled and virgin resin. Tight head drums manufactured with 30% Recycled / 70% Virgin and 100% Virgin Resin were subjected to the following performance tests:

① Drop Tests (2 Orientations)
- In accordance with the Department of Transportation's Title 49 CFR; Section 178.603
- Conditioning: -18°C (0° F)
- Packing Group: II
- Specific Gravity: 1.5
- Drop Height: 1.5 meters (59.06")

② 28-Day Stack Test
- In accordance with the Department of Transportation's Title 49 CFR; Section 178.606
- Conditioning: 40° C (104° F)
- Specific Gravity: 1.8

③ Leakproofness Test
- In accordance with the Department of Transportation's Title 49 CFR; Section 178.604
- Conditioning: Ambient
- Packing Group: II
- Test Pressure: 20 kPa (2.9 psi)
- Duration: 5 Minutes

④ Hydrostatic Pressure Test
- In accordance with the Department of Transportation's Title 49 CFR; Section 178.605
- Conditioning: Ambient
- Packing Group: II
- Test Pressure: 100 kPa (14.5 psi)
- Duration: 30 Minutes

⑤ Repetitive Shock Vibration
- In accordance with the Department of Transportation's Title 49 CFR; Section 178.608
- Duration: 60 Minutes

⑥ Dynamic Compression Testing
- In accordance with ASTM D642 - Standard Test Method for Determining Compressive Resistance of Shipping Containers
- Conditioning: Ambient

⑦ Environmental Stress Crack Resistance Testing
- In accordance with ASTM D 5571 - Standard Test Method for Environmental Stress Crack Resistance (ESCR) of Plastic Tighthead Drums Not Exceeding 60 Gallons (227 L) In Rated Capacity - Procedure A
- Test Pressure: 13.8 +/- 1.4 kPa (2.0 +/- 0.1 psi.)
- Duration: 28 Days
- Conditioning: 50^0 C (122^0 F)

SUMMARY OF TEST RESULTS

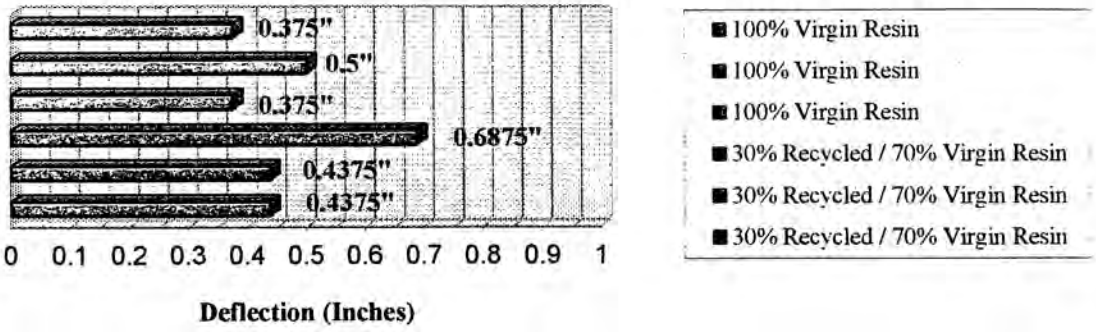
DROP TEST:		30% Recycled / 70% Virgin Resin:	
Packing Group:	II	*Diagonal Top Chime Orientation:*	3 / 3 Passed
Specific Gravity:	1.5	*Flat on Side Orientation:*	3 / 3 Passed
Conditioning:	-18^0 C (0^0 F)	100% Virgin Resin:	
Drop Height:	1.5 Meters (59.06")	*Diagonal Top Chime Orientation:*	3 / 3 Passed
		Flat on Side Orientation:	3 / 3 Passed

LEAKPROOFNESS TESTS:		30% Recycled / 70% Virgin Resin:	3 / 3 Passed
Test Pressure:	20 kPa (2.9 psi)	100% Virgin Resin:	3 / 3 Passed

HYDROSTATIC TESTS:		30% Recycled / 70% Virgin Resin:	3 / 3 Passed
Test Pressure:	100 kPa (14.5 psi)	100% Virgin Resin:	3 / 3 Passed

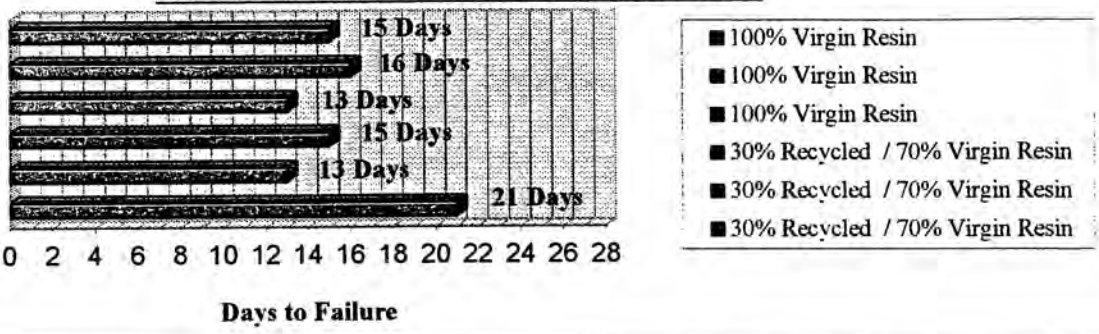
28-DAY STACK TESTS:		30% Recycled / 70% Virgin Resin:	3 / 3 Passed
Specific Gravity:	1.8	100% Virgin Resin:	3 / 3 Passed

TOTAL DEFLECTION FOLLOWING 28-DAY STACK TEST



VIBRATION TESTS:		30% Recycled / 70% Virgin Resin:	3 / 3 Passed
Test Frequency:	4.3 Hz	100% Virgin Resin:	3 / 3 Passed

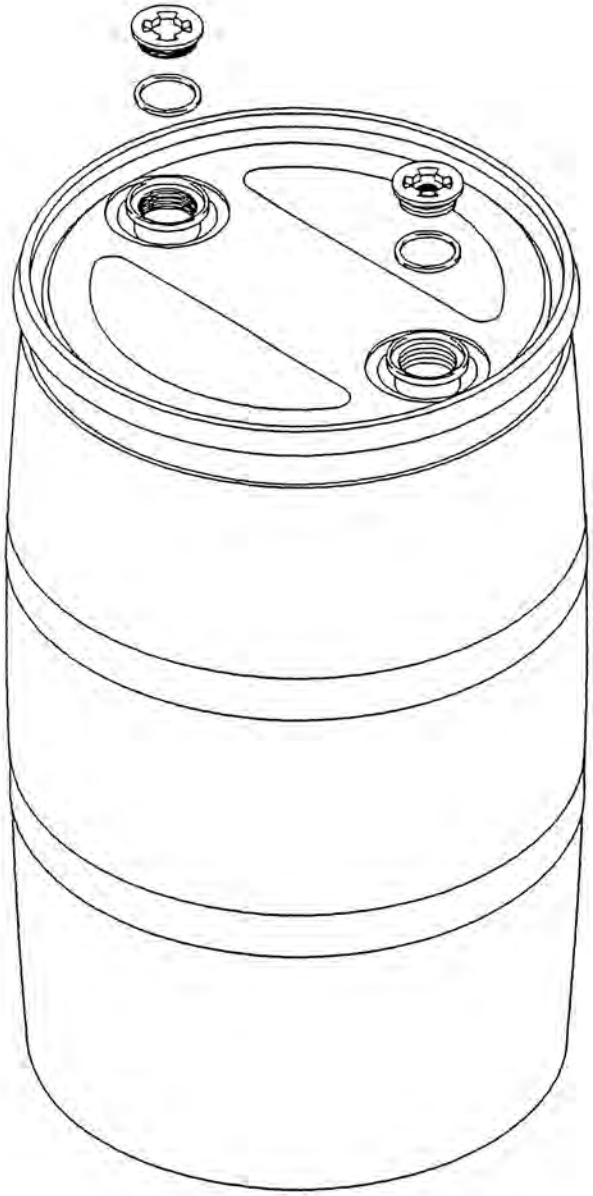
ESCR TESTS - DAYS TO FAILURE



DYNAMIC COMPRESSION TESTS:		30% Recycled / 70% Virgin Resin:	5820 Lbs(.76")
Average Max. Load & (Deflection)		100% Virgin Resin:	6170 Lbs(.64")

TEST SAMPLE DESCRIPTION

210 LITRE TIGHT HEAD DRUM WITH RIEKE® PPA-53-B-5 & PPA-55 PLUGS



PPA-55 PLUG (N.P.S.)

DESCRIPTION:
PPA-55 2" Poly-Visegrip Plug

MATERIAL:
Polyethylene Blend

PIGMENT:
Natural

THREAD STYLE:
NPS

CLOSURE THREAD DIMENSIONS:
- **T:** 2.308" - 2.343"

CLOSURE DIMENSIONS:
- **Height:** 0.785" - 0.835"
- **Diameter:** 2.925" - 2.865"

GASKET:
Santoprene O-Ring Gasket

SUPPLIER / MANUFACTURER:
Rieke® Corporation - Auburn, Indiana

PPA-53-B-5 PLUG (Buttress)

DESCRIPTION:
PPA-53-B-5 2" Buttress Plug

MATERIAL:
Polyethylene Blend

PIGMENT:
Natural

THREAD STYLE:
Buttress

CLOSURE THREAD DIMENSIONS:
- **T:** 2.510" - 2.5540"

CLOSURE DIMENSIONS:
- **Height:** 1.035" (Max)
- **Diameter:** 2.915" (Max)

GASKET:
Santoprene O-Ring Gasket

SUPPLIER / MANUFACTURER:
Rieke® Corporation - Auburn, Indiana

TEST SAMPLE DESCRIPTION

210 LITRE TIGHT HEAD DRUM WITH RIEKE® PPA-53-B-5 & PPA-55 PLUGS

DRUM

DESCRIPTION:
210 Litre Tight Head Plastic Drum

MATERIAL:
High Density Polyethylene

PIGMENT:
Black

CAPACITY (Nominal):
- **Litres:** 210 Litres
- **U.S. Gallons:** 55 U.S. Gallons
- **Imperial Gallons:** 46 Imperial Gallons

OVERALL DRUM DIMENSIONS:
- **Height:** 920 mm (36.25")
- **Top Diameter:** 598 mm (23.50")

TARE WEIGHT (Nominal):
10.9 - 12 Kgs (24 - 26 Lbs)

MARKINGS:
Made In Canada — Date Stamp
Hunter Logo — "2" HDPE Recycling Symbol
2 - 10 HLMI — T.C. - 34 - 210 Litres
D.O.T. - 34 - 55 - Hunter
U.N. - 1H1 - Y1.9 - 150 - 90 - CDN

SUPPLIER / MANUFACTURER & ID:
Hunter Drums Limited

TEST PROCEDURES AND RESULTS

Testing was completed by TEN-E Packaging Services, Inc. on March 10, 1995

EQUIPMENT

Appendix I contains a complete list of equipment used to conduct this program

QUALITY CONTROL AUDIT

PLUGS:

- Description
- Material
- Pigment
- Tare Weight
- Plug Dimensions
- Finish Dimensions
- Markings
- Liner Identification
- Liner Gram Weight
- Liner Thickness

DRUM:

- Description
- Material
- Pigment
- Tare Weight
- Drum Capacity
- Drum Dimensions
- Finish Dimensions
- Melt Index
- Density
- Wall Thickness Profile
- Markings

PACKAGE WEIGHT INFORMATION:

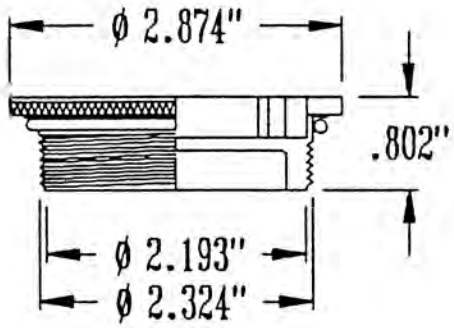
- Overall Package Tare Weight
- Package Test Weight

QUALITY CONTROL AUDIT RESULTS:

TEST PROCEDURES AND RESULTS - QUALITY CONTROL AUDIT

PPA-55 PLUG (N.P.S. Threads)	
• **Description:**	PPA-55 2" Poly-Visegrip Plug
• **Material:**	Polyethylene
• **Pigment:**	Natural
• **Tare Weight:**	24.370 Grams
• **Markings:**	Reike® PPA-55-1

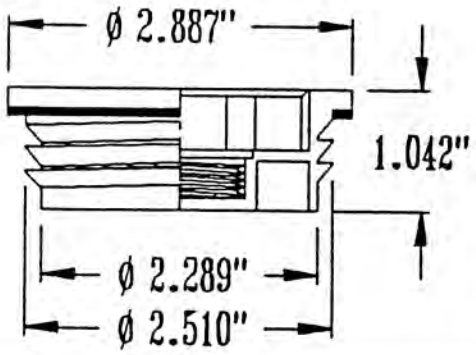
Plug Dimensions



GASKET	
• **Description/Material:**	Santoprene O-Ring Gasket
• **Tare Weight:**	3.278 Grams
• **Thickness:**	0.184"

PPA-53-B-5 PLUG (Buttress Threads)	
• **Description:**	PPA-53-B-5 2" Buttress Plug
• **Material:**	Polyethylene
• **Pigment:**	Natural
• **Tare Weight:**	32.028 Grams
• **Markings:**	Reike® PPA-55-1

Plug Dimensions

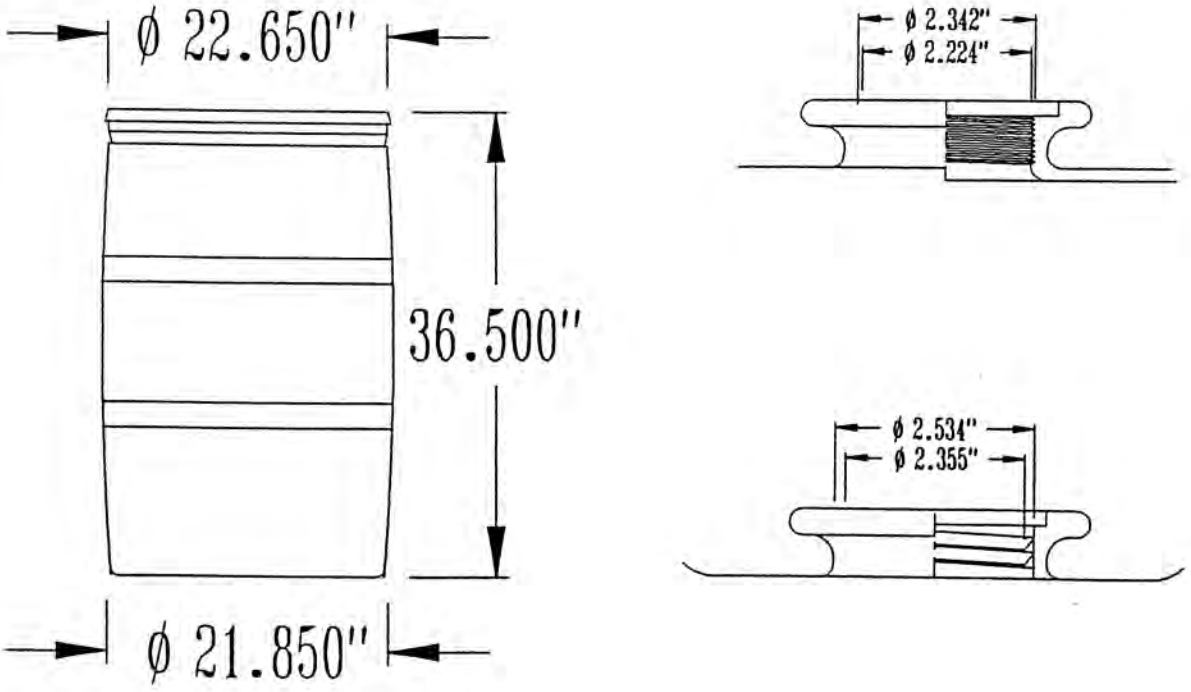


GASKET	
• **Description/Material:**	Santoprene O-Ring Gasket
• **Tare Weight:**	3.284 Grams
• **Thickness:**	0.184"

TEST PROCEDURES AND RESULTS - QUALITY CONTROL AUDIT

DRUM (30% Recycled Resin / 70% Virgin Resin)		
• Description:	210 Litre Tight Head Drum	
• Material:	High Density Polyethylene (30% Recycled / 70% Virgin Resin)	
• Pigment:	Black	
• Openings: (Number & Size)	(1) 2" Buttress (1) 2" NPS	
• Tare Weight:	11.0 Kg (24.2 Lbs)	
• Overflow Capacity:	226.8 Litres (226.8 Kg)	60.0 Gallons (500 Lbs)
• 98% Overflow Capacity:	222.3 Litres (222.3 Kg)	58.8 Gallons (490 Lbs)
• Melt Index (I^{21}):	3.68 g/10 minutes	
• Density:	0.952 g/cc	
• Wall Thickness Profile:	Refer to Next Page	
• Markings:	HUNTER MADE IN CANADA 210 LITRES "2" SPI HDPE Recycling Symbol un 1H1/Y1.9/150/94/CAN/HDL/2-186-2.2mm	Date Code: 12/15/94 55 HUNTER 5-10 HLMI

Drum Dimensions



PACKAGE WEIGHT INFORMATION		
• Overall Package Tare Weight:	11.1 Kg	(24.5 Lbs)
• Package Test Weight:	233.4 Kg	(514.5 Lbs)

TENE
PACKAGING SERVICES INCORPORATED

210 LITRE TIGHT HEAD PLASTIC DRUM (30% Recycled / 70% Virgin Resin)

WALL THICKNESS PROFILE (inches) — **REPORT #: 13465**

TOP HEAD

MSMT	DRUM 1	MSMT	DRUM 1
1	0.201	8	0.199
2	0.183	9	0.191
3	0.191	10	0.171
4	0.189	11	0.171
5	0.191	12	0.182
6	0.196	13	0.190
7	0.187	14	0.185

SIDEWALL

MSMT	DRUM 1	MSMT	DRUM 1
15	0.202	18	0.162
16	0.180	19	0.162
17	0.174	20	0.163

SIDEWALL

MSMT	DRUM 1	MSMT	DRUM 1
21	0.251	24	0.164
22	0.177	25	0.169
23	0.160	26	0.232

SIDEWALL

MSMT	DRUM 1	MSMT	DRUM 1
27	0.184	30	0.156
28	0.172	31	0.160
29	0.167	32	0.149

SIDEWALL

MSMT	DRUM 1	MSMT	DRUM 1
33	0.266	36	0.153
34	0.177	37	0.175
35	0.163	38	0.249

BOTTOM HEAD

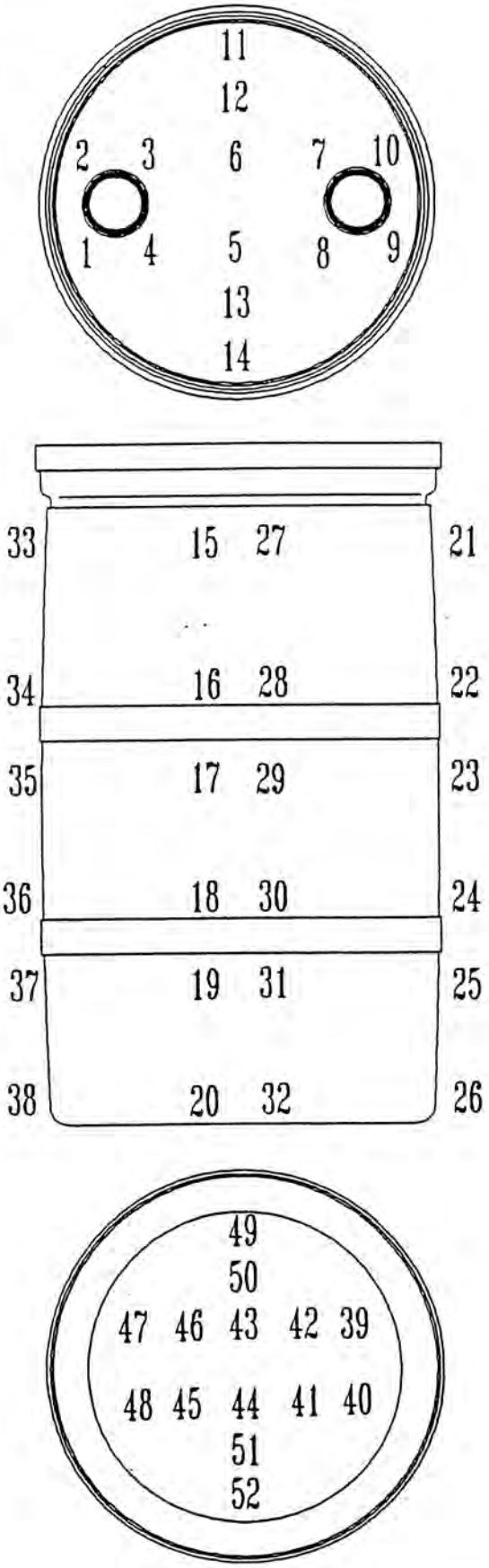
MSMT	DRUM 1	MSMT	DRUM 1
39	0.253	46	0.205
40	0.241	47	0.229
41	0.212	48	0.242
42	0.224	49	0.197
43	0.209	50	0.206
44	0.205	51	0.204
45	0.226	52	0.179

TOP RADIUS (Under Handling Ring)

MSMT	DRUM 1	MSMT	DRUM 1
53	0.265	57	0.265
54	0.226	58	0.212
55	0.236	59	0.260
56	0.230	60	0.204

BOTTOM RADIUS

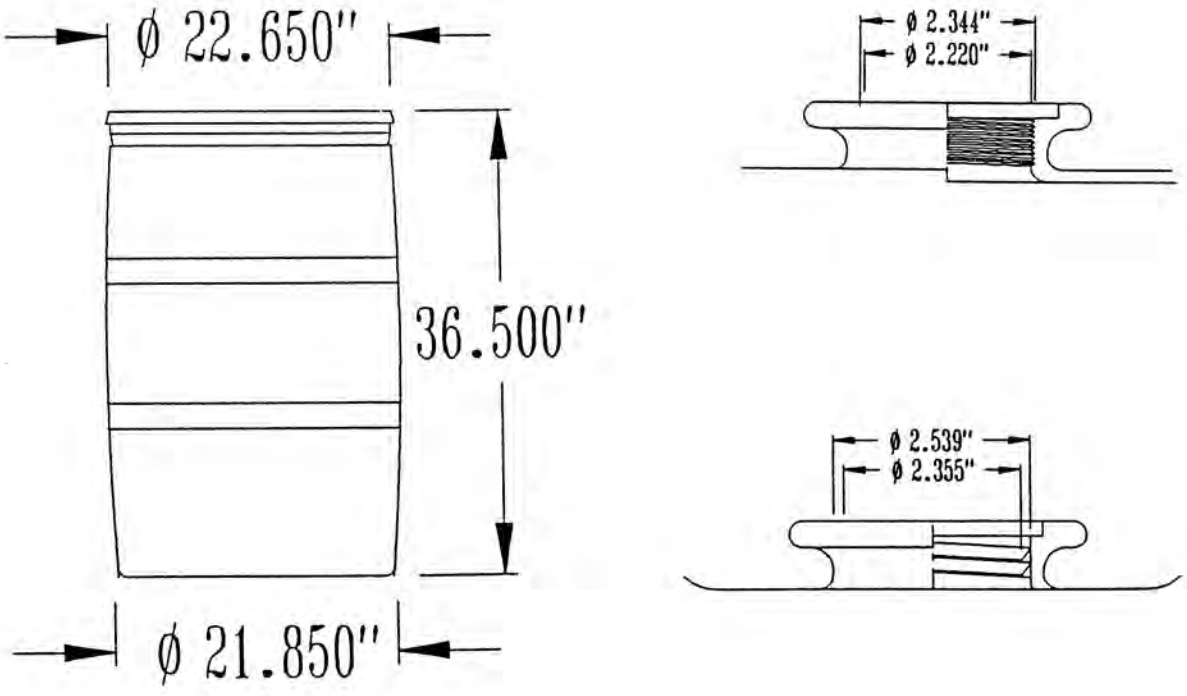
MSMT	DRUM 1	MSMT	DRUM 1
61	0.256	65	0.253
62	0.174	66	0.177
63	0.148	67	0.165
64	0.174	68	0.186



TEST PROCEDURES AND RESULTS - QUALITY CONTROL AUDIT

DRUM (100% Virgin Resin)		
• Description:	210 Litre Tight Head Drum	
• Material:	High Density Polyethylene (100% Virgin Resin)	
• Pigment:	Black	
• Openings: (Number & Size)	(1) 2" Buttress (1) 2" NPS	
• Tare Weight:	11.2 Kg (24.6 Lbs)	
• Overflow Capacity:	226.8 Litres (226.8 Kg)	60.0 Gallons (500 Lbs)
• 98% Overflow Capacity:	222.3 Litres (222.3 Kg)	58.8 Gallons (490 Lbs)
• Melt Index (I^{21}):	3.76 g/10 minutes	
• Density:	0.952 g/cc	
• Wall Thickness Profile:	Refer to Next Page	
• Markings:	HUNTER MADE IN CANADA 210 LITRES "2" SPI HDPE Recycling Symbol un 1H1/Y1.9/150/94/CAN/HDL/2-186-2.2mm	Date Code: 12/15/94 55 HUNTER 5-10 HLMI

Drum Dimensions



PACKAGE WEIGHT INFORMATION		
• Overall Package Tare Weight:	11.1 Kg	(24.5 Lbs)
• Package Test Weight:	233.4 Kg	(514.5 Lbs)

TENE PACKAGING SERVICES INCORPORATED

210 LITRE TIGHT HEAD PLASTIC DRUM (100% Virgin Resin)

WALL THICKNESS PROFILE (inches)

REPORT: 13465

TOP HEAD

MSMT	DRUM 1	MSMT	DRUM 1
1	0.183	8	0.198
2	0.187	9	0.184
3	0.181	10	0.171
4	0.183	11	0.153
5	0.185	12	0.158
6	0.187	13	0.174
7	0.184	14	0.165

SIDEWALL

MSMT	DRUM 1	MSMT	DRUM 1
15	0.220	18	0.172
16	0.185	19	0.167
17	0.184	20	0.164

SIDEWALL

MSMT	DRUM 1	MSMT	DRUM 1
21	0.242	24	0.166
22	0.186	25	0.179
23	0.168	26	0.245

SIDEWALL

MSMT	DRUM 1	MSMT	DRUM 1
27	0.191	30	0.165
28	0.174	31	0.167
29	0.173	32	0.159

SIDEWALL

MSMT	DRUM 1	MSMT	DRUM 1
33	0.242	36	0.160
34	0.178	37	0.170
35	0.161	38	0.243

BOTTOM HEAD

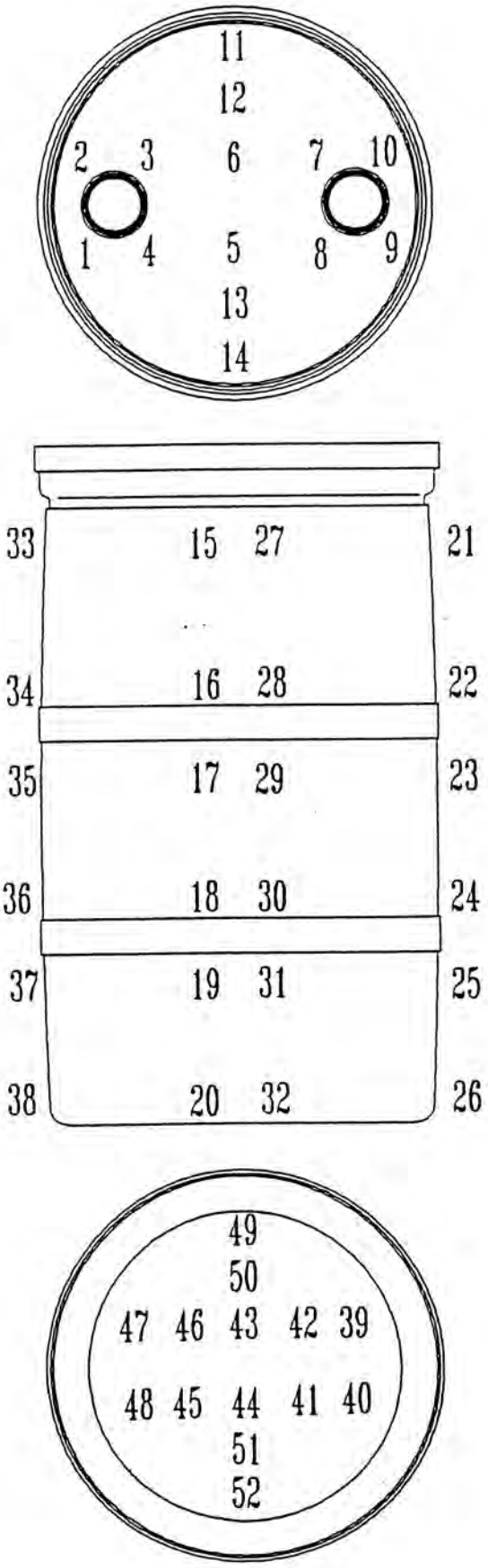
MSMT	DRUM 1	MSMT	DRUM 1
39	0.272	46	0.221
40	0.249	47	0.234
41	0.216	48	0.255
42	0.217	49	0.206
43	0.201	50	0.194
44	0.202	51	0.211
45	0.226	52	0.200

TOP RADIUS (Under Handling Ring)

MSMT	DRUM 1	MSMT	DRUM 1
53	0.265	57	0.267
54	0.182	58	0.171
55	0.251	59	0.233
56	0.185	60	0.175

BOTTOM RADIUS

MSMT	DRUM 1	MSMT	DRUM 1
61	0.266	65	0.288
62	0.182	66	0.184
63	0.159	67	0.172
64	0.187	68	0.183



TEST PROCEDURES & RESULTS - DROP TESTS (30% Recycled Resin / 70% Virgin Resin)

SAMPLE SIZE:
- 3 Samples / Orientation (6 Total)

FILL CAPACITY:
- 98% of Maximum Capacity
- 222.3 Kg (490.0 Lbs) Net / Drum

PLUG APPLICATION:
- PPA-55 (N.P.S.): 20 Ft-Lbs
- PPA-53-B-5 (Buttress): 30 Ft-Lbs

CONDITIONING:
- -18°C (0°F)

DROP TEST EQUIPMENT:
- Quick Release Hook Mechanism

FILLING SUBSTANCE:
- Methanol/Glycol/Water Solution

PACKAGE TEST WEIGHT:
- 233.4 Kg (514.5 Lbs)

DROP HEIGHT CALCULATION:
- Packing Group II Materials
  S.G. Not Exceeding 1.5

DROP HEIGHT:
- 1.5 meters (59.1")

DROP ORIENTATIONS:
- Diagonal Top Chime
- Flat on Side

TEST STANDARD:
- Department of Transportation's Title 49 CFR; Section 178.603
- ASTM D5276 - Standard Test Method for Drop Test of Loaded Containers by Free Fall

DROP TEST RESULTS (30% Recycled Resin / 70% Virgin Resin)

Drop Orientation: Diagonal Top Chime (Fitting in the 6 O'Clock Position)

Sample #	Results	Comments / Observations
1	Pass	There was no leakage of the drums contents present upon completion of the diagonal top chime drop tests. **Note each closure side was impacted at least one time*
2	Pass	
3	Pass	

Drop Orientation: Flat on Side (Fitting in the 6 O'Clock Position)

Sample #	Results	Comments / Observations
4	Pass	There was no leakage of the drums contents present upon completion of the flat on side drop tests. **Note each closure side was impacted at least one time*
5	Pass	
6	Pass	

Refer to Photos 1-2 in Appendix II for Drop Test Set-Ups

CRITERIA FOR PASSING THE TEST

A package is considered to successfully pass the drop tests if for each sample tested:
- For receptacles containing liquid, each receptacle does not leak when equilibrium has been reached between internal and external pressures
- For a drum, any discharge from a closure is slight & ceases immediately after impact with no further leakage

TEST PROCEDURES & RESULTS - DROP TESTS (100% Virgin Resin)

SAMPLE SIZE:
- 3 Samples / Orientation (6 Total)

FILLING SUBSTANCE:
- Methanol/Glycol/Water Solution

FILL CAPACITY:
- 98% of Maximum Capacity
- 222.3 Kg (490.0 Lbs) Net / Drum

PACKAGE TEST WEIGHT:
- 233.4 Kg (514.5 Lbs)

PLUG APPLICATION:
- PPA-55 (N.P.S.): 20 Ft-Lbs
- PPA-53-B-5 (Buttress): 30 Ft-Lbs

DROP HEIGHT CALCULATION:
- Packing Group II Materials S.G. Not Exceeding 1.5

CONDITIONING:
- -18°C (0°F)

DROP HEIGHT:
- 1.5 meters (59.1")

DROP TEST EQUIPMENT:
- Quick Release Hook Mechanism

DROP ORIENTATIONS:
- Diagonal Top Chime
- Flat on Side

TEST STANDARD:
- Department of Transportation's Title 49 CFR; Section 178.603
- ASTM D5276 - Standard Test Method for Drop Test of Loaded Containers by Free Fall

DROP TEST RESULTS (100% Virgin Resin)

Drop Orientation: Diagonal Top Chime (Fitting in the 6 O'Clock Position)

Sample #	Results	Comments / Observations
1	Pass	Upon completion of the diagonal top chime drop tests there was no leakage of the drums contents. **Note each closure side was impacted at least one time*
2	Pass	
3	Pass	

Drop Orientation: Flat on Side (Fitting in the 6 O'Clock Position)

Sample #	Results	Comments / Observations
4	Pass	Upon completion of the flat on side drop tests there was no leakage of the drums contents. **Note each closure side was impacted at least one time*
5	Pass	
6	Pass	

Refer to Photos 1-2 in Appendix II for Drop Test Set-Ups

CRITERIA FOR PASSING THE TEST

A package is considered to successfully pass the drop tests if for each sample tested:
- For receptacles containing liquid, each receptacle does not leak when equilibrium has been reached between internal and external pressures
- For a drum, any discharge from a closure is slight & ceases immediately after impact with no further leakage

TEST PROCEDURES & RESULTS - LEAKPROOFNESS TESTS (30% Recycled / 70% Virgin)

SAMPLE SIZE:
- 3 Samples

CONDITIONING:
- Ambient

TEST PRESSURE:
- 20 kPa (2.9 psi)

PRESSURE TEST EQUIPMENT:
- Regulated Air Source
- Marshalltown G23606 0-60 psi 3½" Gauge
- Marshalltown G16688 0-100 psi 5" Gauge
  ¼ of 1% Accuracy

PLUG APPLICATION:
- PPA-55 (N.P.S.): 20 Ft-Lbs
- PPA-53-B-5 (Buttress): 30 Ft-Lbs

AREA OF PRESSURIZATION:
- Through the top head of the drum

TEST DURATION:
- 5 Minutes

TEST STANDARD:
- Department of Transportation's Title 49 CFR; Section 178.604 and Appendix B to Part 178 - Alternate Test Methods - Solution Over Seams

LEAKPROOFNESS TEST RESULTS (30% Recycled / 70% Virgin)

Sample #	Results	Comments / Observations
7	Pass	No visible leakage of air occurred during the 5 minute leakproofness test.
8	Pass	
9	Pass	

Refer to Photo 3 in Appendix II for Leakproofness Test Set-Up

CRITERIA FOR PASSING THE TEST

- A packaging passes the test if there is no leakage of air from the packaging

TEST PROCEDURES & RESULTS - LEAKPROOFNESS TESTS (100% Virgin Resin)

SAMPLE SIZE:
- 3 Samples

CONDITIONING:
- Ambient

TEST PRESSURE:
- 20 kPa (2.9 psi)

PRESSURE TEST EQUIPMENT:
- Regulated Air Source
- Marshalltown G23606 0-60 psi 3½" Gauge
- Marshalltown G16688 0-100 psi 5" Gauge
  ¼ of 1% Accuracy

PLUG APPLICATION:
- PPA-55 (N.P.S.): 20 Ft-Lbs
- PPA-53-B-5 (Buttress): 30 Ft-Lbs

AREA OF PRESSURIZATION:
- Through the top head of the drum

TEST DURATION:
- 5 Minutes

TEST STANDARD:
- Department of Transportation's Title 49 CFR; Section 178.604 and Appendix B to Part 178 - Alternate Test Methods - Solution Over Seams

LEAKPROOFNESS TEST RESULTS (100% Virgin Resin)

Sample #	Results	Comments / Observations
7	Pass	No visible leakage of air occurred during the 5 minute leakproofness test.
8	Pass	
9	Pass	

Refer to Photo 3 in Appendix II for Leakproofness Test Set-Up

CRITERIA FOR PASSING THE TEST

- A packaging passes the test if there is no leakage of air from the packaging

TEST PROCEDURES & RESULTS - HYDROSTATIC TESTS (30% Recycled / 70% Virgin)

SAMPLE SIZE:
- 3 Samples

FILL CAPACITY:
- Maximum Capacity

CONDITIONING:
- Ambient

TEST PRESSURE:
- 100 kPa (14.5 psi)

PRESSURE TEST EQUIPMENT:
- Regulated Water Source
- Marshalltown G23606 0-60 psi 3½" Gauge
- Marshalltown G16688 0-100 psi 5" Gauge
  ¼ of 1% Accuracy

FILLING SUBSTANCE:
- Water

PLUG APPLICATION:
- PPA-55 (N.P.S.): 20 Ft-Lbs
- PPA-53-B-5 (Buttress): 30 Ft-Lbs

AREA OF PRESSURIZATION:
- Through the top head of the drum

TEST DURATION:
- 30 Minutes

TEST STANDARD:
- Department of Transportation's Title 49 CFR; Section 178.605
- ICAO Technical Instructions on the Safe Transport of Dangerous Goods By Air Part 3; 1.1.16

HYDROSTATIC PRESSURE TEST RESULTS (30% Recycled / 70% Virgin)

Sample #	Results	Comments / Observations
10	Pass	No visible leakage of water occurred during the 30 minute Hydrostatic Pressure test.
11	Pass	
12	Pass	

Refer to Photo 4 in Appendix II for Internal Pressure Test Set-Up

CRITERIA FOR PASSING THE TEST

- Packaging for which retention of liquid is a basic function must be able to withstand, without leakage, the prescribed test pressure.

TEST PROCEDURES & RESULTS - HYDROSTATIC TESTS (100% Virgin Resin)

SAMPLE SIZE:
- 3 Samples

FILL CAPACITY:
- Maximum Capacity

CONDITIONING:
- Ambient

TEST PRESSURE:
- 100 kPa (14.5 psi)

PRESSURE TEST EQUIPMENT:
- Regulated Water Source
- Marshalltown G23606 0-60 psi 3½" Gauge
- Marshalltown G16688 0-100 psi 5" Gauge ¼ of 1% Accuracy

FILLING SUBSTANCE:
- Water

PLUG APPLICATION:
- PPA-55 (N.P.S.): 20 Ft-Lbs
- PPA-53-B-5 (Buttress): 30 Ft-Lbs

AREA OF PRESSURIZATION:
- Through the top head of the drum

TEST DURATION:
- 30 Minutes

TEST STANDARD:
- Department of Transportation's Title 49 CFR; Section 178.605
- ICAO Technical Instructions on the Safe Transport of Dangerous Goods By Air Part 3; 1.1.16

HYDROSTATIC PRESSURE TEST RESULTS (100% Virgin Resin)		
Sample #	**Results**	**Comments / Observations**
10	Pass	No visible leakage of water occurred during the 30 minute Hydrostatic Pressure test.
11	Pass	
12	Pass	

Refer to Photo 4 in Appendix II for Internal Pressure Test Set-Up

CRITERIA FOR PASSING THE TEST
- Packaging for which retention of liquid is a basic function must be able to withstand, without leakage, the prescribed test pressure.

TEST PROCEDURES & RESULTS - STACK TESTS (30% Recycled Resin / 70% Virgin Resin)

SAMPLE SIZE:
- 3 Samples

FILL CAPACITY:
- 98% of Maximum Capacity
- 222.3 Kg (490.0 Lbs) Net / Drum

PLUG APPLICATION:
- PPA-55 (N.P.S.): 20 Ft-Lbs
- PPA-53-B-5 (Buttress): 30 Ft-Lbs

CONDITIONING:
- 40°C (104°F)

STACK TEST DURATION:
- 28 Days

FILLING SUBSTANCE:
- Water

PACKAGE TEST WEIGHT:
- 233.4 Kg (514.5 Lbs)

STACK TEST EQUIPMENT:
- Dead Load Steel Weights
  Guided Load Fixtures

TEST LOAD APPLIED:
- 952.6 Kg (2100 Lbs)

TEST STANDARD:
- Department of Transportation's Title 49 CFR; Section 178.606
- ASTM D4577-Standard Test Method for Compression Resistance of a Container Under Constant Load

STACK TEST LOAD CALCULATION	
• Number of packages in a 3 meter (118") high stack (-1):	2.3 Drums
• Package gross weight (Based on 1.8 Specific Gravity):	411.2 Kg (906.5 Lbs)

2.3 Drums x 411.2 Kg (906.5 Lbs) = 945.8 Kg (2085 Lbs) (Min. Required Load Per Drum)

STACK TEST RESULTS (30% Recycled Resin / 70% Virgin Resin)			
Sample #	**Maximum Deflection After 28 Days**	**One Hour Stack Stability Test Performed**	**Results**
13	7/16"	Yes	Pass
14	7/16"	Yes	Pass
15	11/16"	Yes	Pass

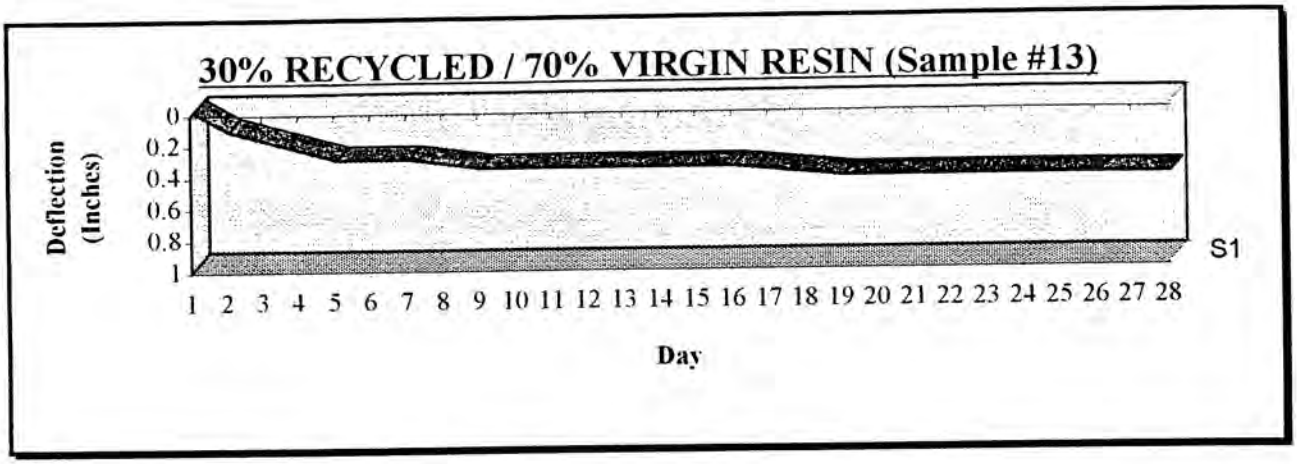
Refer to Photo 5 in Appendix II for Stack Test Set-Ups

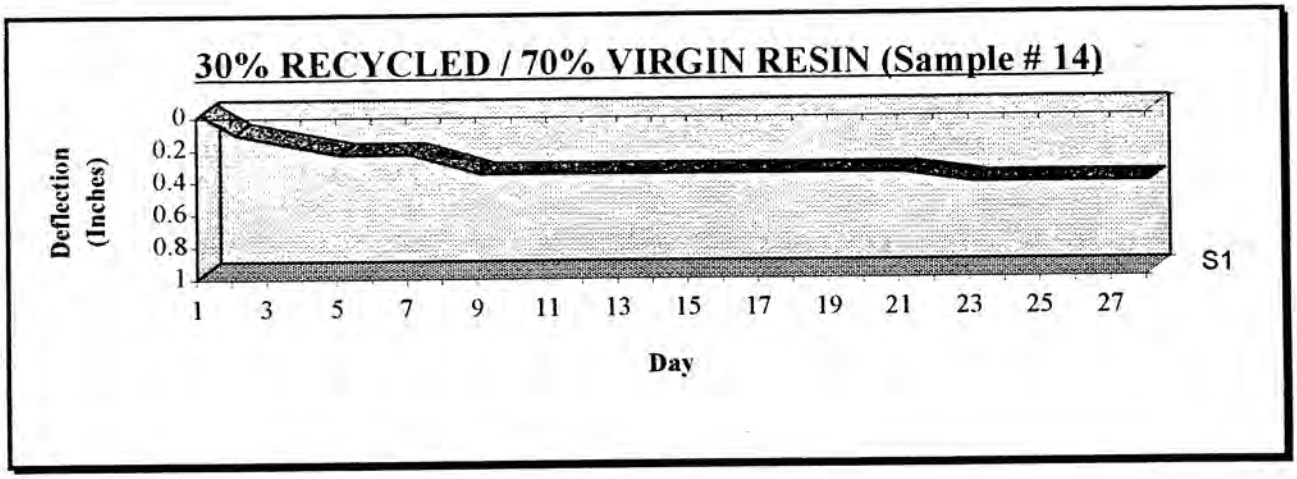
CRITERIA FOR PASSING THE TEST

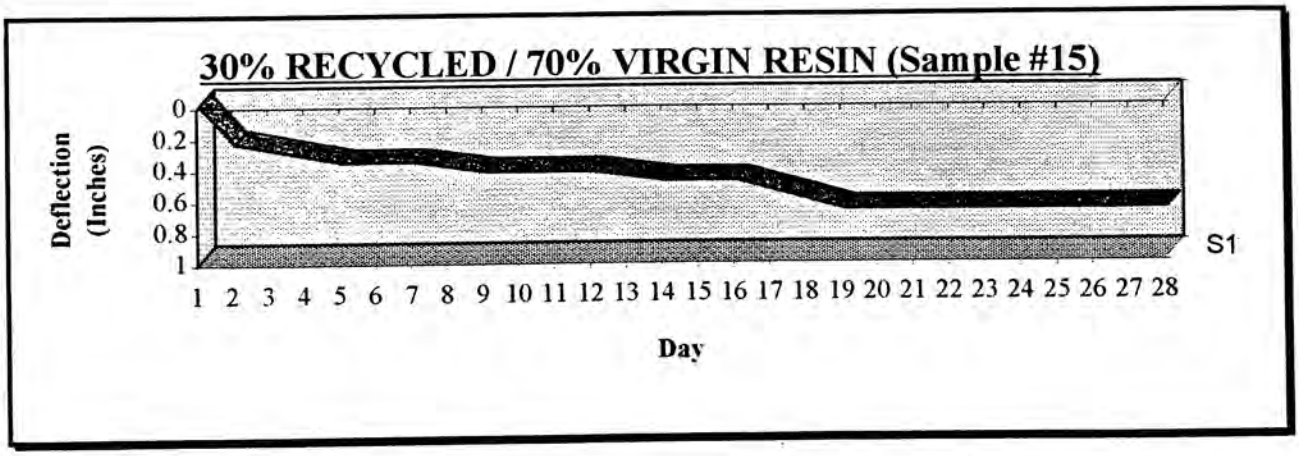
- There is no deterioration that could adversely affect transport safety or any distortion liable to reduce its strength or cause instability in stacks of packages
- No test sample may leak.

TEST PROCEDURES & RESULTS - STACK TESTS (30% Recycled Resin / 70% Virgin Resin)

DRUM DEFLECTION DURING THE 28-DAY STACK TEST







TEST PROCEDURES & RESULTS - STACK TESTS (100% Virgin Resin)

SAMPLE SIZE:
- 3 Samples

FILL CAPACITY:
- 98% of Maximum Capacity
- 222.3 Kg (490.0 Lbs) Net / Drum

PLUG APPLICATION:
- PPA-55 (N.P.S.): 20 Ft-Lbs
- PPA-53-B-5 (Buttress): 30 Ft-Lbs

CONDITIONING:
- 40°C (104°F)

STACK TEST DURATION:
- 28 Days

FILLING SUBSTANCE:
- Water

PACKAGE TEST WEIGHT:
- 233.4 Kg (514.5 Lbs)

STACK TEST EQUIPMENT:
- Dead Load Steel Weights
  Guided Load Fixtures

TEST LOAD APPLIED:
- 952.6 Kg (2100 Lbs)

TEST STANDARD:
- Department of Transportation's Title 49 CFR; Section 178.606
- ASTM D4577-Standard Test Method for Compression Resistance of a Container Under Constant Load

STACK TEST LOAD CALCULATION	
• Number of packages in a 3 meter (118") high stack (-1):	2.3 Drums
• Package gross weight (Based on 1.8 Specific Gravity):	411.2 Kg (906.5 Lbs)

2.3 Drums x 411.2 Kg (906.5 Lbs) = 945.8 Kg (2085 Lbs) (Min. Required Load Per Drum)

STACK TEST RESULTS (100% Virgin Resin)

Sample #	Maximum Deflection After 28 Days	One Hour Stack Stability Test Performed	Results
13	3/8"	Yes	Pass
14	1/2"	Yes	Pass
15	3/8"	Yes	Pass

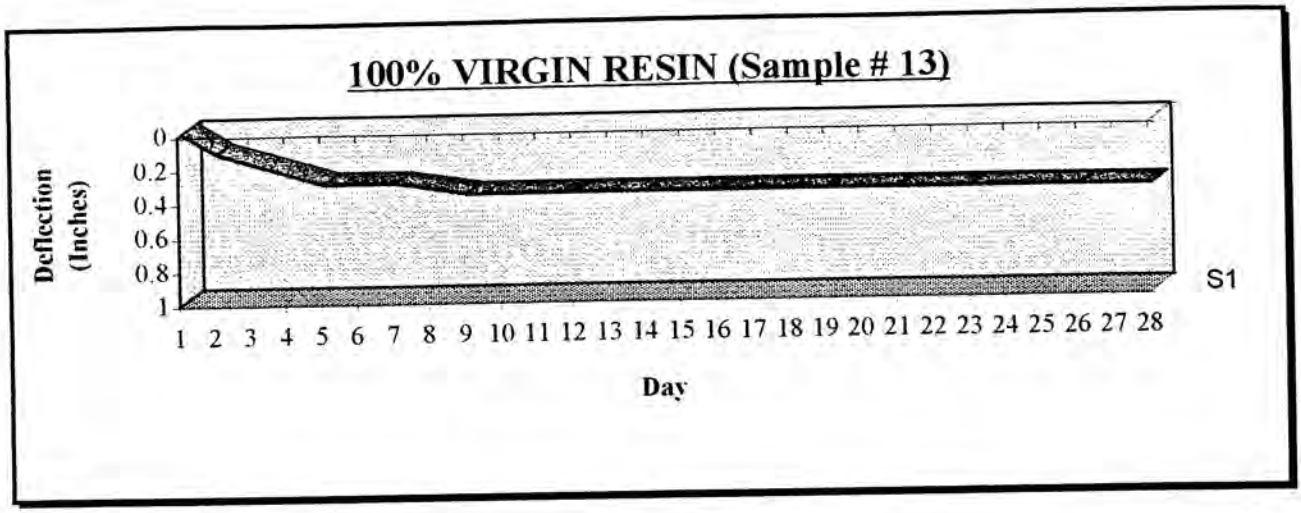
Refer to Photo 5 in Appendix II for Stack Test Set-Ups

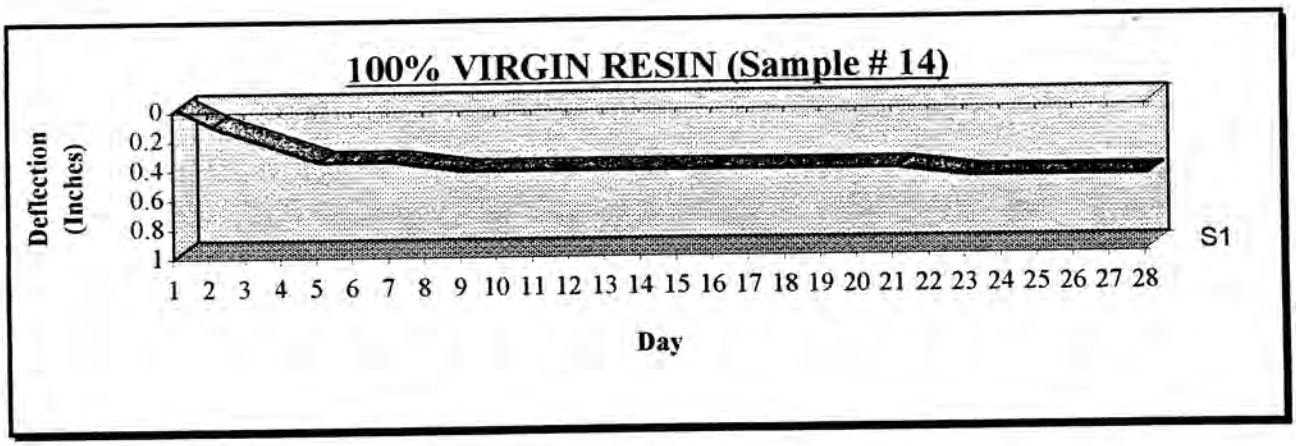
CRITERIA FOR PASSING THE TEST

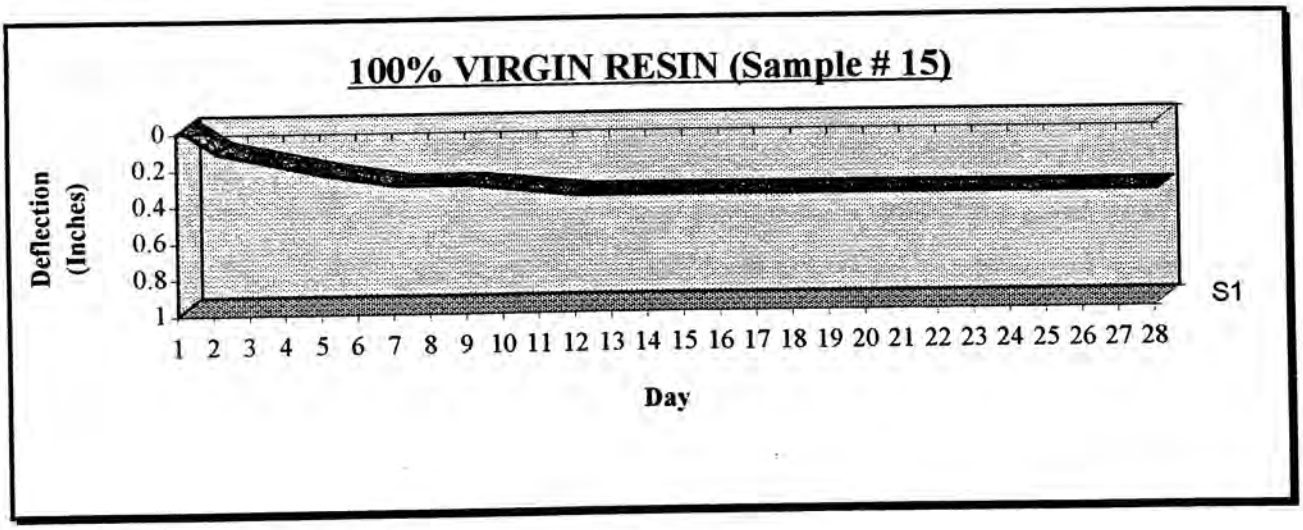
- There is no deterioration that could adversely affect transport safety or any distortion liable to reduce its strength or cause instability in stacks of packages
- No test sample may leak.

TEST PROCEDURES & RESULTS - STACK TESTS (100% Virgin Resin)

DRUM DEFLECTION DURING THE 28-DAY STACK TEST







TEST PROCEDURES & RESULTS - VIBRATION TESTS (30% Recycled / 70% Virgin Resin)

SAMPLE SIZE:
- 3 Samples

FILLING SUBSTANCE:
- Water

FILL CAPACITY:
- 98% of Maximum Capacity
- 222.3 Kg (490.0 Lbs) Net / Drum

PACKAGE TEST WEIGHT:
- 233.4 Kg (514.5 Lbs)

PLUG APPLICATION:
- PPA-55 (N.P.S.): 20 Ft-Lbs
- PPA-53-B-5 (Buttress): 30 Ft-Lbs

CONDITIONING:
- Ambient

TABLE DISPLACEMENT:
- 1"

FREQUENCY:
- 4.3 Hz

TEST DURATION:
- 60 Minutes

VIBRATION TEST EQUIPMENT:
- LAB Model 6000 Transportation Simulator

TEST STANDARD:
- Department of Transportation's Title 49 CFR; Section 178.608
- ASTM D999 - Standard Test Method for Vibration Testing of Shipping Containers - Method A1

REPETITIVE SHOCK VIBRATION TEST RESULTS (30% Recycled / 70% Virgin Resin)

Sample #	Results	Comments / Observations
16	Pass	Upon completion on the 60 minute vibration test there was no leakage or visible deterioration.
17	Pass	
18	Pass	

Refer to Photo 6 in Appendix II for Repetitive Shock Vibration Test Set-Up

CRITERIA FOR PASSING THE TEST

- A packaging passes the vibration test if there is no rupture or leakage from any of the packages.
- No test sample should show any deterioration which could adversely affect transportation safety or any distortion liable to reduce packaging strength

TEST PROCEDURES & RESULTS - VIBRATION TESTS (100% Virgin Resin)

SAMPLE SIZE:
- 3 Samples

FILL CAPACITY:
- 98% of Maximum Capacity
- 222.3 Kg (490.0 Lbs) Net / Drum

PLUG APPLICATION:
- PPA-55 (N.P.S.): 20 Ft-Lbs
- PPA-53-B-5 (Buttress): 30 Ft-Lbs

TABLE DISPLACEMENT:
- 1"

TEST DURATION:
- 60 Minutes

FILLING SUBSTANCE:
- Water

PACKAGE TEST WEIGHT:
- 233.4 Kg (514.5 Lbs)

CONDITIONING:
- Ambient

FREQUENCY:
- 4.3 Hz

VIBRATION TEST EQUIPMENT:
- LAB Model 6000 Transportation Simulator

TEST STANDARD:
- Department of Transportation's Title 49 CFR; Section 178.608
- ASTM D999 - Standard Test Method for Vibration Testing of Shipping Containers - Method A1

REPETITIVE SHOCK VIBRATION TEST RESULTS (100% Virgin Resin)

Sample #	Results	Comments / Observations
16	Pass	Upon completion on the 60 minute vibration test there was no leakage or visible deterioration.
17	Pass	
18	Pass	

Refer to Photo 6 in Appendix II for Repetitive Shock Vibration Test Set-Up

CRITERIA FOR PASSING THE TEST

- A packaging passes the vibration test if there is no rupture or leakage from any of the packages.
- No test sample should show any deterioration which could adversely affect transportation safety or any distortion liable to reduce packaging strength

TEST PROCEDURES & RESULTS -DYNAMIC COMPRESSION

SAMPLE SIZE:
- 3 Samples

CONDITIONING:
- Ambient

PLATEN RATE OF TRAVEL:
- ½" Per Minute

SAMPLE PREPARATION:
- Samples Tested Empty Without Closures

PRELOAD APPLIED:
- 50 Lbs.

TEST EQUIPMENT:
- LAB 5250 Compression System

TEST STANDARD:
- ASTM D642 - Standard Test Method for Determining Compressive Resistance of Shipping Containers

DYNAMIC COMPRESSION TEST RESULTS

Drum Variable: 30% Recycled / 70% Virgin

Sample #	Maximum Load	Deflection at Max. Load	Area of Failure
19	5,250 Lbs	0.71"	Failure occurred when the sidewall of the drum collapsed inward approximately one half of the way up the side.
20	6,370 Lbs	0.82"	
21	5,840 Lbs	0.75"	
Average:	***5,820 Lbs***	***0.76"***	

DYNAMIC COMPRESSION TEST RESULTS

Drum Variable: 100% Virgin Resin

Sample #	Maximum Load	Deflection at Max. Load	Area of Failure
19	6,010 Lbs	0.55"	Failure occurred when the sidewall of the drum collapsed inward approximately one half of the way up the side.
20	6,000 Lbs	0.57"	
21	6,500 Lbs	0.80"	
Average:	***6,170 Lbs***	***0.64"***	

Refer to Photo 7 in Appendix II for Dynamic Compression Test Set-Up

TEST PROCEDURES & RESULTS - ESCR TESTS (30% Recycled / 70% Virgin Resin)

SAMPLE SIZE:
- 3 Samples / Variable

FILL CAPACITY:
- 10% Rated Capacity
- 21.0 Kg (46.3 Lbs) Net / Drum

PLUG APPLICATION:
- PPA-55 (N.P.S.): 20 Ft-Lbs
- PPA-53-B-5 (Buttress): 30 Ft-Lbs

ESCR TEST DURATION:
- 28 Days

INTERNAL SURFACES RECOATED:
- Every 48 Hours

SAMPLES VISUALLY INSPECTED:
- Every 24 Hours

FILLING SUBSTANCE:
- 10% Nonyl Phenoxypoly Ethanol Solution

DRUM TEST WEIGHT:
- 32.1 Kg (70.8 Lbs) (10% of Rated Capacity)

CONDITIONING:
- 50°C (122°F)

INTERNAL TEST PRESSURE:
- 2.0 psi +/- 0.1 psi (13.8 kPa +/- 1.4 kPa)

ESCR TEST EQUIPMENT:
- Marshalltown G23606 3½" Gauge
- Vollrath Walk-In Environmental Chamber

TEST STANDARD:
- ASTM D 5571 - Standard Test Method for Environmental Stress Crack Resistance (ESCR) of Plastic Tighthead Drums Not Exceeding 60 Gallons (227 L) In Rated Capacity

ESCR TEST RESULTS (30% Recycled / 70% Virgin Resin)

Sample #	Days to Failure	Comments \ Observations
22	21 Days	A 2" stress crack occurred on the partline along the top radius of the drum. The failure was located on the partline / NPS side. **Refer to photo # 9 in appendix II*
23	13 Days	Two stress cracks (¾" & 1¼") occurred along the bottom radius of the drum on opposite sides near the partline. **Refer to photo # 10 in appendix II*
24	15 Days	A 2½" stress crack occurred along the bottom radius of drum (near the partline) on the buttress opening side **Refer to photo # 11 in appendix II*

Refer to Photo 8 in Appendix II for Environmental Stress Crack Resistance Test Set-Up

CRITERIA FOR PASSING THE TEST

- If there are no stress cracks evident (which would be indicated by bubbling or leaking of the regent through the points of failure) following the predetermined test duration.

TEST PROCEDURES & RESULTS - ESCR TESTS (100% Virgin Resin)

SAMPLE SIZE:
- 3 Samples / Variable

FILL CAPACITY:
- 10% Rated Capacity
- 21.0 Kg (46.3 Lbs) Net / Drum

PLUG APPLICATION:
- PPA-55 (N.P.S.): 20 Ft-Lbs
- PPA-53-B-5 (Buttress): 30 Ft-Lbs

ESCR TEST DURATION:
- 28 Days

INTERNAL SURFACES RECOATED:
- Every 48 Hours

SAMPLES VISUALLY INSPECTED:
- Every 24 Hours

FILLING SUBSTANCE:
- 10% Nonyl Phenoxypoly Ethanol Solution

DRUM TEST WEIGHT:
- 32.1 Kg (70.8 Lbs) (10% of Rated Capacity)

CONDITIONING:
- 50°C (122°F)

INTERNAL TEST PRESSURE:
- 2.0 psi +/- 0.1 psi (13.8 kPa +/- 1.4 kPa)

ESCR TEST EQUIPMENT:
- Marshalltown G23606 3½" Gauge
- Vollrath Walk-In Environmental Chamber

TEST STANDARD:
- ASTM D 5571 - Standard Test Method for Environmental Stress Crack Resistance (ESCR) of Plastic Tighthead Drums Not Exceeding 60 Gallons (227 L) In Rated Capacity

ESCR TEST RESULTS (100% Virgin Resin)

Sample #	Days to Failure	Comments \ Observations
22	13 Days	A ¾" stress crack occurred along the bottom radius of drum (near the partline) on the NPS opening side **Refer to photo # 12 in appendix II*
23	16 Days	A 1" stress crack occurred along the bottom radius of drum (near the partline) on the NPS opening side **Refer to photo # 13 in appendix II*
24	15 Days	A 1" stress crack occurred on the partline along the top radius of the drum. The failure was located on the partline / NPS side. **Refer to photo # 14 in appendix II*

Refer to Photo 8 in Appendix II for Environmental Stress Crack Resistance Test Set-Up

CRITERIA FOR PASSING THE TEST

- If there are no stress cracks evident (which would be indicated by bubbling or leaking of the regent through the points of failure) following the predetermined test duration.

DISCLAIMER OF WARRANTIES

TEN-E PACKAGING SERVICES, INC. certifies that the previously described testing services have been performed in accordance with standard good laboratory practices. **ALL OTHER WARRANTIES, EXPRESSED OR IMPLIED, INCLUDING ANY WARRANTY THAT THE PACKAGING TESTED IS MERCHANTABLE, FIT FOR A PARTICULAR PURPOSE OR IN COMPLIANCE WITH ANY FEDERAL OR STATE REGULATIONS, ARE DISCLAIMED.** In no event shall TEN-E PACKAGING SERVICES, INC. Liability exceed the total amount paid by the PLASTIC DRUM INSTITUTE for services rendered.

In the event of future changes to the above referenced Test Procedures, It is the responsibility of the PLASTIC DRUM INSTITUTE to determine whether additional testing or updating of past testing is necessary to verify that the packaging we have tested remains in compliance with those standards.

Lewis G. Anderson
Packaging Engineer

APPENDIX I - EQUIPMENT LIST

EQUIPMENT LIST - TEN-E PACKAGING SERVICES, INC.

QUALITY CONTROL AUDIT EQUIPMENT

- Mitutoyo 570-116 Height Gauge
- Panametrics Model 22 DLHR Ultrasonic Thickness Tester
- Magna-Mike 8000 Thickness Tester
- Deltronic MPC-4 Optical Comparator
- Satorius F135S 150 Lb Scale
- Thurman 405 1,000 Lb Scale
- DIGI Model S-PL 5,000 Lb Scale
- Tinius Olsen Extrusion Plastometer
- Electronic Densitometer Model ED-120T
- Precisa 100A-300M Analytical Balance
- Kayeness Galaxy Model 7054 Melt Flow Indexer
- Elektro Physik Mikrotest GMIV Coating Thickness Gauge (0-4 mils)
- Mitutoyo 500-321 Vernier Calipers
- Mitutoyo 293-701 Digital Caliper
- TIF 7000 Digital Pyrometer

CONDITIONING CHAMBERS

- Vollrath Walk-In Heat Chamber (to 140° F) 12'-6" x 16'-4" x 12'-4"
  - Equipped with guided load fixtures and dead load weights for conducting stack tests at elevated temperatures
- Vollrath Walk-In Freezer (to -30° F) 12'-6" x 12'-6" x 8'-6"
- Vollrath Walk-In Heat Chamber (to 140° F) 12'-6" x 12'-6" x 8'-6"
- Vollrath Temperature / Humidity Chamber 5'-10" x 4'-10" x 7'-4"
- ColTech Services Co: Environmental Chamber (to 160° F) 10' x 12' x 8'-3"

EQUIPMENT LIST - TEN-E PACKAGING SERVICES, INC.

DROP / IMPACT TEST EQUIPMENT:

- L.A.B Accu-Drop 160 Tester
- Gaynes Incline Impact Tester
- L.A.B Swing Arm Drop Tester

COMPRESSION / TENSILE EQUIPMENT:

- Sintech 6 Tensile Compression System (10,000 Lb. Capacity)
- L.A.B 5250 Compression Machine (10,000 Lb. Capacity)

VIBRATION TEST EQUIPMENT:

- L.A.B PTV-48 Servo-Hydraulic Vibration System
- Gaynes Rotary Motion Vibration Table
- L.A.B. Model 6000 SVMC Transportation Simulator

PRESSURE TEST EQUIPMENT:

- Baxter D1360-300 Desiccator; Dry Seal
- Marshalltown G23606 3½" Gauge, 0-60 psi
- Marshalltown G16688 5" Gauge, 0-100 psi (¼ of 1% Accuracy)

EQUIPMENT LIST - TEN-E PACKAGING SERVICES, INC.

CLOSING DEVICES:

- AKO, Inc. TSD 1286 Torque Meter
- Harbil Can Closer
- Enercon Industries Induction Sealer
- Tri-Sure LPM-0044 Crimping Tool
- Tri-Sure U6 #1078 Crimping Tool
- Rieke G10-18100 Crimping Tool
- Rieke W-168 PT (20/9) Pre-Set Torque Wrench
- Snap-On Tools TE 12-FUA Torque Wrench
- Atlanta Grotnes 8½" - 12 Lug Manual Closing Tool
- Atlanta Grotnes 11¼" - 16 Lug Manual Closing Tool
- Atlanta Grotnes Semi-Automatic Closing Tool

APPENDIX II - PHOTOGRAPHS

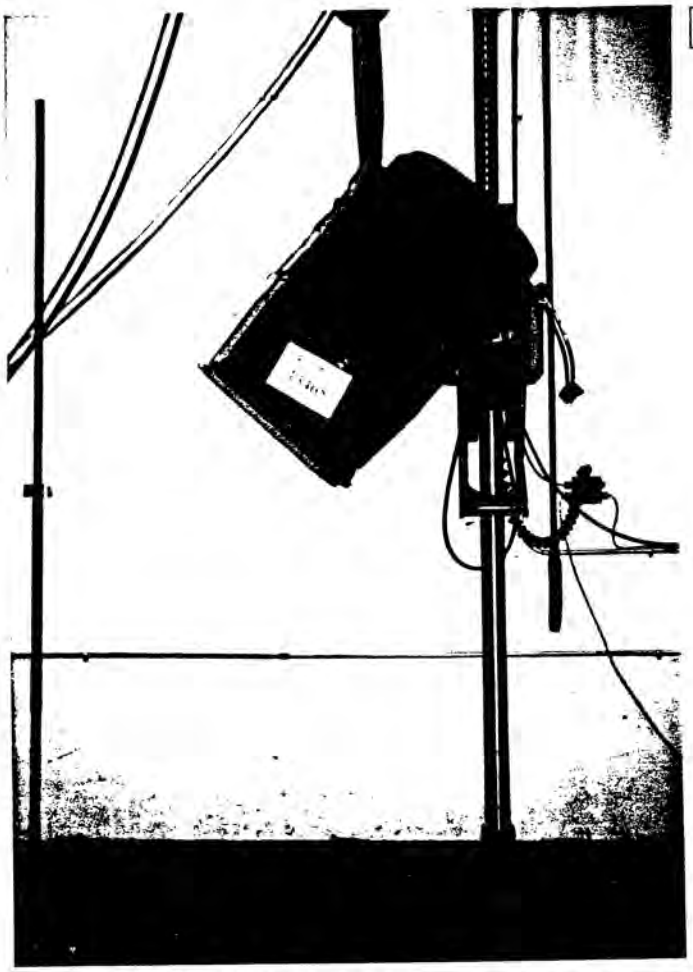


Photo #1 -Diagonal Top Chime Drop Test Set-up

⇦ • Drop Height = 1.5 meters (59.1")
• Conditioning = -18°C (0°F)

Photo # 2 - Flat on Side Drop Test Set-up

⇨ • Drop Height = 1.5 meters (59.1")
• Conditioning = -18°C (0°F)

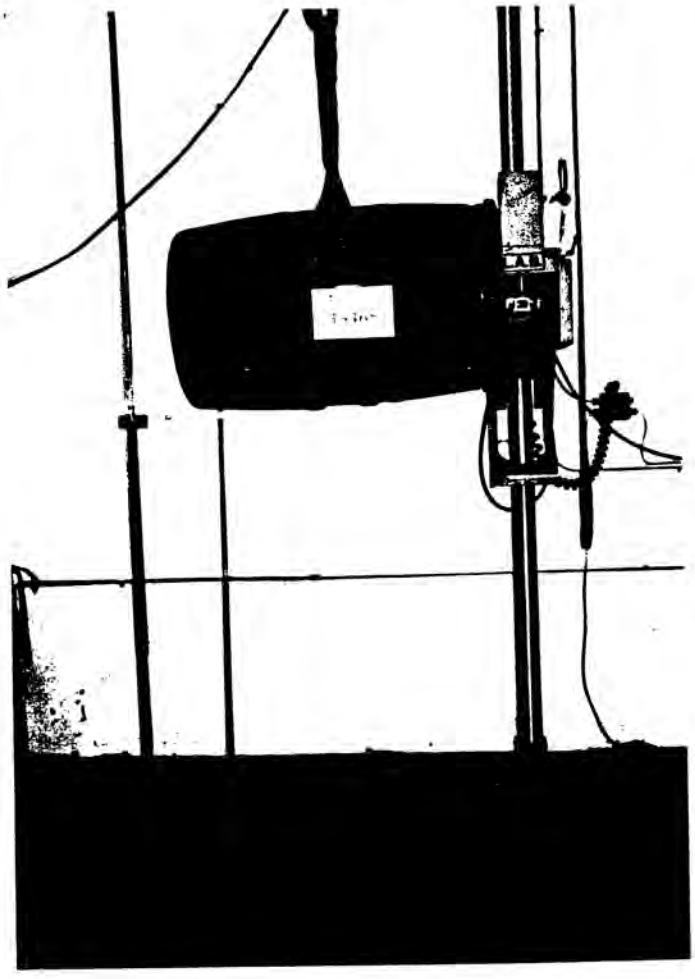


Photo # 3 - Leakproofness Test Set-up

⇦ • Test Pressure = 20 kPa (2.9 psi)
• Test Duration = 5 Minutes

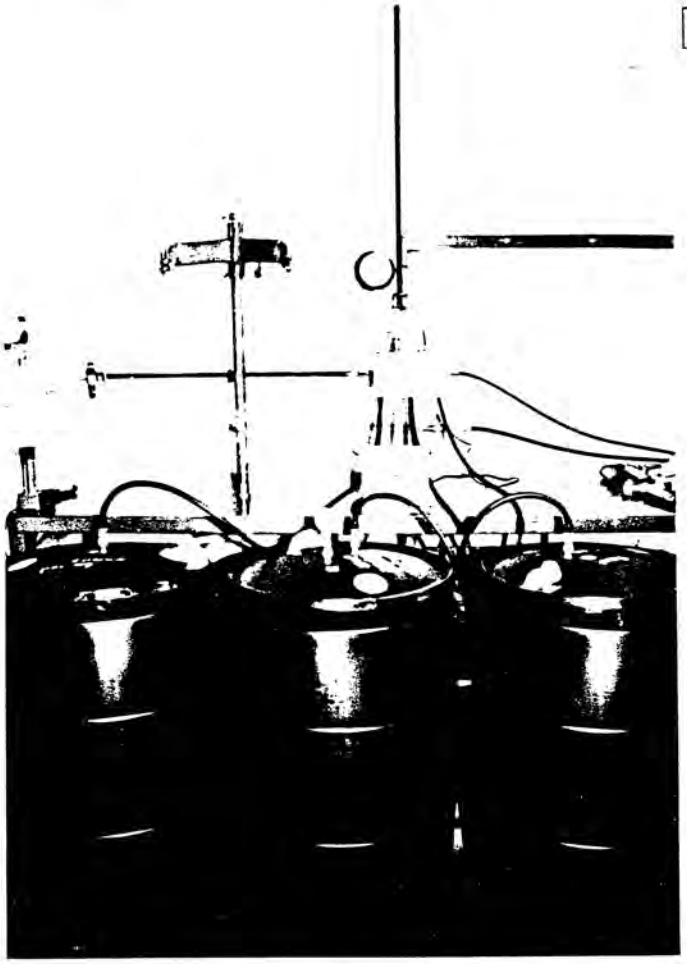


Photo # 4 - Hydrostatic Test Set-up

⇨ • Test Pressure = 100 kPa (14.5 psi)
• Test Duration = 30 Minutes

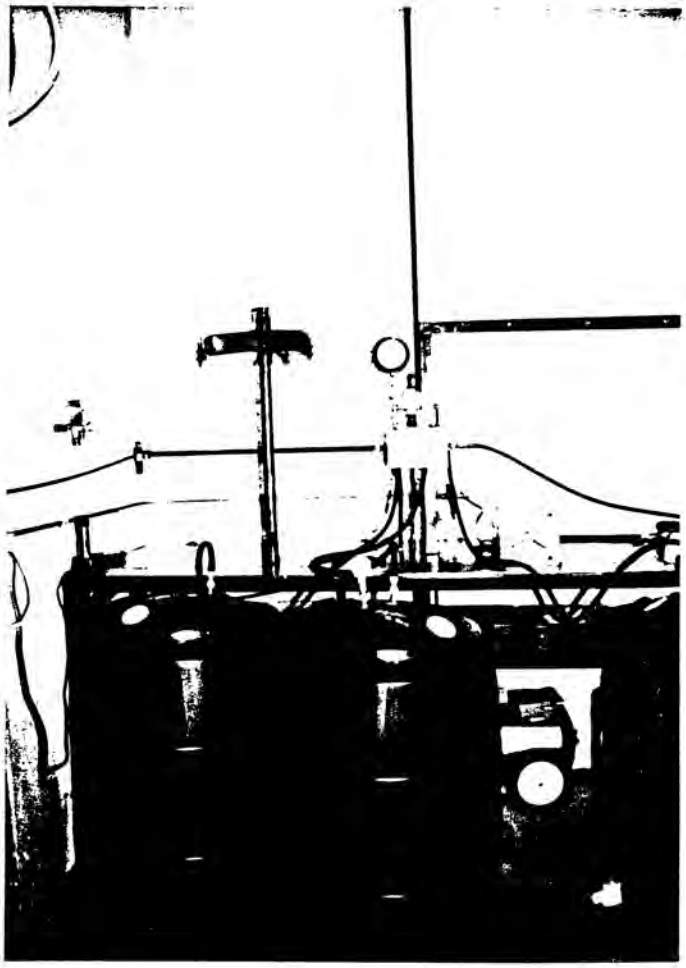


Photo # 11 - ESCR Test Failure

- Drum Variable = 30% Recycled Resin
- Days to Failure = 15 Days

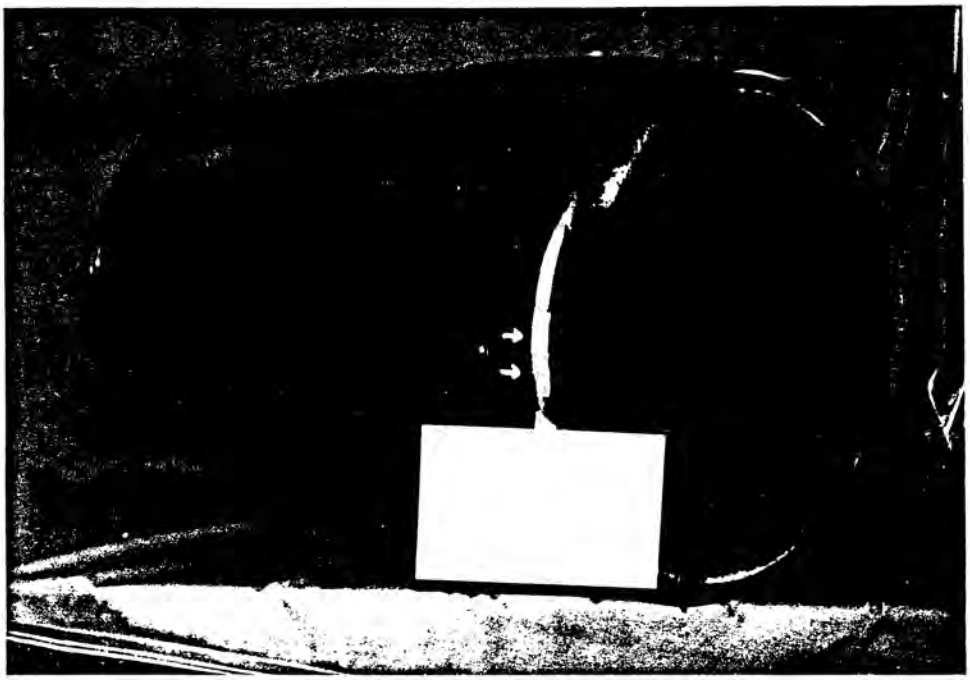


Photo # 12 - ESCR Test Failure

- Drum Variable = 100% Virgin Resin
- Days to Failure = 13 Days

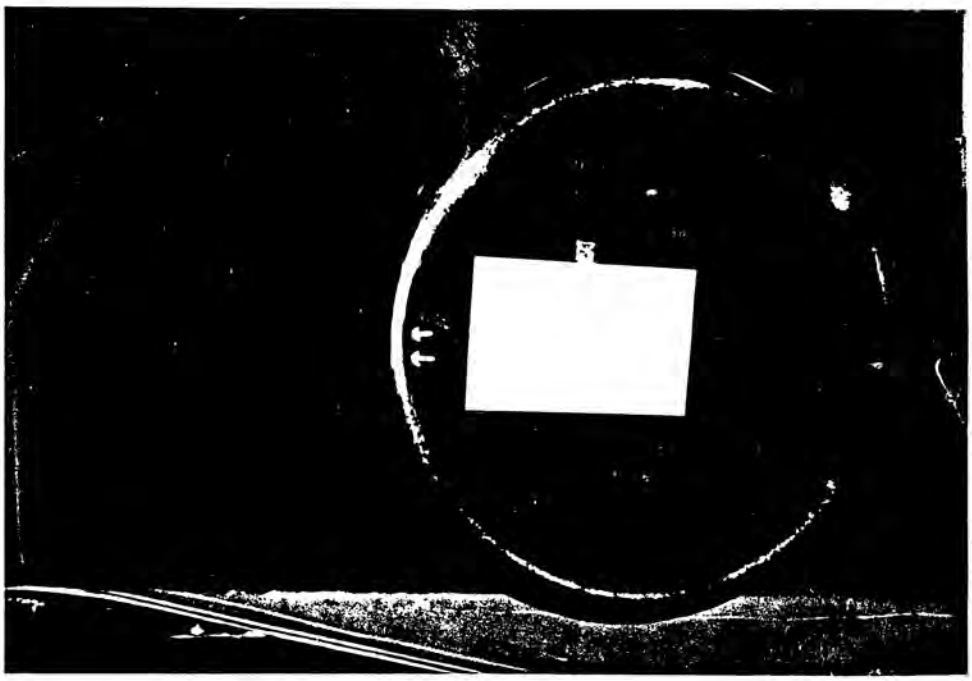


Photo # 13 - ESCR Test Failure

- Drum Variable = 100% Virgin Resin
- Days to Failure = 16 Days

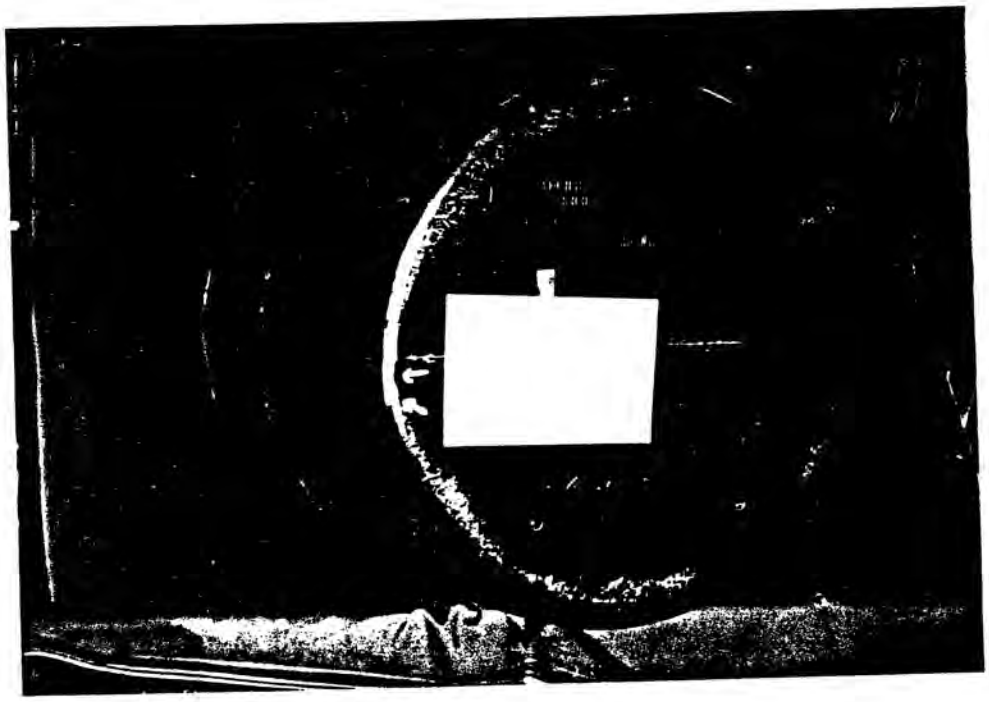


Photo # 14 - ESCR Test Failure

- Drum Variable = 100% Virgin Resin
- Days to Failure = 15 Days

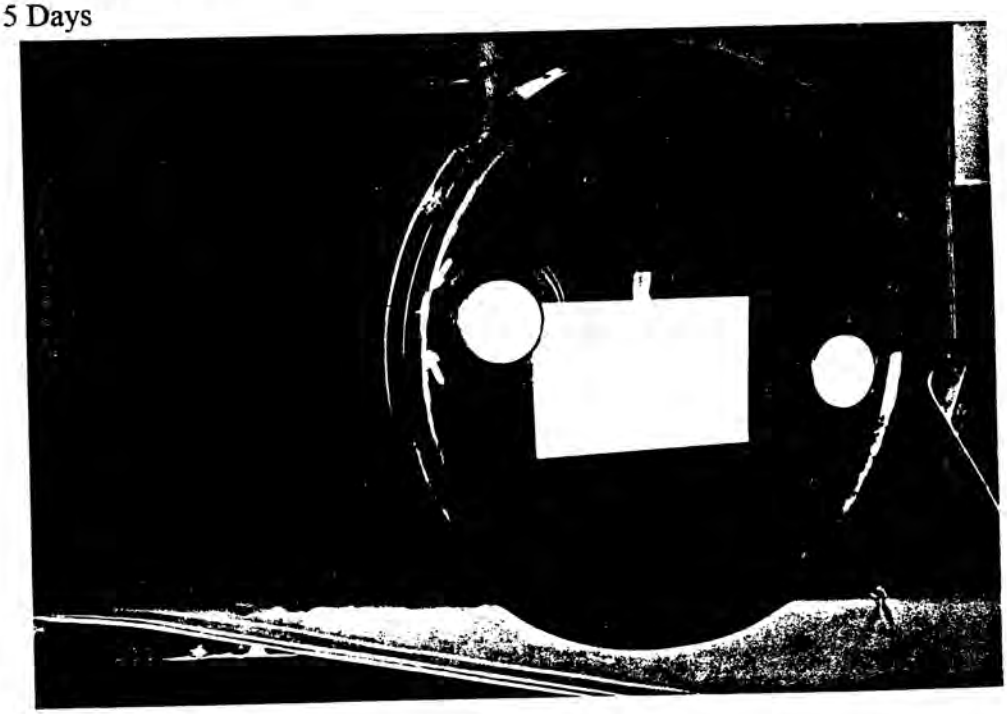


Photo # 5 - 28 Day Stack Test Set-up

- Load Applied = 2100 Lbs / Drum
- Conditioning = 40° C (104°F)

Photo # 6 - Vibration Test Test Set-up

- Test Frequency =4.3 Hz
- Test Duration = 60 Minutes

Photo # 7 - Dynamic Compression Test Set-Up

- Rate Load Applied = ½ Inch / Minute

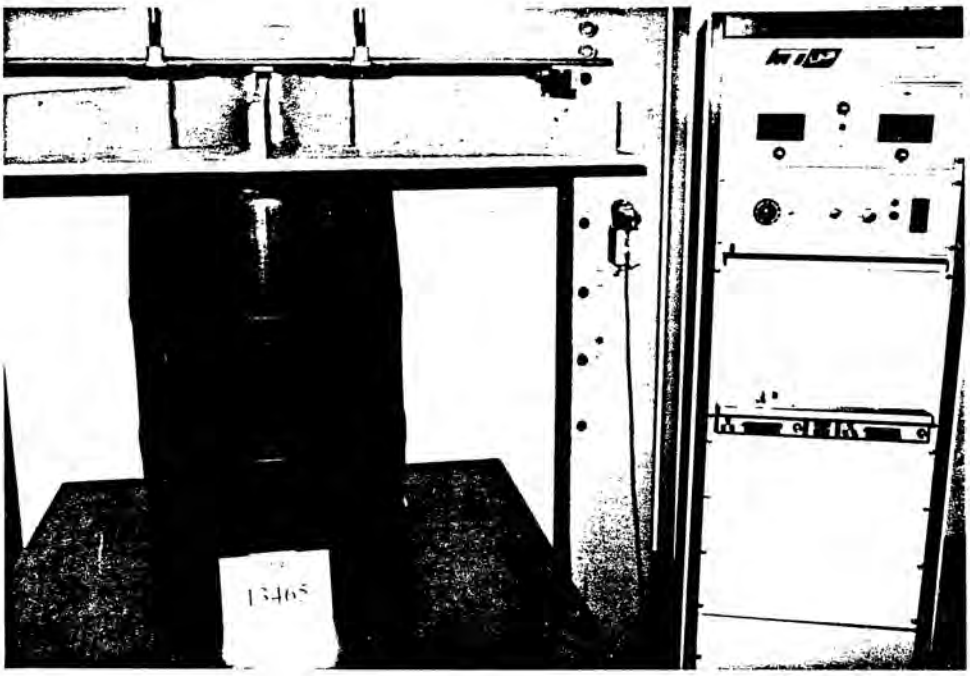


Photo # 8 - ESCR Test Set-up

- Conditioning = 50° C (122°F)
- Test Duration = 28 Days

Photo # 9 - ESCR Test Failure

- Drum Variable = 30% Recycled Resin
- Days to Failure = 21 Days

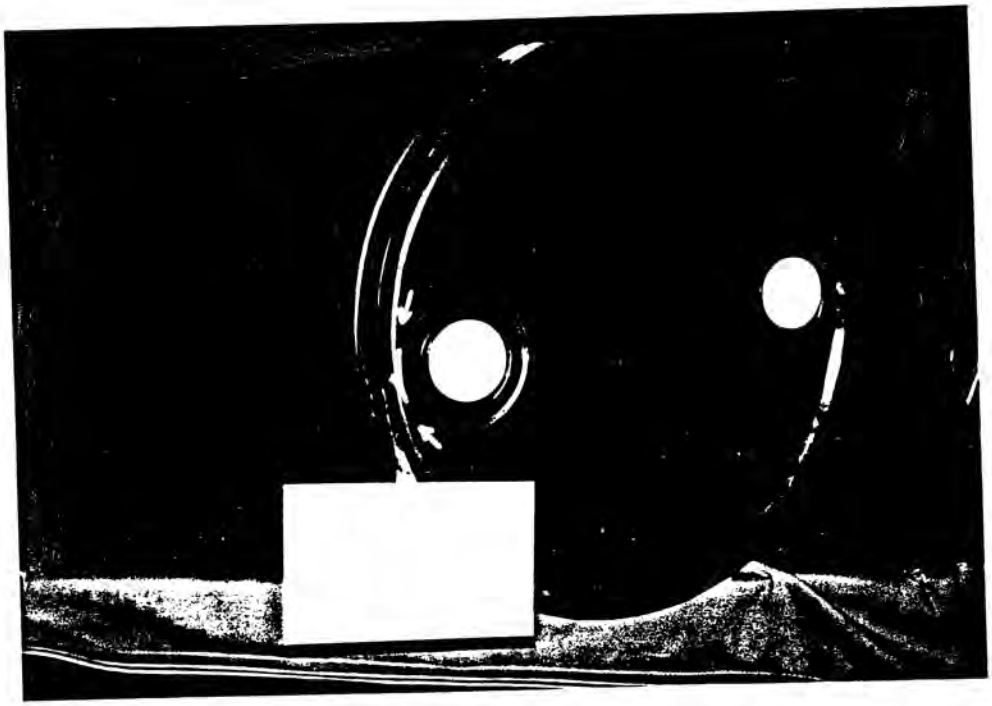


Photo # 10 - ESCR Test Failure

- Drum Variable = 30% Recycled Resin
- Days to Failure = 13 Days