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INTERIM REPORT ON CHEMICAL AND THERMAL ANALYSIS

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SYNOPSIS

Work during the past six months has included significant research in several areas aimed at further clarification of the aging and chemical failure mechanism of thermoplastics (PVDF or Tefzel) for pipes. Among the areas investigated were the crystallinity changes associated with both the Coflon and Tefzel after various simulated environmental exposures using X-Ray diffraction analysis. We have found that significant changes in polymer crystallinity levels occur as a function of the exposures. These crystallinity changes may have important consequences on the fracture, fatigue, tensile, and chemical resistance of the materials. We have also noted changes in the molecular weight distribution of the Coflon material using a dual detector Gel Permeation Analysis. Again these changes may result in variation in the mechanical and chemical properties in the material. We conducted numerous analytical studies with methods including X-Ray Diffraction, Gel Permeation Chromatography, Fourier Transform Infrared Spectroscopy, Thermogravimetric Analysis, and Differential Scanning Calorimetry. We investigated a number of aged samples of both Tefzel and Coflon that were forwarded from MERL. Pressurized tests were performed in a modified Fluid G, which we will call G2. In this case the ethylene diamine concentration was increased to 3 percent in methanol. Coflon pipe sections and powdered Coflon were exposed in pressure cells at 1700 psi at three separate test temperatures, 70 °C, 110 °C, and 130 °C. The primary purpose of the pressure tests in Fluid G2 was to further elucidate the aging mechanism of PVDF degradation.

1.0 High Pressure Aging of Coflon

1.1 Test Apparatus

Tests were performed in a high pressure Atlas cells which was graciously loaned to TRI for the CAPP project by CONOCO. The configuration for chemical exposure cell is indicated in Figure 1. The Atlas cell symmetry axis is horizontal, and the end plates are operated on edge, sealed against the body of the cell with O-rings. For these tests each cell was half filled with the EDA/MeOH liquid and pressurized to 1700 psi with CO₂ gas. Provisions for heating and monitoring the temperature of the liquid are incorporated into the cell. The cell is also plumbed to allow the gas pressure to be monitored and adjusted as necessary. Pressurization of the test chambers was accomplished using a double-piston high pressure intensifier.

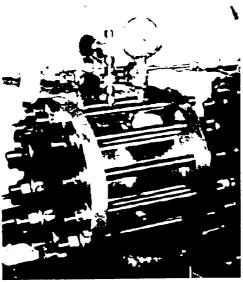


Figure 1 - Atlas Pressure Test Cell

1.2 Sample Preparations

Two types of samples were prepared for exposure in the Atlas pressure cell,. Coflon pipe sections, and powdered Coflon. The following sections describe how the two types were prepared.

1.2.1 Pipe Sections

Pipe sections were cut in approximately 2 mm lengths. These thin sections were chosen so that maximum deplasticization and diffusion of the ethylenediamine/methanol mixture could occur during the seven day test. Two pipe section specimens each were included in the 110 °C and 130 °C experiments. One of the two ring specimens was cut perpendicular to the pipe axis in order to allow free contraction. The other specimen was left intact to observe constrained contraction after exposure. Figure 1.2 shows both the cut and un-cut versions before exposure.

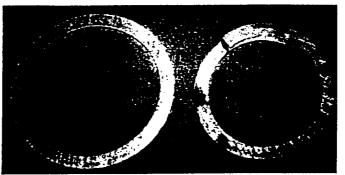


Figure 1-2. Unaged Coflon pipe sections. Section on the right was cut prior to exposure.

1.2.2 Powdered Materials

Powdered Coflon material was also used in order to facilitate deplasticization and diffusion of fluids into the polymer during the pressurized tests. The pipe section rings, described in Section 1.2.1, were cut into smaller sections and placed into a liquid nitrogen freezer mill for ten minutes. This process was repeated until enough powder was collected to perform the aging experiments (~ 20 g).

1.3 Test Conditions

Three temperatures were used in the high pressure Atlas cell tests, 70°C, 110°C, and 130°C. The Atlas cells were partially assembled and the powdered Coflon and Coflon pipe sections were placed inside. The cell was then completely bolted together. The cell was then filled half-way with a mixture of 3% EDA/ 97% MeOH. After reaching the appropriate temperature, 70°C, 110°C, or 130°C, CO₂ was introduced into the cell and the pressure raised to 1700 psi (117 bar). The cell was insulated with fiberglass and left for seven days. At the end of the seven day exposure the cell was drained and opened. Fluid samples were retained for analyses. The powdered Coflon was rinsed with methanol and dried in an oven for one hour at 70°C. Figure 1-3 shows the Atlas test cell after 70 °C test with coflon powder.

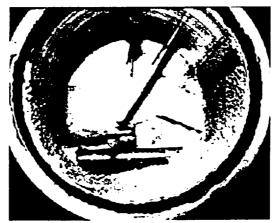


Figure 1-3. Atlas test cell after 70 °C test with Coflon powder.

2.0 Chemical Analysis Assessment of Aging Effects

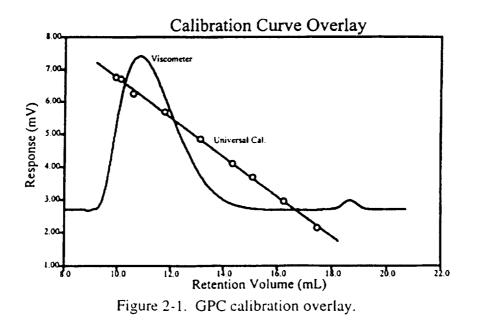
We have conducted a variety of chemical analysis to detect changes in Coflon and Tefzel when aged. We have detected significant changes in molecular weight and percentage crystallinity for the materials. Using infrared spectroscopy we have noted decreases in plasticizer and formation of double bond character in Coflon. We have derived an activation energy in association with the crystallinity change of Coflon.

2.1 Gel Permeation Chromatography of Aged Coflon

The samples were run on a Model 100 Viscometer and Viscotek LR40 Refractometer using one 15 μ m and one 10 μ m mixed bed gel American Polymer Standards GPC column. The solvent was NMP (No Lithium Bromide was Required) and the flow rate was 0.6 mL/minute. The injection volume was 100 μ L. The Detector Temperature was set at 35 °C and the columns were run at 80°C (in order to reduce solvent viscosity and maintain good resolution). Three hours dissolution time was initially allowed with all of the samples.

A universal calibration detector system was used for the analysis. This consists of a refractometer, and viscometer combination. Concentration distribution was calculated through the refractive index signal, and the viscosity was calculated directly through the Differential Wheatstone Bridge Viscometer. The molecular weight is calculated via the Universal Calibration technique. This allows us to measure absolute molecular weight and molecular size independent of the polymer calibration type.

The universal calibration curve is given with the viscometer chromatogram overlaid in Figure 2-1. The maximum error based upon the standards is 8.0% and the average error is 4%



Signal to noise was excellent on the Viscometer. There was some linear drift present on the Refractometer. Despite this drift, the signal to noise was applicable for good Universal Calibration reproducibility and accuracy.

The moderate intrinsic viscosity values coupled with linear Mark-Houwink plots (Figure 2-2). (over most of the molecular weight range) present in these samples indicate that the samples have a general linear structure. (There is no evidence of any aggregated or crosslinked material in these samples that seemed to be present in the first set of samples that was run. However, samples T73, T7D70, and T7D110 had different Mark-Houwink plot than that of samples T68, T70, and T72. This indicates that there is either the presence of a backbone modification or short chain branching difference between these samples, or that there is the presence of a contaminant in one of the two sample lots as shown by the Mark-Houwink Plot

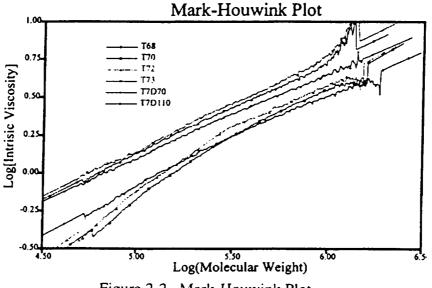


Figure 2-2. Mark-Houwink Plot.

Samples run in duplicate gave similar findings. As in the case of the previous samples, dissolution percentage varied between sample lots with the worst recovery (estimated between 40-50% from samples T68, T73, T7D70, and T7D110.

Overlaying these samples (Figure 2-3) with the past samples that were run, confirm no aggregation compared to but continuity of structure with samples with T73, T7D70, and T7D110. T68, T70, and T72 seem to be different in structure somehow (i.e.) less compact or lighter on the backbone.

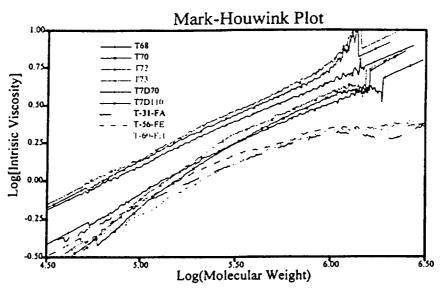


Figure 2-3. Mark-Houwink Plot overlaied with previous samples.

Molecular Weight Distributions							
Sample ID	Mn	Mw	Mz	Exposure			
Control Coflon	81,700	538,900	3,217,000	No Exposure			
T-31 Coflon	92,900	636,100	2,060,000	Fluid A 140 °C 13 days			
T-43 Coflon	81,100	779,500	10,600,000	Fluid F 120 °C 4 weeks			
T-56 Coflon	80,700	461,200	2,403,000	Fluid E 120 °C 4 weeks			
T-58 Coflon	44,300	3,742,000	143,940,000	Fluid G 120 °C 4 weeks			
T-68	115,400	379,600	814,500	Fluid A 120 °C 126 days			
T-70	101,900	350,300	762,800	Fluid A 100 °C 126 days			
T-72	85,600	332,800	784,400	Fluid F 100 °C 1 month			
T-73	105,300	281,800	503,100	Fluid F 100 °C 3 months			
T-7D70	115,800	310,500	606,900	Fluid G2 70 °C I week			
T7D110	116,000	285,400	497,800	Fluid G2 70 °C 1 week			

Table 2-1. Molecular weight distributions for Coflon samples

2.2 X-Ray Diffraction - Crystallinity Changes

X-Ray Diffraction was conducted on both samples from the exposed Tefzel and Coflon samples. The exposure fluids for the environmentally aged samples are detailed below.

(1) Fluid A- 100% Methanol

- (2) Fluid B- 97/3 CH4/CO2 with saturated water vapor
- (3) Fluid C- 97/3 CH4/CO2
- (4) Fluid D- 94/5/1 CH4/CO2/ H₂S
- (5) Fluid E- 94/5/1 CH4/CO2/H2S with saturated water vapor
- (6) Fluid F- As fluid E plus 1% ethylene diamine
- (7) Fluid G- As fluid A plus 1% ethylene diamine
- (8) Fluid H- As Fluid B plus 1% ethylene diamine

Percent crystallinity determinations on 16 polymer samples of PVDF were conducted. The percent crystallinity is calculated as the integrated area of the crystalline portion of the X-Ray diffraction pattern, as determined after background removal, divided by the total integrated area of the patterns. Correction factors for air scattering are also applied.

Results:

The percent crystallinities are very close between many of these samples. The X-Ray diffraction patterns show that samples T68, T70 and T77 have almost identical X-Ray diffraction patterns, and resulting percent crystallinity calculations. Samples T74 and T75 have only slightly lower percent crystallinity. Data plots are included in Appendix one.

Sample	% Crystallinity	Exposure
Control Tefzel	26.0	Laboratory Ambient
Control Coflon	41.5	Laboratory Ambient
T-66 Coflon	25.5	Fluid A 120 °C 22 days
T-68 Coflon	29.1	Fluid A 120 °C 126 days
T-70 Coflon	28.8	Fluid A 100 °C 126 days
T-73 Coflon	43.0	Fluid F 100 °C 3 months
T-74 Coflon	25.0	Fluid F 120 °C 2 weeks
T-75 Coflon	25.8	Fluid F 140 °C 3 days
T-76 Coflon	27.2	Fluid F 140 °C 5 days
T-77 Coflon	29.3	Fluid F 140 °C 8 days
T-79 Tefzel	18.0	Fluid F 100 °C 1 month
T-84 Tefzel	20.2	Fluid F 140 °C 8 days
T-88 Cotlon	42.0	Fluid F 120 °C 1 week
T-89 Coflon	41.0	Fluid F 120 °C 3 week
T-90 Coflon	39.0	Fluid F 140 °C 2 week
T-92 Coflon	32.0	Fluid G 120 °C 2 week

Table 2-2. percent crystallinity for Coflon and Tefzel samples.

Also included are the percent (%) Crystallinity on three powdered Coflon samples. percent crystallinity (microdiffraction) on cross section of Coflon tube. Three samples are Coflon Baseline Powder (CBP) (unaged), CP70 (aged at 70°C) and CP110 (aged at 110°C), and CP130 (aged at 130°).

Coflon Powder Samples

The results on four powder samples, three aged in the Atlas cell, are shown below in Table 2-3

Sample	% Crystallinity	Exposure
CBP	41%	Laboratory Ambient
CP70	37%	Fluid G2 70 °C 1 week
CP110	32%	Fluid G2 110 °C 1 week
CP130	18%	Fluid G2 130 °C 1 week

Table 2-3. percent crystallinity results for Coflon powder samples

The results show a decrease in crystallinity after aging (See Appendix One ((Exhibit I and 6))).

The changes in crystallinity were used to construct an Arrhenius relationship as displayed in Figure 2-4. An activation energy of 3 kcal/mole was obtained for the process. This is a rather low activation energy indicating that the energy needed to initiate the degradation reaction is relatively small. Likewise, within the range tested, the effect of temperature on acceleration of the process is also relatively small.

We attempted to model the data considering the decrease in crystallinity to be a first order reaction. The reaction rate was taken to have an Arrhenius temperature dependence. With the data available this led to three equations and three parameters, two of which were known previously. The three equations were solved for the third parameter leading to three fairly different values for the reference reaction rate. We concluded that the model considered did not constitute an adequate representation of the data. In order to further define the mechanism additional testing at other temperatures will be needed.

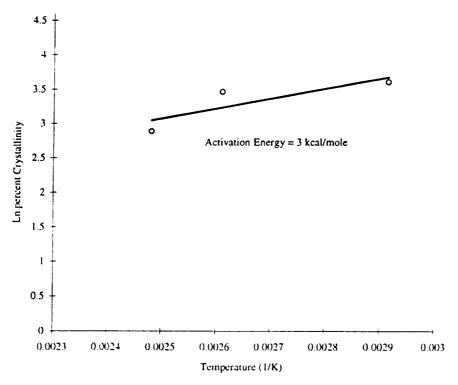


Figure 2-4. Plot of Arrhenius relationship of aged Coflon powder.

The most interesting result of this analysis was the fact that, in addition to crystallinity changes upon heating, a structural change is also observed upon heating. The raw data composite plot containing the baseline, 70 °C, and 110 °C Coflon powder in Appendix One, shows the peak shift occurring at the higher 20 range (lower d-spacing). In Table 2-4 we see the d-spacings in this region increasing upon heating.

Table 2-4. Shif	ted Peak D-Spacing for Coflon po	wder samples
Sample	D-Spacing for Shifted Peak	Δd
CBP	2.19A	
CP70	2.20A	+0.01 A
CP110	2.25A	+0. 05A
CP130	Peak Not Significant	+0. 05A

In the 2 θ range between 15° and 35°, the peak positions remain stationary (see exhibit 1). The 2 θ range between 35° and 60°, however, shows significant peak shifts to lower d-spacings upon heating. The 2 θ range below 5° shows a large peak developing upon heating to 110°C.

It should be noted that although the bulk of CP130 sample showed less crystallinity and a greater amorphous area, there were tiny individual crystallites identified in this sample (but not in the other samples). See data image in Appendix One for 130 °C aged Coflon powder.

The peak shifts indicate structural changes occurring upon heating. One theory for this is that the peaks between 15° and 35° (stationary peaks) represent regular inter-chain distances which do not change upon heating. In contrast, the region between 35° and 60° represents a secondary structure based upon the dipole moment (and arrangement) of the fluorine atoms. These bonds have a typical disassociation energy of between 1.5 to 3 kcal/mole. The shift to higher d-spacing appears to be related to an increase in chain length, (or increase in distance between chain folds in the structure of the polymer) caused from the relaxing of the interchain or intrachain structure, in a way similar to a compressed spring relaxing. Notice that the intensity of the peaks in this region also decrease, which also indicates a decrease in crystalline structure associated with these d-spacings. From the X-Ray diffraction pattern in the region between 35° and $65^{\circ} 2\theta$, we see both a chain length increase from the peak position change and a decrease in crystalline structure from the decrease in peak intensity.

Tube Cross Section

X-Ray diffraction patterns were taken by stepping from the outer to the inner wall of the tube cross section. The tube cross section wall thickness is 8 mm. The percent crystallinity results are as follows.

% Crystallinity	Exposure
37%	Laboratory Ambient
9%	Laboratory Ambient
11%	Laboratory Ambient
15%	Laboratory Ambient
18%	Laboratory Ambient
	37% 9% 11% 15%

Table 2-5. percent crystallinity through a Coflon pipe section.

The crystallinity is highest at the extreme outer wall. It falls off quickly and then increases slowly to the inner wall.

Again, we are seeing structural changes as well as crystallinity changes over this cross section (see Appendix One). The pattern taken at the extreme outer wall of the tube shows a completely different and more ordered structure, as seen by the crystalline peaks emerging between 35° and 60° 20. This increased order could be due to differences in the fluorine dipole moments and atomic arrangement as discussed in the section above. The amorphous scattering is also increasing as shown by the increasing background intensity.

This experiment represents a classic skin-core X-Ray diffraction result. It does suggest that the structural change is due to a difference in the cooling rate between the two walls.

Sample ID %C %Н %N %F Exposure CBP Coflon 40.77 3.91 < 0.02 54.64 Ambient Baseline Powder T-66 Coflon 37.91 3.13 < 0.02 59.05 Fluid A 120 °C 22 days T-68 Coflon 38.01 3.09 0.08 56.88 Fluid A 120 °C 126 days T-72 Coflon 39.96 3.48 0.09 56.26 Fluid F 100 °C 1 month T-76 Cotlon 40.06 3.65 0.19 54.20 Fluid F 140 °C 5 days T-79 Tefzel 36.30 2.75 0.03 60.29 Fluid F 100 °C 1 month T-84 Tefzel 36.52 2.80 < 0.02 60.55 Fluid F 140 °C 8 days T-85 Coflon 40.63 3.82 0.11 53.79 Unaged T-86 Coflon 40 49 3.75 0.22 55.45 Unaged extruded bar T-91 Coflon 38.04 3.24 0.15 58.62 Fluid F 140 °C 5ksi 4 weeks T-92 Coflon 38.31 3.35 0.32 58.12 Fluid G 120 °C 5ksi 2 weeks CP70 Coflon 37.23 3.10 0.08 58.62 Fluid G2 70 °C 1 week CP110 Coflon 37.72 3.04 0.07 59.61 Fluid G2 110 °C 1 week CP130 Cotlon 49.72 4.30 7.29 36.98 Fluid G2 130 °C 1 week

2.3 Elemental Analysis

Table 2-6. Elemental Analysis of Coflon and Tefzel samples.

Two samples. T79 and T92 showed (initial results) total percentages (CHNF) of <95%. Carbon results were confirmed in reanalysis. Repeating fluorine using a smaller sample size resulted in a correspondingly greater value such that total percentages showed >99%. Fluorine recovery tended to be more difficult in the analysis. There are several samples (T68, T76, T85) whose total percentage values are <99%. The fluorine value is slightly low due to some

incomplete recovery of fluorine. Combustion difficulties also can result in slightly excessive nitrogen. Values greater than 0.1 are suspect. Reanalysis of samples whose initial nitrogen is >0.2 usually resulted in a lower value. We have not reanalyzed T76 and T86. The nitrogen value for T92 was the only one which consistently showed >0.3%.

2.4 FTIR and Gas Chromatography/Mass Spectroscopy

We investigated accelerated high temperature aging of the Coflon material and analyzed the liquid exudate using gas chromatography coupled with mass spectrometry. The gas chromatography/mass spectrum of the liquid is provided in Appendix 5. The database search in many instances provided the best match with a dibutyl ester of decanedioic acid. The search routine can only match those compounds catalogued in the library that is approximately seven hundred thousand compounds. The tests were conducted on liquids (methanol) after Atlas cell aging.

We have conducted FTIR analyses of Coflon and Tefzel test pieces after aging for selected times, temperature, and exposure conditions. We are interested in the chemical functionalities or changes the samples exhibited upon aging. We were also interested whether the recrystallized PVDF deposits exhibited a change from the α or the type 2 form to the β or type 1 crystalline form when the PVDF dissolved under strain. The FTIR results are contained in Appendix 5. One factor that was evident was a band that appeared at about 1650 cm⁻¹ and can be indicative of double bond formation. We have enclosed FTIR result on samples that were not included in the last interim report. Dr. Baye and Dr. Casisdy also conducted infrared testing included in this report.

3.0 Thermal Stability and Compositional Analysis

We have conducted thermal studies to examine the migration of residual volatile material from aged Coflon test pieces. This includes aged Coflon pipe sections, power, and specimens aged at MERL. After aging we would expect some if not all the plasticizer to be removed or displaced by other components in the case of liquid exposure and to simply diffuse out of the polymer in gaseous environments. The following information describes how some of these processes occur in plasticized PVDF.

3.1 Analyses of Aged Coflon Powder.

Several aspects of the aged powdered Coflon material were analyzed. First, the residual volatile content, including plasticizer, is shown in Table 3.1. The volatile content decreased upon aging at 70°C and 110°C. However, at 130°C the residual content increases significantly. This may be due to the presence of low molecular weigh products of the degradation reaction. In addition, we see from the DSC results, also in Table 3-1, that the number of components appearing after aging is increasing with exposure temperature.

Sample	Exposure	Residual Volatiles Including Plascticizer	Weight Retention at 500 °C (%)	Weight Retention at 600 °C (%)	Melt Onset °C	Melt Peak °C	Heat of Fusion (J/g)
Baseline Coflon	None	<u>(%)</u> 10.97	41	30	144	168	51
Powder CP70	EDA/Methanol 7d at 70°C	3.52	52	34	162	172	77
CP110	EDA/Methanol 7d at 110°C	1.86	44	33	159 175	171 171	56
CP130	EDA/Methanol 7d at 130°C	9.13	55	50	51 165	86 171	224 · 7.3

Table 3-1. Aged Powdered Coflon Pipe Results

Another outstanding feature is the weight retention at 500°C which remains fairly constant at around 33% except for the 130°C exposure where an increases to 50% was observed. This appears to be a verification that some degree of crosslinking is occuring resulting in a more stable char.

3.2 Analyses of Aged Coflon Pipe Sections

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Again with the 130 °C aged Coflon pipe sections the weight retention at 600°C is greater than observed at the lower exposure temperatures. As shown in Table 3-2 we also detected, as with the powder, an increase in the number of endothermic events in the DSC data indicating conversion to a multi-component material.

Sample	Exposure	Residual Volatiles Including Plascticizer (%)	Weight Retention at 500 °C (%)	Weight Retention at 600 °C (%)	Melt Onset °C	Melt Peak °C	Heat of Fusion (J/g)
Baseline	None	11	41	33	159	170	49 - 67
PS110	7d 110°C 3%	3	53	35	161	166	100
PS130	EDA/Methanol 7d 130°C 3%	7	48	44	169 88	172 134	318
	EDA/Methanol		.0		192	195	2.5
					212	217	2.7

3.3 Compositional Profile of Aged Coflon Permeation Specimens

In order to determine how high pressure permeation affects the composition of Coflon pipe, thermogravimetric analysis (TGA) was performed. This analysis provides information regarding how plasticizer and other volatile materials migrate through the pipe wall. High

pressure permeation testing was performed at MERL using 8 mm thick sections cut from Coflon pipe. After permeation testing the specimens were sent to TRI for analysis. An example of this behavior is shown in Table 3-3. Shown here are the results obtained from the high and low pressure side of a test specimen that was exposed to Fluid F for 4 weeks at 120 °C and 5 ksi. Note that the high pressure side contains less residual volatile material than the low pressure side.

Sample	Exposure	Residual Volatiles Including Plascticizer (%)	Weight Retention at 500 °C (%)
T-51 HP Side	Fluid F 120°C 5 ksi 4 weeks	8	37
T-51 LP Side	Fluid F 120°C 5 ksi 4 weeks	12	N/A
T-74	Fluid F 120°C 5 ksi 2 weeks	7	35
T-75	Fluid F 140°C 5 ksi 3 days	8	36
T-76	Fluid F 140°C 5 ksi 5 days	9	36
T-77	Fluid F 140°C 5 ksi 8 days	8	37

Table 3-3. TGA test result for aged Coflon samples.

3.3.1 Long-Term Coflon Methane Permeation Test Specimen

Thermogravimetric analysis was performed on a Coflon specimen, T98 (MERL C22), after long term methane permeation testing at 140°C and 5000 psi. This specimen was not exposed to any fluid so that all residual volatile material within the sample should be plasticizer. Sections one mm in thickness were cut from the surface of the high and low pressure sides of the specimen. These were then placed in the TGA sample compartment where a nitrogen atmosphere was maintained while ramping the temperature 10°C per minute.

The results of the TGA indicated that the high pressure side had a residual plasticizer content of 1.9 percent. In comparison the low pressure side had a plasticizer content of 0.95 percent. This may be due to some molecular "packing" phenomenon occurring on the high pressure side.

3.3.2 Long Term Coflon Methane/Methanol Permeation Test Specimen

An additional thermogravimetric analysis was performed on a Coflon specimen which was exposed to methane permeation testing at 140°C and 5000 psi. In this case the high pressure side was covered with methanol to investigate any synergy between methane and methanol. Figure 3-1 shows the cross sectional profile of the specimen. Eight 1 mm thick sections were cut from the specimen. These sections were cut from the surface of the high and low pressure sides of the specimen. These were then placed in the TGA sample compartment where a nitrogen atmosphere was maintained while ramping the temperature 10°C per minute. This allows the determination of a residual plasticizer profile through the wall thickness of the Coflon pipe permeation specimen. The onset temperature observed indicates that the residue coming off is primarily plasticizer and not methanol.



Figure 3-1. Cross sectional profile of Methanol/Methane permeation specimen, 140 °C and 5ksi.

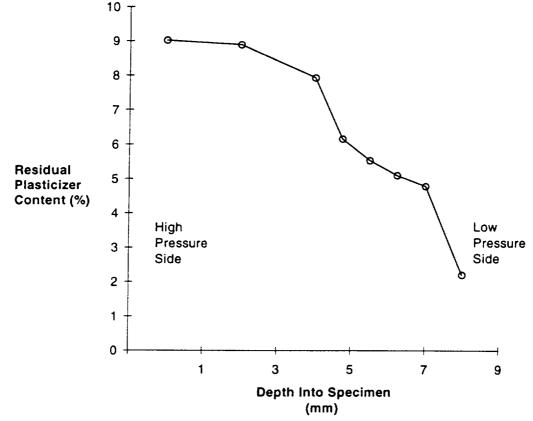


Figure 3-2 - Residual plasticizer content in a high pressure permeation specimen.T99. (MERL C28) CH₄ permeation through Methanol 5000 psi gas 140°C

As shown in Figure 3-1, the highest residual plasticizer content was again found on the high pressure side. A transition point appears to occur midway through the specimen For comparison a similar crossectioning for determination of plasticizer concentration profile was performed on unaged Coflon pipe. The results of this test indicated that the plasticizer concentration is very consistent through the wall thickness of the pipe. Differential Scanning Calorimetry (DSC) tests were also performed at the same wall depth locations. The DSC and TGA test results are summarized in Table 3-4.

	Melt Onset (°C)	Melt Peak (°C)	Heat of Fusion (J/g)	Residual Plasticizer (%)	Crystallinity (%)*
1 mm From I.D.	161	170	49.8	11.98	18
Interior of Pipe Wall	163	170	55.2	10.98	10
I mm From OD	159	168	67.1	10.36	37
Average	161	169	57	11	22

Table 3-4. DSC and TGA test results from unaged Cotlon Pipe

* performed using X-Ray crystallography

As would be expected the heat of fusion and crystallinity appear to correlate. The highest crystallinity and heat of fusion were found on the O.D. of the pipe. The plasticizer concentration is relatively constant through the thickness of the unaged pipe but there does seem to be trend toward higher concentrations near the I.D.

3.4 Residual Strain Before and After Aging Coflon Pipe Sections

Additional pipe sections were cut and placed into the high pressure test chambers along with the powdered Coflon for the 110°C and 130°C tests. Three sections were placed in the pressure cell during each experiment. Additional specimens were exposed to 110°C at atmospheric pressure with no fluid for comparison. After the one week aging cycles the sections were removed from the test cells and measured to determine shrinkage effects caused by exposure to the high temperature and pressure in the EDA/methanol mixture. Dimensional changes were primarily obtained for the 110°C test. Sufficient pipe section material was not available for the 70°C test. Pipe sections were placed in the 130°C test but were deformed and degraded so severely that only a few measurements were possible.

3.4.1 Residual Strain in Pre-Cut Pipe Sections

Two of the three specimens that were placed in the cells were cut perpendicular to the axis of the pipe prior to the test to allow for free contraction of the specimen. Immediately after cutting, the specimens contracted circumferentially approximately three percent corresponding to a residual hoop stress of 4500 psi. This residual stress is primarily due to thermal drawing history of the pipe during the extrusion process. Knowing the residual hoop stress we can calculate the internal pipe pressure needed to compensate for the residual stress by:

$$q = \frac{qt}{r}$$

where:

r = radius t = wall thickness

q = internal pressure

After exposure, the additional contraction of the cut sections was measured and found to have a total circumferential shrinkage of 17 percent for the specimens exposed to the EDA/methanol fluid at 110°C. Similarly an identical pre-cut specimen that was simply deplasticized for one week at 110°C had a circumferential shrinkage of 15 percent. It was also interesting to note that this specimen shrank 9.2% during the first ten minutes of the oven exposure indicating that most of the dimensional change is a result of thermal processing history. One dimension was measured for the specimen that was exposed to the EDA/methanol fluid at 130°C. The wall thickness was found to shrink by 7.2% after the seven day exposure. This was significantly higher than any of the other specimens tested. Table 3-5 summarizes the dimensional changes occuring in the various pipe sections tested.

			nges in Coflon Pi	pe Sections	
	Wall Shrinkage (%)	Length Shrinkage (%)	Initial Circumferential Shrinkage Prior To Exposure (%)	Circumferential Shrinkage After Aging (%)	Weight Loss (%)
Aged 7d 110°c 3% EDA/Methanol; Pre-Cut	2.45	6.55	3.00	17.00	3.40
Deplasticized 7d 110°c Pre- Cut	1.70	< 1%	3.00	15.00	4.80
Aged 7d 130°c 3% EDA/Methanol	7.20	N/A	N/A	N/A	N/A
	Wall Shrinkage (%)	Length Shrinkage (%)	Circumferential Shrinkage After Aging (%)	Circumferential Shrinkage After Aging The Specimen Then Cutting (%)	Weight Loss (%)
Aged 7d 110°c 3% EDA/Methanol; Cut After Exposure	2.51	4.44	1.48	2.59	2.80
Deplasticized 7d 110°c Cut After Exposure	1.88	< 1%	2.60	10.70	4.57

3.4.2 Residual Strains in Pipe Sections Cut After Exposures

The Coflon pipe specimens that were not cut perpendicular to the axis of the pipe were exposed to either an elevated temperature fluid or air atmosphere as described in Table 3-5. These specimens were intact and not as free to contract compared to the pre-cut specimens. Because of this constraint, we observed a significant decrease in the amount of circmferential shrinkage after aging (~1-3%). After making the dimensional measurements subsequent to aging, the specimens were then cut and measured. In this case the fluid exposed specimen had a

much smaller residual strain compared to the specimens conditioned in air at 110°C, (2.6% vs. 10.7% respectively). Figures 3-3(a-c) show the severe cracking observed in the 130 °C aged Coflon pipe sections. Figure 3-3 (c) is a comparison of unaged and aged Coflon.

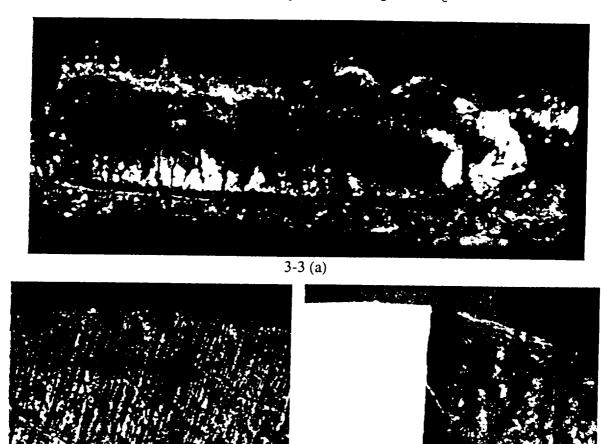


Figure 3-3 (a-c). Cross section of 130 °C aged Cotlon pipe section.

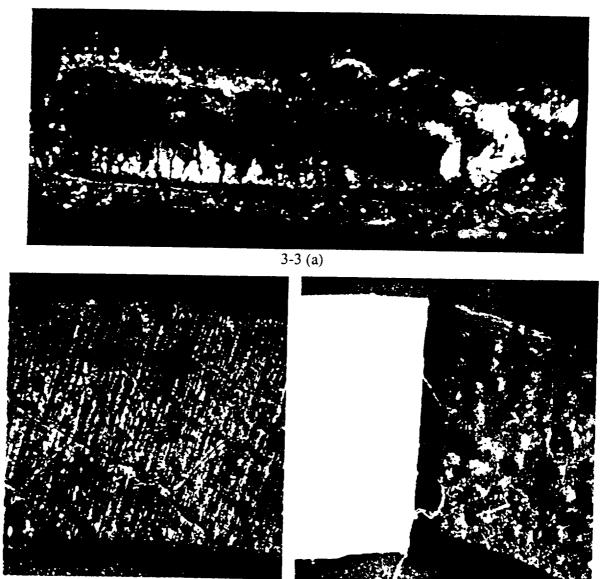
3-3 (c)

4.0 Degradation Kinetics and Mechanisms

3-3 (b)

Both old and new PVDF pipe were dissolved in NMP and films were cast on glass plates. After drying, small $(1^{\circ}X1^{\circ})$ film sections were used for aging studies. In a typical experiment, a 100 mL, three neck, round-bottom flask was fitted with reflux condenser and argon inlet.. The flask was charged with methanol (50 mL) and ethylenediamine (0.5 mL), and the film was added

much smaller residual strain compared to the specimens conditioned in air at 110° C, (2.6% vs. 10.7° c respectively). Figures 3-3(a-c) show the severe cracking observed in the 130 °C aged Cotlon pipe sections. Figure 3-3 (c) is a comparison of unaged and aged Cotlon.



3-3(b)

3-3 (c)

Figure 3-3 (a-c). Cross section of 130 °C aged Cotlon pipe section.

4.0 Degradation Kinetics and Mechanisms

Both old and new PVDF pipe were dissolved in NMP and films were cast on glass plates. After drying, small $(1^{"}X1^{"})$ film sections were used for aging studies. In a typical experiment, a 100 mL, three neck, round-bottom flask was fitted with reflux condenser and argon inlet. The flask was charged with methanol (50 mL) and ethylenediamine (0.5 mL), and the film was added

to this mixture and heated to reflux. After a specified time interval the film was removed, washed with methanol, dried and analyzed by infrared spectroscopy.

PVDF films were aged under several different conditions and the results are summarized in Table 4-1. All of the films were discolored by the aging process, and films subjected to prolonged aging acquired a dark color and became brittle.

The infrared spectra of old PVDF films showed that peaks at 3370, 2149 and 1642 cm-1 appeared after aging. A comparison of the infrared spectra of new PVDF film before and after aging showed that the absorption at 1642 cm-1 moved to 1599 cm-1. This may be due to reaction of the plasticizer.

Chemical analysis of aged old PVDF films indicated that composition is dependent on aging conditions (Table 4-1). In run 1 (10% ED, reflux, 24h), the film suffered a drastic reduction in percent fluorine (from 59,38% F in PVDF to 16.19%) accompanied by an increase in C, H, and N percentage. They also became extremely dark (black). Run 3 (1% ED, reflux, 16h) also resulted in some decomposition (lowered %F and raised values for % C, H and N). However, runs 2 and 4 (much milder conditions) left the PVDF analyses unchanged.

Future Plans:

- 1. The PVDF powder will be extracted with methanol, ethyl acetate, THF or 10% NaHCO₃ and aging studies will be repeated.
- 2. These films will be analyzed by infrared and NMR.

	Agin	g			E	lementa	l Analy	'sis
Run No	Solvent	Temp. (°C)	Time (hr.)	IR (cm ⁻¹)	%C	%H	%N	%F
1	MeOH + 10% EDA	Reflex	24.0	3372(b), 2945(s), 2149(w), 1642(b)	56.34	4.27	7.16	16.19
2	MeOH + 1% EDA	Reflex	1.5	3378,(b) 3021(s), 2979(s), 1642(w), 1407(s), 1208(w), 1027(b), 882(s)	37.79	2.90	0.04	59.58
3	MeOH + 1% EDA	Reflex	16.0	3384(b), 3021(s), 2979(s), 1601(b), 1453(s), 1410(b), 1264(b), 878(s), 837(s)	42.81	3.17	1.03	51.34
4	MeOH + 1% EDA	Room	16.0	3380(b), 3021(s), 2979(s), 1452(s), 1388(b), 1214(b), 1027(b), 886(s), 837(s)	37.86	2.87	0.13	59.60

Table 4-1. Chemical analysis of aged PVDF	Table 4-1.	sis of aged PVDF film	ns –
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Aging				
Solvent	Temp. (°C)	Time (hr.)	IR (cm ⁻¹)	Elemental Analysis
MeOH + 1% EDA	Reflex	22.0	2945(b), 1599(s), 1400(s), 1182(b), 879(s), 838(s)	NA
MeOH + 1% EDA	Reflex	15.0	Almost Same as Above	NA
MeOH + 1% EDA	Room	22.0	Almost Same as Above	NA

5.0 Literature Search

A literature search was conducted using NERAC on flexible PVDF pipes. Below are the results of this search.

1. FLEXIBLE METAL PIPE LINED WITH >POLYVINYLIDENE< >FLUORIDE< AND HAVING HIGH BENDABILITY

CAS SECTION- 156 CAS SUBSECTION- BBE - CAS 121-11121-261450 CA 121:261450 NDN- 010-0572-4587-3

NO-AUTHOR

JPN. KOKAI TOKKYO KOHO, AUG 02 1994, 4 PP. PATENT NUMBER- 94213377 PATENT APPLICATION NUMBER- 93-249954 GATE FILED- SEP 10 1993 DOCUMENT

TYPE-PATENT CDDEN-JKXXAF LOCATION OF WORK-FR. PATENT CLASS-F16LB111400BA; B32B0150800BB; F16L0111000B, B29D0231800-PATENT ASSIGNEE(S) - ELF ATOCHEM S. A. SUBFILE CODE- APP PATENT APPLICATION PRIORITY-920910FR92-10010 ORIGINAL PATENT APPLICATION COUNTRY- JP LANGUAGE-JAPANESE NO-ABSTRACT

Chemical Enqineering Abstracts (CEA)

2. Surface treatment of chemical equipment with fluorinated plastics. - CEA 87-ORB CEB1802006024 NDN- 141-0009-3451-3

Mieschke, W.

ABBREVIATED JOURNAL TITLE- Chemie-Technik VOL. 15 NO. 11 1986 PP. 33-34,36,39 DOCUMENT TYPE- Journal ISSN- 0340-9961 CODEN- CMTKAT LANGUAGE OF ABSTRACT- English ORIGINAL LANGUAGE- German

The >lining< of steel >pipes< and reactors with Fluorinated plastics considerably reduces corrosion problems in chemical environments., The range of fluorinated plastics now available enables selection of material to suit particular process conditions., The most commonly encountered fluoropolymer is PTFE, which has exceptional chemical resistance., Other compositions now available include >PVDF<, FEP, and TFA.

3. Choosing fluoropolymers for >lining< >pipes< - CEA 85-00 CEB1612015743 NDN-141-0006-6025-5

anonym

ABBREVIATED JOURNAL TITLE- Processing, London NO. 11 1984 PP. 26 DOCUMENT TYPE- Journal ISSN- 0305-439 CODEN- PCSNA4 LANGUAGE OF ABSTRACT -German ORIGINAL LANGUAGE- English

Fuer die Auskleidung van Stahlrohren stehen eine Reihe von Fluorpolymeren (PTFE, FEP, PFA, ETFE, ECTFE, >PVDF<) zur Verfuegung., Dem Artikel ist zu entnehmen, welcher Kunststoff unter den Aspekten Chemikalienbestaendigkeit, Temperaturbestaendigkeit, Verarbeitbarkeit und mechanische Eigenschaften zu bevorzugen ist., (Materne)

Engineering Index

4. Thermal insulation for >flexible< >pipe< development, and testing - EIX
94-27 EIX94271289170 NDN-017-0204-6436-1

Kalman, M.D.; Alexander, H L.; Belcher, J.R.; Loper, C.W.

Offshore and Arctic Oper ations American Society of Mechanical Engineers, Petroleum Division (Publication) PO v 51 1993. Publ by ASME, New York, NY, USA. p 49-SS 1993 DOCUMENT TYPE- CA, Conference Artie MONOGRAPH TITLE- American Society of Mechanical Engineers, Petr oleum Division (Publication) PD ISBN- 0-7318-0946-3 CODEN-ASMPEX AUTHOR AFFILIATION- Wellstream Corporation, Panama Cit, y, FL, USA SPONSDR- ASME

CONFERENCE DATE- 19930131-19930204 CONFERENCE TITLE- Proceedings of the 16th Annual Energy - Sources Technology Conference and Exhibition CONFERENCE LOCATION-Houston, TX, USA CONFERENCE CODE NO. - 19938 LANGUAGE- English

This paper presents the method of calculating non-bonded >flexible< >pipe< thermal resistance, the results of efforts to develop suitable insulation materials to increase the thermal resistances of non-bonded >flexible< >pipe< and the results of tests to verify the thermal resistance of an insulated > flexible< >pipe< section. The thermal resistance of a pipe (expressed in units of m degree C/M) is a function of the >flexible< >pipe< layer materials thermal conductivities and thicknesses. The effects on thermal resistance of flooding the >flexible< >pipe< annuls with seawater resulting from outer sheath damage and burial of the pipe in soil are also considered. Mater ials that were investigated include the thermoplastics normally used for >Flexible< >pipe< (HDPE), Nylon, >PVDF<), other solid thermoplastics (PVC, PP), and syntactic foams. The purpose of the investigations was to verify the thermal resistance of the conventional >flexible< >pipe< thermoplastics and develop a suitable material for providing additional thermal insulation that would be economical, manufacturable, chemically and thermally stable, and withstand the long term effects of hydrostatic pressure and moisture absorption at design water depth without significantly affecting the thermal performance of the material. The results of tests conducted to verify the thermal conductivity of the conventional >flexible< >pipe< materials and the mater ials being considered For thermal insulation are presented. Tests were conducted to verify the thermal resistance of a >flexible< >pipe< section by maintaining a constant temperature differential between the inside and outside of the >flexible< >pipe< and measuring the heat input

required to maintain the temperature differential. The test methodology and results are presented. (Author abstract) 5 Refs.

5. Advanced >flexible< >pipe< materials for aggressive hydrocarbon service EIX 93-38 EIX93381046180 NDN- 017-0191-0373-0

Hill, R.T.; Measamer, J.C.

Proceedings of the Third (1993) International Offshore and Polar Engineering Conference Proc Third 93 Int Offshore Polar Eng Conf 1993. Publ by Int Soc of Offshore and Polar Engineer ns (ISOPE), P.O.Box 1107, Golden, CO, USA. p 359-364 1993 DOCUMENT TYPE- CA, Conference Artie ISBN- 1-880653-09-S AUTHOR AFFILIATION- Wellstr earn Corp, Panama City, FL, USA CONFERENCE DATE- 19930606-19930611 CONFERENCE TITLE-Proceedings of the Third (1993) International Offshore and Polar Engineering Conference CONFERENCE LOCATION- Singapore, Singapore CONFERENCE CODE NO. - 18719 LANGUAGE- English

The increasing development of mar ginal offshore hydrocarbon deposits has resulted in production of increasingly corrosive fluids This has increased the need for pipelines capable of operating at elevated temperatures in the presence of high concentrations of HSUB2S and COSUB2 gases. Conventional pipelines requires the use oF stainless steels or corrosion resistant alloys which drives up the cost of materials and fabrication/installation of the system. The use of >flexible< >pipe< systems is assuming as important role in these applications where stainless steel and CRA carcass materials may be economically utilized. This paper discuses testing of >flexible< >pipe< carcass materials and a new >PVOF< hornopolymer fluid barrier material. (Author abstract) 7 Refs.

6. Acid resistance of thermoplastics. - EIX 92-04 EIX92040042607 NDN- 017-0166-3323-8

Patuska, G.; Just, U.

Kunstst Ger Plast v 81 n 5 May 1991 p 20-22 1991 DOCUMENT TYPE- JR, Journal Article ISSN- 0723-0192 CODEN- KSGPA7 MONTHLY PUBLICATION NO. - 054883 JOURNAL NAME- Kunststoffe - German Plastics LANGUAGE- German, english

For estimating the service performance of polyethylene (PE), polypropylene (PP),

polyvinylchloride (PVC) and polyvinylidenfluoride (>PVDF<) as >lining< materials for >pipes< and tanks for corrosive chemicals, the test specimens cited in Table 1 have been star ed in contact with dif f erent oxidizing acids. The per iod of storage was up to 24 months, the temperatur e of the chemicals has been adjusted to 23 or 40 degree C. 30% chromic acid, 80% sulphuric acid and 65% nitric acid have been chosen as oxidizing acids. All chemicals were of analytical grade pro analysis. During the period oF storage, the test specimens did not come into contact wit,h metals. At the end of storage in chemicals, the molecular weight distributions have been determined by gel permeation chromatography. In this connection, the test conditions indicated in Table 2 have been maintained. (Author abstract) 3 Refs. In German, English.

7. QUALITY IMPROVEMENTS, INNOVATIONS, AND SUCCESSFUL APPLICATIONS OF >PVDF< (PVFSUB2) LINED SYSTEMS. - EIX 85-04 EIX85040050387 NDN- 017-0044-1560-0

Werthmueller, Ernst

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Publ by NACE, Houston, TX, USA p 89-97 1983 DOCUMENT TYPE- CA, Conference Paper AUTHOR AFFILIATION- Symalit Co, Lenzburg, Switz SPONSOR- AIChE, New York, NY, USA; NACE, Houston, TX, USA; Soc of Plastics Engineers, Brookfield, CT, USA, SPI, New York, NY, USA; SPI of Canada, Don Mills, Ont, Can; TAPPI, Atlanta, GA, USA CONFERENCE TITLE- Managing Cor rosion With Plastics, Volume V. CONFERENCE CODE NO. - 04932 LANGUAGE- English

>Polyvinylidene< >fluoride< (>PVDF<) is a material distinguished by the tested, excellent corrosion and temperature resistance of a fluorine resin, without losing the technological advantages of a thermoplastic. The thermoplastic properties as well as the outstanding chemical resistance of >PVDF< led to this material being used more and more in lining metal containers, as well as in connection with glass Fiber reinforced plastics (FRP) as dual laminates. 4 refs.

Rubber K Plastics Research Abstracts

8. ASTM F 491-. SPECIFICATION FOR POLY (VINYLIDENE FLUORIDE) (>PVOF<) PLASTIC-LINED FERROUS METAL PIPE & FITTINGS RPA 95-05 538774 NDN-131-0107-5151-8

NO-AUTHOR

1993 Philadelphia, Pa, 1993. 1/8/93. NALOAN 6H210 DOCUMENT TYPE- Specification (Standard) CORPORATE AUTHOR- American Society for Testing & Materials SUBFILE CODE- RR ISSUE OF ORIGINATION- 9504 LANGUAGE- English

Details are given of >PVDF< plastic lined metal pipe and fittings, for use with corrosive gases and liquids. Photocopies and loans of this document are not available from Rapra. It may be purchased from BSI. Please contact Rapra for further details. NON POLYMER- METAL

9. GET THE MOST OUT OF PLASTIC LINEO PIPE - RPA 94-23 528177 NDN- 131-0106-5173-1

Bu x t on, L. W.; Hent horn, G. V.

ABBREVIATED JOURNAL TITLE- Chem.Engng. 1934 101, No.9, Sept. 1994, p. 133-7 DOCUMENT TYPE- Journal CODEN- CHEEA3 CORPORATE AUTHOR- DuPont Co.; Dow Chemical Co. NAMED COMPANY- AMERICAN SOCIETY FOR TESTING & MATERIALS SUBFILE CODE- RR ISSUE OF ORIGINATION- 9412 LANGUAGE-English

The advantages of Fluoropolymer >linings< in metallic >pipes< are explained. Common fluoropolymers used in lined-pipe installations are listed. The use of fluoropolymer linings is than discussed in detail, including standards, permeation mechanics, physical properties of the liner, laboratory programme, laboratory results, field test results, and design considerations. 10 refs. NON POLYMER- BENZENE; CHLDRIOE; CHLORINE; HYDROCHLORIC ACID; METAL; METHYL ETHYL KETONE; METHYLENE CHLORIDE; NITRIC ACID; STAINLESS STEEL; SULPHURIC ACID; WATER

10. MECHANICAL BEHAVIOUR OF COMPOSITE TUBES AFTER EXPOSURE TO CORROSIVE ENVIRONMENTS - RPA 94-07 505159 NDN- 131-0104-2191-9

Dudley, N.; Hoa, S. V.

1990 Washington, DC, 12th-15th Feb.1990, paper 2-E. pp.8. 627 DOCUMENT TYPE-Conference Paper CORPORATE AUTHOR- Montreal, Concordia University CORPORATE EDITOR- SPI, Composites Institute SUBFILE CDDE- RR CONFERENCE INFORMATION-SPI Composites Institute 45th Annual Conference 1990 Conference Proceedings ISSUE OF ORIGINATION- 9404 LANGUAGE- English

Several hundred filament wound tubes made of fibreglass/vinyl ester with veil and PVC liners were exposed to hydrochloric acid, sulphuric acid, and water at room temperature and at 145F and 180F for up to 3 years. At different intervals of time, the tubes were emptied of their contents and observed visually for any degradation. Also, the tubes were subjected to axial tests, internal pressure to create hoop stress and internal pressure tests. High temperature (180F) had detrimental effect on the long-term performance of fibre reinforced plastic materials. Thermoplastic lined (PVC, >PVDF<) tubes had better environmental resistance than veil lined tubes. Photographs show modes of failure. 1 ref.

11. DEMYSTIFY VACIIUM CAPABILITIES OF PLASTIC-LINED PIPING - RPA 92-19 454292 NDN- 131-0084-7610-0

Jeglic, M. F.; Lindley, N. L.

ABBREVIATED JOURNAL TITLE- Chem.Engng.Prog. 1992 88, No.<, April 1992.p.SB-BB DOCUMENT TYPE- Journal CORPORATE AUTHOR- DOW CHEMICAL CO. SUBFILE CODE- RR ISSUE OF ORIGINATION- 9211 LANGUAGE- English (DEF)

A description is given of various operating conditions that can create vacuum within process piping, the theoretical and empirical capabilities of plastic-lined piping in vacuum service are presented, and guidelines are suggested which the chemical engineer should consider when designing and specifying plastic-lined piping systems capable of withstanding possible vacuum conditions. 5 reft.

12. ECONOMICAL, SAFE PLASTIC >LINING< FOR STEEL >PIPES< - RPA 92-11 445175 NDN- 131-0083-9510-0

NO-AUTHOR

ABBREVIATED JOURNAL TITLE- Plast.S.Africa 1992 21,No.7,Jan. 1992,p.44 DOCUMENT TYPE- Journal NAMED COMPANY- CHEMPLAST INC. SUBFILE CODE-BR TRADE NAME(S) - ECONOSAFE; TEFLON ISSUE OF ORIGINATION- 9207 LANGUAGE- English (DEF)

Chemplast Mare Etters new Econosafe range of plastics materials for the >lining< of steel >pipes< is claimed to ofhcer significant savings over pipe systems of exotic materials and rubber linings. Products are available in a wide range of plastic liner materials, all extruded to inteinational standards, which are less costly than coated and stainless steel pipes. BrieF details are given. NON POLYMER- STAINLESS STEEL; STEEL

13. REDUCE LEAKS AND CORROSION WITH PLASTIC-LINED PIPING - RPA 91-15 424035 NDN- 131-0081-8381-8

Lednicky, R.; Lindley, N. L.

ABBREVIATED JOURNAL TITLE- Chem.Engng.Prog. 1991 87,No.3,March 1991,p.45-8 DOCUMENT TYPE- Journal CORPORATE AUTHOR- DOW CHEMICAL CO. SUBFILE CODE- RR; MM ISSUE OF ORIGINATION- 9109 LANGUAGE- English (DEF)

The capabilities and limitations of plastic-lined metallic piping are described and guidelines for its consideration, including liner selection, are presented. Techniques for improving sealability, such as system design, installation and maintenance practices, are discussed. Lining materials considered are PP, >PVDF< and PTFE. NON POLYMER- METAL

14. LINING UP TO RESIST CORROSION - RPA 91-03 411282 NDN- 131-0080-5632-8

Jones, M. E.

ABBREVIATED JOURNAL TITLE- Chem.Engng. 1990 97, No.10, Oct.1990, p.104/11 DOCUMENT TYPE- Journal CORPORATE AUTHOR- PERFORMANCE PLASTICS INC. SUBFILE CODE- RR, MM ISSUE OF ORIGINATION- 9103 LANGUAGE- English (DEF)

Plastic linings For piping and equipment are reviewed. Assistance is given n choosing t, he type of lining for a particular application. NON POLYMER- STAINLESS STEEL; STEEL

15. TABLES OF CHEMICAL RESISTANCE OF SOLEF - RPA 90-17 400630 NDN-131-0079-4980-7

NO-AUTHOR

1988 Brussels, 1988, pp.23. 8xl2ins. 30/1/89. 42C386-935 DOCUMENT TYPE library Edition CORPORATE AUTUOR SOLVAY & CIE SA SUBFILE CODE RR; MM TRADE NAME(S)- SQLEF 1008; SOLEF 1010; SOLEF 1012; SOLEF 5008 ISSUE OF ORIGINATION- 9009 LANGUAGE- English (DEF)

(Also in German and Dutch). A series of tables are presented to illustrate the chemical resistance of Solef >PVDF< virgin grades 1008, 1010, 1012 and 5008. The tables are divided into two parts. The left hand side gives the chemical resistance of solid Solef as it is used for tubes, fittings, linings, pumps etc., and the right hand side refers solely to powder coatings applied by electrostatic spraying or by fluidised bed. Methods of testing are described and symbols and evaluation criteria are explained.

16. FMS 1 - RPA 90-11 395865 NDN- 131-BB79-0215-3

NO-AUTHOR

1989 (Burton-on-Trent), c. 1989, pp.13. 12ins. 21/2/89. 6H210 DOCUMENT TYPE- Library Edition CORPORATE AUTHOR- BTR SILVERTOWN LTO., FLUOROLINE DIV. SUBFILE CODE- RR TRADE NAME(S) - FLUOROLINE ISSUE OF ORIGINATION- 9007 LANGUAGE- English (DEF)

Details are given of the specifications, quality and performance of the Fluoroline range of plastic lined ferrous pipework as supplied by BTR Silvertown Ltd. The plastic linings include PTFE, PFA, FEP, >PVDF< and PP. Design details are given oF the pipes and fittings, and properties of the linings. NON POLYMER- STEEL

17. FLUOROLINE PIPE MEETS MAXIMUM SERVICE REQUIREMENTS AT MINIMUM COST - RPA 90-11 395842 NDN- 131-0079-0192-6

NO-AUTHOR

1981 Burton-on-Trent, 1981, pp.22. 12ins. 21/2/89. 6H21-935 DOCUMENT TYPE- Library Edition CORPORATE AUTHOR- BTR SILVERTOWN LTD. SUBFILE CODE- RR TRADE NAME(S) - FLUOROLINE-F; FLUOROLINE-K, FLUOROLINE-P; FLUOROLINE-T ISSUE OF ORIGINATION- 9007 LANGUAGE- English (DEF)

Details are given of the manufacture and properties of Fluoroline lined pipework. They consist of a seamless liner formed into Schedule 40 steel pipe by an exclusive process developed by Resistoflex Corp., which compensates for thermal expansion and contraction between the liner

and the steel. Four types of liner are available, i.e. Fluoroline T (PTFE/PFA), Fluoroline-F (FEP), Fluoroline-K (>PVDF<) and Fluoroline-P (PP). NON POLYMER- STEEL

18. GAFLON PTFE. LINED CHEMICAL EQUIPMENT - RPA 89-26 386720 NDN-131-0078-1071-4

NO-AUTHOR

1989 Telford, c. 1989, pp. 30. 12ins. 11/1/89. 42C38(10)-6H2 DOCUMENT TYPE- Library Edition CORPORATE AUTHOR- PLASTIC OMNIUM UK LTD. SUBFILE CODE- BR TRADE NAME(S) - GAFLON ISSUE OF ORIGINATION- 9001 LANGUAGE- English (DEF)

Gafton PTFE pipes and Fittings are described. The products can be lined with a selection of corrosion resistant materials including PTFE, PFA, FEP, >PVDF< and PP. Products include lined flexible hoses, lined dip pipe, lined sight glasses, expansion bellows and PTFE sheet, rod and tube. Specifications For pipes and fittings are given with details oF linings and steel components NON POLYMER- STEEL

19. INVESTIGATION OF DAMAGE TO ORGANIC MATERIALS USED IN CHEMICAL PLANT - RPA 85-28 301182 NDN- 131-0069-8893-3

Schindler, H.; Graefen, H.

ABBREVIATED JOURNAL TITLE- Chem. Ing. Tech. 1985 57, No.7, July 1985, p. 597-602 DOCUMENT TYPE- Journal ISSUE OF ORIGINATION- 8601 LANGUAGE- German

By use of thirteen case histories, the authors discuss types of damage which can occur in polymeric components such as >linings<, >pipes<, pumps, fittings and the like used in chemical plant. Polymers covered are PTFE, >PVDF<, HDPE, PP, PVC, rubber and GRP. Reasons For damage are analysed, causes considered include incorrect construction, installation, unsuitability or overloading of materials

20. FLUOROPOLYMER LININGS. SELECTION FOR PERFORMANCE AND RELIABILITY - RPA 84-21 256500 NDN- 131-0067-2933-2

Jones, A. P.

ABBREVIATED JOURNAL TITLE- Pipes Pipelines Int. 1984 29,No.4,July/Aug-.1984,p.7/25 DOCUMENT TYPE- Journal CORPORATE AUTHOR- FLUOROCARBON CO.LTD. ISSUE OF ORIGINATION- 8411 LANGUAGE- English (DEF)

Details are given of the use of Fluor opolymers f or >pipe< >linings<. Materials generally available are identified and the design factors which influence their use, performance and reliability over the range of pipeline requirements are discussed. Information is given about

the various fluoropolymers including PTFE, perfluoro alkoxy, FEP, ETFE, ECTFE and >PVDF<. Fluorocarbon Ltd., is discussed with reference to its activities in >lining< of >pipes< and vessels.

21. FLUOROLINE CORROSION-PROOF LININGS - RPA 84-17 253518 NDN-131-0066-9952-2

NO-AUTHOR

1976 Burton-on-Trent, 1976-, looseleaf. 15/12/83. 42C38-6H-935 DOCUMENT TYPE-Library Edition CORPORATE AUTHOR- BTR SILVERTOWN LTD. TRADE NAME(S) -FLUOROLINE ISSUE OF ORIGINATION- 8408 LANGUAGE- English (DEF)

Presented in this f older is inf or mat ion on f luoridine products, which include corrosion-prooF >pipes<, fittings and vessels, vessel >linings<, PTFE hose and end fittings and pumps. These products are fluoroplastic lined and are designed to withstand the corrosive influences of virtually all corrosive Fluids. Tabulated data, specificat, ions, and diagrams are given.

22. >PVdF< LININGS OUTLAST EXOTIC METALS - RPA 83-22 238210 NDN- 131-0065-4673-0

NO-AUTHOR

ABBREVIATED JOURNAL TITLE- Eur.Plast.News 1983 10, No.10, Oct.1983, p.63 DOCUMENT TYPE- Journal NAMED COMPANY- BRITISH STEEL CORP.; BTR SILVERTOWN LTD. TRADE NAME(S)- FLUOROLINE ISSUE OF ORIGINATION- 8311 LANGUAGE- English (DEF)

A solution to the problem oF pumping hot hydrochlor ic acid through pipework is briefly discussed. British Steel are using Fluoroline pipes lined with >PVdF< by BTR Silvertown NON POLYMER- HYDROCHLORIC ACID

23. ANTICORROSION: PIPING LINED WITH >PVDF< OR TEFLON FEP - RPA 83-16 233486 NDN- 131-BBB4-9955-7

NO-AUTHOR

ABBREVIATED JOURNAL TITLE- Rev.G.Caout.Plast. 1982 59, No. 624, Oct.1982-, p.65-8 DOCUMENT TYPE- Journal NAMED COMPANY- SYMALIT SA TRADE NAME(S)-ARMALON; MAXAR; TEFLON; TEFLON FEP ISSUE OF ORIGINATION- 8308 LANGUAGE- French

An account is given of the use oF >PVQF< or Teflon FEP >linings< for >pipes< for corrosion resistant piping, particular reference being made to the products of Symalit SA. Topics discussed include the use of Armalon composite Film for >lining< >pipes<, Maxar composite film, glass

fibre reinforced >PVDF< pipes and tubes, films of >PVDF< with films of an elastomer. In particular piping for chemical plants and similar applications is considered.

24. MILD STEEL PIPING WITH LOOSE THERMOPLASTIC LINERS - RPA 77-02 101164 NDN- 131-0052-9325-0

SMITH, F.

1976 PRI, NORTH EASTERN SECTION; IME. PLASTICS LININGS & COATINGS IN PREVENTION OF CORROSION. NEWCASTLE UPON TYNE, MAR. 1976, PAPER 16, PP.7. PREPRINT.935 DOCUMENT TYPE- Conference NAMED COMPANY- ICI ISSUE OF ORIGINATION- 8201 LANGURGE- English (DEF)

(SYMPOSIUM) PROBLEMS ASSOCIATED WITH >PIPE< >LININGS< OF >PVDF<, PTFE AND FEP ARE DISCUSSED. NDN POLYMER- STEEL; METAL

25. BTR FLUOROLINE CORROSION-PROOF LININGS: THE DEVELOPMENT IDEA THAT FOUNDED AN INDUSTRY. BURTON-ON-TRENT, C.1974 - RPA 74-11 10342 NDN- 131-0001-7369-1

NO-AUTHOR

PP.9. 12INS. 28/5/74. 42C38(10)-BA312 DOCUMENT TYPE- Library Edition CORPORATE AUTHOR- BTR SILVERTOWN LTD., CHEMICAL PLANT DIV. NAMED CQMPANY- BTR SILVERTQHN LTD., CHEMICAL PLANT DIV. TRADE NAME(S) - FLUOROLINE ISSUE OF ORIGINATION- 8201 LANGUAGE- English (DEF)

THE RANGE OF HOSE, >PIPE<, >PIPE< FITTINGS AND >PIPE< >LININGS< AVAILABLE IN PTFE, FEP, POLYPROPYLENE AND >POLYVINYLIDENE< >FLUORIDE< ARE OUTLINED.

26. FLUOROPLASTIC LININGS FOR CORROSIVE SERVICE - RPA 73-02 26090 NDN- 131-0000-8425-6

ATKINSON, H. E.

ABBREVIATED JOURNAL TITLE- CHEM.ENGNG. 1972 79, NO.29, 25TH DEC.1972, P.76/80 DOCUMENT TYPE- Journal ISSUE OF ORIGINATION- 8201 LANGUAGE- English (DEF)

CHARACTERISTICS OF THE MAJOR VARIETIES OF CORROSION RESISTANT FLUOROPLASTICS (I.E. PTFE, FEP, >POLYVINYLIDENE< >FLUORIDE<, PCTFE AND (PERFLUOROALKOXY) FLUOROCARBON RESIN ARE GIVEN. METHODS OF >LINING< >PIPES<, PIPE FITTINGS, VALVES, PUMPS,) AND TANKS AND COLUMNS, ARE DESCRIVED. TYPICAL CASE HISTORIES FOR >LININGS< >PIPES< ARE LISTED. FACTORS WHICH DETERMINE THE TYPE OF MATERIAL TO BE 'JSED ARE CONSIDERED. THEY INCLUDE CHEMICAL ENVIRONMENT, TEMP., PRESSURE, COMPONENT DESIGN, MECHANICAL ENVIRONMENT, FABRICATION TECHNIQUE AND QUALITY CONTROL INSTALLATION PRECAUTIONS, COMMERCIALLY AVAILABLE PRODUCTS AND INSTALLATION COST ARE ALSO DISCUSSED.

World Surface Coatings Abstracts

27. >Polyvinylidene< >fluoride<: polymer suitable for fluid handling under extremely corrosive conditions. - WSC 78-00 61860 78/01860 NDN- 138-0001-8300-7

DILLEY, E. R.

ABBREVIATED JOURNAL TITLE- BHRA 2nd Internat. Conf. on Internal & External Protection of Pipes, Canterbury 1977, Paper H2, 10 pp. DOCUMENT TYPE- Journal; etc. MAIN HEADING- PIPELINE COATINGS; PIPES; POLYVINYLIDENE FLUORIDE; POWDER COATINGS ISSUE OF ORIGINATION- 7803 LANGUAGE- English (DEF)

>Polyvinylidene< >fluoride< may be used for lining steel pipe, as solid pipe and as a powder coating For pipe. It is highly corrosion-resistant and suitable for chemical plant.

Second Search

1. DEGRADATION OF >POLYVINYLIDENE< >FLUORIDE< CAPACITORS DURING >ACCELERATED< >TESTS< CAS SECTION- 076 CAS SUBSECTION- 003 - CAS 090-08 090-131368 CA 090:131368 NDN- 154-0469-7845-5

BUROUGH, S. W.; BRAMMER, H. G.; BURNHAM, JOHN

ANNU. PROC, RELIAB. PHYS. (SYMP.), 1978, VOL.16, PP.219-23 DOCUMENT TYPE-JOURNAL CODEN-ARLPBI LOCATION OF WORK-HUGHES AIRCR. CO., TECH. SERV. OIV., CULVER CITY, CALIF. SUBFILE CODE-PHY LANGUAGE-ENGLISH

NO-ABSTRACT

DESCRIPTOR(S) - >POLYVINYLIDENE< >FLUORIDE< CAPACITOR DEGRDN; >LIFE< TEST >POLYVINYLIDENE< >FLUORIDE< DEGRDN; ALUMINUM FLUORIDE FORMATION CAPACITOR.

2. >POLYVINYLIDENE< >FLUORIDE< FOR PIPING OPERATING UNDER HIGHLY CORROSIVE CONDITIONS. - EI1 80-82 07350 NDN- 115-0079-7275-8

E. R. Dilley

ITEM NUMBER- 016121 Plast Pipes, Int Conf, 4th, Univ of Sussex, Falmer, Brighton, Engl, Mar 28-38 1979 Publ by Plast and Rubber Inst, London, Engl, 1979 p 13. 1-13. 8 LOCATION OF WORK- Laporte Ind Ltd. Luton, Bedfordshire, Engl MONTHLY PUBLICATION NO. -014835

The high crystal energy of polyvinylidene fluoride (PVDF) with the strength of the C-F bond make it probably the strongest of the fluoropolymers. Chemical resistance is outstanding, as one would expect of a fluoropolymer, so the material has a useful combination of properties to offer in the field of piping. A brief summary of PVDFs properties relevant to piping is given. It is concluded that PVDF is clearly a polymer with high potential for the protection or manufacture of piping systems where resistance to aggressive environment and/or where long >life< is required.

DESCRIPTOR(S) - APPLICATIONS; CONDITIONS; CORROSION; FLUOROPOLYMERS; LI; LINING, PIPE, PLASTIC; PIPE, STEEL; PIPING SYSTEMS; PLASTICS APPLICATIONS, PLASTICS, REINFDRCEO; POLYVINYLIDENE FLUORIOE SECTIONAL CLASSIFICATION CODE- CAL539, CAL545; CAL619, CAL817.

Engineered Materials Abstracts (EMA)

3. Cracking of >Poly(Vinylidene<>Fluoride<) Due to Chemical Attack. - EMA 93-12 199312-Cl-P-2648 NDN- 104-0018-222S-9

Stirling, C-D., Van Tilburg, V. S. M., Miller, N-A.

JOURNAL- NAME- Polymers and Polymer Composites 1, (3) 1993 PP. 167-174 reference (s) FIGURES- Photomicrographs. DOCUMENT TYPE- Journal Article ISSN- 0967-3911 AUTHOR AFFILIATION- Industrial Research (New Zealand), (All Authors) PUBLICATION COUNTRY- UK LANGUAGE- English

The effects of exposure of poly(vinylidene fluoride) (PVDF) to various chemical solutions have been investagated. Scanning electron microscopy (SEM) was used to examine the exposed samples for singns of chemical attack and cracking, while tensile and flexural bend tests were used to determine the effects of exposure on mechanical properties. Samples exposed to 58% sodium hydroxide exhibited both chemical attack and cracking together with a significant loss in ducti Iiy. Exposure to 16% sodium hypochlorite and concentrated sulphuric acid did not give either chemical attack or cracking but exposure to sulphuric acid did cause significant loss of mechanical properties. From the results obtained it was concluded that the cause of cracking in 58% sodium hydroxide was chemical embrittlement, whereby a brittle layer was formed on the surface of the material. This brittle layer subsequently cracked under applied tensile stress The actual mechanism by which the brittle layer was formed was not established. The cracking was not considered to be due to environmental stress cracking as this requires the simultaneous action of tensile stress and cracking medium. DESCRIPTOR(S) - Chemical attack; Embrittlement, Exposure; Journal Article; Liners --Service >life<;- Polyvinylidene fluorides --Mechanical properties; Stress cracking SECTION HEADING CODE- Cl SECTION HEADING- Mechanical Properties.

4. Semi-Fabrics From >Polyvinylidene< >Fluoride< (>PVDF<) Showing Different Morphologies--Long-Term Behaviour in Contact With Liquids. - EMA 90-00 9009F1-P-0724 NDN- 104-0007-2015-7

Henkhaus, R.

JOURNAL NAME- Werkst. Korros. 41 (4) Apr. 1990 PP. 162-169 ISSN-0043-2822 AUTHOR AFFILIRTION- Dechema-Institut SUBFILE- Polymers LANGUAGE- German

Two types of pipe were extruded from one special kind of granulate. Long-term tests with internal pressure were performed with water, p-xylene and acetic acid, in one case with pressure maintained at a constant level in the other case with constantly increasing pressure rates. In addition the uni-axial creep behaviour of injection-moulded and pressed material was examined. According to the degree of crystallinity of the semi-Fabrics and the kind of configuration within the different crystalline types it was found that creep compliances and failure times vary. For polymers like PVDF which show very flat time curves until rupture vs. applied stress, long-term tests with slowly increasing stresses and strains prove advantageous. Graphs, Photomicrographs.-AA

DESCREPTOR(S) - Creep (materials) --Pressure effects; Creep >life< --Microstructural effects; Creep tests; Pipe --Materials substitution; Polyvinylidene fluorides --Materials substitution SECTION HEADING CODE- Fl SECTION HEADING- Engineering Components and Structures.

5. Reinforced >PVDF< Pipe--Thermal Stress and Failure Mechanisms. - EMA 89-00 8904F1-P-0172 NDN- 104-0004-6582-0

Echalier, B.; Bluet, P.; Lefebvre, F.

JOURNAL NAME- Compos. Polym. 1, (4) 1988 PP. 265-279 ISSN- 0952-6919 AUTHOR AFFILIATION- Atochem SUBFILE- Polymers LANGUAGE- English

A comparison of the mechanical, thermal and chemical properties of polyvinylidene fluoride (PVDF) with those of other thermoplastics shows that PVDF is often the best material for hightemperature applications (100 deg C to 140 deg C) in contact with corrosive fluids, e.g. mineral acids, saline solutions or oxidising agents. The reinforcment of PVDF tube by encasing it in thermosetting resin based GRP increases resistance to internal pressure. Mowever, the difference in coefficient of linear expansion between the reinforcing material and the PVDF causes very high internal stresses to be set up as the temperature rises. Analysis of failures which have occurred during the service >life< of pipework shows that these were due to a combination of two factors: creep under compression of the PVDF at high temperature and tensile stress set up when returning to ambient temperature. The analytical basis of the problem is presented and the mechanical and thermal properties of each material are related to the overall behaviour of the composite material. 4 ref.--AA

DESCRIPTOR(S) - Corrosion resistance; Creep (materials), Failure; Pipe --Materials substitution; Polyvinylidene fluor ides --Materials substitution, Thermal expansion, Thermal stresses SECTION HEADING CODE- Fl SECTION HEADING- Engineering Components and Structures.

6. Development of Kynar >Polyvinylidene< >Fluoride<. - EMA 88-00 8803G1-P-0049 NDN- 104-0002-9543-4

Dohany, J. E

CONFERENCE PROCEEDINGS- High Performance Polymers: Their Origin and Development 1986 PP. 287-296 AUTHOR AFFILIATION- Pennwalt SUBFILE- Polymers PUBLISHER-Elsevier Science Publishing Co. Inc. PUBLICATION PLACE- 52 Vanderbilt Avenue, New York, New York 10017, USA CONFERENCE DATE- 15-18 Apr. 1986 CONFERENCE LOCATION- New York, USA TRADE NAME(S) - Kynar, PAGG; Kynar 500, PAGG; Kyna, 460, PAGG; Kynar Flex 2800, PAGG; Kynar SL, PAGG; Kynar ADS, PAGG LANGUAGE- English

Kynar polyvinylidene fluoride was introduced in 1960. First commercial production began in 1965. For the first five years of its >life<, Kynar resin was used almost exclusively in the electrical and electronic market. In 1965, the architectural finish, Kynar 500, was introduced followed by first plan expansion in 1969. In 1975, Kynar 460 was introduced for high speed wire extrusion. In 1981, the Underwriter laboratories listed Kynar resins for use in plenum cables that can be used without conduit. In 1983, a new homopolymer series was introduced known as the series 700 and was Followed by Kynar Flex 2800 in 1984. To meet the demands of the growing markets, a new plant for Kynar resins was built and placed in operation in 1985. Also that year, new coating resins, Kynar SL and Kynar ADS, were introduced. Kynar polyvinylidene fluoride is a crystalline polymer that is produced by the emulsion polymerization technique. Typical properties are discussed. 3 ref.--AA

DESCRIPTOR(S) - End uses: Historical; Polyvinylidene fluorides --Development; Properties (attributes) SECTION HEADING CODE- G1 SECTION HEADING- General and Nonclassified.

Rubber K Plastics Research Abstracts

7. DEVELOPMENTS IN FLUOROPOLYMER COATINGS - RPA 95-10 546171 NDN-131-0108-2543-5

Scheirs, J.; Burks, S.; Locaspi, A.

ABBREVIATED JOURNAL TITLE- Trends in Polymer Science 1995 3, No.3, March 1995, p.74-82 DOCUMENT TYPE- Journal ·CORPORATE AUTHOR- EcelPlas Australia: Ausimont USA Inc.; Ausimont SpA SUBFILE CODE RR ISSUE OF ORIGINATION- 9506 LANGUAGE- English

A review is presented of developments in Fluoropolymer coatings as the demand increases for outdoor durability. The new systems are based on copolymers such as polyfluoroethylene-co-vinyl ether and perfluoropolyethers, which can be used to coat temperature-sensitive substrates. This new generation of Fluorocarbon-based coatings are soluble in common organic solvents, curable at ambient temperatures, and have increased compatability with hardeners. Automotive topcoat applications are described which upgrade the service >life< of engineering plastics, speciality paints for graffiti and corrosion resistance, and water-based dispersions for environmental compliance are all discussed, as well as advanced techniques for monitoring the weathering performance of these coatings. 53 refs.

DESCRIPTOR(s)->ACCELERATED<>TEST<; AUTOMOTIVE APPLICATION; BLEND; BUILOING APPLICATION; COATING; COIL COATING; COLOUR RETENTION; COMPANY; COMPATIBILITY; COPOLYMER; CORROSION RESISTANCE; DATA; DEGREE OF POLYMERISATION; DURABILITY; ELECTRON SPECTROSCOPY FOR CHEMICAL ANALYSIS; ENVIRONMENTAL LEGISLATION; ESCA, ESR; ESR SPECTROSCOPY; FLUOROETHYLENE COPOLYMER; FLUOROPOLYMER; FOURIER TRANSFORM INFARED SPECTROSCOPY, FTIR; FUNCTIONALISATION, GLOSS; GRAPH; HARDENER; MECHANICAL PROPERTIES; MOLECULAR STRUCTURE; MOLECULAR WEIGHT; PAINT; PERFLUORINATION; PERFLUOROPOLYETHER; PLASTIC; PMMA; POLYETHER; POLYMETHYL METHACRYLATE; >POLYVINYLIDENE< >FLUORIDE<; PROPERTIES; >PVDF<; REVIEW, ROOM TEMPERATURE CURING; RTV; SOLUBILITY; SDLVENTLESS; TABLES; TECHNICAL; TENSILE STRENGTH; TEST METHOD; TESTING, TETRAFLUOROETHYLENE COPOLYMER; THEORY, THERMOPLASTIC; THERMOSET; WEATHER RESISTANCE; WEATHERING RESISTANCE IDENTIFIER(S) - COATINGS --fluoropolymers, FLUOROPOLYMERS --coatings GEOGRAPHIC DESERIPTOR(S) - AUSTRALIA; EUROPEAN COMMUNITY; EUROPEAN UNION; ITALY; USA; WESTERN EUROPE SECTIONAL CLASSIFICATION CODE-42C38; 6A311 SECTION HEADING CODE- QB; KM.

8. GET THE MOST OUT OF PLASTIC LINED PIPE - RPA 94-23 528177 NDN- 131-0106-5173-1

Buxton, L. W., Henthorn, G. V

ABBREVIATED JOURNAL TITLE- Chem.Engng. 1994 101, No. 9, Sept. 1994, p. 133-7 OOCUMENT TYPE- Journal CODEN- CHEEA3 CORPORATE AUTHOR- DuPont Co.; Dow Chemical Co. NAMED COMPANY- AMERICAN SOCIETY FOR TESTING & MATERIALS SUBFILE CODE- RR ISSUE OF ORIGINATION- 9412 LANGUAGE-English The advantages of fluoropolymer linings in metallic pipes are explained. Common fluoropolymers used in lined-pipe installations are listed. The use of fluoropolymer linings is then discussed in detail, including standards, permeation mechanics, physical properties of the liner, laboratory programme, laboratory results, field test results, and design considerations. 18 refs. NON POLYMER- BENZENE; CHLORIDE; CHLORINE; HYDROCHLORIC ACID; METAL; METHYL ETHYL KETONE; METHYLENE CHLORIDE; NITRIC ACID; STAINLESS STEEL; SULPHURIC ACID; WATER

DESCRIPTOR(S)- CHEMICAL; COMPANY, CORROSION; CORROSION RESISTANCE, COST; CRYSTALLINITY; DATA; DESIGN; ETHYLENE-CHLOROTRIFLUOROETHYLENE COPOLYMER; ETHYLENE-TETRAFLUOROETHYLENE COPOLYMER; FAILURE; FLUOROPOLYMER; GAS; GRAPH; HAZARDOUS MATERIAL; MECHANICAL PROPERTIES; PERFLUOROALKOXYALKANE COPOLYMER, PERFLUOROETHYLENE COPOLYMER, PERMEATION; PHYSICAL PROPERTIES; PIPE; PIPE LINING; PLASTIC; POLYPROPYLENE; POLYTETRAFLUOROETHYLENE; POLYVINYLIDENE CHLORIDE; >POLYVINYLIDENE< >FLUORIDE<; PP, PROPERTIES; PROPYLENE COPOLYMER; PTFE; PVDC; >PVDF<; SERVICE >LIFE<; STANDARD; STRESS CRACKING; TABLES; TEMPERATURE; TEST; THERMOPLASTIC; THICKNESS IDENTIFIER(S) -FLUOROPOLYMERS --pipe linings, LININGS --fluoropolymer linings; LININGS --pipes; PIPES --fluoropolymer linings; GEOGRAPHIC DESCRIPTOR(S) - USA SECTIONAL CLASSIFICATION CODE- 42C38; 6L12; 6H21 SECTION HEADING CODE- KM; QL; QI.

9. SOLEF >PVOF< ENGINEERING POLYMER STANDS THE TEST OF TIME...APPLICATIONS IN THE CHEMICAL AND ASSOCIATEO INDUSTRIES - RPA 92-23 458024 NDN- 131-0085-1342-9

NO-AUTHOR

1991 Hemel Hernpstead, 1991, pp.26. 12ins. 5/3/92. 42C386-935 DOCUMENT TYPE-Library Edition CORPORATE AUTHOR- SOLVAY CHEMICALS LTD. SUBFILE CODE-RR TRAOE NAME(S) - SOLEF ISSUE OF ORIGINATION- 9301 LANGUAGE- English (DEF)

Solef PVOF is a Fluorinated semi-crystalline thermoplastic material which offers chemical resistance to a wide spectrum of chemical products. It guarantees high reliability during a long service >life< and requires no special maintenance. It is lighter than steel and can reduce the weight and cost of a plants superstructure if used for solid structures or in conjunction with reinforced polyester. Examples are described and illustrated of the use of Solef PVDF in a variety of corrosive situations. NON POLYMER- BROMINE

DESCRIPTOR(S) - ACID RESISTANCE; ACID RESISTANT; APPLICATION; CASE HISTORY; CHEMICAL INDUSTRY; CHEMICAL RESISTANCE; COMPANIES;

COMPANY; COMPOSITE; CORROSION RESISTANCE; CORROSION RESISTANT; DATA. ENGINEERING APPLICATION; ENGINEERING PLASTIC; GRAPH; HEAT EXCHANGER; PLASTIC, >POLYVINYLIDENE< >FLUORIDE<; PRODUCT ANNOUNCEMENT; >PVDF<, REINFORCED PLASTIC; STORAGE TANK; TABLES; TANK: TECHNICAL; THERMOPLASTIC; VINYLIDENE FLUORIDE POLYMER IDENTIFIER(S) CHEMICAL RESISTANCE - -PVDF; VINYLIDENE FLUORIDE POLYMERS --chemical resistance GEOGRAPHIC DESCRIPTOR(S)- EUROPEAN COMMUNITY; UK; WESTERN EUROPE SECTIONAL CLASSIFICATION CODE-42C38E-935 SECTION HEADING- KM; UE.

10. FLUOROPOLYMERS AS COATING MATERIAL - RPA 89-03 367944 NDN- 131-0076-2303-3

Munekata, S.

ABBREVIATED JOURNAL TITLE- Prog.Org.Coatings 1988 16, No.2, 1st Aug. 1988, p.113-34 DOCUMENT TYPE- Journal CORPORATE AUTHDR- ASAHI GLASS CO.LTD SUBFILE CODE- RR; MM TRADE NAME(S)- KYNAR-1120; KYNAR-1140; TEDLAR ISSUE OF ORIGINATION- 8903 LANGUAGE- English (DEF)

The weather resistance of PVF and PVDF as coatings and the characteristics of such coatings are discussed and compared with those of recently developed fluoroethylene/vinyl ether copolymers (FEVE)s, which are soluble in organic solvents and curable over a wide range of temps. Data are provided on the properties of PVF, including Tedlar, PVDF, including Kynar-1120 and 1140, compatibility of FEVE with curing agents and pigments, adhesiveness of FEVE finish to various substrates, such as metals and polymers, and the mechanical properties and gas permeability of FEVE exposed in a Sunshine Weather-O-Meter. 14 refs. NON POLYMER- METAL

DESCRIPTOR(S) - >ACCELERATED< >TEST<; ADHESION; BONDING; COATING, COMPANIES, COMPANY; COMPATIBILITY; CURE, CURING, CURING AGENT; DATA; FLUOROETHYLENE COPOLYMER; FLUOROPOLYMER, GAS PERMEABILITY; MECHANICAL PROPERTIES; PIGMENT; PROPERTIES, >PVDF<; PVF; SOLUBILITY; SOLVENT; SUBSTRATE; TECHNICAL; TEST; TESTING; THERMOPLASTIC; ULTRAVIOLET IRRADIATION; UV LIGHT; VINYL ETHER COPOLYMER; VINYL FLUORIDE POLYMER; VINYLIDENE FLUORIDE POLYMER; WEATHERING IDENTIFIER(S)- FLUOROPOLYMERS, --properties; COATINGS --fluoropolymers; COATINGS, --properties; FLUOROPOLYMERS --coatings GEOGRAPHIC DESCRIPTOR(S) - JAPAN SECTIONAL CLASSIFICATION CODE- 42C38-6A3-9T SECTION HEAOING-QB; KM; UA.

11. UNIQUE COMPLEX - RPA 89-01 366393 NDN- 131-0076-0758-1

NO-AUTHOR

ABBREVIATED JOURNAL TITLE- Rev.G.Caout.Plast 1988 65, No.678, May 1988, p.71-5 DOCUMENT TYPE- Journal NAMED COMPANY- ATOCHEM SA; ELKAPLAST SA NV SUBFILE CODE- RR TRADE NAME(S)- ELKALITE; FORAFLON ISSUE OF ORIGINATION- 8902 LANGUAGE- French

Details are given of the activities of Elkaplast of France in the coextrusion of ABS and PVDF. Working under licence from Atochem, the company is the worlds only manufacturer of such coextrusions. The laminates obtained are trade named Elkalite, and consist of 1 to 9 mm thick ABS sheet covered with a 50 to 300 microns layer of Foraflon PVDF. Applications include vehicle components and equipment housings. Some company information is presented on Elkaplast.

DESCRIPTOR(S) - ABS; >ACCELERATED< >TEST<; AGEING; APPLICATION; CASING; CHEMICAL RESISTANCE IDENTIFIER(S)- COEXTRUSION --ABS, COEXTRUSION, --PVDF; COMPANY INFORMATION --Elkaplast GEOGRAPHIC DESCRIPTQR(S)- FRANCE SECTIONAL CLASSIFICATION CODE- 42C21C391D11-623, 42C386-623; 06 SECTION HEAOING- OI; SC, KF, KN, KO, KM; CS.

12. FORAFLON >POLYVINYLIDENE< >FLUORIDE< - A MATERIAL FOR CHEMICAL PLANT CONSTRUCTION - RPA 86-21 317104 NDN- 131-0071-4755-7

NO-AUTHOR

ABBREVIATED JOURNAL TITLE- Plastverarbeiter 1986 37, No.2, Feb. 1986, p.3-8-2 DOCUMENT TYPE- Journal TRADE NAME(S) - FQRAFLON ISSUE OF ORIGINATION-8611 LANGUAGE- German

Foraflon is a semi-crystalline homopolymer containing 59% fluorine and has a melting point of 170C. Properties of this PVDF are discussed, mention being made of thermoplasticity, heat stability, chemical resistance, wear, service >life<, mechanical behaviour, electrical characteristics and solubility. Information on processing character istics is given and application areas are outlined. Mention is made of its use in pipes, packaging, cables and wires and chemical plant.

DESCRIPTOR(S) - ABRASION RESISTANCE; CABLE, CHEMICAL PLANT; CHEMICAL RESISTANCE; CHEMICALLY RESISTANT APPLICATION, ELECTRICAL PROPERTIES; HEAT STABILITY; MECHANICAL PROPERTIES; MELTING POINT; PACKAGING; PIPE; PLI-"ISTICITY; PROCESSING; >PVDF<; SEMI-CRYSTALLINE; SERVICE >LIFE<; SOLUBILITY; THERMAL STABILITY; VINYLIDENE FLUORIDE POLYMER; WEAR; WIRE IDENTIFIER(S) - CHEMICAL RESISTANCE --PVDF; VENYLIDENE FLUORIDE POLYMERS GEOGRAPHIC OESCRIPTOR(S) - FRANCE SECTIONAL CLASSIFICATION CODE- 42C386; 42C386-935 SECTION HEADING- KM; UE.

13. PROGRAMME ON POLYMER AGEING EFFECTS - RPA 86-06 30603 NDN-131-0070-3720-0 NO-AUTHOR

ABBREVIATED JOURNAL TITLE- Chem. Engng.News 1985 63, No.48, 2nd Dec. 1985, p.13 DOCUMENT TYPE- Journal NAMED COMPANY- NEW YORK, POLYTECHNIC INSTITUTE ISSUE OF ORIGINATION- 8604 LANGUAGE- English (DEF) A major research programme on the ageing of polymer blends has begun at the Polytechnic

Institute of New York. Very brief details are provided.

DESCRIPTOR(S) - AGEING; BLEND; BUTADIENE-ACRYLONITRILE COPOLYMER; DEGRADATION; ETHYLENE POLYMER, LIGHT DEGRADATION; METHYL METHACRYLATE POLYMER; NITRILE RUBBER; PE; PHOTOOXIDATION; PMMA; POLYVINYL METHYL ETHER; POLYVINYL METHYL KETONE; >POLYVINYLIDENE< >FLUORIDE<; PS; PVC; RESEARCH; RUBBER-MODIFIED; SERVICE >LIFE<, STYRENE POLYMER; THERMAL DEGRADATION, THERMOOXIDATION; VINYL CHLORIDE POLYMER, VINYLIDENE FLUORIDE POLYMER IDENTIFIER(S) - AGEING --blends; DEGRADATION --blends GEOGRAPHIC DESCRIPTOR(S) - USA SECTIONAL CLASSIFICATION CODE- 03; 6125-93 SECTION HEADING-UE; OD.

World Surface Coatings Abstracts

Forming flexible coatings on metal plate. - WSC 91-03 323670 91/03670 NDN- 138-14. 0014-6154-4

NO-AUTHOR

6 pp: Jap. Pat. Abs (Unexamined) 1998, Vol 98 No 33, Gp G, 25. PATENT NUMBER- JP 02/174977 PATENT CODE JU DOCUMENT TYPE- Patent COUNTRY- Japanese Unexamined Patent MAIN HEADING- Acrylic Polymers, Durability; Flexible Coatings; Fluoropolymers; Polyvinylidene Fluoride PATENT ASSIGNEE(S)- NIPPON OILS & FATS CO ISSUE OF ORIGINATION- 9105 LANGUAGE- Japanese

A coating system having a claimed service >life< of 28 years and comprising undercoat/intermediate coat/topcoat containing polyvinylidene fluoride, has an intermediate coat cornpsn. consisting of a mixture of polyvinylidene fluoride with an acrylic polymer of MW 50,000 -200,000.

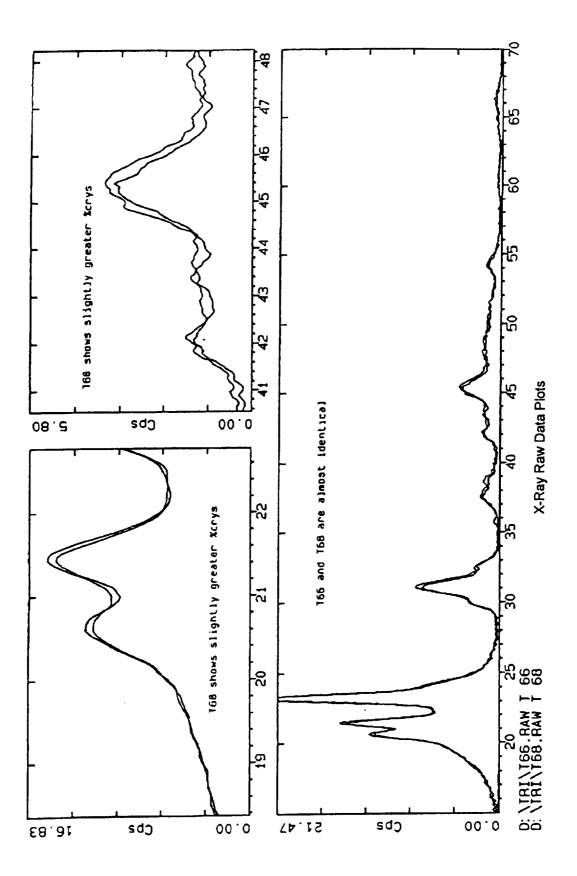
DESCRIPTOR(S) - Acrylic Polymers --coatings, fluoropolymers &; Durability --paint systems, intermediate coats, fluoropolymers/acrylics, Flexible Coatings -- for metals, intermediate coats, fluoropolymers etc; Fluor opolymers --coatings, acr ylics &; >Polyvinylidene<>Fluoride< -coatings, acrylics & CHEMICAL INDEXING- ACRYLIC; METHYL METHACRYLATE; BUTYL METHACRYLATE: POLYVINYLIOENE FLUORIOE; PROPYL

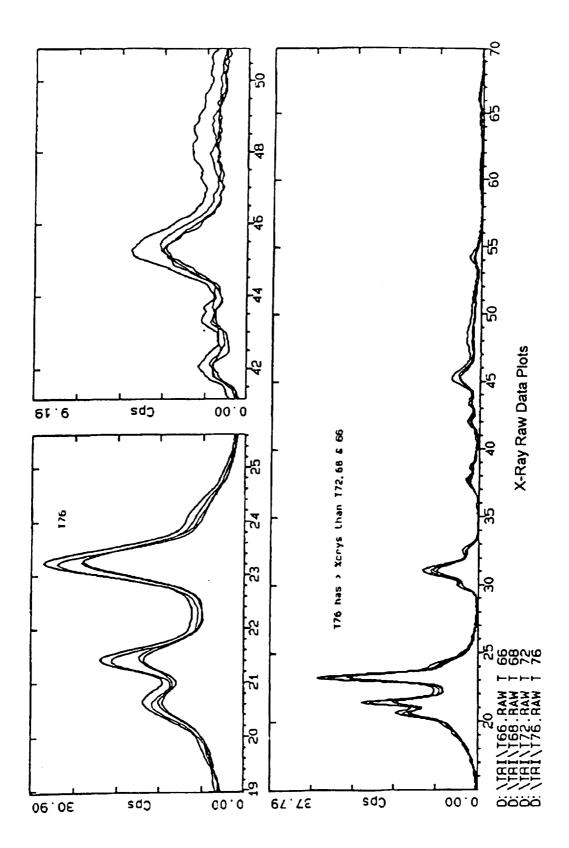
METHACRYLATE; OCTYL METHACRYLATE; FLUOROPOLYMER; ETHYL METHACRYLATE; HEXYL METHACRYLATE SECTIONAL CLASSIFICATION CODE- 64 SECTION HEADING- Paints, etc. for other Specific Uses.

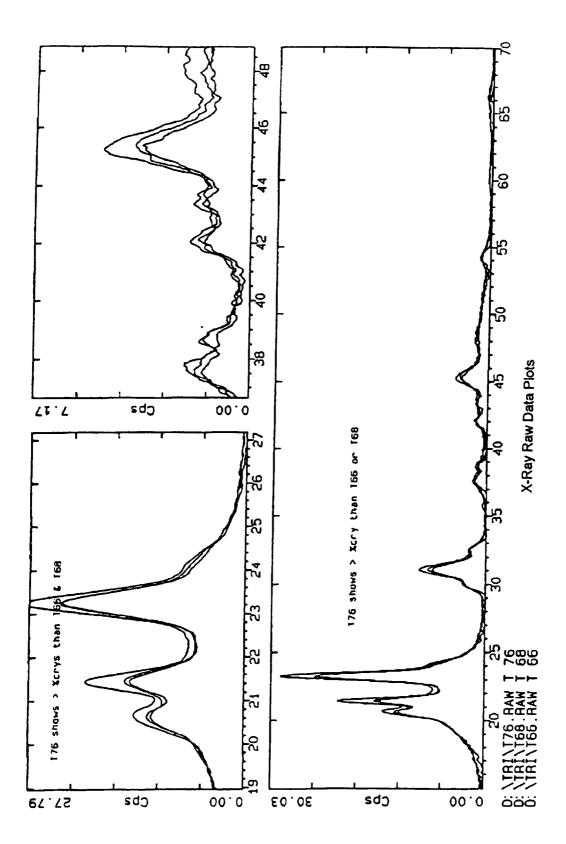
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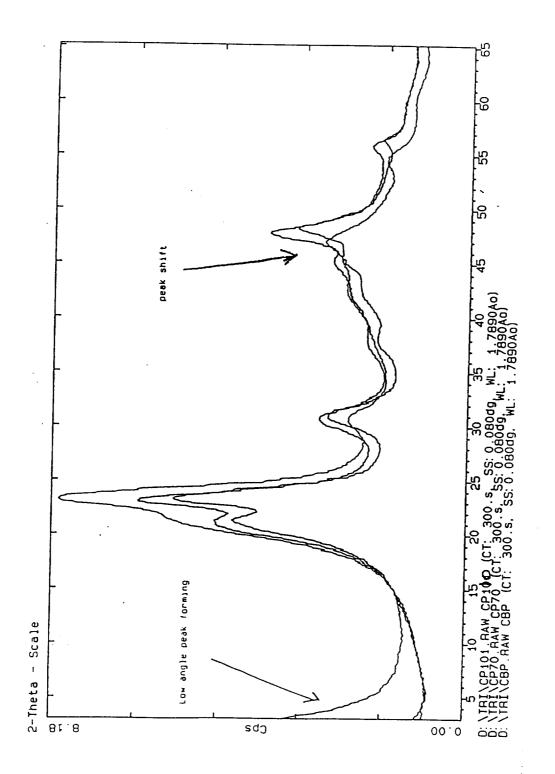
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X-Ray Diffraction Results

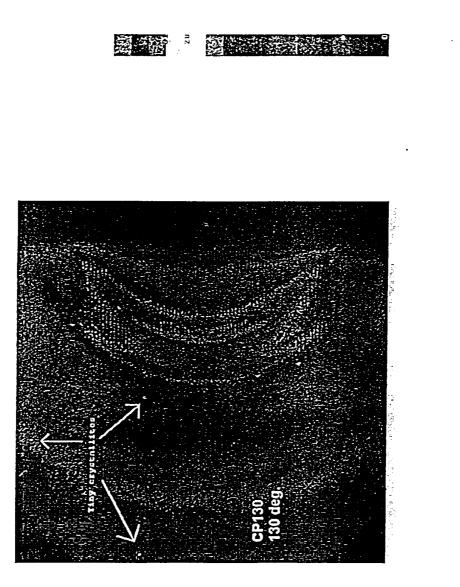




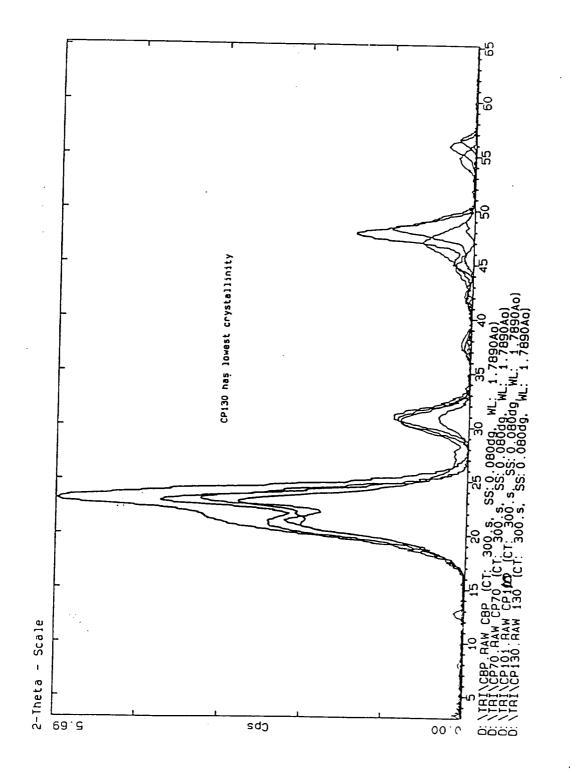




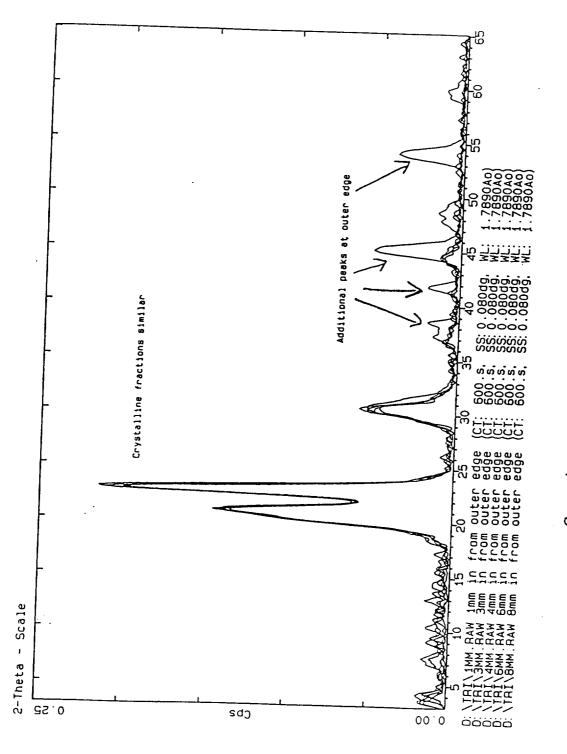


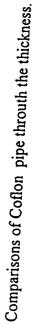


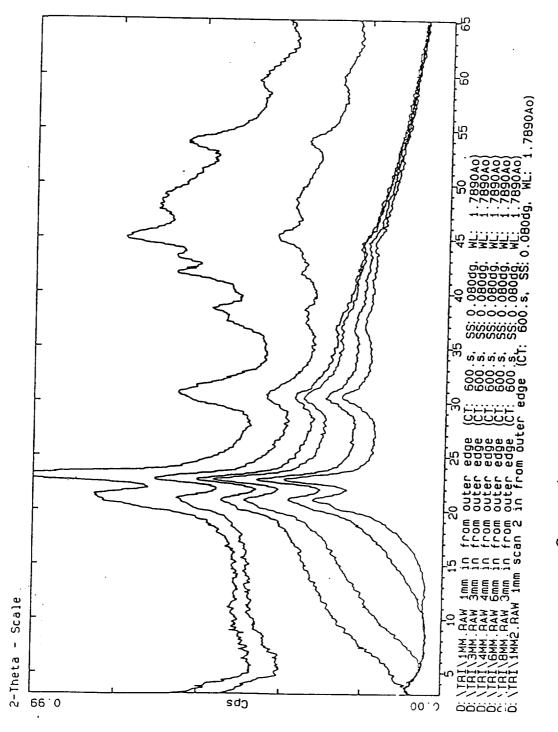
Data from images indicationg tiny crystallites in the 130 °C aged Coflim powder.





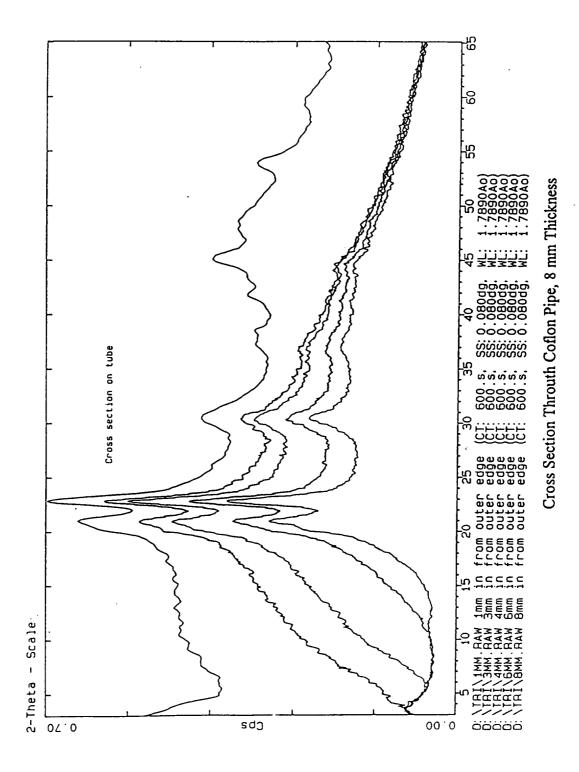




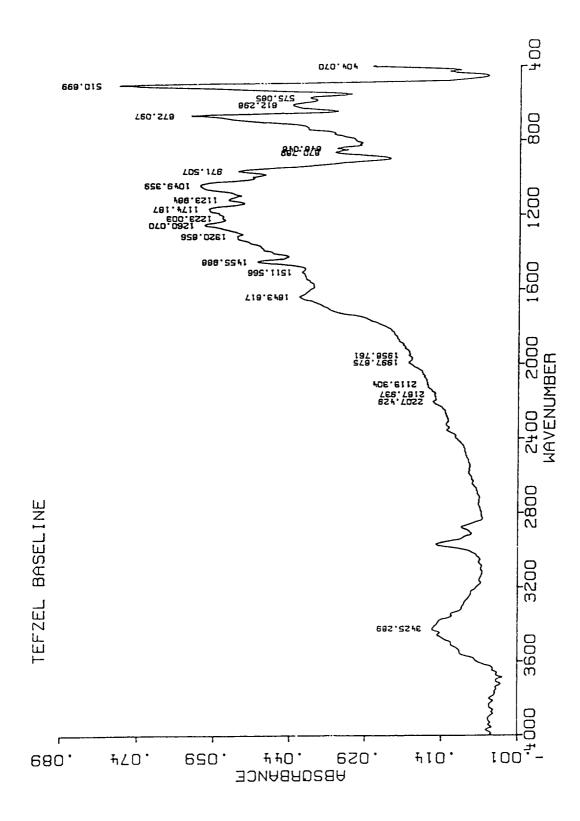


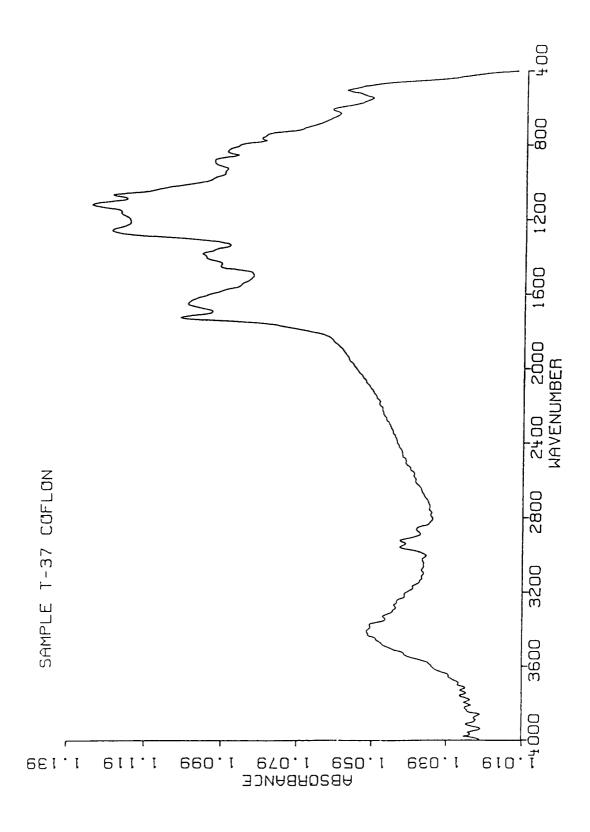
Cross section through coflon pipe, 1mm scan 25.4mm from outer edge.

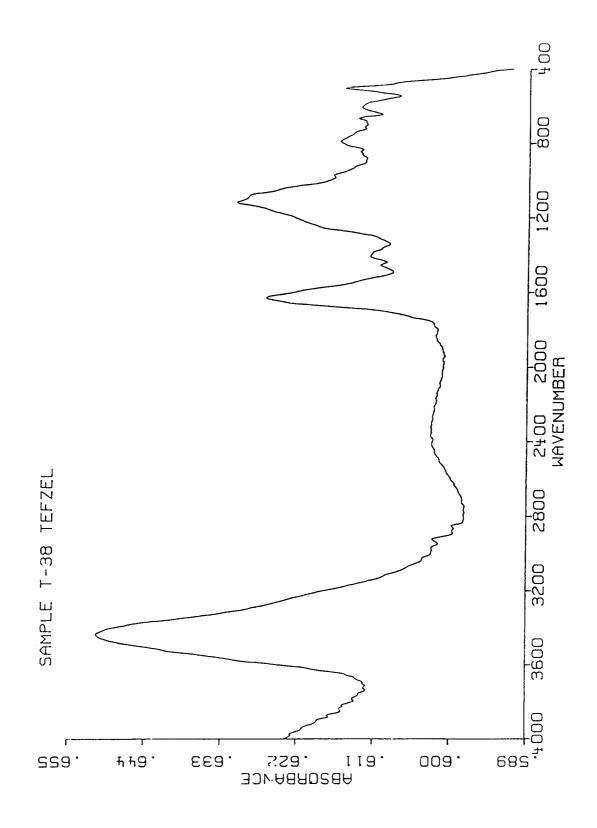
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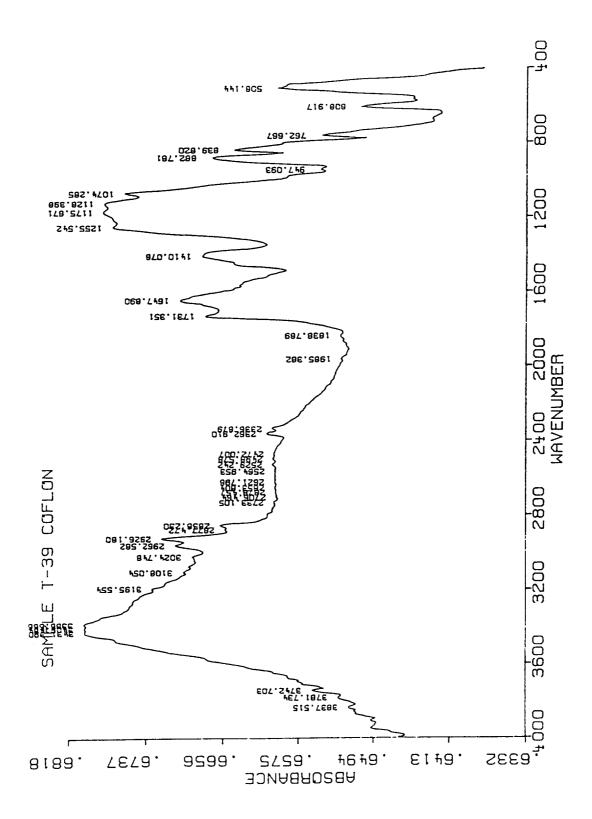


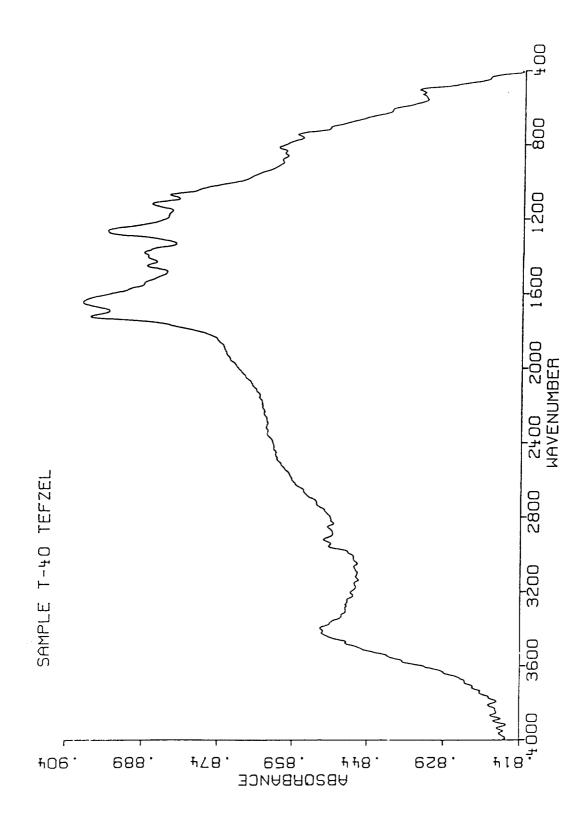
Appendix Two FTIR Results

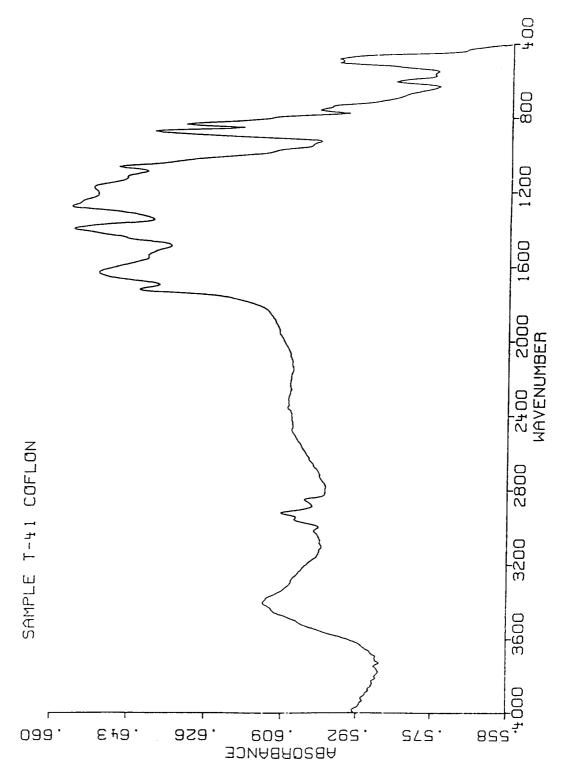


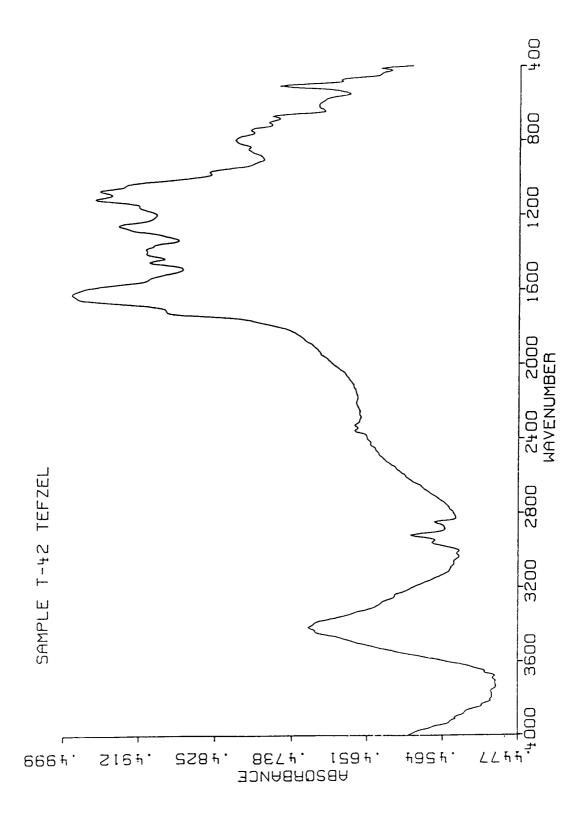




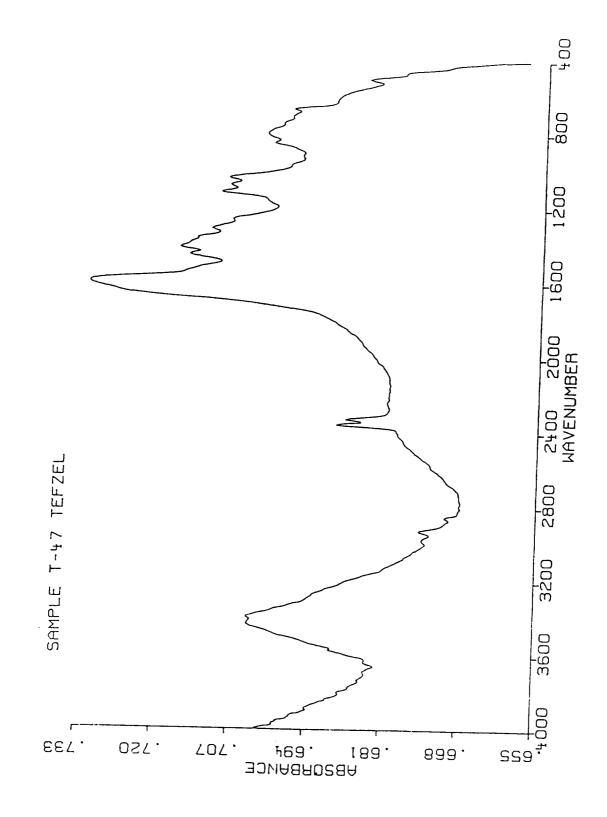


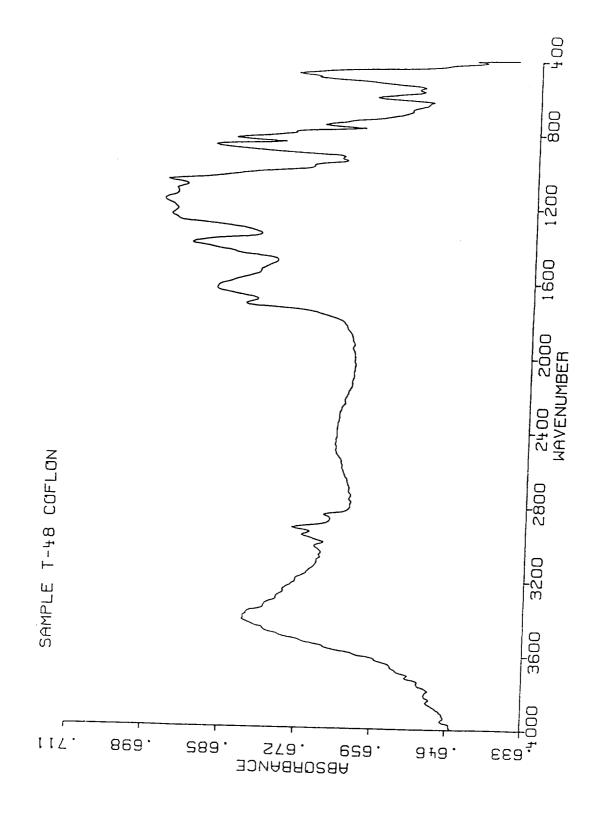


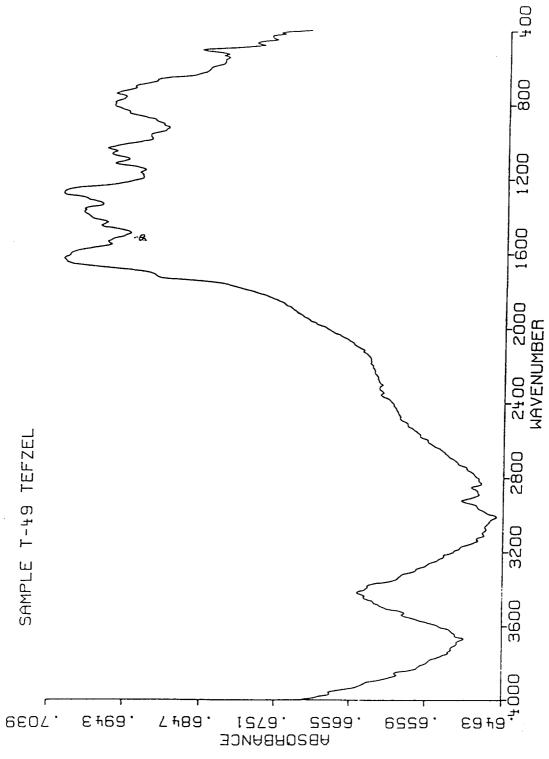


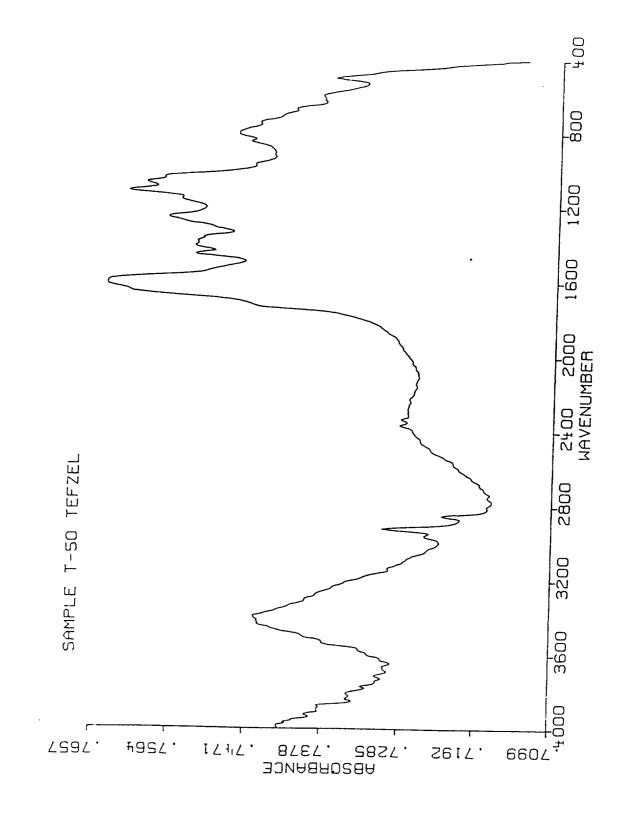


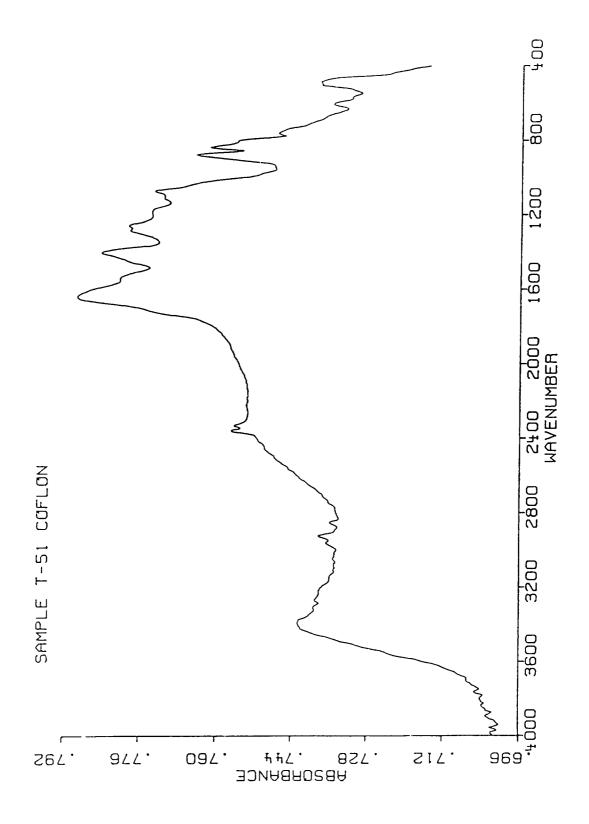
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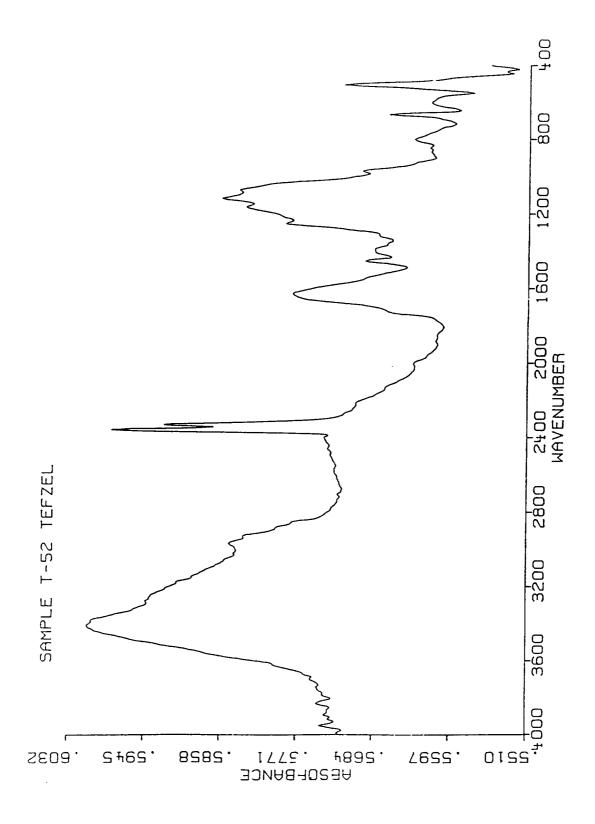




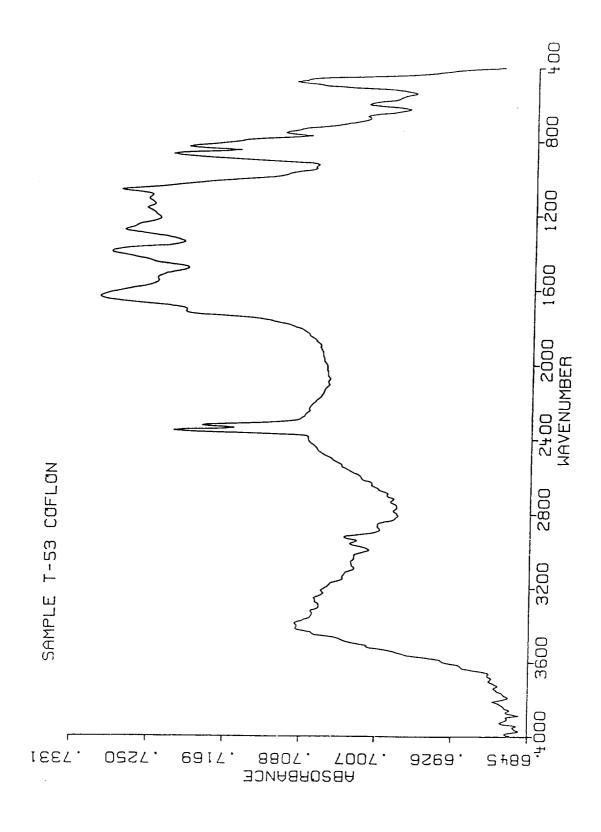


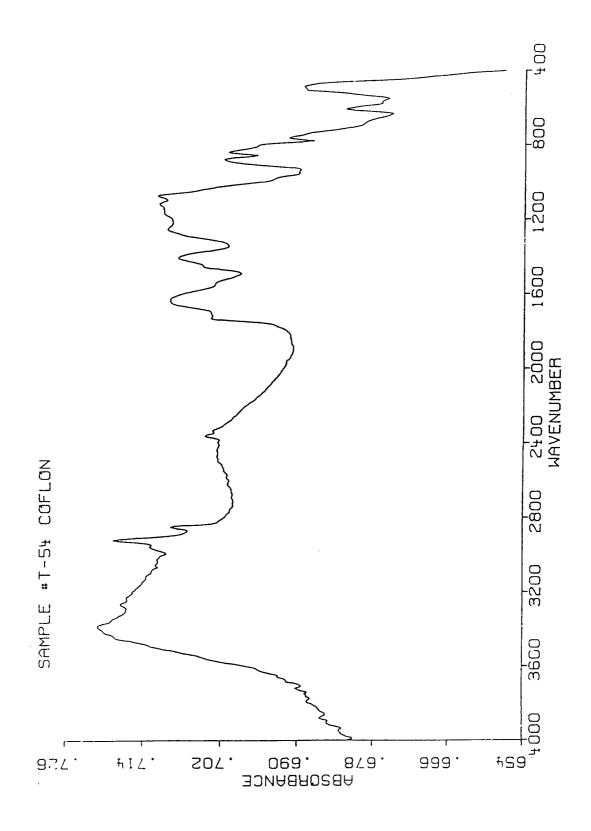


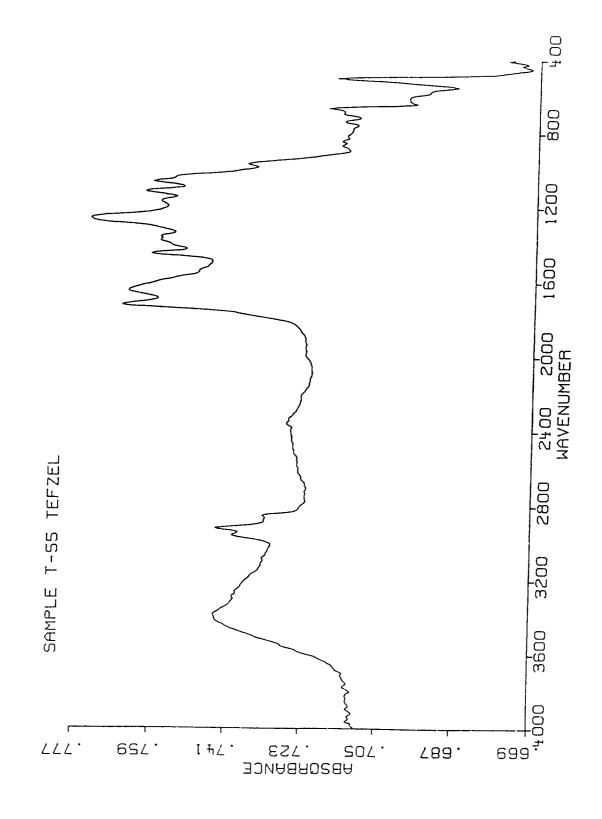


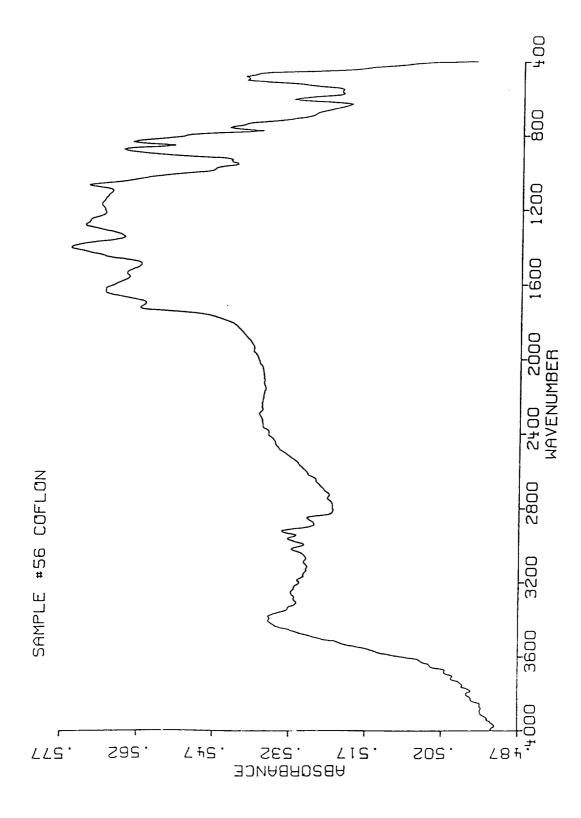


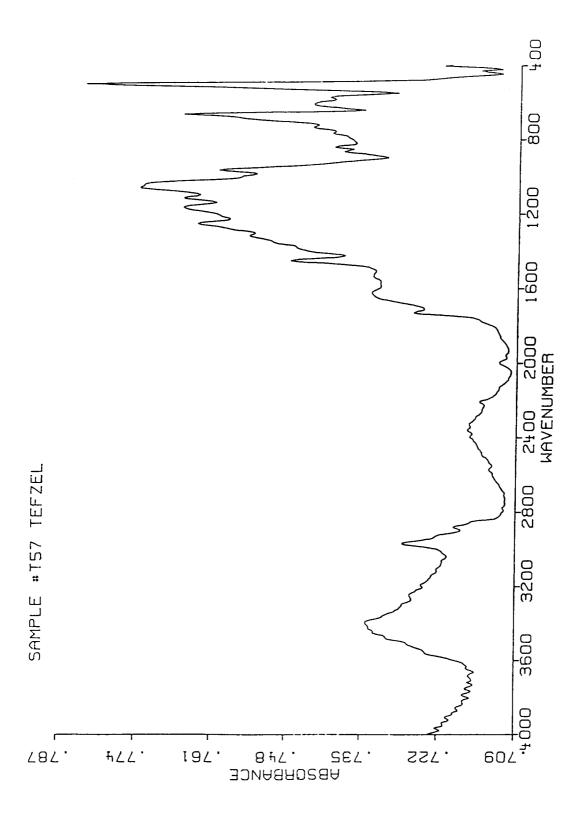
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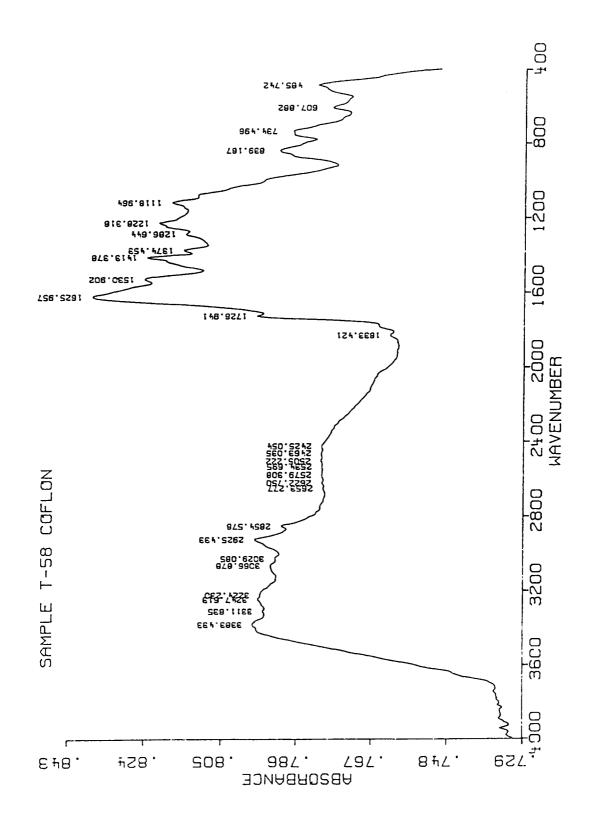


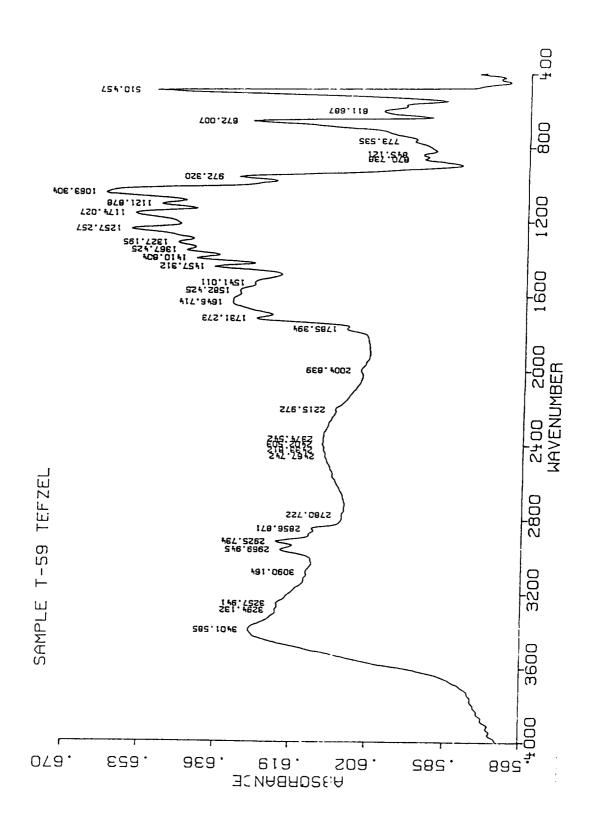




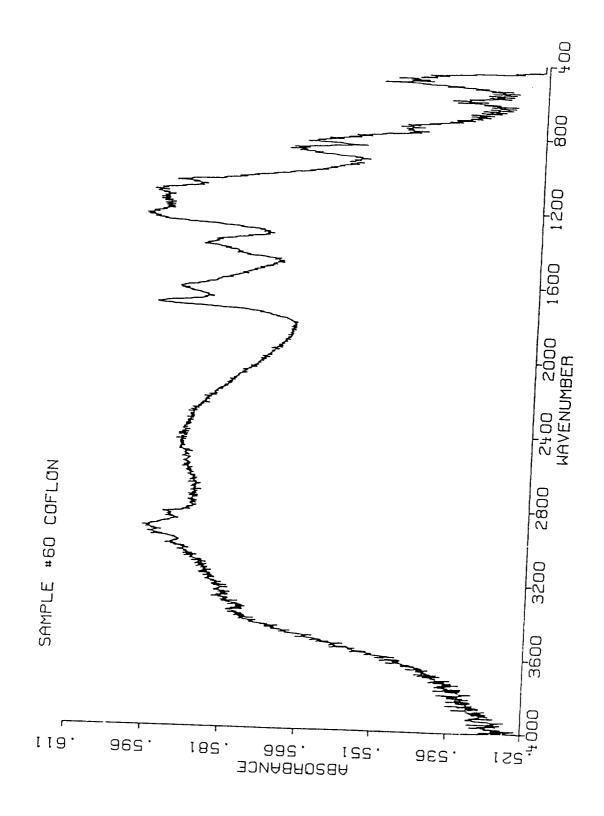




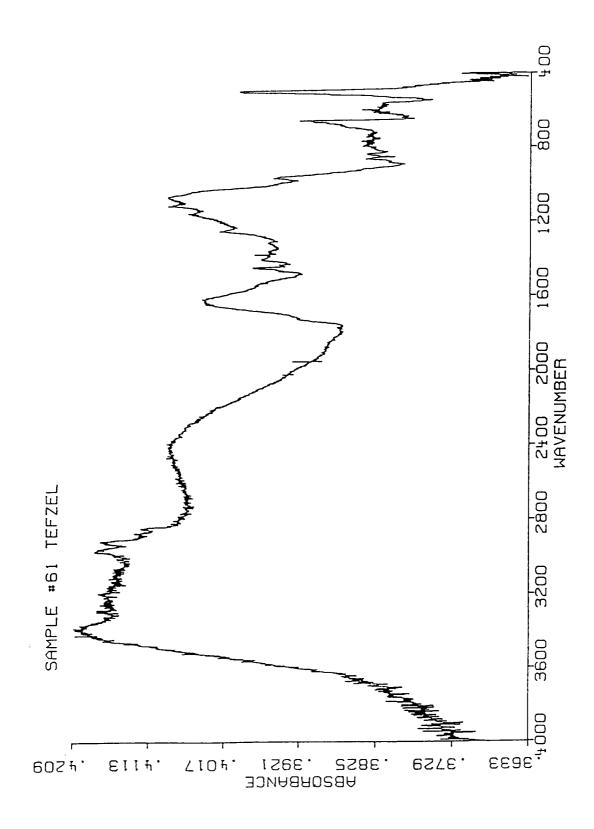


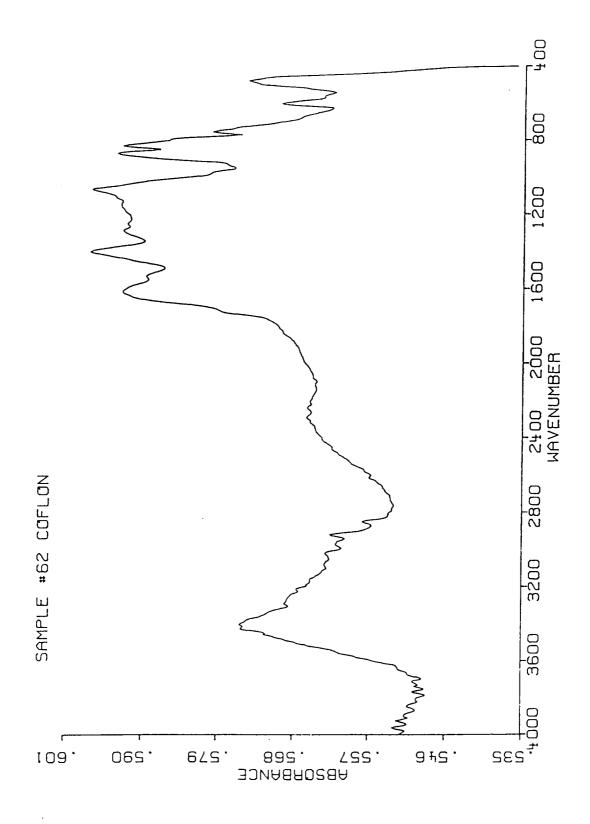


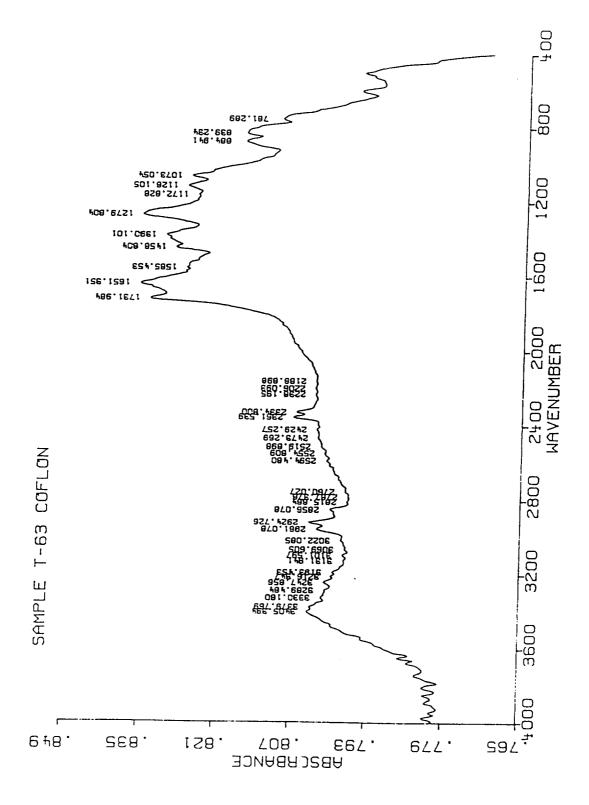
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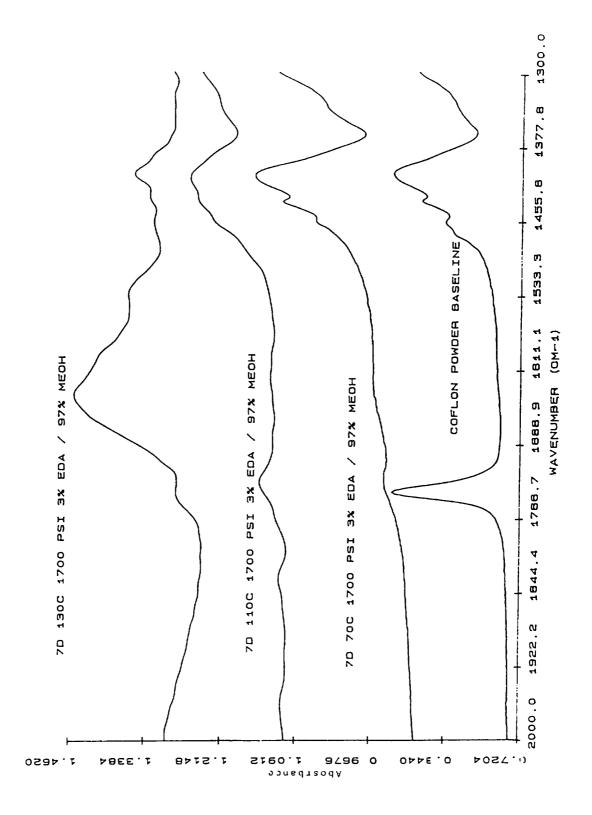
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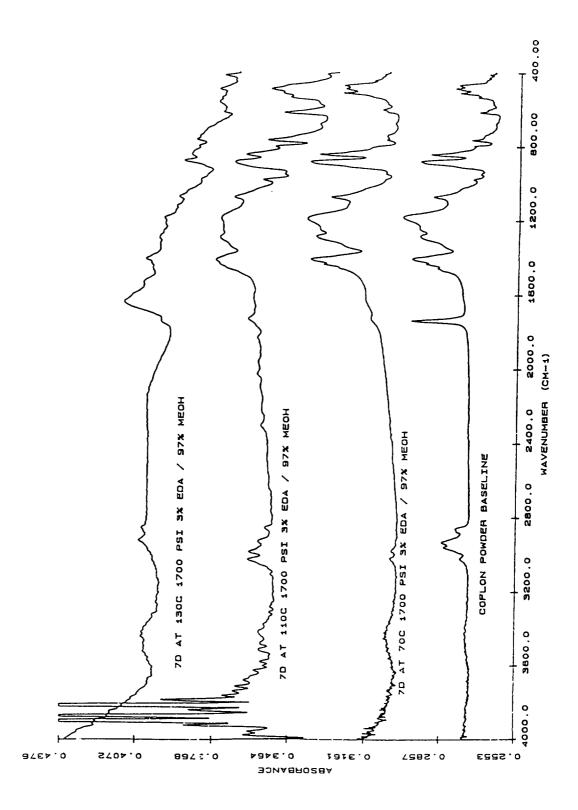


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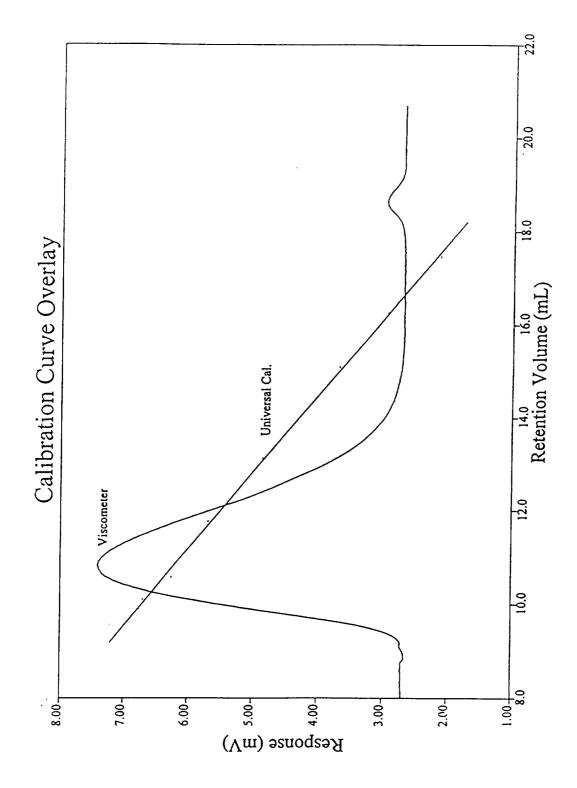
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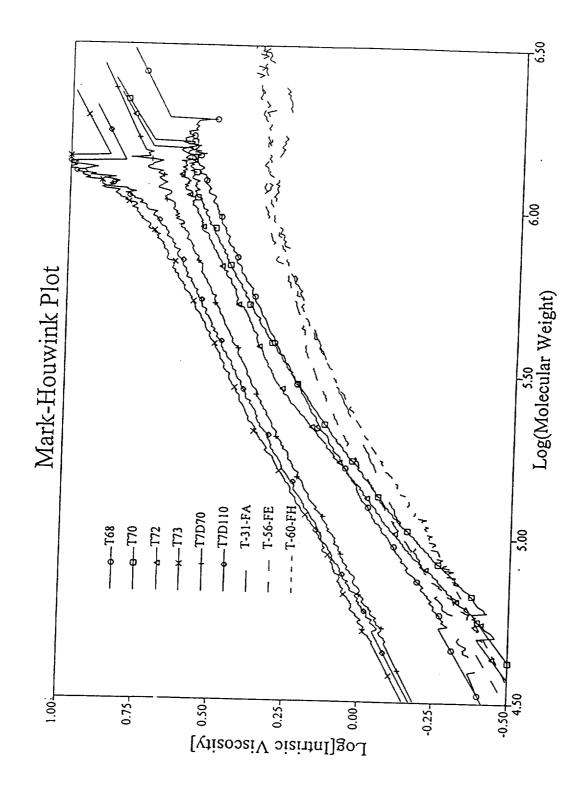
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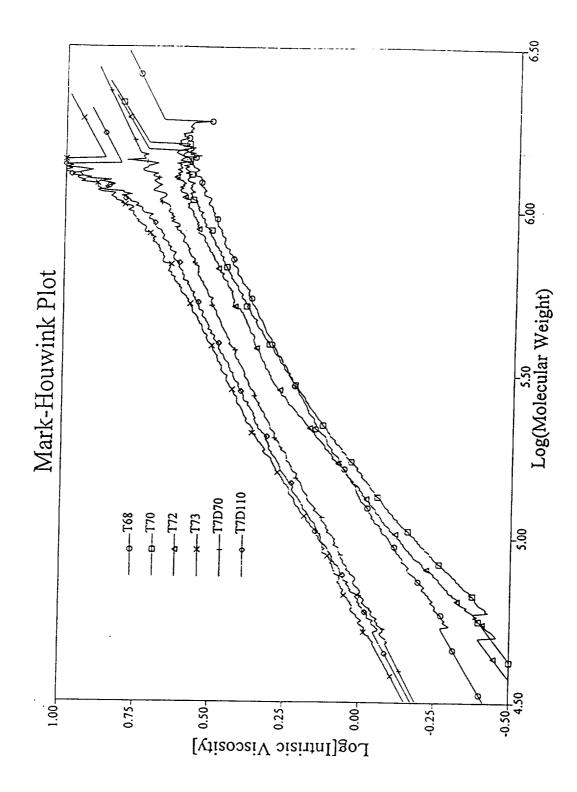


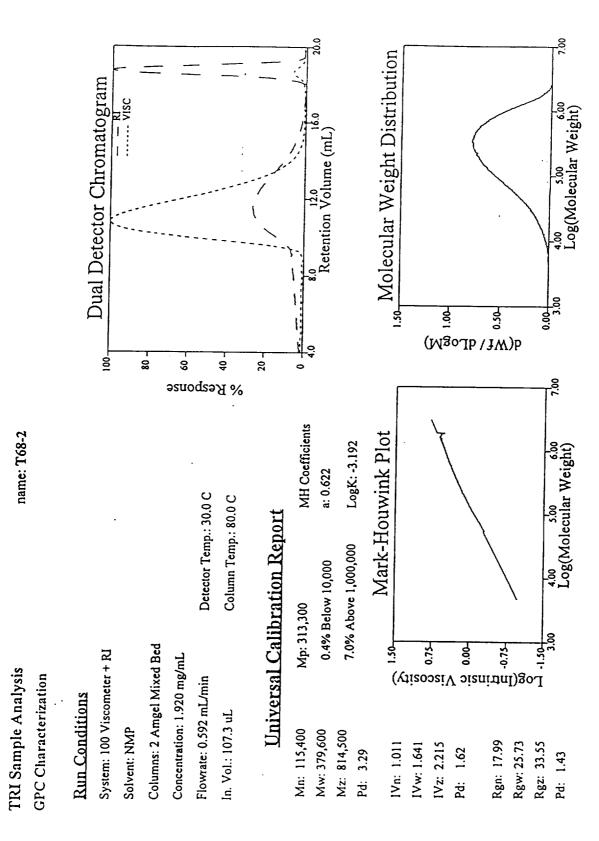
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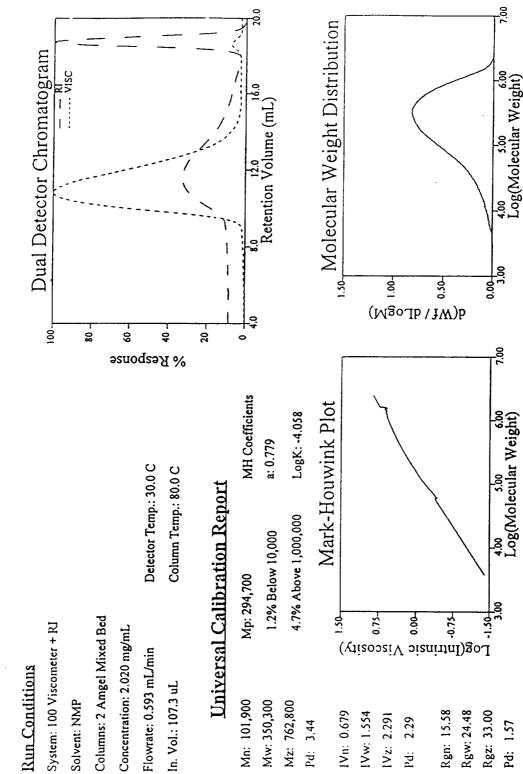
Appendix Three Gel Permeation Chromatography Results





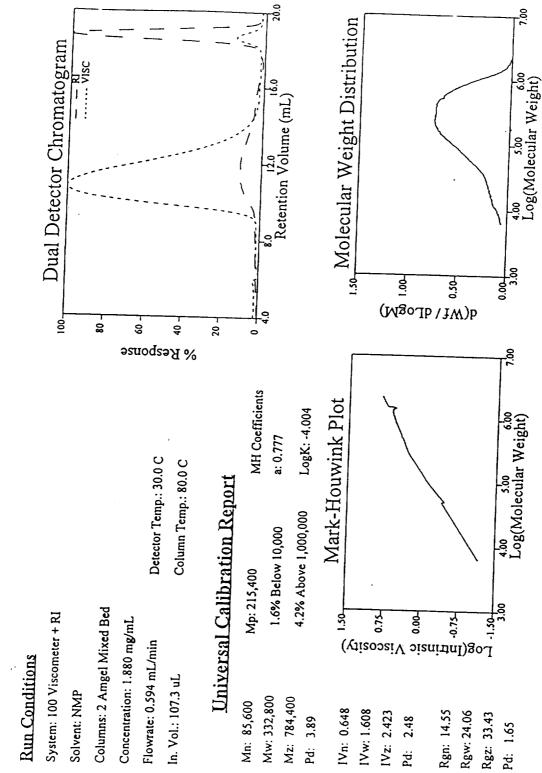






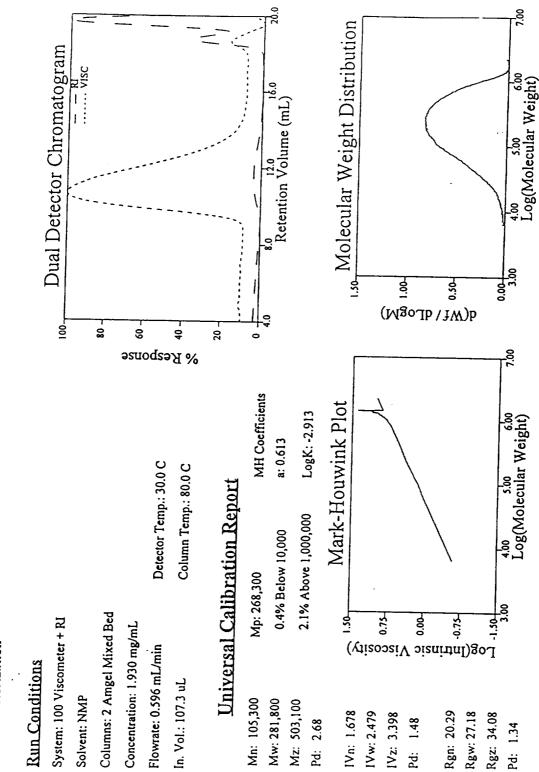
name: T70-2

TRI Sample Analysis GPC Characterization



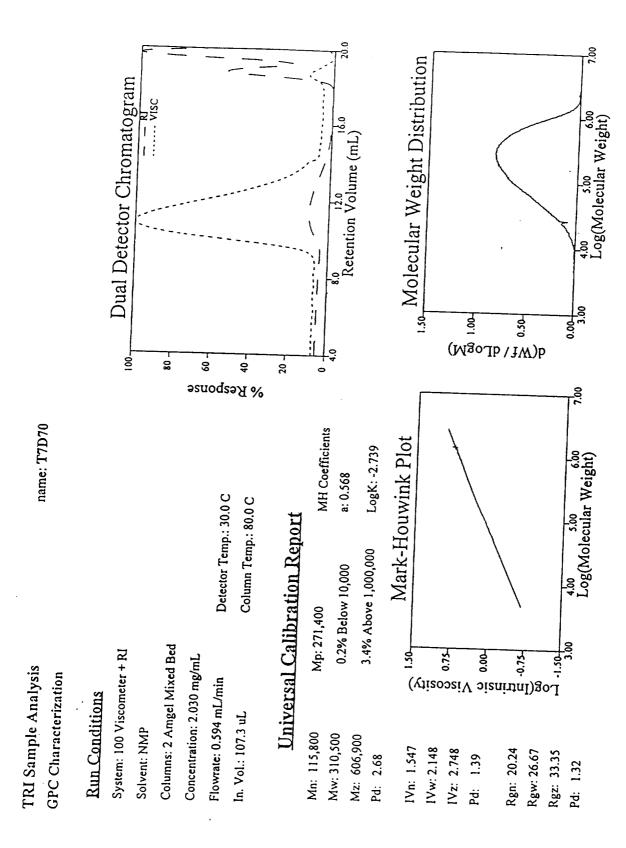
name: T72-1

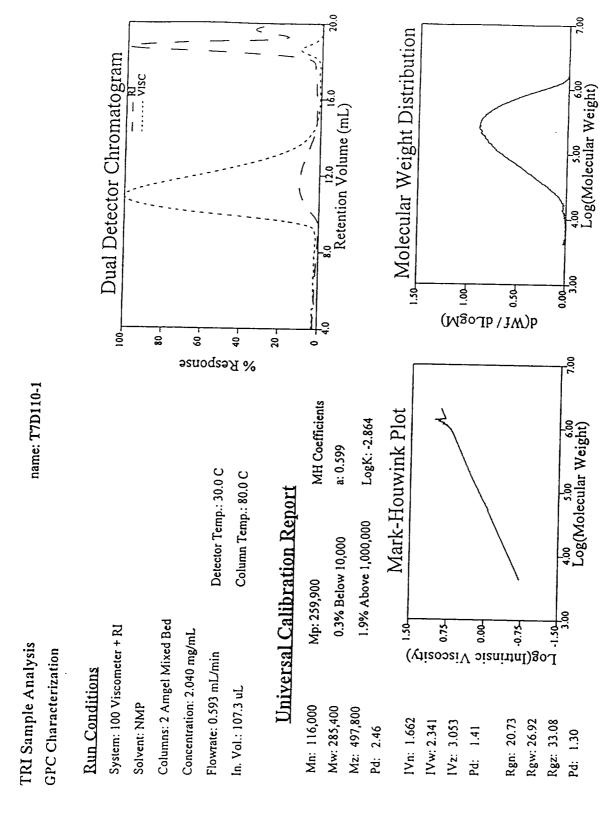
TRU Sample Analysis GPC Characterization



name: T73-1

TRI Sample Analysis GPC Characterization



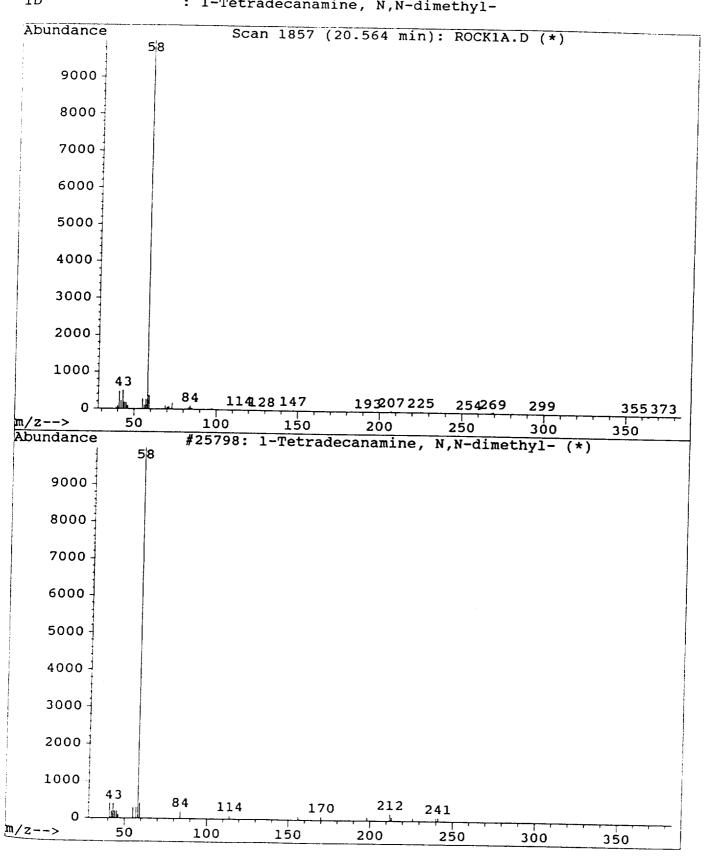


Appendix Four Gas Chromatography / Mass Spectroscopy Results

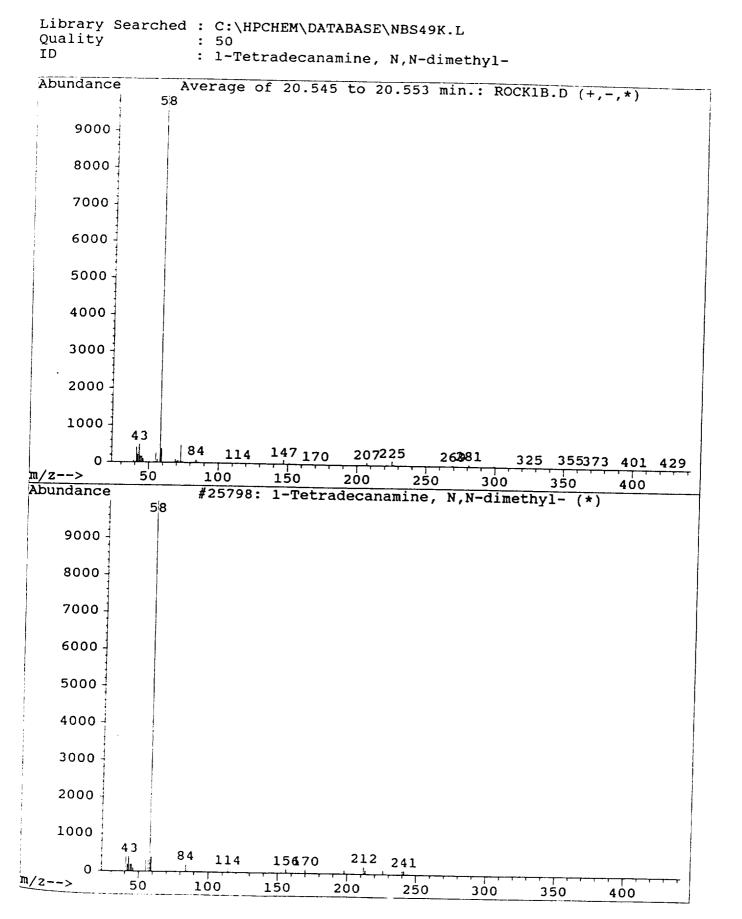
Library Search Results			Sample CP-7D70
Probable Compound Identity		Peak	Concentration
	CAS #	Time	ug/mL
ISTD 20 ng/mL		5.91	
ISTD 20 ng/mL		13.48	
unidentified		22.65	0.10
Decanedioic acid, dibutyl ester	109-46-3	24.64	1.81
unidentified		25.95	0.04
Hexanedioic acid, dioctyl ester	123-79-5	26.15	0.02
Decanedioic acid, bis(2-ethylhexyl	122-62-3	30.02	0.02

Sample CP- 7D70

Library Sea	arched :	C:\HPCHEM\DATABASE\NBS49K.L
Quality		64
ID	:	1-Tetradecanamine N N-dimethyl-

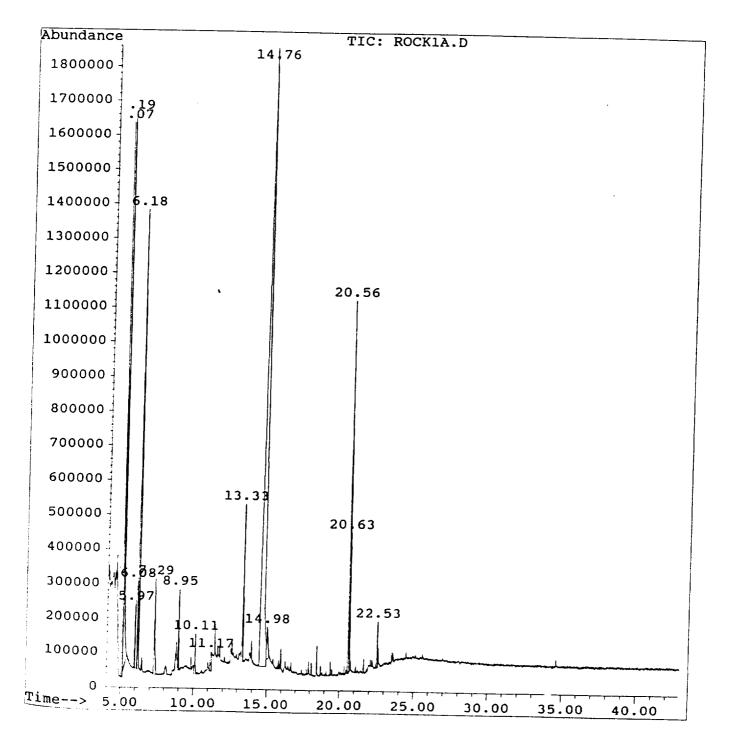


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File : C:\HPCHEM\1\DATA\MAY31PAH\ROCK1A.D
Operator : Ken McFerren
Acquired : 2 Jun 96 12:34 am using AcqMethod MEOHSCAN.M
Instrument : 5971 - In
Sample Name: EDA METHANOL 130C 1:100
Misc Info : w/ 125 ng/mL VOA Int Stnds
Vial Number: 16

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-5%

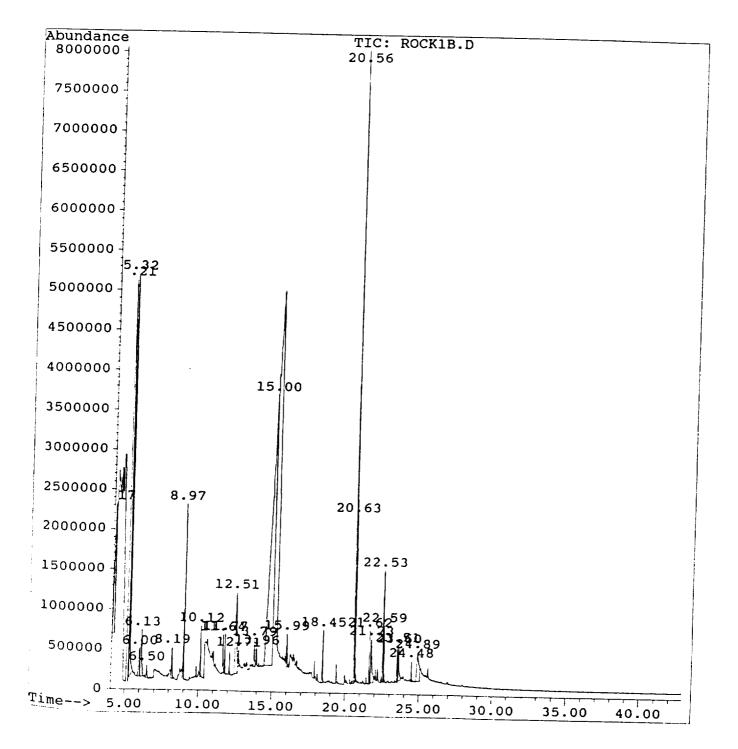
Fil Ope Acq Sam Mis	.e rator uired ple Nam	: C: : Ken : 2 ne: ED2 : W/	m Data File: \HPCHEM\1\DATA\MAY31PAH\ROCK1A.D n McFerren Jun 96 12:34 am using AcqMethod MJ A METHANOL 130C 1:100 125 ng/mL VOA Int Stnds	EOHSCAN.M
	rch Lib		C:\HPCHEM\DATABASE\WILEY.L	Minimum Quality: 50 Minimum Quality: 0
Unk Int	nown Sp egratio	ectrum n Para	n: Apex minus start of peak ms: current RTEINT parameters	
Pk#	RT	Area%	Library/ID	Ref# CAS# Qual
1	5.07	7.93	C:\HPCHEM\DATABASE\WILEY.L No matches found	
2	5.19	9.69	C:\HPCHEM\DATABASE\NBS49K.L 1-Butanol Furan, 2-propyl-	321 000071-36-3 86 2056 004229-91-8 78
3	5.97	1.43	C:\HPCHEM\DATABASE\NBS49K.L Silane, trimethoxymethyl- Silane, butyltrimethoxy- Silane, ethyltrimethoxy-	5444 001185-55-3 86 14112 001067-57-8 59 8117 005314-55-6 50
4	6.08	3.89	C:\HPCHEM\DATABASE\NBS49K.L Silane, trimethoxymethyl- Silane, trimethoxy[3-(oxiranylmeth Silane, ethyltrimethoxy-	5444 001185-55-3 83 24866 002530-83-8 53 8117 005314-55-6 45
5	6.18	11.47	C:\HPCHEM\DATABASE\NBS49K.L Silane, trimethoxy[3-(oxiranylmeth Silane, ethyltrimethoxy- Silane, (3-chloropropyl)trimethoxy	8117 005314-55-6 50
6	7.29	2.06	C:\HPCHEM\DATABASE\NBS49K.L Silicic acid (H4SiO4), tetramethyl Benzeneethanol, 2-methoxy- Benzenecarbothioic acid, O-methyl	8520 000681-84-5 93 8649 007417-18-7 38 8572 005873-86-9 32
7	8.95		C:\HPCHEM\DATABASE\WILEY.L N-Ethyl-N-methyl-4-phenetidine 1,1,2-TRIMETHOXY-1,2,2-TRIMETHYL-D 1,1,1-Trimethoxytrimethyldisilane	25603 067274-57-1 83 31649 018107-31-8 64 31651 018000-25-4 40
8	10.11		C:\HPCHEM\DATABASE\WILEY.L 2(3H)-Benzothiazolone, hydrazone 10,11-DIHYDRO-5H-DIBENZ(B,F)AZEPIN 1,1,2,2-TETRAMETHOXY-1,2-DIMETHYLD	19439 000615-21-4 43 123396 000000-00-0 43 124240 018107-32-9 37
9	11.17		C:\HPCHEM\DATABASE\NBS49K.L 1H-Pyrazole, 4,5-dihydro-3-methyl- 2H-Pyran, 3,4-dihydro-	501 001911-30-4 50 512 000110-87-2 28
)CK1	A.D		Sun Jun 02 11:02:39	1996 Page 1

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2K#	RT	Area%	Library/ID	Ref#	CAS# (Qual
10	13.33	2.58	C:\HPCHEM\DATABASE\NBS49K.L Silane, dimethoxymethylphenyl-	15032	003027-21-2	2 58
11	14.76	48.08	C:\HPCHEM\DATABASE\NBS49K.L 2-Imidazolidinone	594	000120-93-4	4 50
12	14.98	1.22	C:\HPCHEM\DATABASE\WILEY.L Ethanamine, N-butylidene- Hexanoic acid, 2-methylpropyl este Propane, 1-(ethenyloxy)-2-methyl-	122092	001611-12-7 000105-79-3 000109-53-5	36
13	20.56	5.93	C:\HPCHEM\DATABASE\NBS49K.L N,N-DIMETHYL-5-BENZYLOXYPENTYLAMIN 1-Tetradecanamine, N,N-dimethyl- 1-Hexadecanaminium, N,N,N-trimethy	25798	071126-71-1 000112-75-4 000057-09-0	64
14	20.63		C:\HPCHEM\DATABASE\WILEY.L 2-(METHYL-D3)-CYCLOUNDECANONE 2-(METHYL-D3)-CYCLODECANONE Carbamic acid, (4-chlorophenyl)-,	21221	032475-99-3 032454-56-1 002621-80-9	25
15	22.53		C:\HPCHEM\DATABASE\WILEY.L Decanedioic acid, dibutyl ester Methyl trans-7-methoxy-9-methylhex 1H-Imidazole-2-methanol	28393	000109-43-3 000000-00-0 003724-26-3	27

File : C:\HPCHEM\1\DATA\MAY31PAH\ROCK1B.D Operator : Ken McFerren Acquired : 2 Jun 96 2:41 am using AcqMethod MEOHSCAN.M Instrument : 5971 - In Sample Name: EDA METHANOL 130C 1:20 Misc Info : w/ 125 ng/mL VOA Int Stnds Vial Number: 18



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Information from Data File: File : C:\HPCHEM\1\DATA\MAY31PAH\ROCK1B.D Operator : Ken McFerren Acquired : 2 Jun 96 2:41 am using AcqMethod MEOHSCAN.M Sample Name: EDA METHANOL 130C 1:20 Misc Info : w/ 125 ng/mL VOA Int Stnds Vial Number: 18

Search Libraries:C:\HPCHEM\DATABASE\NBS49K.L
C:\HPCHEM\DATABASE\WILEY.LMinimum Quality: 50
Minimum Quality: 0Unknown Spectrum:Apex minus start of peak
current RTEINT parameters

:

Pk#	RT	Area%	Library/ID	Ref#	CAS# Ç	Qual
1	4.17	6.37	C:\HPCHEM\DATABASE\NBS49K.L Silane, dimethoxydimethyl- Silane, dimethoxymethyl-		001112-39-6 016881-77-9	
2	5.21	17.99	C:\HPCHEM\DATABASE\WILEY.L N-METHYL-D3-AZIRIDINE 1-D1-AZIRIDINE		040244-76-6 005381-40-8	
3	5.32	7.69	C:\HPCHEM\DATABASE\NBS49K.L 1-Butanol Furan, 2-propyl-		000071-36-3 004229-91-8	
4	6.00	0.98	C:\HPCHEM\DATABASE\NBS49K.L Silane, trimethoxymethyl- 1-Propanethiol, 3-(trimethoxysilyl Silane, trimethoxy{3-(oxiranylmeth	17682	001185-55-3 004420-74-0 002530-83-8	72
5	6.13	1.99	C:\HPCHEM\DATABASE\NBS49K.L Silane, trimethoxymethyl- Silane, butyltrimethoxy- 1-Propanethiol, 3-(trimethoxysilyl	14112	001185-55-3 001067-57-8 004420-74-0	64
6	6.50	0.35	C:\HPCHEM\DATABASE\NBS49K.L 1,3-Diazine	393	000289-95-2	64
7	8.19	0.76	C:\HPCHEM\DATABASE\NBS49K.L Cyclotrisiloxane, hexamethyl- Arsenous acid, tris(trimethylsilyl		000541-05-9 055429-29-3	
8	8.97	2.51	C:\HPCHEM\DATABASE\WILEY.L 1,1,2-TRIMETHOXY-1,2,2-TRIMETHYL-D 1,1,2-TRIMETHOXY-1,2,2-TRIMETHYL-D sym-Tetramethyldimethoxydisiloxane	31649	018107-31-8 018107-31-8 018187-24-1	72
9	10.12		C:\HPCHEM\DATABASE\WILEY.L 2(3H)-Benzothiazolone, hydrazone 10,11-DIHYDRO-5H-DIBENZ(B,F)AZEPIN 1,1,2,2-TETRAMETHOXY-1,2-DIMETHYLD	123396	000615-21-4 000000-00-0 018107-32-9	38

Sun Jun 02 11:32:41 1996

Page 1

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ROCK1B.D

₽k#	RT	Area%	Library/ID	Ref#	CAS#	Qua	1
10	11.64	0.67	C:\HPCHEM\DATABASE\NBS49K.L 1-Hexanol, 2-ethyl- 2-Decene, 5-methyl-, (Z)- 1-Decene, 2,4-dimethyl-	9304	000104-76- 074645-86- 055170-80-	6 43	3
11	11.77	0.50	C:\HPCHEM\DATABASE\WILEY.L 2(1H)-Quinolinone, 3-hydroxy-4-(3- 2(3H)-Oxazolethione, 4,5-diphenyl- 4-Hexen-2-yn-1-one, 1-phenyl-5-(1-	56780	014484-44- 006670-13- 029743-43-9	9 10)
12	12.51	4.02	C:\HPCHEM\DATABASE\WILEY.L Butanoic acid, 1-methylethyl ester 1-Butanamine, N-methyl-N-nitro- Butanoic acid, 3-hydroxy-, ethyl e	7696	000638-11-9 052330-07-3 005405-41-4	L 8	}
13	12.71	0.36	C:\HPCHEM\DATABASE\WILEY.L 1-TRIMETHYLSILYLOXY-1-OXO-2-(3',4'	62179	000000-00-0) 8	i
14	13.79	0.68	C:\HPCHEM\DATABASE\WILEY.L PIPERAZINTRIONE 1-METHYL-5-AMINO-D2-1,2,4-TRIAZOLE Propanal, 2-methyl-, methylhydrazo	1646	000000-00-0 053778-60-2 016713-37-4	2 38	
15	13.96	0.33	C:\HPCHEM\DATABASE\NBS49K.L Silane, ethylmethyl[[5-methyl-2-(1 Butyric acid, 4-methoxy-, trimethy Oxazole, 5-bromo-4-phenyl-	16557	074841-60-4 021273-18-7 020756-97-2	50	
16	15.00	31.48	C:\HPCHEM\DATABASE\NBS49K.L 2-Imidazolidinone 1,2-Ethanediamine, N,N,N',N'-tetra		000120-93-4 000150-77-6		
17	15.99	0.65	C:\HPCHEM\DATABASE\WILEY.L 3-HEXENE, 3,4-DIDEUTERO-2,2,5,5-TE 3-Heptanone, 5-methyl- 1-Piperazinecarboxamide, N,N-dieth	117806	000541-85-5	40	
18	18.45		C:\HPCHEM\DATABASE\WILEY.L Decanedioic acid, dimethyl ester Undecanedioic acid, dimethyl ester Cyclohexanebutanoic acid, .alpha.,	125729	000106-79-6 000000-00-0 054852-77-6	43	
19	20.56		C:\HPCHEM\DATABASE\NBS49K.L 1-Tetradecanamine, N,N-dimethyl- 1-Hexadecanaminium, N,N,N-trimethy		000112-75-4 000057-09-0		
20	20.63		C:\HPCHEM\DATABASE\WILEY.L 1-Tetradecanol 4,5-DICYANO-2-PENTADUETEROPHENYL-I 3-UNDECENE, 8-METHYL-, CIS=TRANS	31878	000112-72-1 000000-00-0 000000-00-0	22	
21	21.62		C:\HPCHEM\DATABASE\WILEY.L Trisiloxane, 1,1,1,5,5,5-hexamethy 1,1,3,3,5,5,7,7,9,9,11,11,13,13,15 Phosphonic acid, [1-[(trimethylsil	109649	000000-00-0	25	
ROCKI	LB.D		Sun Jun 02 11:33:48	1996	Pa	age	2

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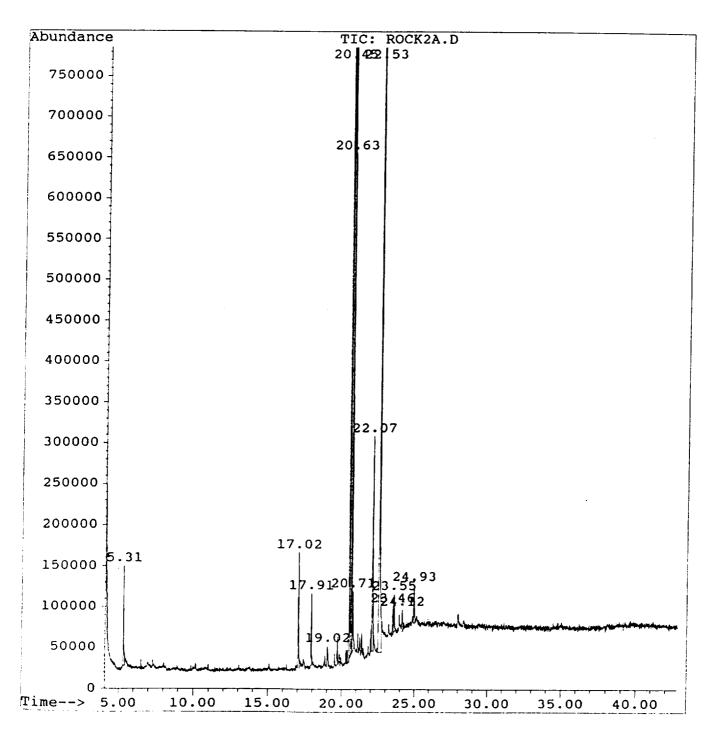
22

Pk#		Area	Dibialy/10	Ref#	CAS#	Qual
22	21.73	1.91	C:\HPCHEM\DATABASE\WILEY.L 4-Hexenoic acid, 5-amino-3-oxo-, m 1-Dodecanone, 1-cyclopropyl- Phosphonic acid, [1-[(1-methylethy	45632	052812-86-9 019873-44-0 055108-64-0	2 38
23	22.53	1.77	C:\HPCHEM\DATABASE\NBS49K.L Decanedioic acid, dibutyl ester Hydrazinecarbothioamide, 2-(2-meth Cyclopropane, 2-butyl-1,1-dichloro	15710	000109-43-3 056324-61-9 005685-44-9	18
24	22.59	0.78	C:\HPCHEM\DATABASE\WILEY.L Trisiloxane, 1,1,1,5,5,5-hexamethy 1,1,3,3,5,5,7,7,9,9,11,11,13,13-TE 1,1,3,3,5,5,7,7,9,9,11,11,13,13,15	92911	003555-47-3	38
25	23.51	0.49	C:\HPCHEM\DATABASE\WILEY.L Trisiloxane, 1,1,1,5,5,5-hexamethy VANILLYLPYRUVIC ACID 2TMS SILICATE ANION TETRAMER	87187	003555-47-3 000000-00-0 000000-00-0	27
26	23.60	1.13	C:\HPCHEM\DATABASE\WILEY.L Cyclopentanecarboxylic acid, 1-ami Pyrrolidine, 2-butyl-1-methyl- BUTYL ESTER OF N-METHYL-PROLINE	10584	000052-52-8 003447-03-8 000000-00-0	38
27	24.48		STITCARE ANTON REPRESENTS	106098 99975	054284-45-6 000000-00-0 000000-00-0	43 35
28	24.89		C:\HPCHEM\DATABASE\WILEY.L 6H-8-METHOXYINDOLO(2,3-B)QUINOXALI 2-Ethyl-4,5-diphenyloxazole 1-(1-Methylcyclohexyl)-1,2-diazeti	55176 55238 (067986-24-7 053833-30-0 080351-05-9	50 43

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File : C:\HPCHEM\1\DATA\MAY31PAH\ROCK2A.D
Operator : Ken McFerren
Acquired : 2 Jun 96 1:38 am using AcqMethod MEOHSCAN.M
Instrument : 5971 - In
Sample Name: EDA METHANOL 110C 1:100
Misc Info : w/ 125 ng/mL VOA Int Stnds
Vial Number: 17



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Information from Data File: : C:\HPCHEM\1\DATA\MAY31PAH\ROCK2A.D File : Ken McFerren Operator Acquired : 2 Jun 96 1:38 am using AcqMethod MEOHSCAN.M Sample Name: EDA METHANOL 110C 1:100 Misc Info : w/ 125 ng/mL VOA Int Stnds Vial Number: 17 Search Libraries: C:\HPCHEM\DATABASE\NBS49K.L Minimum Quality: 50 C:\HPCHEM\DATABASE\WILEY.L Minimum Quality: 0 Unknown Spectrum: Apex minus start of peak Integration Params: current RTEINT parameters Pk# RT Area% Library/ID Ref# CAS# Qual 1 5.31 0.57 C:\HPCHEM\DATABASE\WILEY.L No matches found 0.78 C:\HPCHEM\DATABASE\NBS49K.L 2 17.02 Undecane, 2,3-dimethyl-15585 017312-77-5 78 Hexadecane 23535 000544-76-3 72 Tridecane 15584 000629-50-5 59 3 17.91 0.43 C:\HPCHEM\DATABASE\NBS49K.L 5-Eicosene, (E)-31393 074685-30-6 58 1-Hexadecanol 25992 036653-82-4 52 9-Eicosene, (E)-31392 074685-29-3 52 19.02 0.21 C:\HPCHEM\DATABASE\WILEY.L 4 No matches found 5 5.36 C:\HPCHEM\DATABASE\NBS49K.L 20.45 2-Propenamide, 2-methyl-N-phenyl-10662 001611-83-2 52 Dodecane, 1-chloro-19373 000112-52-7 50 Tritetracontane 48304 007098-21-7 50 20.57 63.26 C:\HPCHEM\DATABASE\NBS49K.L 6 Cyclohexaneethanamine, N,.alpha.-d 9445 000101-40-6 80 1-Hexadecanaminium, N,N,N-trimethy 39973 000057-09-0 47 Silacyclopentane 632 000288-06-2 43 7 20.63 2.62 C:\HPCHEM\DATABASE\WILEY.L Decanedioic acid, dimethyl ester 47856 000106-79-6 37 Decanedioic acid 123842 000111-20-6 17 Decanedioic acid 123840 000111-20-6 16 8 20.71 0.56 C:\HPCHEM\DATABASE\NBS49K.L 2-Naphthalenol, 8-amino-10246 000118-46-7 50 Tetradecanoic acid, 12-methyl-, me 28100 005129-66-8 39 9-Octadecenoic acid, 12-(acetyloxy 39276 000140-03-4 38 9 22.07 1.77 C:\HPCHEM\DATABASE\WILEY.L N, N-DIMETHYL-TRIDECYLAMINE 46844 000000-00-0 72 **V,N-DIMETHYL-HENEICOSANYLAMINE** 76288 00000-00-0 72 N,N-DIMETHYL-UNDECYLAMINE 34458 00000-00-0 72

ROCK2A.D

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Page 1

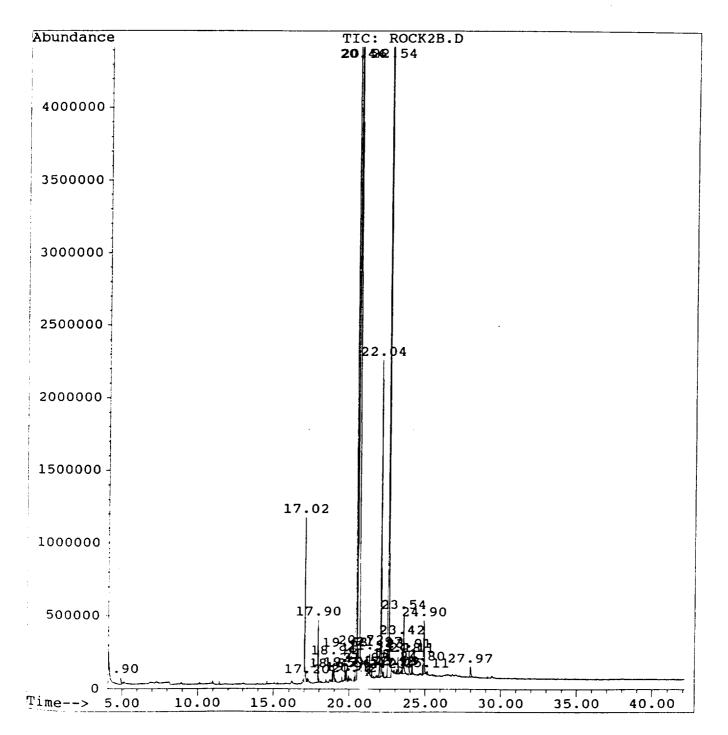
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Pk;		Area	LIDEARY/ID	Ref#	
10	22.53	23.4	5 C:\HPCHEM\DATABASE\NBS49K.L	Rel#	CAS# Qual
11			Cyclohexane Cyanic acid, 2-methylpropyl ester	209	00C109-43-3 72 000110-82-7 14 001768-25-8 14
		0.20	C:\HPCHEM\DATABASE\WILEY.L		- 11
			N,N-DIMETHYL-HEPTADECYLAMINE N,N-DIMETHYL-PENTADECYLAMINE N,N-DIMETHYL-TRIDECYLAMINE	3/020	000000-00-0 72 000000-00-0 56
12	23.55	0.32		46844	000000-00-0 56
		_	C:\HPCHEM\DATABASE\WILEY.L 1-Butanamine, N-(1-methylethyl)- N-ETHYL-N-METHYL-3-BUTENYLAMINE Butanal	3/67	039099-23-5 58 000000-00-0 42
13	24.12	0.16	C:\HPCHEM\DATABASE\WILEY.L	11400/	000123-72-8 42
1.4			Hexanedioic acid, dioctyl ester Hexanedioic acid, dioctyl ester Hexanedioic acid, dioctyl ester	300/9	000123-79-5 47 000123-79-5 43 000123-79-5 38
14	24.93	0.32	C: \HPCHEM\DATABASE\ MEG (300123-79-5 38
			N,N-DIMETHYL-3-PHENOXYPROPYLAMINE 1-Hexanamine, 2-ethyl-N,N-dimethyl 1-Tetradecanamine, N,N-dimethyl-	9854 C)20904-57-8 59)28056-87-3 39)00112-75-4 39

ROCK2A.D

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File : C:\HPCHEM\1\DATA\MAY31PAH\ROCK2B.D Operator : Ken McFerren Acquired : 2 Jun 96 3:45 am using AcqMethod MEOHSCAN.M Instrument : 5971 - In Sample Name: EDA METHANOL 110C 1:20 Misc Info : w/ 125 ng/mL VOA Int Stnds Vial Number: 19



Fi Op AC Sau Mis	le erator quired mple Nam	: C : Ke : 2 me: EI : W/	om Data File: :\HPCHEM\1\DATA\MAY31PAH\ROCK2B.D en McFerren 2 Jun 96 3:45 am using AcqMethod M DA METHANOL 110C 1:20 / 125 ng/mL VOA Int Stnds	IEOHSCAN.	. M	
	arch Lib		C:\HPCHEM\DATABASE\WILEY.L	Minim Minim	uum Quality uum Quality	7: 50 7: 0
Unk Int	nown Sp egratic	on Par	m: Apex minus start of peak ams: current RTEINT parameters			
Pk#		Area		Ref#	CAS#	Qual
1	4.90	0.1	4 C:\HPCHEM\DATABASE\NBS49K.L 1-Butanol Cyanic acid, propyl ester	321	000071-36 001768-36	-3 64
2	17 00	1 0	Pentane, 1-bromo-3,4-dimethyl-	14111	006570-92	-1 56 -9 36
2	17.02	1.94	4 C:\HPCHEM\DATABASE\NBS49K.L Decane, 2,3,6-trimethyl- Undecane, 5-ethyl- Tetratetracontane	15631	062238-12 017453-94- 007098-22	-0 72
3	17.20	0.08	C:\HPCHEM\DATABASE\NBS49K.L N,N-DIMETHYL-3-PHENOXYPROPYLAMINE 1,3-Dioxolane-4,5-dimethanamine, N Propanal	14433 21533	020904-57- 074764-43- 000123-38-	-8 50 -5 50
4	17.90	0.70	C:\HPCHEM\DATABASE\NBS49K.L 1-Hexadecanol 5-Octadecene, (E)- 5-Eicosene, (E)-	25992 27498	036653-82- 007206-21- 074685-30-	4 83 5 80
5	18.85	0.14	C:\HPCHEM\DATABASE\NBS49K.L Iron, tricarbonyl[N-(phenyl-2-pyri Decane, 2,3,5-trimethyl- Octane, 2,4,6-trimethyl-	42337 (15638 (772372
6	18.94	0.48	C:\HPCHEM\DATABASE\NBS49K.L 1-Tetradecanamine, N,N-dimethyl-		000112-75-	
7	19.68	0.31	C:\HPCHEM\DATABASE\NBS49K.L Undecane, 3-methyl- Pentadecane Undecane, 2,7-dimethyl-	12793 (21020 (001002-43-3 000629-62-9 017301-24-5	372 964
8	19.79		C:\HPCHEM\DATABASE\WILEY.L 1-Tetradecanamine, N,N-dimethyl- 1-Octadecanamine, N,N-dimethyl- N,N-DIMETHYL-TRIDECYLAMINE	52350 0 71989 0	00112-75-4 00124-28-7 00000-00-0	50 50

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	# RT	Area		Ref#	CAS#	Qual
	9 19.9	93 0.0	8 C:\HPCHEM\DATABASE\NBS49K.L Decanoyl chloride	16609		
			Nonanoic acid, 7-methyl-, methyl e Dodecanoic acid, 10-methyl-, methy	12365	3 000112-13 3 005129-63 2 005129-65	3-5 56
10	0 20.3	2 0.0	C:\HPCHEM\DATABASE\NBS49K.L			
			2-Propenoic acid, 2-methyl-, decyl Cyclobutanecarboxylic acid, 2-meth Cyclohexane, 1,2,3-trimethyl- (1		003179-47	'-3 56
			$\gamma = \gamma = \gamma = \gamma = \alpha = \alpha = \alpha = \alpha = \alpha = \alpha = $	4301 4032	014132-44 007667-55	-6 42 -2 35
11	20.4	4 12.25	C:\HPCHEM\DATABASE\NBS49K.L Tritetracontane	48304	007000 01	.
			Decane, 2,4,6-trimethyl-	15627	007098-21 062108-27	-7 50
12	20.56	5 35 00	1-Decanol, 2-ethyl-	16022	021078-65	-9 43
	20.50	5 55.09	C:\HPCHEM\DATABASE\NBS49K.L			
			Cyclohexaneethanamine, N,.alphad 1-Hexadecanaminium, N,N,N-trimethy	9445	000101-40	-6 80
1.5	00 50		eyerobucane, metnoxy-		000057-09- 018593-33-	-0 47 -4 40
13	20.72	0.20	C:\HPCHEM\DATABASE\NBS49K.L			
			rencauecanolc acid, methyl ester	28101	007132-64-	1 64
			2 haphchalenol, 8-amino-	10246	000118-46-	7 64
	ı		Cyclopentaneundecanoic acid, methy	29784	025779-85-	5 56
14	21.07	0.15	C:\HPCHEM\DATABASE\WILEY.L			
			UNDECANAL DIMETHYL ACETAL	41814	000000-00-	0 64
			Dodecane, 1,1-dimethoxy- DECANAL DIMETHYL ACETAL	48014	014620-52-	1 50
			A A A A A A A A A A A A A A A A A A A	35668	000000-00-	0 39
15	21.18		C:\HPCHEM\DATABASE\WILEY.L No matches found			
16	21.34	0 27				
	21.34	0.27	C:\HPCHEM\DATABASE\WILEY.L			
			N,N-DIMETHYL-UNDECYLAMINE N,N-DIMETHYL-HEPTADECYLAMINE	34458 (00000-00-0) 64
			N N-DIMERUVI DENER	67476 ()00000-00-c) 56
17	21 47			57626 0	00000-00-0	50
-,	21.4/	0.08	C:\HPCHEM\DATABASE\WILEY.L 2-Dodecanol			
			-Pentadecanol -	28894 0	10203-28-8	43
		e	5,6-(D2)MENTH-2-ENE	25135 0	00629-76-5	42
18	21 07			9703 0	00000-00-0	41
-0	21.97	0.42 (C:\HPCHEM\DATABASE\WILEY.L			
		Г .	Pentadecane 13	80188 0	00630-06-8	45
		T	letradecano 12	24388 0	00629-62-9	43
19	22 -		12	3696 00	00629-59-4	30
• 3	22.04	Ť	:\HPCHEM\DATABASE\NBS49K.L -Tetradecanamine, N,N-dimethyl- 2	5700 01		
•		1	-Hexadecanaminium, N,N,N-trimethy 3	9973 OC	00112-75-4	78
20	22.23	0.08 C	: \ HPCHEM \ DATABASE \ WITEV T			
		0	ecanedioic acid 3 6-dimethicit i a	0 ()		
]	1,1'-Biphenyl]-4-ol, 3-amino- 28	8380 00	1134-36-7	25
50CK51	B.D	Me	12F	5252 00	0064-55-1	22
	-		Sun Jun 02 11:48:03 19	996		14 ge 2
						y - 4

Pk#	RT	Area	<pre>% Library/ID</pre>	Ref#	CAS#	Qual
21	22.5	4 39.3	7 C:\HPCHEM\DATABASE\NBS49K.L			
			Decanedioic acid, dibutyl ester	3544	6 000109-43	
			Cyclonexanone, 4-methyl-, thiosemi	1671	1 022397 - 22	-3 90
			1,2,4-Trioxolane-2-octanoic acid,		1 055398-23	-4 12
22	22.93	2 0 07				-/ 12
22	22.9.	5 0.07	C:\HPCHEM\DATABASE\WILEY.L			
			Silane, trimethyl(1-methyl-1-prope	e 6831	010111-13	-4 11
			SILANE, 2-BUTENYLTRIMETHYL-, TRANS	6833	3 000000-00	-0 10
			3-Dodecanol, 3,7,11-trimethyl-	47378	007278-65	-1 10
23	23.09	0.09	C:\HPCHEM\DATABASE\NBS49K.L			
			Cyclopentanemethylamine, 2-isoprop			
			2-Butanone, 4-(dimethylamino)-3-me		017943-85-	-0 53
			Benzeneethanamine, N,.alphadimet		022104-62-	-7 45
			all all and a set and a set all all all all all all all all all al	25083	054833-35-	-1 42
24	23.18	0.28	C:\HPCHEM\DATABASE\WILEY.L			
			Uridine, 2'-O-methyl-	58449	002140-76-	2 4 2
			2-Nonadecanone, O-methyloxime	76263	036379-39-	· 3 40
			N, N-DIMETHYLSTEARAMIDE	76265	000000-00-	2 38
25	<u> </u>	o				0 12
20	23.42	0.45	C:\HPCHEM\DATABASE\NBS49K.L			
			1-Hexanol, 6-(dimethylamino)-	7329	001862-07-	3 72
			1-Tetradecanamine, N,N-dimethyl-	25798	000112-75-	4 56
26	23.54	1.00	C:\HPCHEM\DATABASE\WILEY.L			
			1-Butanamine, N-(1-methylethyl)-			
			1,1,3-TRIMETHYL-1-SILACYCLOBUTANE	4234	039099-23-	5 52
			3,3,5,5-D4-CIS-1,2-CYCLOHEXANEDIOL	3999	002295-13-	840
			, , , , , , , , , , , , , , , , , , ,	4451	053897-44-2	240
27	23.91	0.37	C:\HPCHEM\DATABASE\WILEY.L			
			Cyclononanone	119126	003350-30-9	
			-/- CICIOPENLANEUIONE, 4-1SOpontvl	20912	00000-30-30-9	# 50 5 ac
			Hexadecanoic acid	126168	000057-10-3	0 J 5
28	24.11	0 95			000007 10-2	5 35
20	24.11	0.25	C:\HPCHEM\DATABASE\NBS49K.L			
			HEXANEDIOIC ACID, MONO(2-ETHYLHEXY	28340	004337-65-9	64
			Hexanedioic acid, dioctyl ester	40566	000123-79-5	45
		-	Hexanedioic acid, dicyclohexyl est	34998	000849-99-0	32
29	24.80	0.16	C:\HPCHEM\DATABASE\WILEY.L			
			Methyl trans-7-methoxy-9-methylhex			
]	LAURIC ACID, N-OCTYL ESTER	28393 (00000-00-0	35
		1	Decane, 1-(ethenyloxy)-	/6668 (00000-00-0	30
2.0				28060 (000765-05-9	27
30	24.90	0.89 (C:\HPCHEM\DATABASE\WILEY.L			
		· N	N-DIMETHYL-HEPTADECYLAMINE	67476 0	00000-00-0	70
		Ν	N-DIMETHYL-HENEICOSANYLAMINE	76288 0	00000-00-0	72
		N	,N-DIMETHYL-PENTADECYLAMINE	57626 0	00000-00-0	12
31	75 11					oc
	25.11	U.07 C	:\HPCHEM\DATABASE\WILEY.L			
		P	Pyridine, 3-(1-methyl-2-pyrrolidin	31850 0	02055-29-0	12
		2	-ryrroilainone, 1-(2-0x)-4-(1-ny)	45364 0	35022-19-6	12
		В	utanoic acid, 3,4-dihydroxy-2-met	7682 0	24923-78-2	1.2

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Page 3

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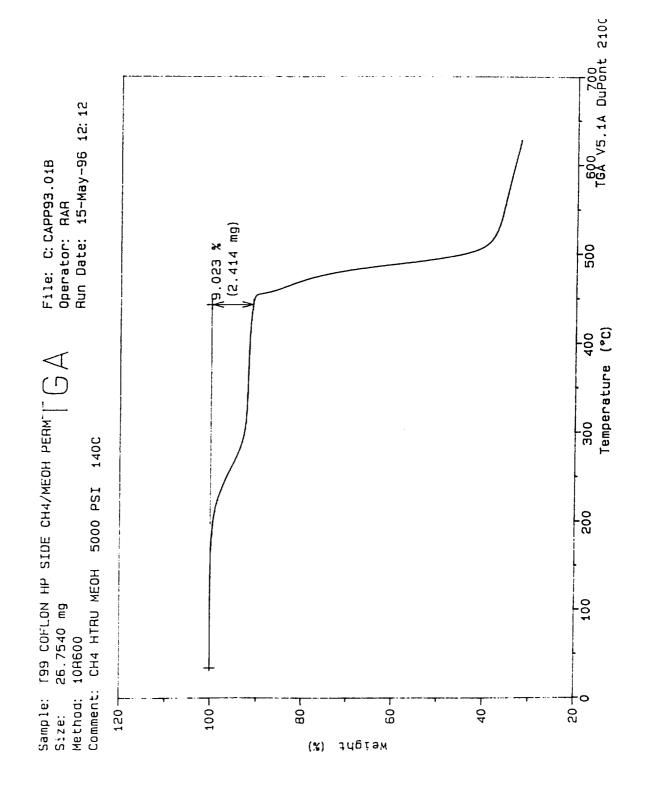
Appendix Five Thermal Analysis Results

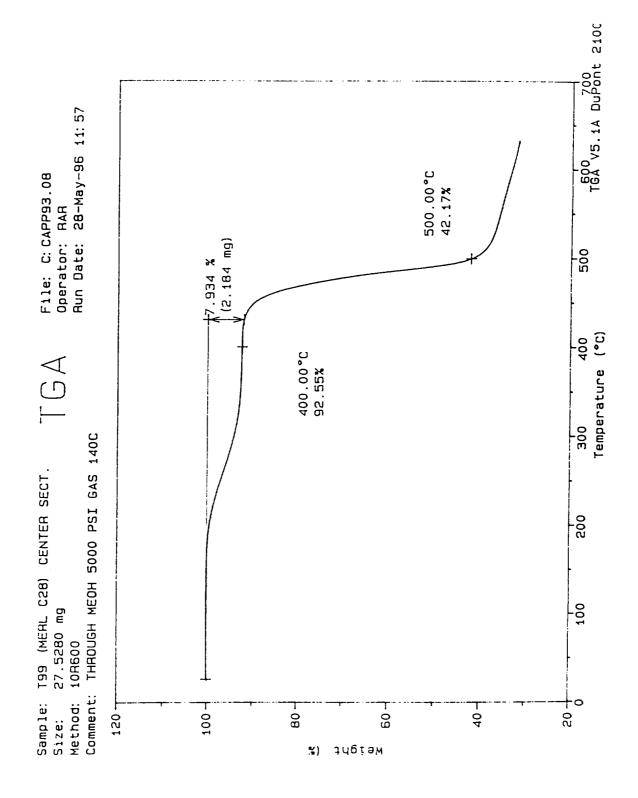
	RT	Area%	Library/ID	Ref#	CAS# Qual
32	27.97		C:\HPCHEM\DATABASE\WILEY.L Decanedioic acid, bis(2-ethylhexyl 2-Nonanol, 5-ethyl- N-ISO-BUTYL BARBITAL	122138	

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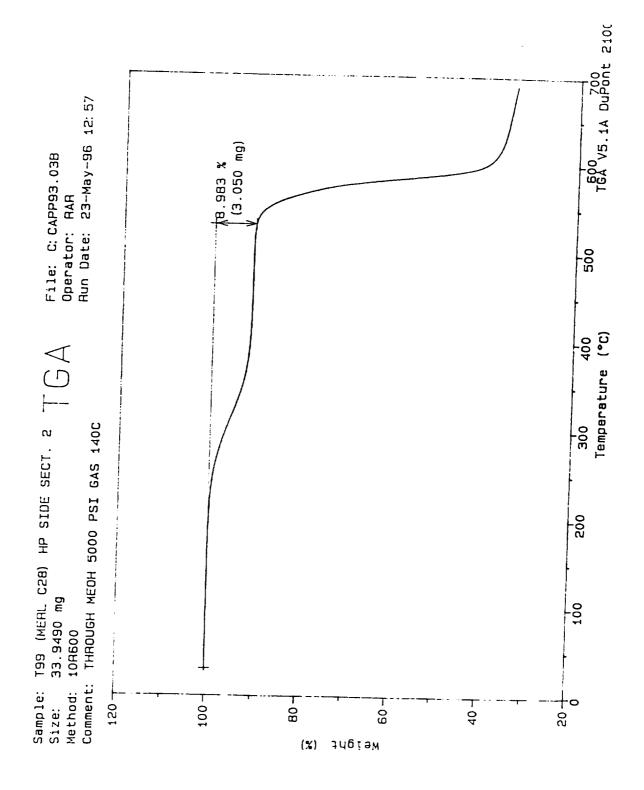
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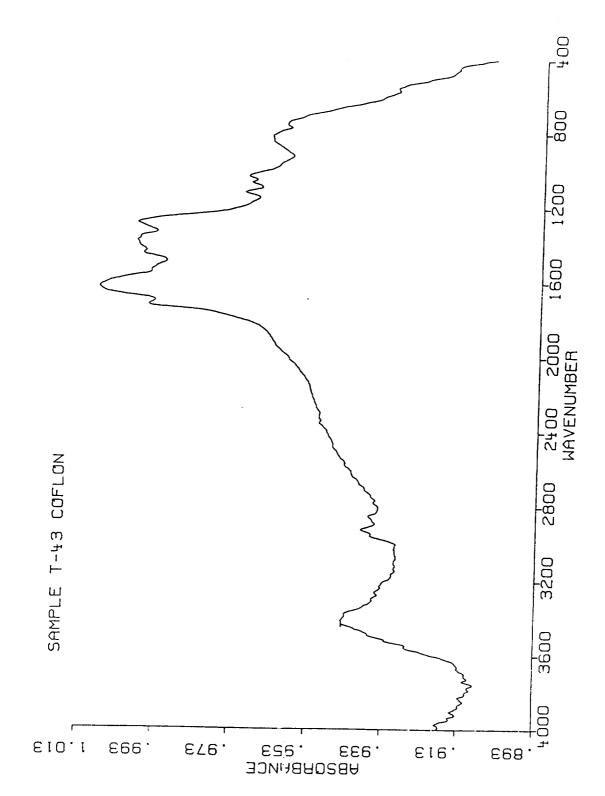
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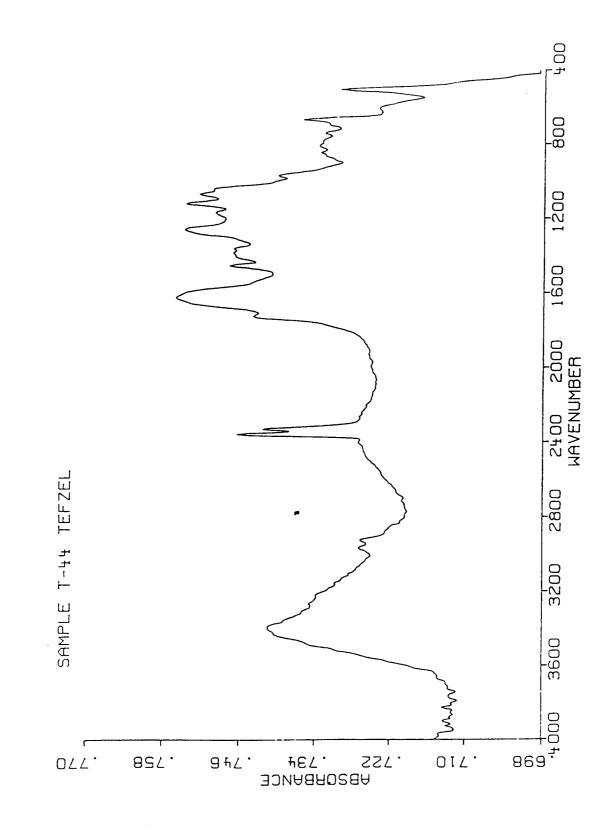


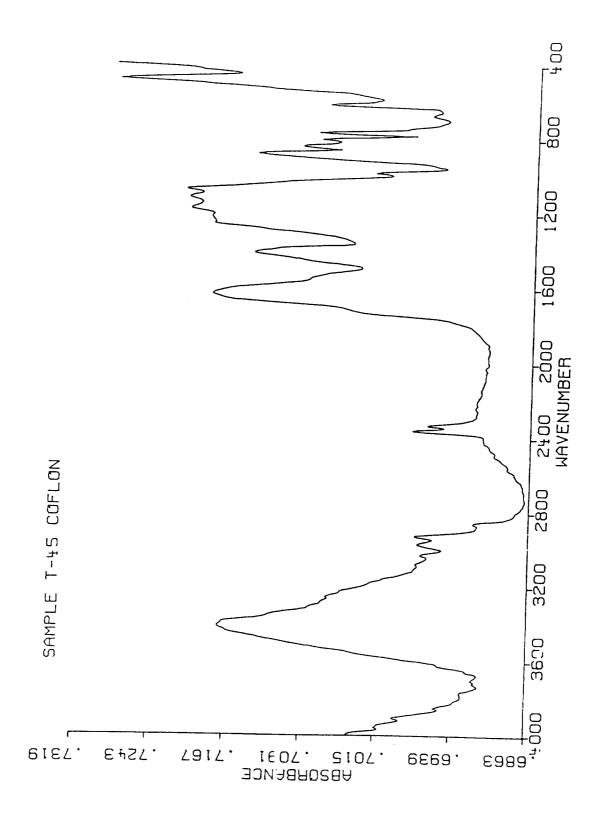


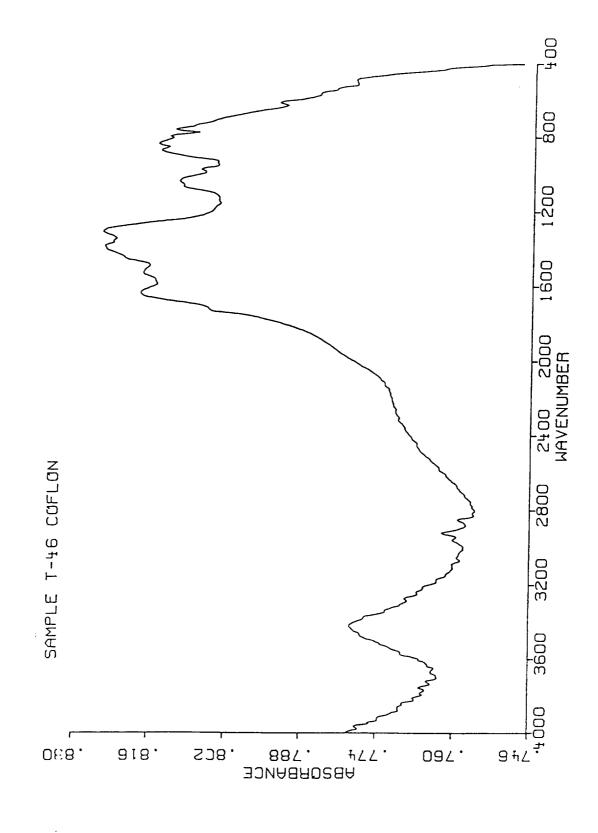
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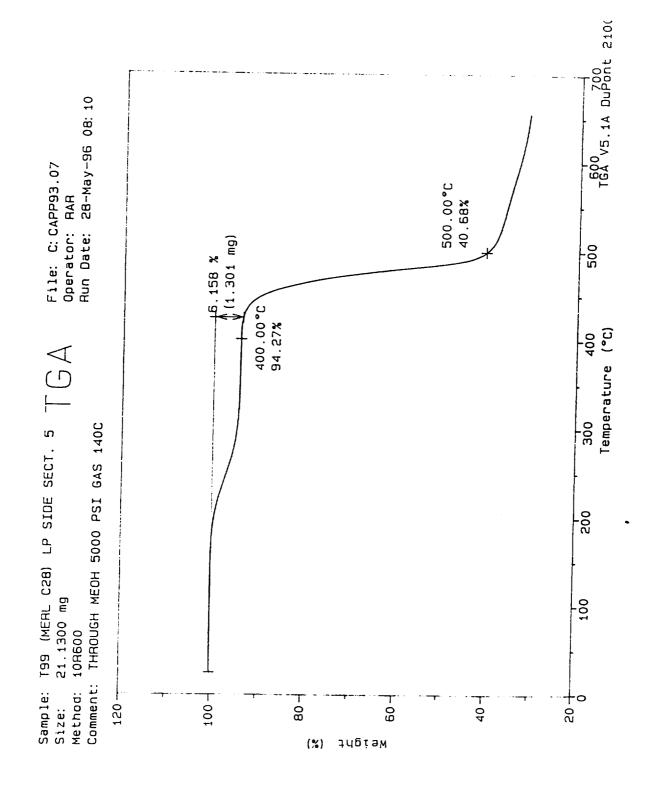


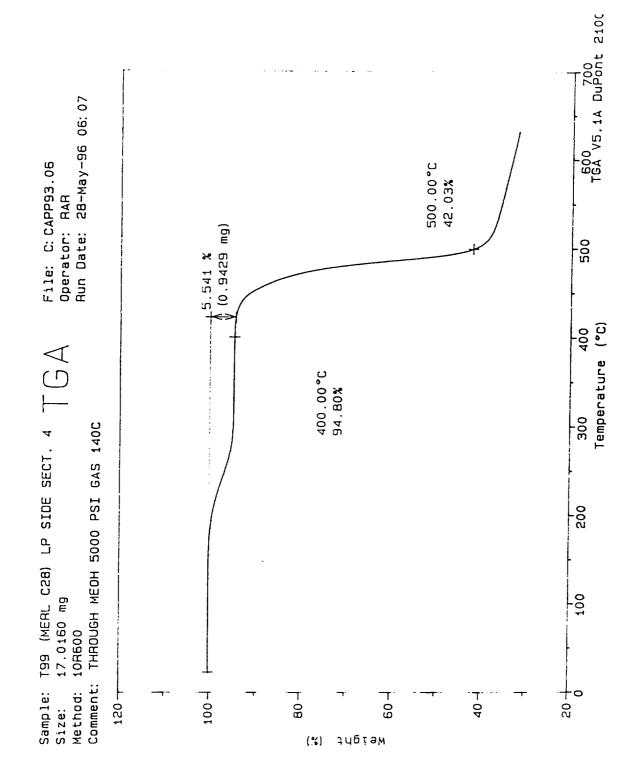






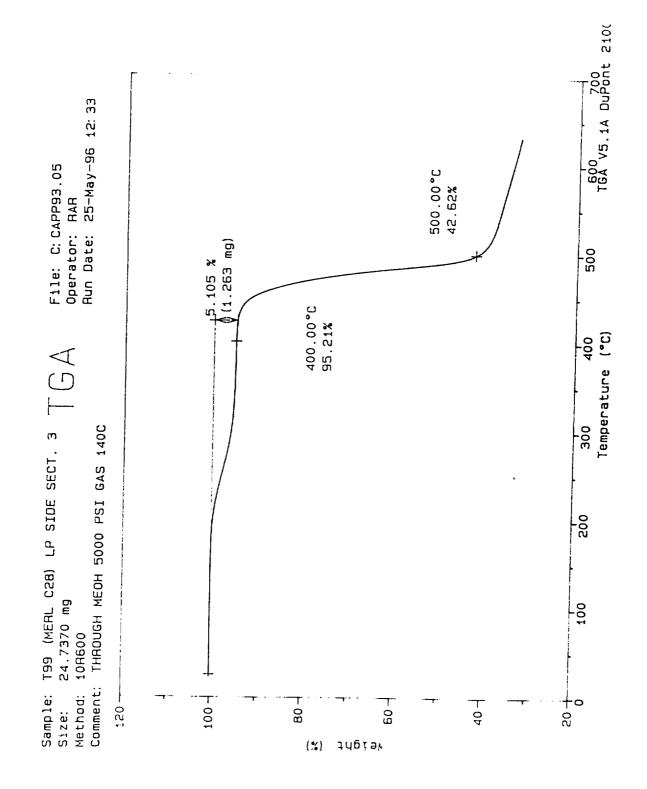
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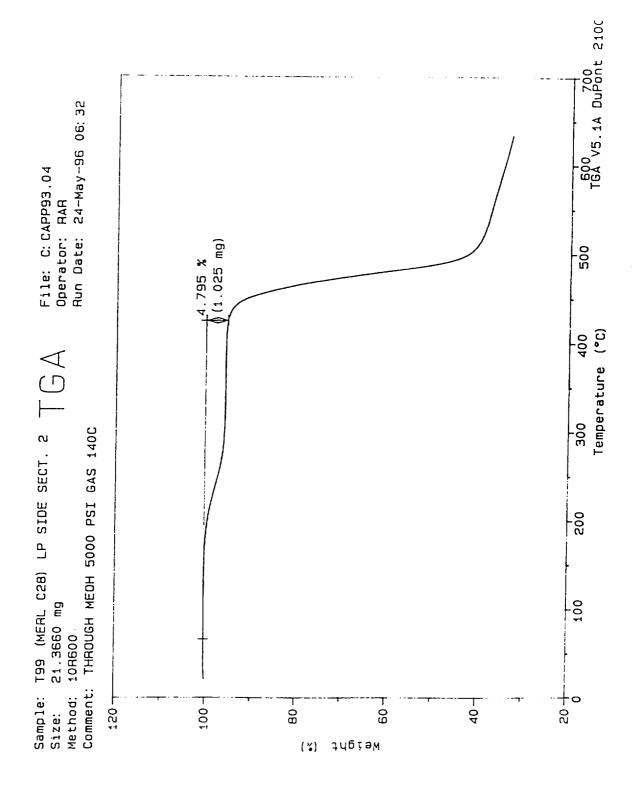




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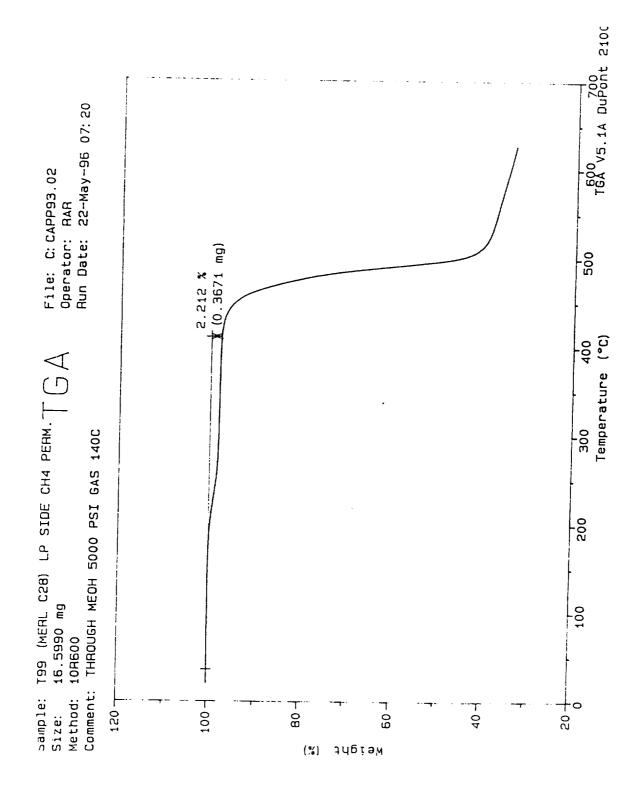


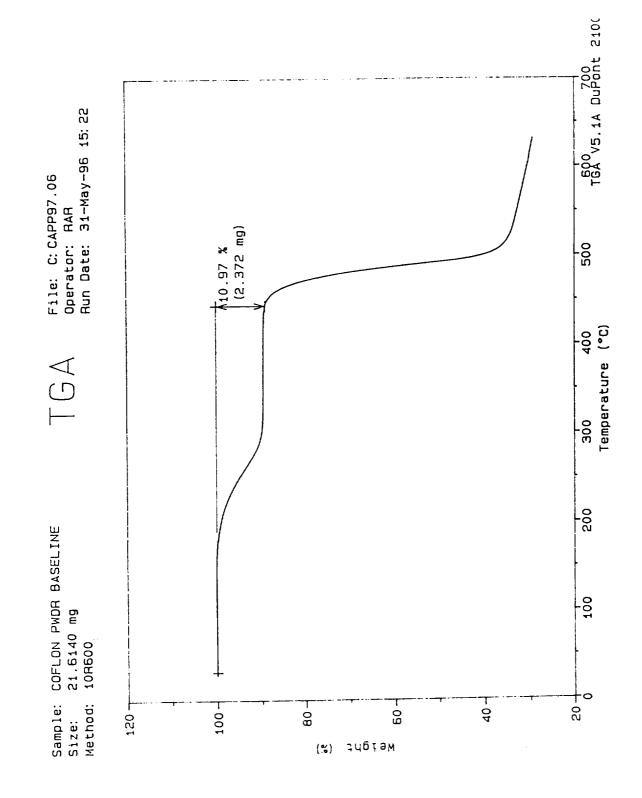


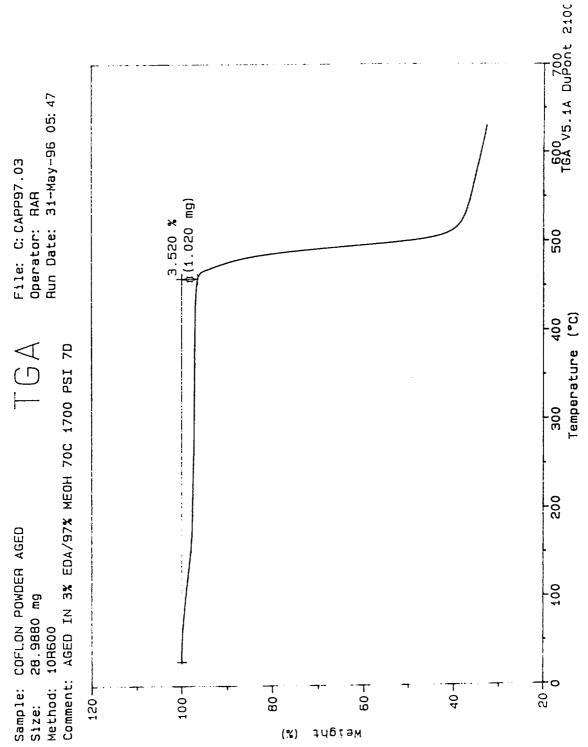
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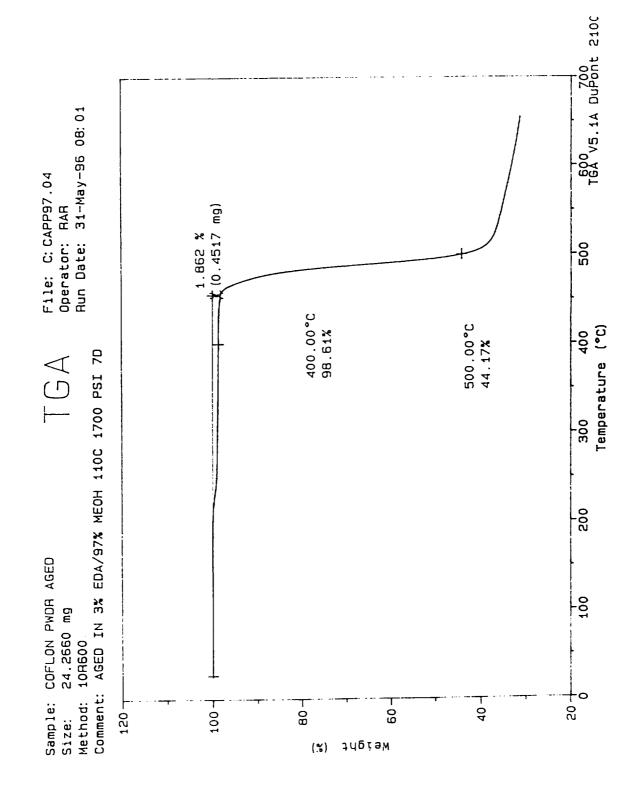
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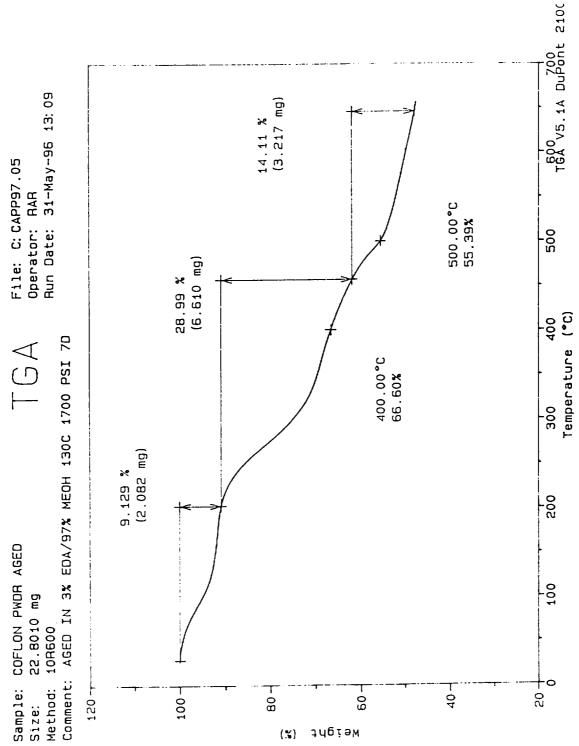




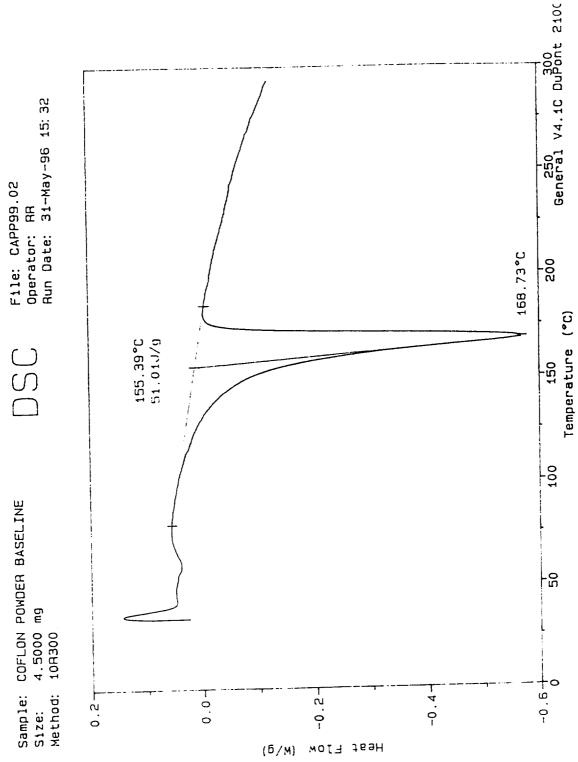


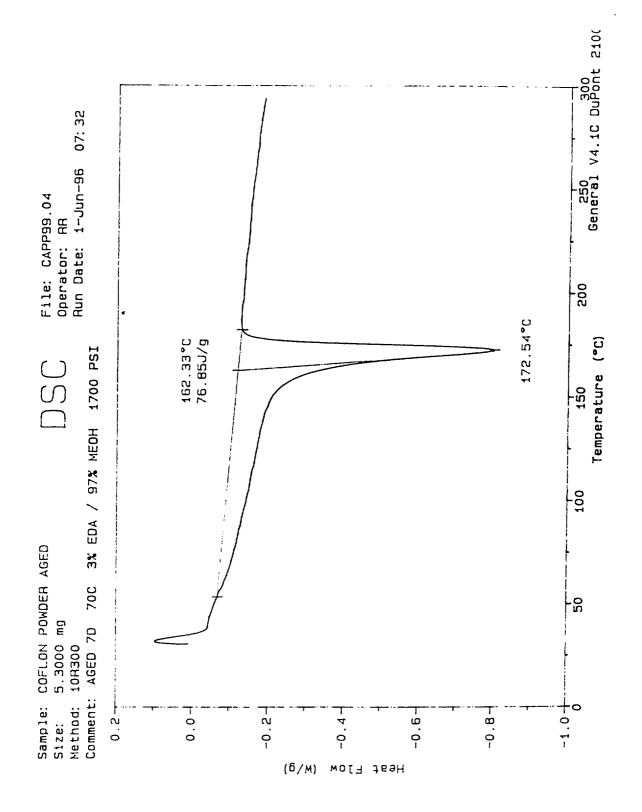


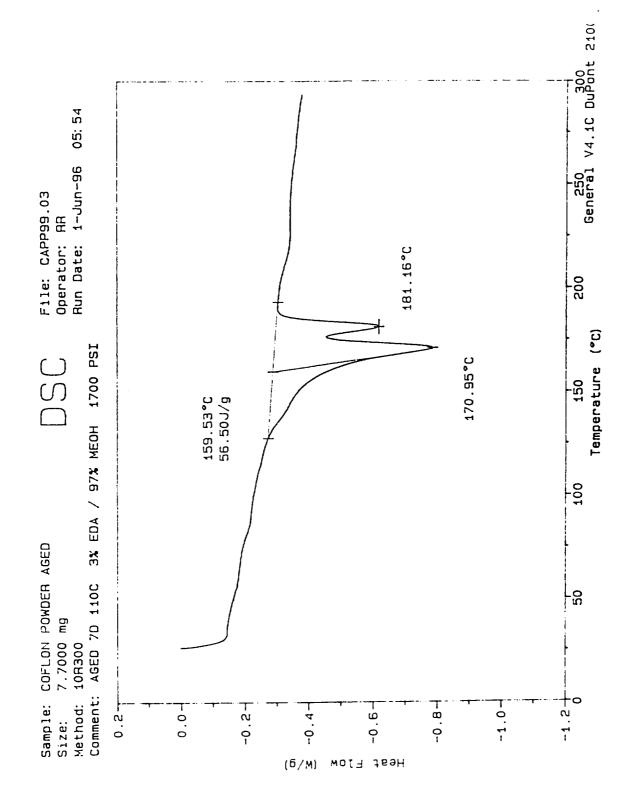
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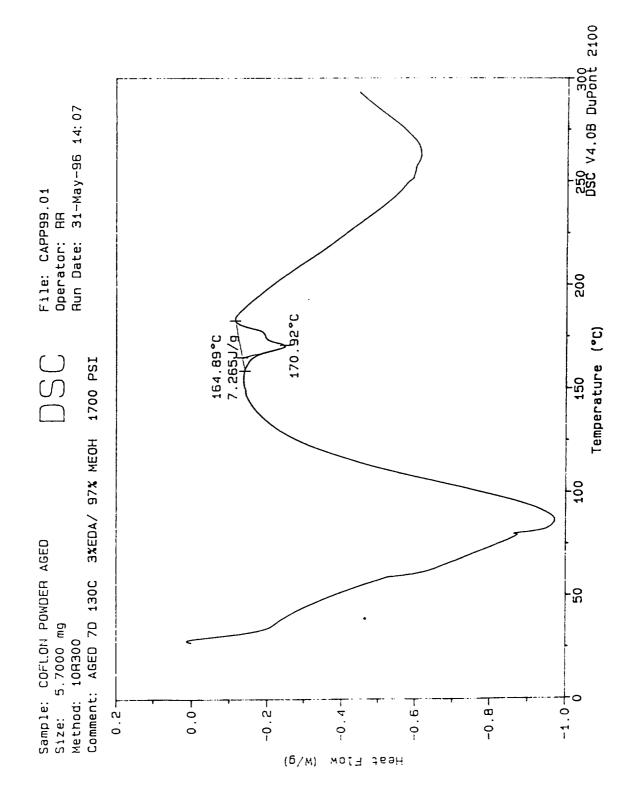
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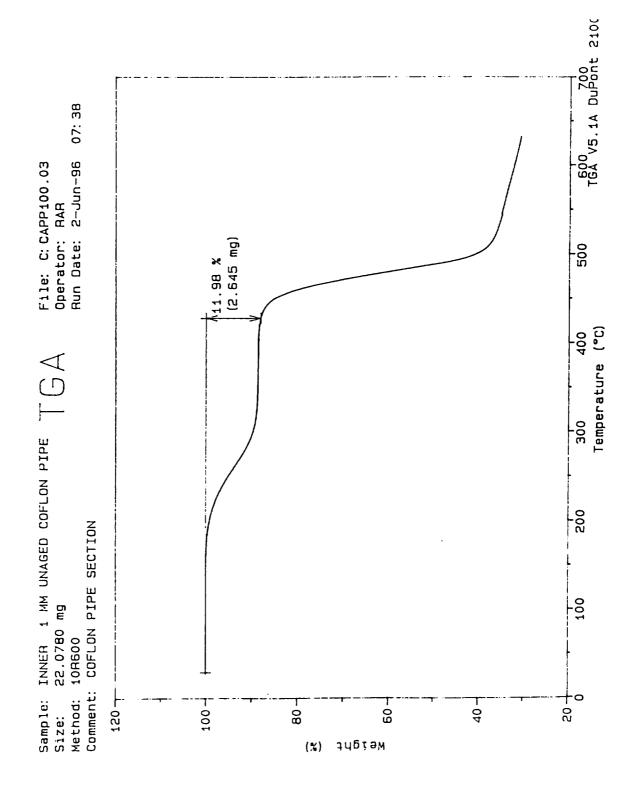


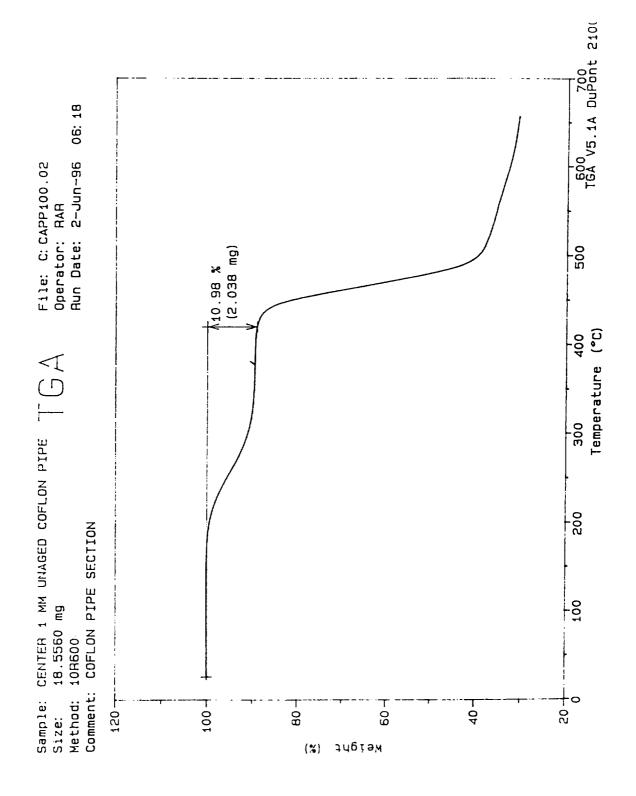


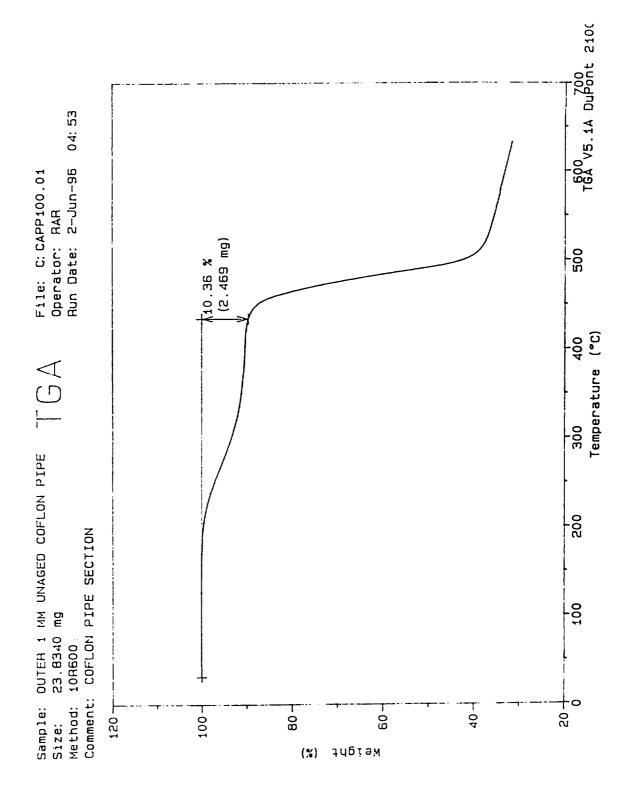


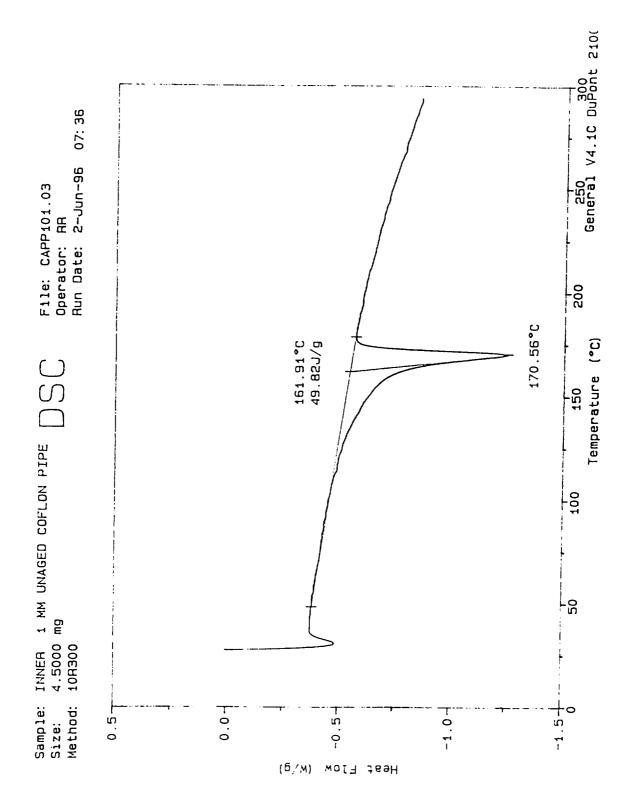
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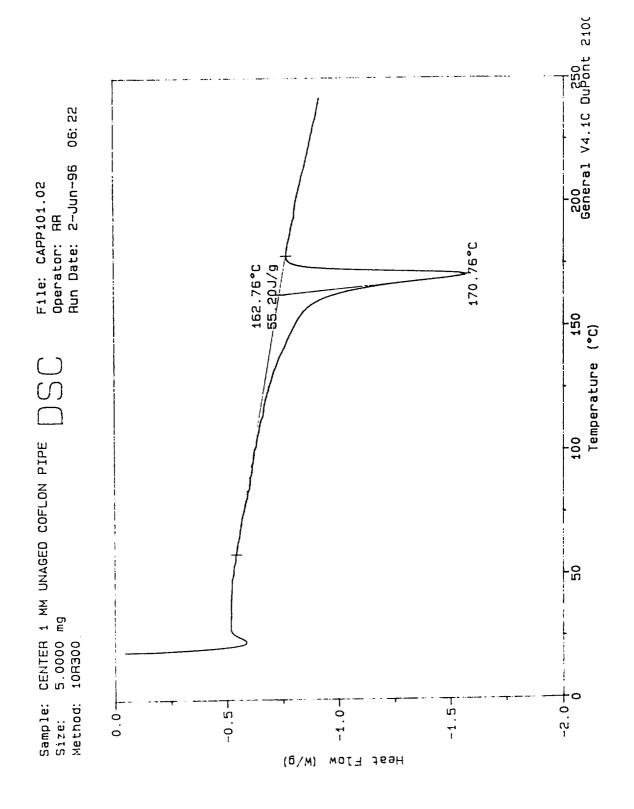


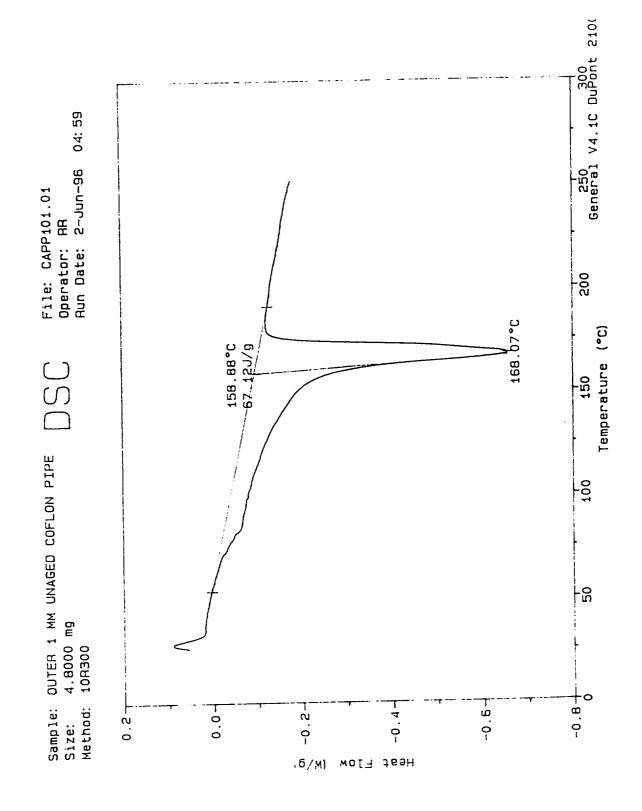






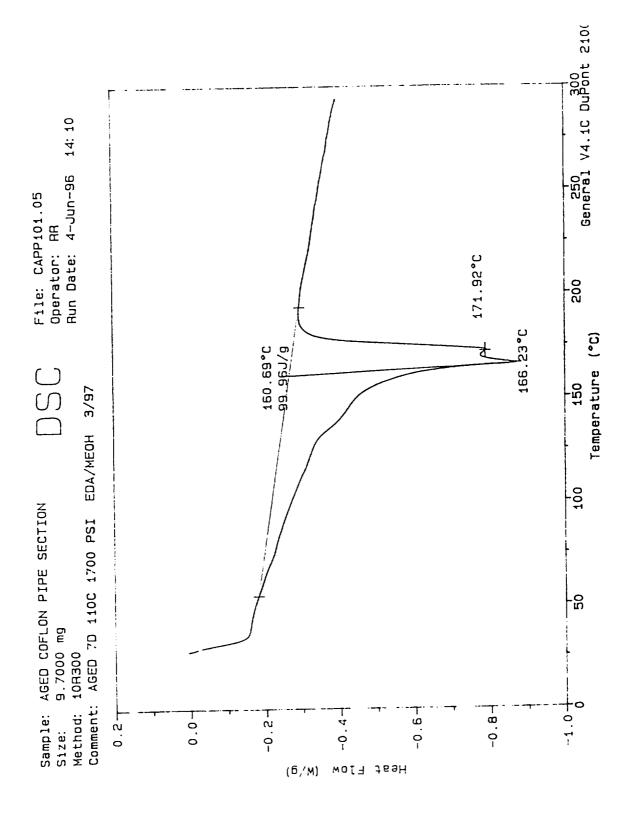
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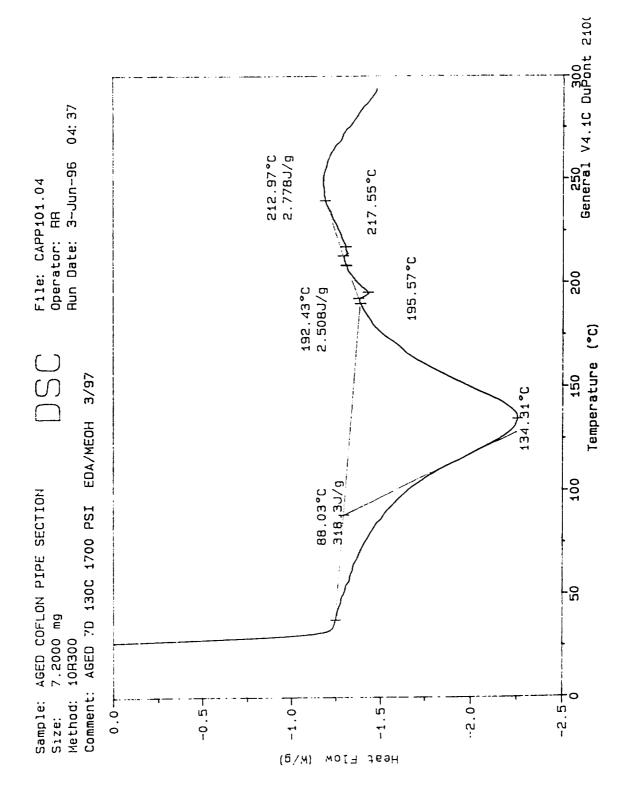


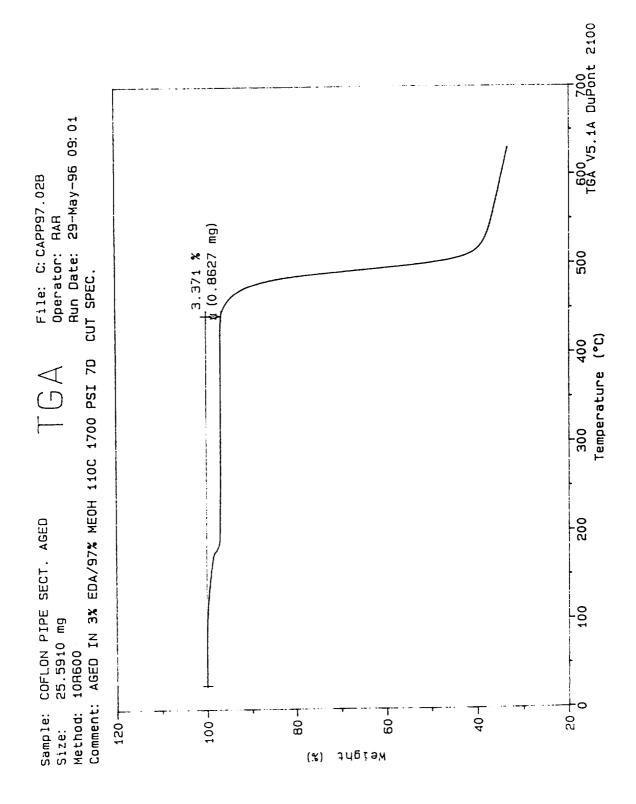


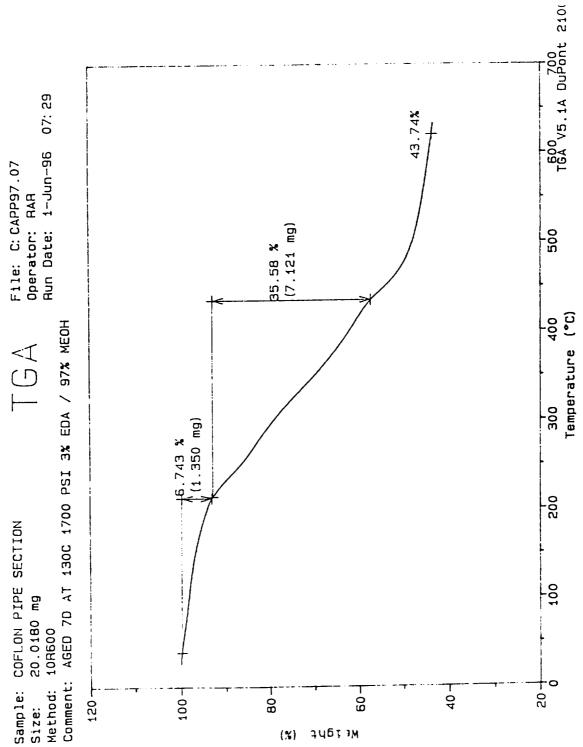
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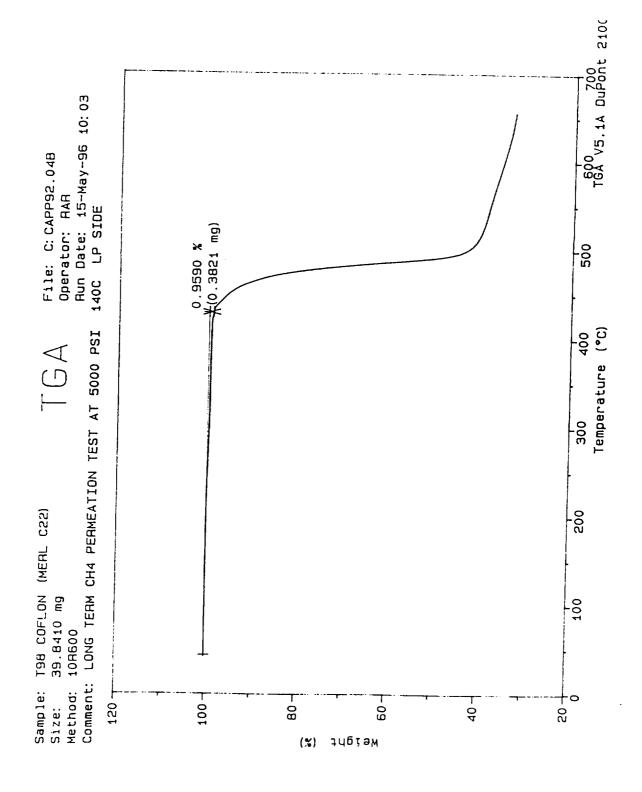
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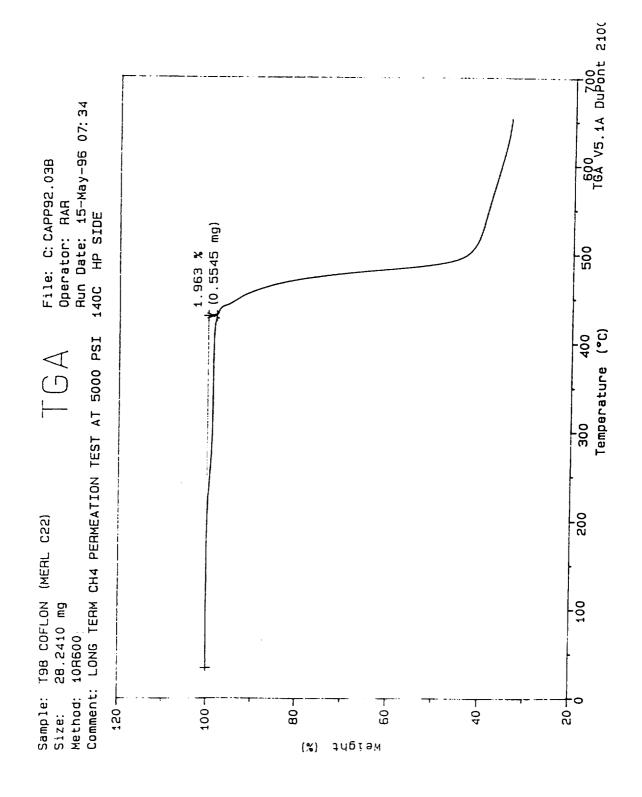


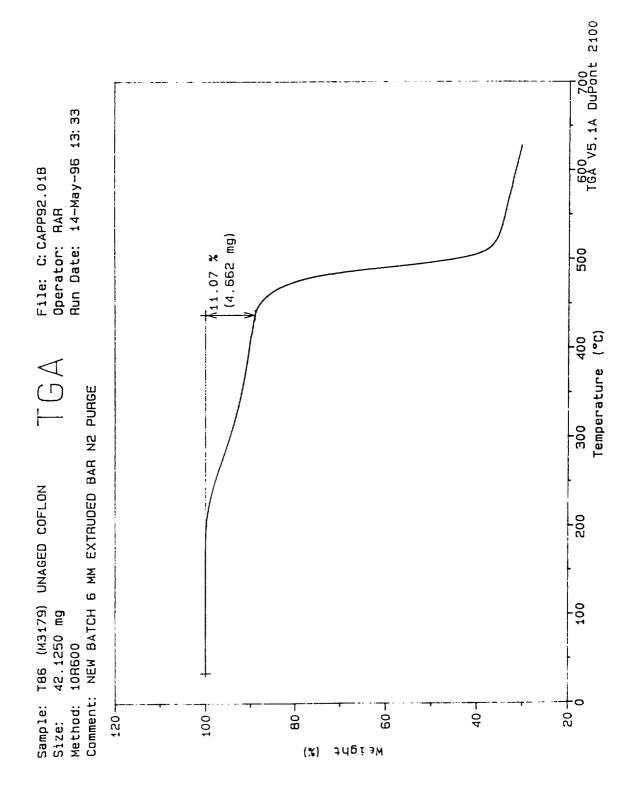




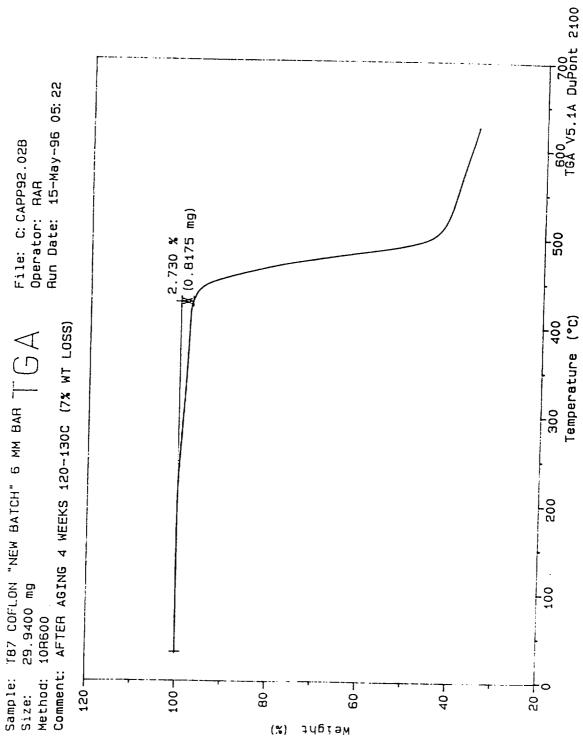


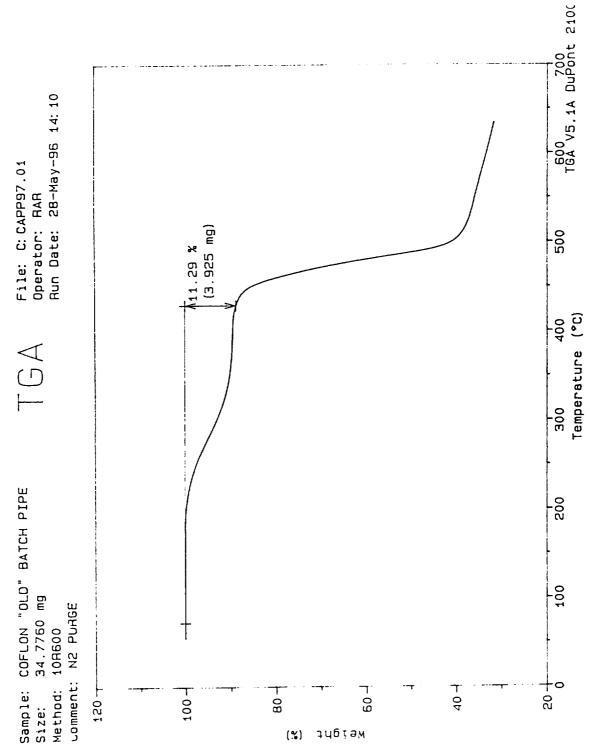






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