CAPP

International Research Project on the Effects of Chemical Ageing of Polymers on Performance Properties

Interim Report on **Chemical and Thermal Analysis**



9063 Bee Caves Road, Austin Texas 78733-6201, U.S.A. Tel: 1-512-263-2101

Fax: 1-512-263-3530



Tamworth Road, Hertford SG13 7DG, England Tel: 44-(0) 992-500120 Fax: 44-(0) 992-586439

Interim Report On Chemical And Thermal Analysis

Prepared for: CAPP International Consortium

Prepared by: J.W. Bulluck & R.A. Rushing

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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) 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 small changes in the molecular weight distribution. Again these changes may result in variations 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, Ultra-Violet Scanning Analysis, GC/Mass Spectrometry, Differential Scanning Calorimetry and Thermomechanical Analysis. In the ultra-violet analysis we noted the presence of an absorption band indicative of triene formation. We investigated a number of aged samples of both Tefzel and Coflon that were forwarded from MERL. We also cast films at SWT and subjected these films to a refluxing methanol 1% ethylene diamine solution. An updated literature search was conducted using Dialog and DROLLS to identify any new papers that may have been published in the open literature since the start of this project. The updated literature search and abstracts are contained in the Appendix section of this report.

1.0 HIGH TEMPERATURE AGING OF COFLON FOR MECHANISTIC ANALYSIS

1.1 <u>Test Apparatus</u>

The apparatus for the experimentation involved a round bottom flask fitted with a reflux condenser for aging of the Coflon film. Into the reflux condenser the diamine containing methanol solution was charged, and the Coflon film was added.

1.2 <u>Sample Preparations</u>

The virgin Coflon was dissolved in hot DMAC and cast into thin films. The solvent was allowed to evaporate prior to the beginning of the aging experiments.

1.3 Test Conditions

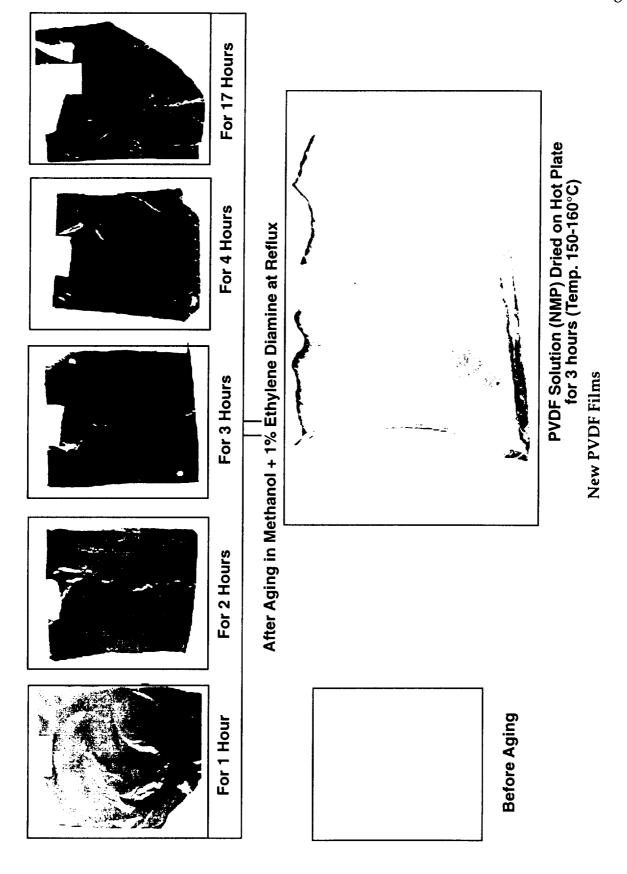
The tests were conducted by immersing the Coflon films in the refluxing methanol ethylene diamine solution. The test films were removed periodically for later chemical analysis. Photographs that exhibit the visual changes of the Coflon upon aging follow on the next page.

2.0 CHEMICAL ANALYSIS ASSESSMENT OF AGING EFFECTS

2.1 Gel Permeation Chromatography of Aged Coflon

Gel Permeation Chromatography was conducted on a number of environmentally aged Coflon test pieces. The solvent was NMP and the flow rate was 2mL/ minute. The injection volume was 100 uL the detector temperature was set at 30°C and the columns were run at 30°C.

Sample Identification	Visual Appearance	Molecular Weights Mn,Mw, Mz	Exposure
Control	White	81,700-538,900-	Laboratory Exposure and
Coflon	Translucent	3,217,000	Conditions
T-75 Coflon	White	177,460-	Fluid F, 140°C 3 day
		536,509-	
		1,062,095	
T-77 Coflon	Brown	170,504-	Fluid F, 140°C 8 days
		532,717-	
		1,052,564	
T-74 Coflon	White	152,905-	Fluid F, 120°C 2 weeks
		534,361-	
		1,080,943	
T-87 Coflon	White	171,265-	27 days 120-135°C air oven
		538,897-	
		1,067,315	
T-88 Coflon	White	177,880-	Fluid F, 120°C 5 ksi 1 weeks
		549,827-	
		1,084,282	
T-89 Coflon	White	173,803-	Fluid F, 120° C 5 ksi 3 weeks
		539,103-	
		1,073,977	
T-76 Coflon	White	173,329-	Fluid F, 140° C 5 day
		534,908-	
		1,059,213	
T-53 Coflon	Brown	158,990-	Fluid F, 120° C 5 ksi 4 weeks gas
		519,823-990,690	phase



2.2 X-Ray Diffraction - Crystallinity Changes

X-ray Diffraction was conducted on both samples from the exposed Tefzel and Coflon thermoplastics. 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
- (9) Fluid I- MERL Formulated aromatic and aliphatic hydrocarbon solution

The percentage crystallinity was calculated on the samples. Twelve polymer samples were analyzed, ten PVDF (Coflon) samples and two Tefzel samples. The degree of crystallinity ranges from as low as 9% to as high as 48%. The Coflon samples were more crystalline. Below are the results of the analysis including the fluids the samples were exposed to prior to testing. Color changes were slight with both the Tefzel and Coflon materials in this batch of samples.

Sample Identification	Visual Appearance	Percentage Crystallinity	Exposure
Control	White	26.0%	Laboratory Temperature and
Tefzel	Translucent		Conditions
T-105 Tefzel	White	9%	Fluid A, 6 days 140°C vapor
	Translucent		pressure
Control	White	41.5%	Laboratory Exposure and
Coflon	Translucent		Conditions
T-73 Coflon	Beige	39%	Fluid F, 3 months 100°C
T-74 Coflon	Beige	35	Fluid F, 120° C, 2 weeks
T-75 Coflon	Beige	31% prev. 26%	Fluid F, 140° C 3 days
T-77 Coflon	Beige	34% prev. 29%	Fluid E, 140° C 8 days
T-87 Coflon	White	40%	After 27 days at 120-130° C in air
	Translucent		oven
T-90 Coflon	Beige	29% prev. 39%	Fluid F, 140° C 5 ksi 2 weeks
T-91 Coflon	Beige	37%	Fluid F, 140° C 5 ksi 4 weeks
T-100 Coflon	Brown	39%	Fluid G, 65° C, reflux 2 weeks
T-102 Coflon	Beige	48%	Fluid I, 140° C, 5 ksi 10 weeks

We can seen clearly that all of the exposures for both materials had an effect on the percentage crystallinity. The greatest decrease in crystallinity for both polymers was observed with the Tefzel Fluid G exposure in methanol with ethylene diamine. We also note that the percentage of crystallinity for Coflon increases or decreases depending on the exposure. At the end of this report in Appendix we have attached a number of plots pertaining to the X-ray diffraction analysis of the Coflon and Tefzel test pieces.

2.3 FTIR and Ultraviolet Analysis

The Coflon films described in Section 1.0 were analyzed using Fourier Transform Infrared Spectroscopy (FTIR) and Ultraviolet (UV) spectroscopy. Changes in the molecular structure of PVDF were therefore determined as a function of exposure time. The aging media for the films was methanol with 1% ethylene diamine (EDA). Exposure times were 0, 1, 2, 3, 4 and 17 hours.

Film thicknesses were 0.01 mm for the all tests. FTIR analyses were performed on a Nicolet 20 SX spectrophotometer in the transmission mode. A Varian DMS 200 UV/Vis spectrophotometer was used for the ultraviolet measurements.

FTIR Results

Appendix 4.1 displays the complete FTIR spectra for each Coflon film tested. Figure 2-1 compares an enlarged section of the baseline and 17 hour aged specimens. A clear indication of C=C formation is observed at 1700 cm⁻¹. The decrease in the intensity of the peak at 1400 cm⁻¹ may be due to loss of plasticizer (carbonyl) or carbon-fluorine.

Ultraviolet Results

The UV absorption at 290 nm of the PVDF films aged in methanol/EDA were recorded at room temperature. Absorption at this frequency is known to be associated with C=C double bonds, particularly conjugated trienes. The figure displayed in Appendix 4.2 shows the results for the five aged specimens and the baseline sample. An increase in the absorbance was found after aging of the PVDF films. This growth in absorbance translates into a decrease in percent transmittance in the samples. The absorbance change was found to increase with time of exposure to the methanol/amine mixture.

2.4 Gas Chromatography couples with Mass Spectrometry

We conducted GC/Mass Spec on Sample T-89 extracted with methanol. The extract showed primarily the presence of the dibutyl ester of decanedioic acid. Secondarily evident was the free acid decanedioic acid. The decanedioic acid is a degradation product of the plasticizer. The chromatograms are included in Appendix 4.4.

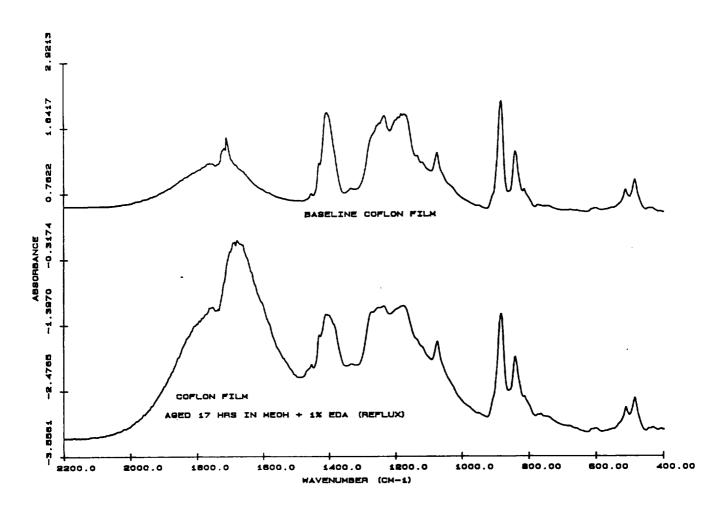


Figure 2-1. FTIR Spectra of Baseline and Aged Coflon Films

3.0 THERMAL STABILITY AND COMPOSITIONAL ANALYSIS

3.1 <u>Creep Analysis using TMA</u>

The Coflon films described in Section 1.0 were subjected to creep testing in TRI's 943 Thermomechanical Analyzer (TMA) after aging in methanol/EDA. The films were cut into 0.15 inch (3.8 mm) widths. The specimens were mounted in a tensile fixture. A dead load was applied sufficient to impose a stress of 500 psi. The TMA furnace was then placed around the sample compartment. The sample temperature was ramped up 10°C per minute to 150°C. In this manner the creep behavior was observed while the specimen was undergoing an increase in temperature.

Appendix Figure 4.1 displays the results for the unaged Coflon and three of the aged specimens. The specimen which was aged for 17 hours was to brittle to

fixture and is not indicated in Appendix Figure 4.1 for this reason. A significant increase in the resistance to creep with temperature was noted in the aged specimens. This behavior may be a result of dehydrofluorination and possible subsequent crosslinking within the polymer.

3.2 DSC of Aged Materials

A separate set of PVDF films was prepared for the DSC tests. The films were prepared as described in Section 1.0 with the exception of having a slightly thicker crossection. These specimens were again exposed to methanol/EDA for 0.25, 1.0, and 15 hours respectively. After exposure, sections of the films were subjected to Differential Scanning Calorimetry (DSC) testing. Sections were cut from the dry aged films and placed into tared DSC sample pans. The sample temperature was raised at a constant rate of 10°C per minute to a maximum of 200°C. Melt onset onset, peak temperature, and heat of fusion were thus determined.

DSC Results

DSC plots are displayed in Appendix 4.2. Tabular data describing the melt aspects and heats of fusion is shown in Table 3.1.

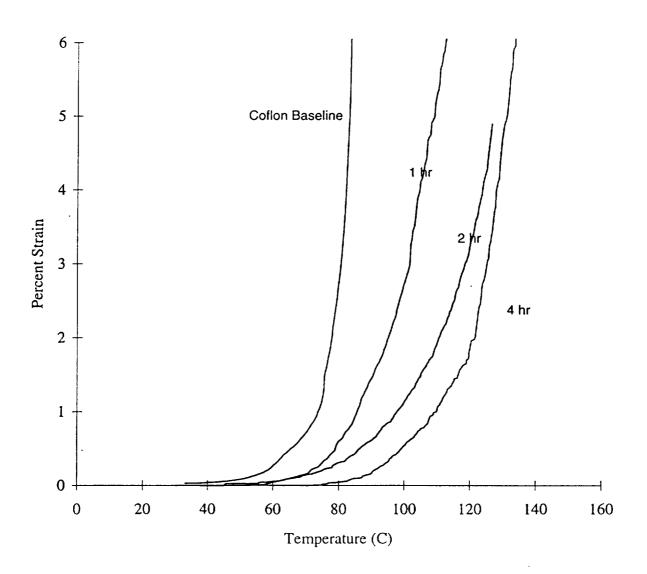
Coflon Film Sample	Melt Onset (°C)	Melt Peak (°C)	Heat of Fusion (J/g)
Baseline	161.45	173.74	58.49
Aged 0.25 hrs	162.11	177.12	56.89
Aged 1.0 hrs	160.76	174.55	68.21
Aged 15 hrs	153.87	169.39	40.17

A slight increase in melt temperatures was noted with the 0.25 hour aged specimen. At the 1 hour and 15 hour exposure level the temperatures appear to be decreasing. Most notable are the values obtained from the specimen which was aged 15 hours. Here the melt onset and peak temperatures dropped significantly as did the heat of fusion. This results lends credibility to the hypothesis that initial aging of PVDF in this fluid causes some crosslinking and at longer exposure times lower molecular weight species begin to appear which may lower the melt points and heats of fusion.

4.0 Appendix

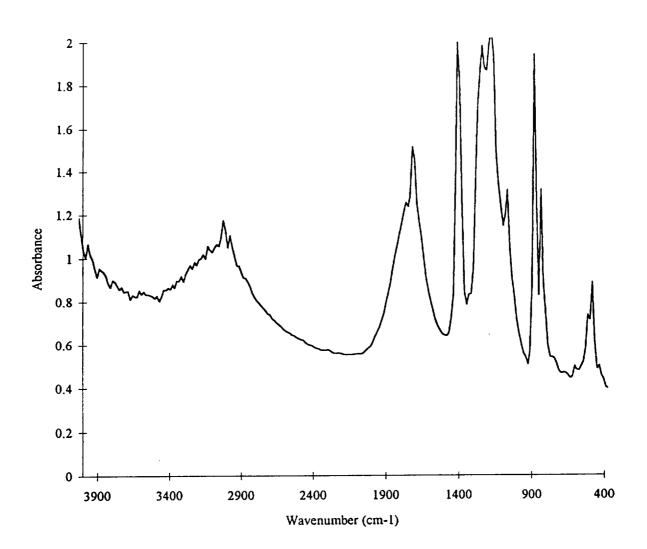
4.1 TMA

Creep Behavior of Coflon Films Aged in Methanol + 1% EDA



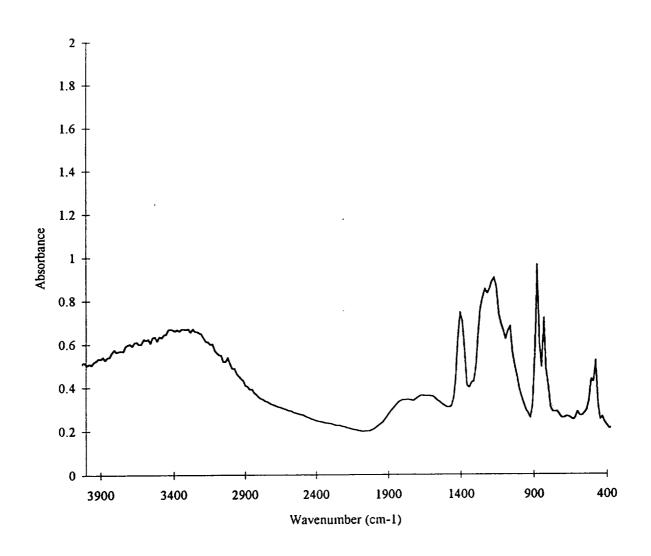
4.2 FTIR and Ultraviolet Absorption Plots

FTIR Spectra Coflon Film Unaged Baseline

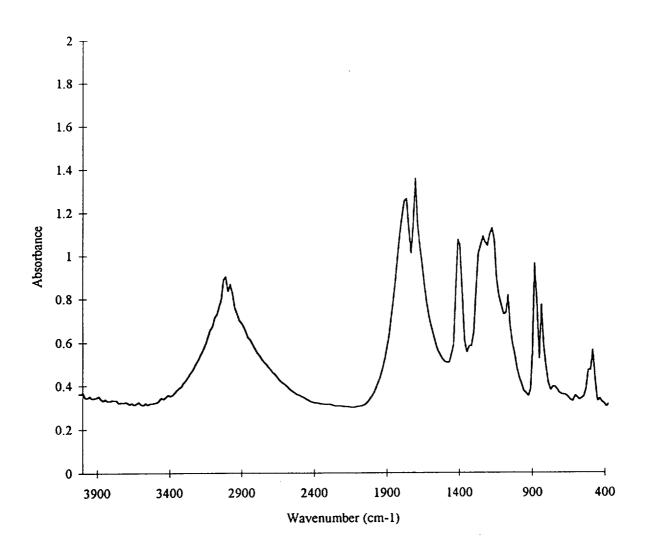


FTIR Spectra

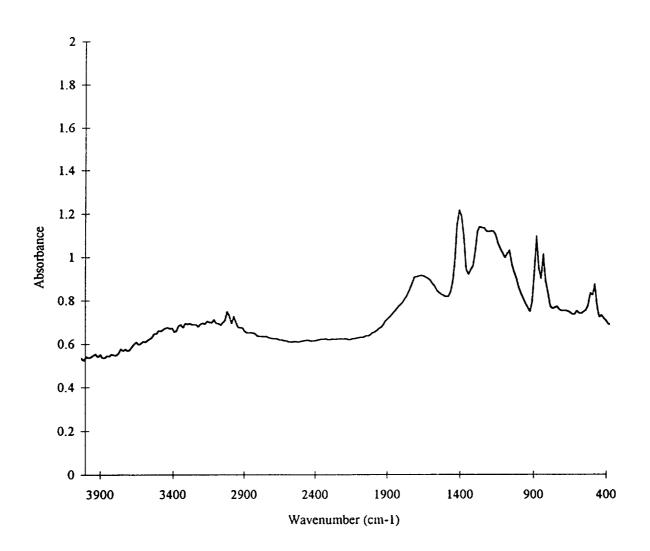
Coflon Film Aged 1 hr MeOH + 1% EDA Reflux



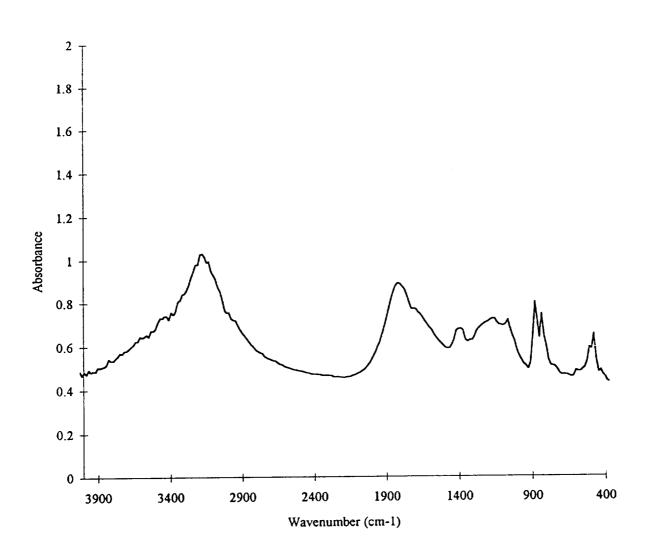
ITIR Spectra
Coflon Film Aged 2 hrs MeOH + 1% EDA Reflux



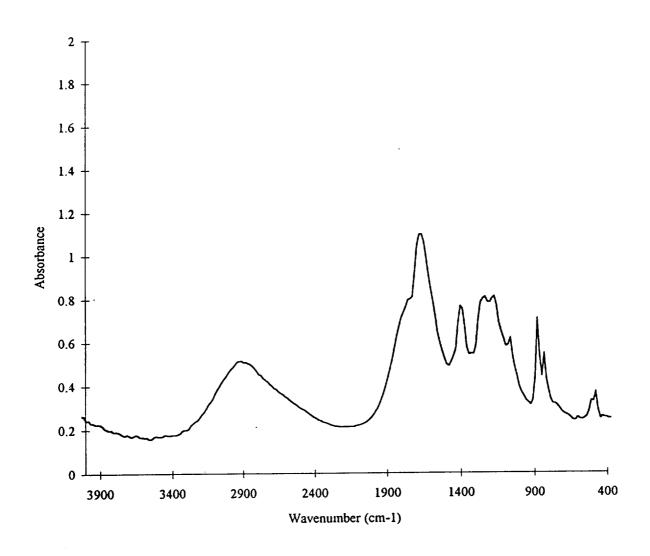
FTIR Spectra Coflon Film Aged 3 hrs MeOH + 1% EDA Reflux



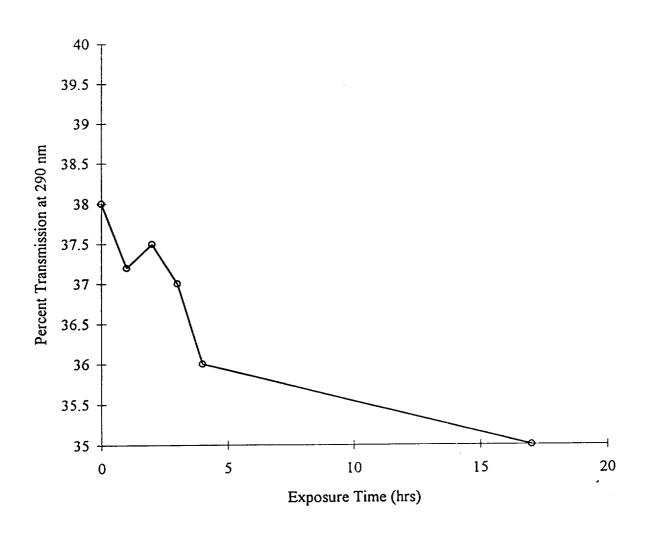
FTIR Spectra
Coflon Film Aged 4 hrs MeOH + 1% EDA Reflux



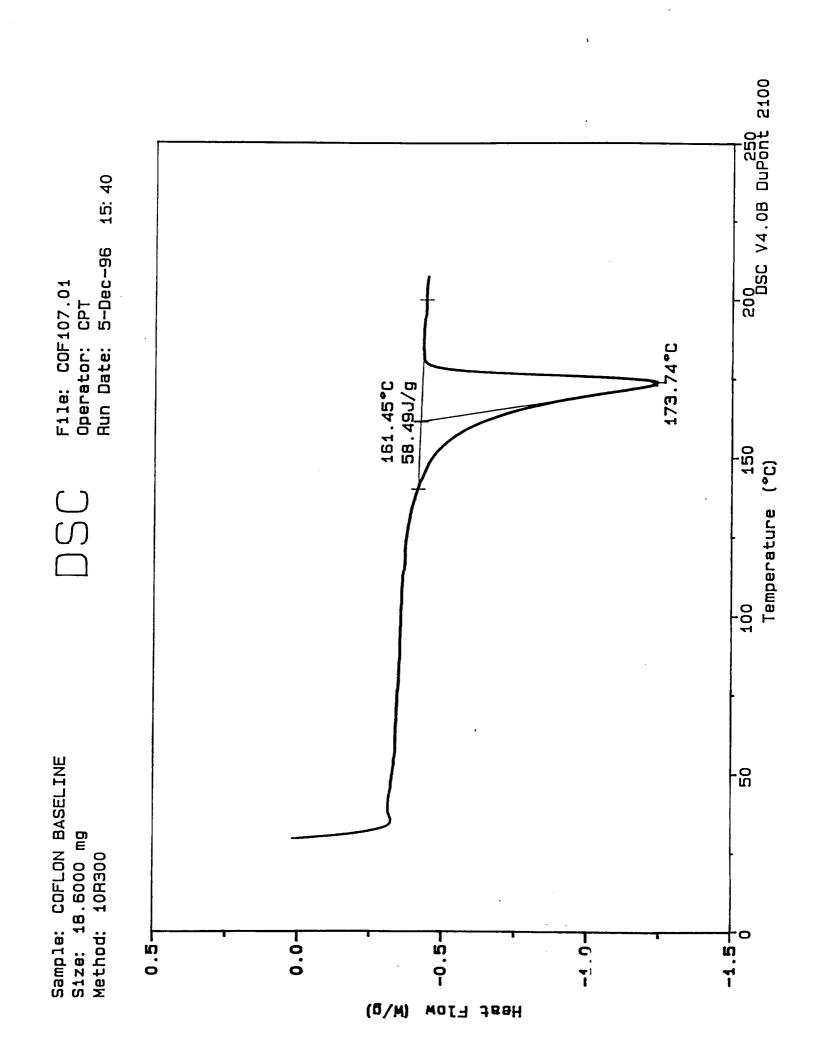
FTIR Spectra
Coflon Film Aged 17 hrs MeOH + 1% EDA Reflux

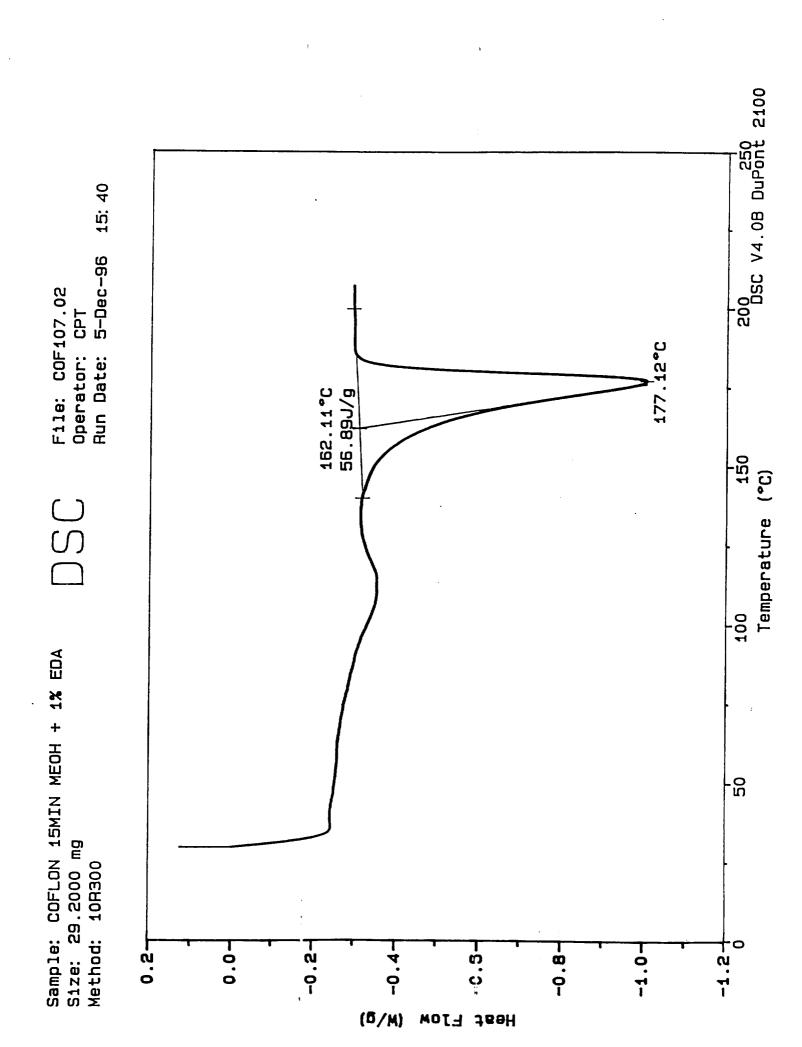


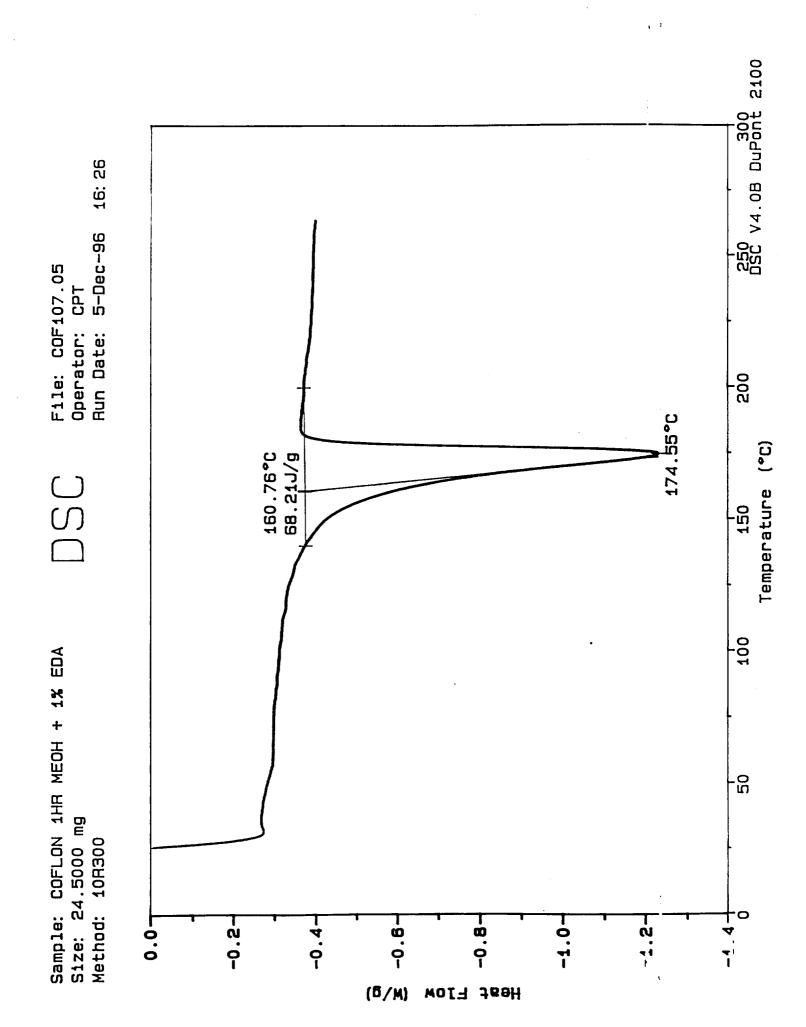
Aged Coflon Films
Changes in UV Absorption at 290 nm
After Exposure to Methanol + 1% EDA

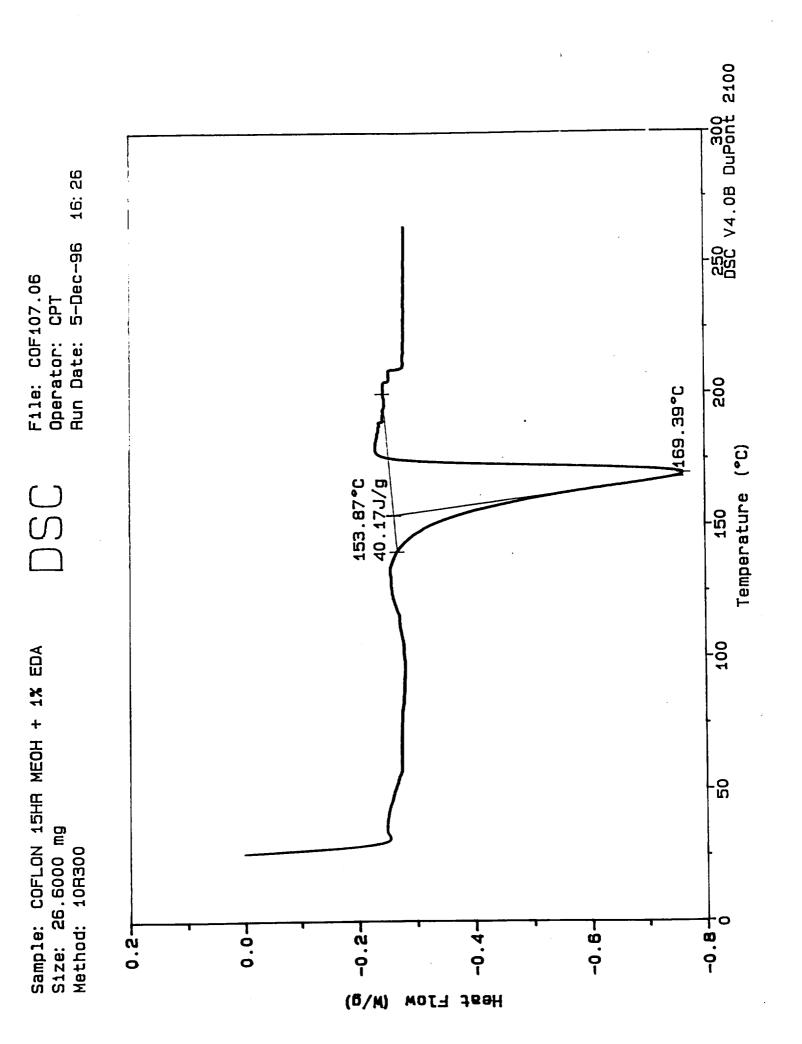


4.2 DSC Plots

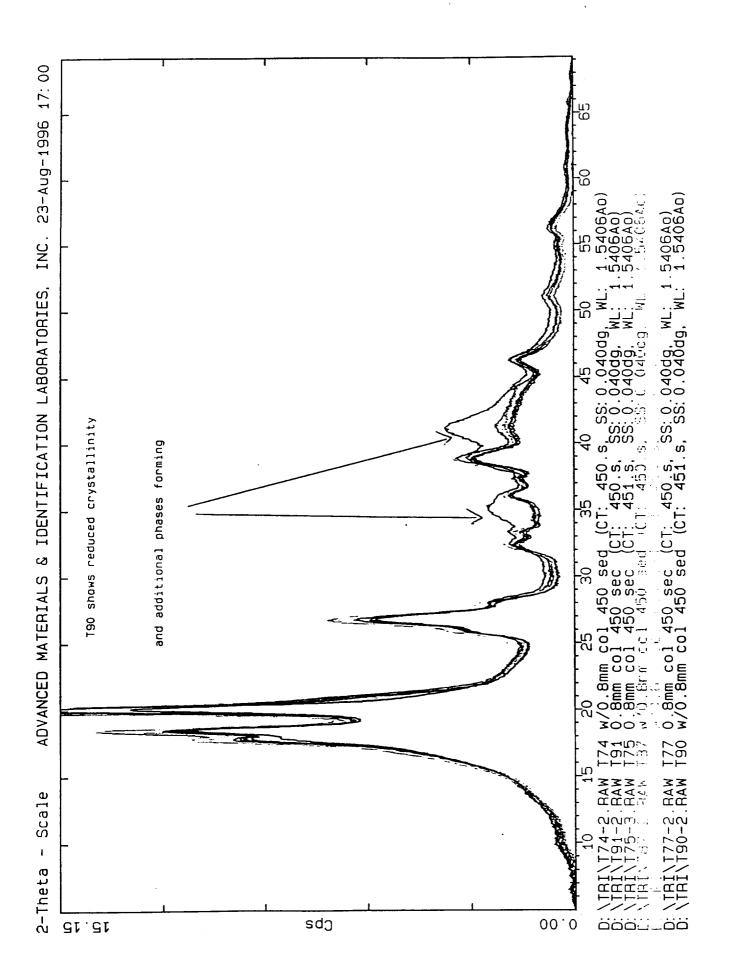


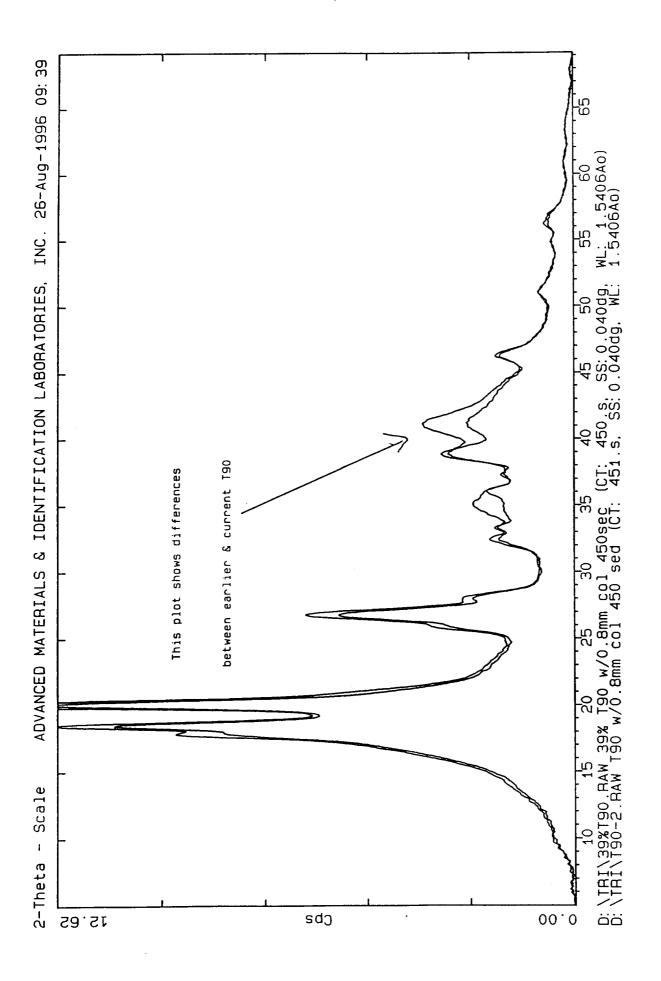






4.3 X-Ray Diffraction Plots





4.4 GC/Mass Spectrometry Chromatograms T-89

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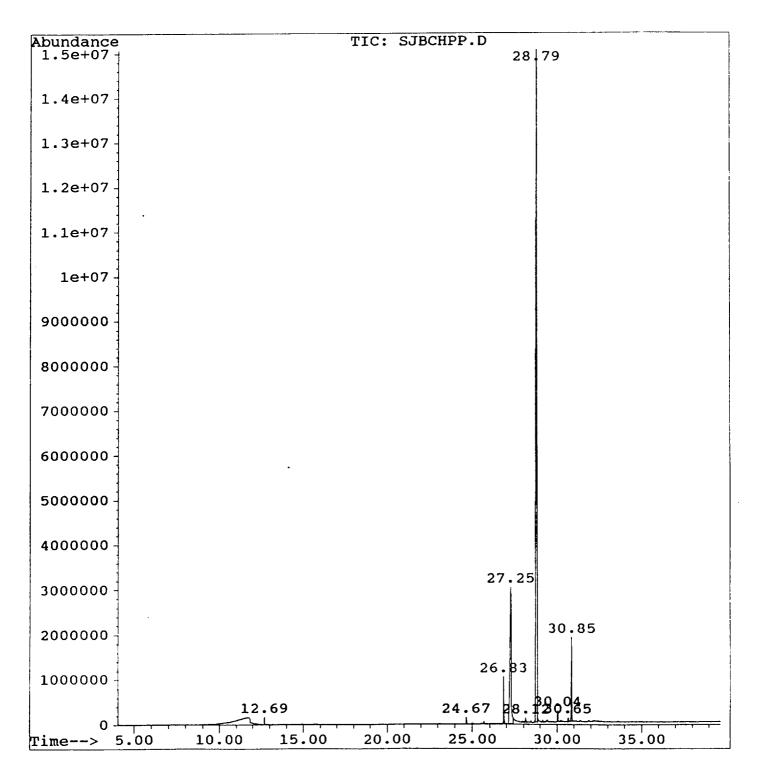
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Acquired: 4 Dec 96 5:09 pm using AcqMethod MEOHSCAN.M

Instrument: 5971 - In

Sample Name: MeOH extract of PVDF 12/04/96

Misc Info : Vial Number: 1



Information from Data File:

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Operator : Don Clay

Acquired : 4 Dec 96 5:09 pm using AcqMethod MEOHSCAN.M

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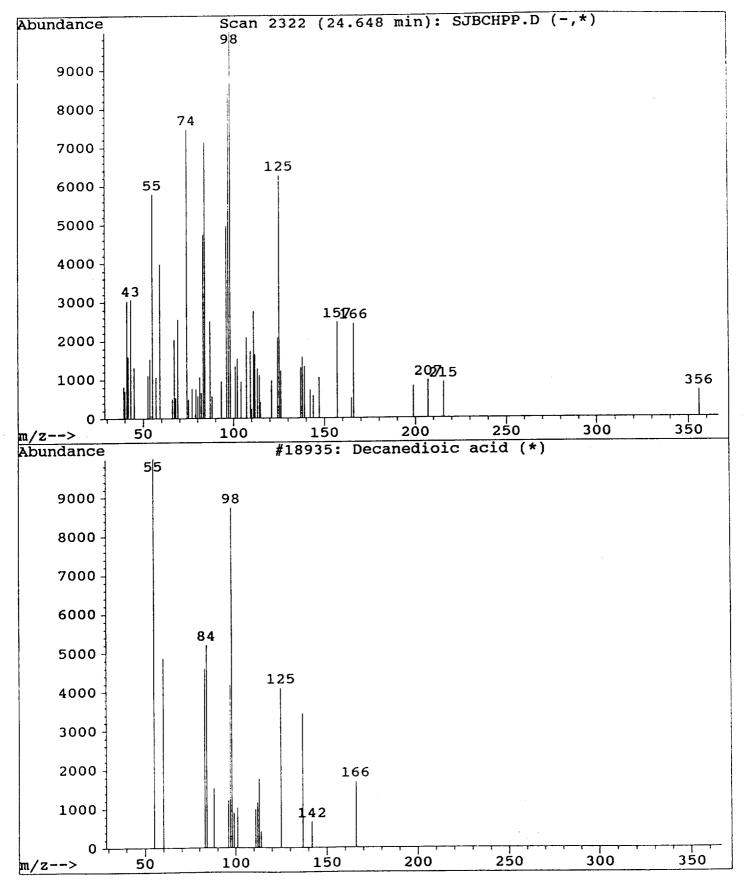
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2	24.67	0.35	C:\HPCHEM\DATABASE\NBS49K.L Decanedioic acid, dimethyl ester 1,2-Butanediol, 1-(2-furyl)- 1H-Imidazole-2-methanol	9571	000106-79-6 004208-60-0 003724-26-3	27
3	26.83	2.41	C:\HPCHEM\DATABASE\NBS49K.L Thiophene, 2-nonyl- Carbofenothion sulfoxide 1,2-Butanediol, 1-(2-furyl)-	39492	057754-07-1 017297-40-4 004208-60-0	32
4	27.25	19.60	C:\HPCHEM\DATABASE\NBS49K.L 1,3-Cyclopentanedione 2(1H)-Pyridinone, 4-hydroxy-1,6-di 4-PENTYNOIC ACID	6085	003859-41-4 006052-75-1 006089-09-4	16
5	28.12	0.35	C:\HPCHEM\DATABASE\NBS49K.L Hexanedioic acid, bis(2-methylprop 2,6-Piperazinedione, monooxime HEXANEDIOIC ACID, MONO(2-ETHYLHEXY	4477	000141-04-8 056700-84-6 004337-65-9	27
6	28.79	70.71	C:\HPCHEM\DATABASE\NBS49K.L Decanedioic acid, dibutyl ester Silacyclopent-3-ene, 3-methyl- 1H-1,2,4-Triazol-3-amine, N-methyl	1113	000109-43-3 054077-65-5 015285-16-2	20
7	30.04	0.60	C:\HPCHEM\DATABASE\NBS49K.L Cyclohexanone, 2-(1-methylethyl)- Oxacyclotetradecane-2,11-dione, 13 Cyclohexanone, 2-ethyl-	25629	001004-77-9 074685-36-2 004423-94-3	32
8	30.65	0.29	C:\HPCHEM\DATABASE\NBS49K.L Hexadecanedioic acid, dimethyl est Cyclohexanone, 2-(1-methylethyl)- Aziridine, 1,2-diisopropyl-3-methy	6384	019102-90-0 001004-77-9 006124-84-1	14

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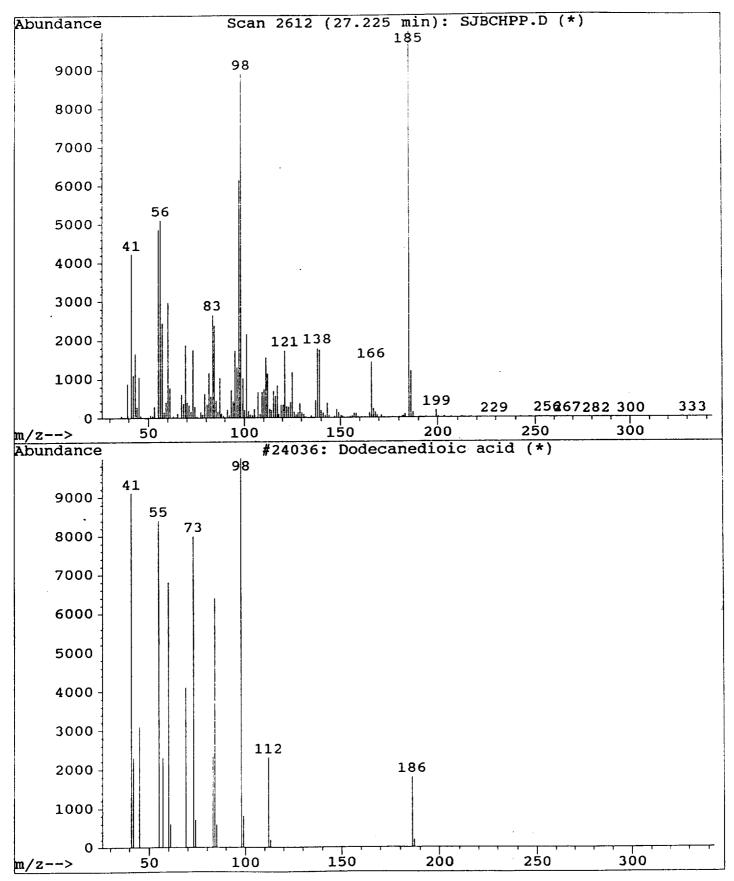
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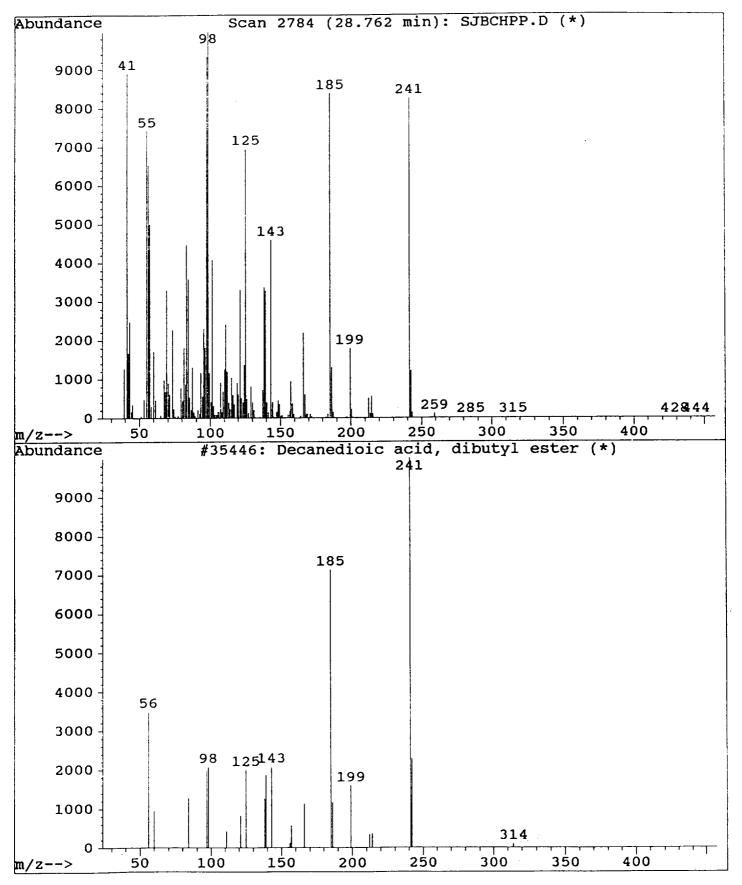
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4.5 Literature Search

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-- 3 - ENTRY CLASSIFICATION: UNCLASSIFIED
-- 5 - CORPORATE AUTHOR: MATERIALS RESEARCH LABS ASCOT VALE (AUSTRALIA)
-- 6 - UNCLASSIFIED TITLE: CHEMICAL DESEALING OF A FLUOROSILICONE
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-- 8 - TITLE CLASSIFICATION: UNCLASSIFIED
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--10 - PERSONAL AUTHORS: HANELA, PETER J.; HUANG, ROBERT H.; MAZUREK,
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--11 - REPORT DATE: MAK , 1990
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--10 - PERSONAL AUTHORS: BROADWAY, N. J.; PALINCHAK, S.
--11 - REMERT DATE: JUN 30, 1959
--12 - PAGINATION: 21P MEDIA COST: $ 6.00 PRICE CODE: AA - REPORT NUMBER: REIC-M-17
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        POLYESTER PLASTICS, RADIATION DOSAGE, BILICONES, WEAR RESISTANCE,
        COBALL, SILASTIC COMPOUNDS, CHEMICAL A TACK (DEGRADATION),
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        TETRAFLUOROETHYLENE RESINS, PLASTIC COATINGS, ELECTRICAL INSULATION,
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        AND KEL-F ARE ESSENTIAL MATERIALS FOR VALVES, HOSES, AND ELECTRICAL
        INSULATION. THEIR HEAT RESISTANCE HAS MADE THEM INVALUABLE FOR HIGH-
        TEMPERATURE APPLICATIONS. THIS RESISTANCE TO HEAT AND CHEMICALS IS
        DUE TO THE CARBON-FLUOKINE BOND WHICH IS CHEMICALLY ONE OF THE MOST
        STABLE OF ORGANIC LINKAGES. BECAUSE OF THEIR IMPORTANT APPLICATIONS,
        THESE MATERIALS WERE INVESTIGATED FOR USE IN A RADIATION
        ENVIRONMENT. IT WAS FOUND THAT THE RADIATION RESISTANCE OF
       FLUOROPOLYMERS IS NOT AS GOOD AS THEIR CHEMICAL AND HEAT RESISTANCE.
        TEFLON HAS VERY POOR RADIATION RESISTANCE, WHILE THE OTHER
       FLUOROPOLYMERS ARE ONLY SLIGHTLY BETTER. THIS MEMORANDUM SUMMARIZES
_ -
        THE AVAILABLE DATA ON THE RADIATION STABILITY OF TEFLON AND KEL-F
       PLASTICS, AND KEL-F, POLYFLUOROBUTYL ACRYLATE (POLYFBA)
        HEXAFLUOROPENTAMETHYLENE ADIPATE (A POLYESTER), AND SILASTIC LS-53
        (A FLUORINATED SILICONE) ELASTOMERS.
--28 -
       ABSTRACT CLASSIFICATION:
                                   UNCLASSIFIED
--29 -
       INITIAL INVENTORY:
--33 - LIMITATION CODES: 12
                         293900
--35 - SOURCE CODE:
--36 - ITEM LOCATION:
                         OITO
--40 - GEOFOLITICAL CODE:
--41 - TYPE CODE: W
--43 - IAC DOCUMENT TYPE:
--*****
                               EXPORT CONTROL
                                *******
        0F
-- 1 - AD NUMBER: 8132607
-- 2 - FIELDS AND GROUPS: 11/1, 11/3, 6/13
  3 - ENTRY CLASSIFICATION: UNCLASSIFIED
      CORPORATE AUTHOR: TECHNICAL RESEARCH ASSOCIATES INC SALT LAKE CITY
-- 5 -
       Uì
                               THE BIODEGRADATION OF FLUOROSILICONE. MHASE
- -- 🔬 -
       UNCLASSIFIED TITLE:
__
       TITLE CLASSIFICATION:
                               - UNCLASSIFIED
-- 8 -
-- 9 - DESCRIPTIVE NOTE: FINAL REPT. JUL 88-FEB 89,
- 10 - PERSONAL AUTHORS: BOWERS-IRONS, GAIL; PRYOR, ROBERT; IRAN, OUYNH
--11 - REPORT DATE: MAY , 1989
--12 - PAGINATION: 71P MEDIA COST: $ 6.00 FRICE CODE: AA
--15 - CONTRACT NUMBER: F33615-38-C-5462
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--16 - PROJECT NUMBER: 3005
--17 - TASK NUMBER:
-- 18 - MONITOR ACRONYM: WRDC
--19 - MONITOR SERIES: 1R-89-4042
--20 - REPORT CLASSIFICATION* UNCLASSIFIED
-- 21 - SUPPLEMENTARY NOTE: ORIGINAL CONTAINS COLOR PLATES: ALL DITC
         REPRODUCTIONS WILL BE IN BLACK AND WHITE.
--
--22 - LIMITATIONS (ALPHA): DISTRIBUTION AUTHORIZED TO U.S. GOV'T.
-- AGENCIES ONLY; SPECIFIC AUTHORITY, PROPRIETARY INFO.; FEB 89. OTHER
         REQUESTS SHALL BE REFERRED TO WRDC/MLBC, WRIGHT-PATTERSON AFB, OH
        45433-6533. THIS OCCUMENT CONTAINS EXPORT-CONTROLLED FECHNICAL DATA.
-- 45433-6533. THIS DOCUMENT CONTAINS EXPORT-CONTROLLED TECHNICHC DATA.
--23 - DESCRIPTORS: *BIODETERIORATION, *RUBBER COATINGS, *ELASTOMERS,
-- *MICROORGANISMS, *PAINTS, *SEALING COMPOUNDS, AGAR, AIR FORCE,
-- AIRCRAFT, BIOLOGY, CHEMICALS, COMPOSITE MATERIALS, CORROSION
-- INHIBITION, DAMAGE, DEGRADATION, DETERIORATION, EFFICIENCY, ENZYMES,
-- FLUORINE COMPOUNDS, FUNGICIDES, GERMICIDES, HAZARDS, MAINTENANCE,
-- METABOLISM, NUTRIENTS, POLYSULFIDES, REMOVAL, REPAIR, SILICONES,
-- SOUP, SURFACES, TETRAFLUOROETHYLENE RESINS, FLUOROFOLYMERS.
--24 - DESCRIPTOR CLASSIFICATION: UNCLASSIFIED --25 - IDENTIFIERS: PE65502F, WUWRDC30055090, EXPORT CONTROL,
--
         FLUOROSILICONE POLYSULFIDE, POLYSULFIDE ELASTOMERS.
--26 - IDENTIFIER CLASSIFICATION: UNCLASSIFIED
--29 - INITIAL INVENTORY:
--33 - LIMITATION CODES: 3
--34 - SOURCE SERIES:
                               397355
--35 - SOURCE CODE:
--36 - ITEM LOCATION: DTIC
--40 - GEOPOLITICAL CODE: 4902
--41 - TYPE CODE: 4
--42 - IAC ACCESSION NUMBER: PL-053019
--43 - TAC DOCUMENT TYPE:
                               PLASTEC-MICROFICHE
--44 - IAC SUBJECT TERMS: P--(U)CORROSION RESISTANCE, MICKOBIOLOGICAL
-- DEFERIORATION, BIODEGRADATION, POLYSULFIDES, FLUOROSILICONES,
         SEALANTS, FLUOROCARBONS, MONITORING, DEGRADATION, FOURIER TRANSFORM
         IR, SPECTROSCOPY, PAINTS, ENZYME DEGRADATION, TEFLON, ELASTOMERS,
- .
         PIFE, COATINGS, ZZ CONTROLLED USGO.,;
--******
--- 4 OF
-- 1 - AD NUMBER: A294734
-- 2 - FIELDS AND GROUPS: 7/3, 7/4, 7/6, 8/8
-- 3 - ENTRY CLASSIFICATION: UNCLASSIFIED
-- 5 - CORPORATE AUTHOR: COLD REGIONS RESEARCH AND ENGINEERING LAB
        HANOVER NH
 - 6 - UNCLASSIFIED TITLE: SUSCEPTIBILITY OF ABS, FEF, FRE, FRF, PTFE,
-- AND PUC WELL CASINGS TO DEGRADATION BY CHEMICALS.
-- 8 - TITLE CLASSIFICATION: UNCLASSIFIED
-- 9 - DESCRIPTIVE NOTE: SPECIAL REPI.,
--10 - PERSONAL AUTHORS: RANNEY, THOMAS A.; PARKER, LOUISE V.
--11 - REPORT DATE: JAN , 1995

--12 - PAGINATION: 24F MEDIA COST: $ 6.00 PRICE CODE:

--14 - REPORT NUMBER: CRREL-SR-95-1
                                                                                AA
--18 - MONITOR ACRONYM: SFIREALCHLT, XA
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--19 - MONITOR SERIES: CR-94Ø71, SFIM-AEC-EF
--20 -
       REPORT CLASSIFICATION: UNCLASSIFIED
       DESCRIPTORS: *FIBERGLASS, *ACRYLON([RILE POLYMERS, *PROPENES.
--23 -
       *BUTADIENES, *WATER WELLS, *FLUORINATED HYDROCARBONS, *ETHYLENES,
       *POLYVINYL CHLORIDE, *REINFORCED PLASTICS, DEGRADATION, MONITORING,
       CHEMICALS, PHYSICAL PROPERTIES, RESISTANCE, TEST EQUIPMENT, GROUND
       WATER, PARTICLES, GLASS FIBERS, WEIGHT, GAIN, ORGANIC COMPOUNDS,
       INERT MATERIALS, ENVIRONMENTAL PROTECTION, DETERIGRATION,
       FLUDROPOLYMERS, FLUDRINATION, ALKALINITY.
       DESCRIPTOR CLASSIFICATION: UNCLASSIFIED
- -24 -
       IDENTIFIERS: *FLUORINATED ETHYLENE PROPYLENE,
       *POLYTETRAFLUOROETHYLENE, FEP(FLUORINATED ETHYLENE PROPYLENE),
       FRE(FIBERGLASS REINFORCED EPOXY), FIBERGLASS REINFORCED EPOXY,
       FRP(FIBERGLASS REINFORCED PLASTICS), FIBERGLASS REINFORCED PLASTICS,
       PIFE(POLYTETRAFLUOROETHYLENE), PVC(POLYVINYL CHLORIDE), NEAT
       ORGANIC COMPOUNDS, WELL CASINGS
--26 -
       IDENTIFIER CLASSIFICATION:
                                    - UNCLASSIFIED
       ABSTRACT: THIS STUDY COMPARES THE CHEMICAL RESISTANCE OF FOUR
--27 -
       LESS COMMONLY USED MATERIALS FOR CASING GROUNDWATER MONITORING
       WELLS: ACRYLONITRILE BUTADIENE STYRENE (ABS), FLUORINATED ETHYLENE
       PROPYLENE (FEP), FIBERGLASS-REINFORCED LPOXY (FRE), AND FIBERGLASS-
       REINFORCED PLASTIC (FRP), WITH TWO MORE COMMONLY USED CASING
       MATERIALS: POLYVINYL CHLORIDE (PVC) AND POLYTETRAFLUOROEIHYLENE
        (PTFE). THE SIX MATERIALS WERE EXPOSED TO 28 NEAT ORGANIC COMPOUNDS
       (INCLUDING ONE ACID) AND 10 EXTREMELY ACIDIC AND ALKALINE
       CONDITIONS FOR UP TO 112 DAYS. THIS WAS DONE TO SIMULATE SOME OF
       THE MOST AGGRESSIVE ENVIRONMENTS THAT MONITURING WELL CASINGS MAY
       BE EXPOSED TO. THE CASINGS WERE OBSERVED FOR CHANGES IN WEIGHT AND
       SIGNS OF PHYSICAL DEGRADATION (SWELLING, SOFTENING, DECREASE IN
       STRENGTH, DETERIORATION, OR DISSOLUTION). AS EXPECTED, THE TWO
       FLUORINATED POLYMERS (FEP AND PIFE) WERE THE MOST INERT MATERIALS
       TESTED. THEY WERE NOT DEGRADED BY ANY OF THE TEST CHEMICALS,
       ALTHOUGH SAMPLES EXPOSED TO A FEW ORGANIC CHEMICALS DID SHOW A
       SLIGHT WEIGHT GAIN (APPROX. 1%). AMONG THE NONFLUORINATED PRODUCTS
       TESTED, FRE WAS THE MOST INERT. THREE ORGANIC CHEMICALS CAUSED
       PARTICLES TO FLAKE FROM THE FRE SURFACE, FOLLOWED BY SEPARATION OF
       THE GLASS FIBERS, AND TWO ORGANIC CHEMICALS CAUSED WEIGHT GAINS
       EXCEEDING 10%. ALSO, HIGHLY ACIDIC CONDITIONS (PH<1) DEGRADED THIS
       MATERIAL, AND THIS MAY LIMIT THE USE OF THIS MATERIAL IN ACIDIC
       ENVIRONMENTS.
                      JG
--28 -
       ABSTRACT CLASSIFICATION: UNCLASSIFIED
--29 - INITIAL INVENTORY:
--33 - LIMITATION CODES:
                          1
--35 - SOURCE CODE:
                         037100
--36 - ITEM LOCATION:
                        EllM\Olld
--40 - GEOPOLITICAL CODE: 3302
--41 - TYPE CODE: A
- 43 - IAC DOCUMENT TYPE:
---
    5
         Œ۴
-- 1 - AD NUMBER: A234188
-- 2 - FIELDS AND GROUPS: 11/9
-- 3 - ENTRY CLASSIFICATION: UNCLASSIFIED
-- 5 - CORPORATE AUTHOR: NATIONAL CENTER FOR COMPOSITE MATERIALS RESEARCH
--
       URBANA IL
```

-- 6 - UNCLASSIFIED TITLE: CHARACTERIZATION STUDIES OF FLUORINATED

emonation.

```
EPOXY RESINS: NAVAL EXPERIMENTAL RESIN C8/1SA AS A STRUCTURAL
        MATERIAL AND FOR USE IN BLENDS AND COMPOSITES.
-- 8 -
        TITLE CLASSIFICATION: UNCLASSIFIED
-- 9 - DESCRIPTIVE NOTE: TECHNICAL REPT.,
--10 - PERSONAL AUTHORS: TWARDOWSKI, F. E.; GEIL, P. H.
--11 - REPORT DATE: JUN Ø1, 199Ø
--12 - PAGINATION: 78P MEDIA COST: $ 6.00 PRICE CODE: AA
--14 - REPORT NUMBER: UTUC-NCCMR-89-0013
--18 - MONITOR ACRONYM: XN
--19 - MONIFOR SERIES: ONR
-- 20 - REPORT CLASSIFICATION: UNCLASSIFIED
--23 - DESCRIPTORS: , ADHESION, COMPOSITE MATERIALS, CONSTRUCTION
       MATERIALS, DEGRADATION, ENVIRONMENTS, EPOXY COMPOUNDS, EPOXY RESINS,
       FILLERS, FLAMES, FLUOROPOLYMERS, LIGHTWEIGHT, LOW ANGLES, MATERIALS,
___
       MEASUREMENT, MODIFICATION, POLYMERS, PROCESSING, RESISTANCE,
-- SIZES(DIMENSIO
-- RESINS, WATER.
        SIZES(DIMENSIONS), STABILITY, THERMAL STABILITY, THERMOPLASTIC
--24 - DESCRIPTOR CLASSIFICATION:
                                      UNCLASSIF1ED
--25 - IDENTIFIERS: #EPOXY REGINS.
--26 - IDENTIFIER CLASSIFICATION: UNCLASSIFIED
--27 - ABSTRACT: EPOXY THERMOSETS ARE CURRENTLY THE MATERIAL OF CHOICE
        FOR HIGH PERFORMANCE COMPOSITE MATERIALS. THEY HAVE HIGH MODULUS,
        LOW WEIGHT, EXCELLENT ADHESION TO THE FILLER COMPONENT, AND HIGH
___
        DIMENSIONAL AND THERMAL STABILITY AS WELL AS PROCESSING
        CHARACTERISTICS MUCH MORE TRACTABLE THAN THERMOPLASTICS.
--
        UNFORTUNATELY, EPOXIES ARE OFTEN ATTACKED BY ENVIRONMENTAL ELEMENTS,
        ESPECIALLY WATER, RESULTING IN A DEGRADATION OF PROPERTIES. AS SUCH,
--
___
        MODIFICATION OF EPOXIES TO IMPROVE THEIR ENVIRONMENTAL RESISTANCE
        IS VALUABLE CONSIDERATION. EPOXY RESINS INCORPORATING LARGE FLUORINE CONTENTS HAVE MET THIS CHALLENGE, SHOWING LOW CONTACT
        ANGLE, LOW MEASURE UPTAKE AND IMPROVED FLAME RESISTANCE. WHAT
        REMAINS IS THE NEED TO INVESTIGATE THE SUITABILITY OF SUCH RESINS
        FOR USE IN STRUCTURAL MATERIALS.
--28 - ABSTRACT CLASSIFICATION:
                                       UNCLASSIFIED
--29 -
       INITIAL INVENTORY:
--33 -
        LIMITATION CODES: 1
--35 - SOURCE CODE: 422821
--36 - 11EM LOCATION: D11C/N11S
--4Ø - GEOPOLIFICAL CODE: 1/19
--41 - TYPE CODE: Ø
--42 - IAC ACCESSION NUMBER:
                                 PL-Ø55137
--43 - IAC DOCUMENT TYPE:
                           PLASTEC-MICROFICHE
       TAC SUBJECT TERMS: P--(U)MODIFIER EFFECTS, INTERFACE STRENGTH,
--44 -
        CHARACTERIZATION, FLUORINATED EPOXY, GLASS TRANSITION, DSC.
        MOISTURE ABSORPTION, BLENDS, COMPOSITES, EPON 828, SWELLING, EPOXY
        828, AGING, STRESS STRAIN, ADHESION, THERMAL STABILITY, PROPERTY
```

DEGRADATION, WATER IMMERSION, TENSILE PROPERTIES, ZZ UNLIMITED.;

<<ENTER NEXT COMMAND>>

--***

Status: Path 1 of [Dialog] ### Status: Initializing Port COM2 using (Baud 19200 Handshake XON/XOFF DataBits 7 Pari ty Even StopBits 1) ### Status: Initializing modem ... ATE1Q0V1X4&C1&D2 OK ### Status: Dialing primary number (448-4611)... ATDT448-4611 PROTOCOL: LAP-M COMPRESSION: NONE CONNECT 19200 ### Status: Connection established at 19200 baud /ARQ please type your terminal identifier -3523:01-005please log in: DIALOG DIALOG: call connected ### Status: Connected DIALOG INFORMATION SERVICES PLEASE LOGON: ****** HHHHHHHH SSSSSSSS? ### Status: Signing onto Dialog ****** ENTER PASSWORD: ****** HHHHHHH SSSSSSS? ******* Welcome to DIALOG ### Status: Connected Dialog level 42.10.06D Last logoff: 22nov96 09:36:40 Logon file405 22nov96 10:54:50 Banner display set OFF. HILIGHT set on as '*' KWIC is set to 5. BLIP set on NOTICE set ON to \$25.00 You will be prompted to confirm each TYPE or PRINT request where format charges exceeds \$25.00 COST = ONESEARCH. Please enter SUBACCOUNT name/number: ?bulluck/n7402 Is BULLUCK/N7402 the SUBACCOUNT you want to use? (Y/N) Subaccount is set to BULLUCK/N7402 SYSTEM: HOME Menu System II: D2 version 1.7.8 term=ASCII Terminal set to DLINK *** DIALOG HOMEBASE(SM) Main Menu *** Information: 1. Announcements (new files, free connect time, price changes, etc.) 2. Database, Rates, & Command Descriptions 3. Help in Choosing Databases for Your Topic

4. Customer Services (telephone assistance, training, seminars, etc.)

5. Product Descriptions

```
Connections:
  6. DIALOG Menus (SM)
  7. DIALOG Business Connection(R) and DIALOG Headlines(SM)
  8. KR SourceOne(SM) Document Delivery
     Data Star(R)
  9.
 10. Other Online Menu Services & Files (MoneyCenter(R), OAG, TNT, etc.)
              (c) 1995 Knight-Ridder Information, Inc.
      /H = Help
                           /L = Logoff
                                                /NOMENU = Command Mode
Enter an option number to view information or to connect to an online
 service. Enter a BEGIN command plus a file number to search a database
(e.g., B1 for ERIC).
?b411
       22nov96 10:54:55 User036172 Session D803.1
       Sub account: BULLUCK/N7402
                    0.001 Hrs FileHomeBase
            $0.00
           Estimated cost FileHomeBase
     $0.00
     $0.01 TYMNET
     $0.01 Estimated cost this search
     $0.01 Estimated total session cost 0.001 Hrs.
File 411:DIALINDEX(R)
DIALINDEX (R)
   (c) 1996 Knight-Ridder Info
*** DIALINDEX search results display in an abbreviated ***
*** format unless you enter the SET DETAIL ON command. ***
?sf chemlit, chemeng, plastics, material
   You have 43 files in your file list.
   (To see banners, use SHOW FILES command)
?s ((pvdf or coflon or polyvinylidene()fluoride or tefzel) and (flexible()pip? or degra
dation))/ti,de,id
Your SELECT statement is:
   s ((pvdf or coflon or polyvinylidene()fluoride or tefzel) and
(flexible()pip? or degradation))/ti,de,id
           Items
                   File
               4
                    2: INSPEC 1969-1996/Nov W3
                    8: Ei Compendex(R) 1970-1996/Dec W4
              10
                    31: World Surface Coatings Abs 1976-1996/Oct
              3
              12
                   94: JICST-EPlus 1985-1996/Oct W4
>>>Term "ID" is not defined in file 144 and is ignored
              14
                  144: Pascal 1973-1996/Oct
>>>Term "ID" is not defined in file 322 and is ignored
                   322: Polymer Online
                   323: RAPRA Rubber & Plastics 1972-1996/Dec B2
>>>Term "DE" is not defined in file 340 and is ignored
>>>Term "ID" is not defined in file 340 and is ignored
                   340: CLAIMS(R)/US PATENTS ABS 1950-1996/SEP
>>>File 399 processing for PIP? stopped at
         PIPERAZINYLALKANOYLDIHYDROBENZOFURANS
                   399: CA SEARCH(R)_1967-1996/UD=12522
              84
                   434: Scisearch(R) Cited Ref Sci 1974-1996/Nov W2
                     6: NTIS 64-1996/Dec W5
                   293: Eng Materials Abs(R) 1986-1996/Dec
>>>Term "ID" is not defined in file 108 and \overline{i}s ignored
                   108: Aerospace Database 1962-1996/Nov
   13 files have one or more items; file list includes 43 files.
```

One or more terms were invalid in 26 files.

```
?save temp
Temp SearchSave "TD228" stored
?b hits
       22nov96 10:57:23 User036172 Session D803.2
       Sub account: BULLUCK/N7402
            $1.50 0.050 Hrs File411
     $1.50 Estimated cost File411
     $0.60 TYMNET
     $2.10 Estimated cost this search
     $2.11 Estimated total session cost 0.051 Hrs.
SYSTEM:OS - DIALOG OneSearch
*File 399: Use is subject to the terms of your user/customer agreement.
For format prices, including formats 6 & 8, see HELP RATES 399.
You have 13 files in your file list.
(To see file names, coverage dates, and copyright notices, enter SHOW FILES.)
      Set Items Dscription
      --- -----
?exs td228
Hilight option is not available in file(s) 6, 293, 399
HILIGHT set on as '*'
KWIC is set to 5.
KWIC option is not available in file(s) 6, 293, 399
>>>Term "ID" is not defined in one or more files
>>>Term "DE" is not defined in one or more files
>>>File 399 processing for PIP? stopped at PIPERAZINYLALKANOYLDIHYDROBENZO
  FURANS
Processed 10 of 13 files ...
Completed processing all files
            4969 PVDF/TI, DE, ID
               3 COFLON/TI, DE, ID
           12502 POLYVINYLIDENE/TI, DE, ID
          194611 FLUORIDE/TI, DE, ID
            9055 POLYVINYLIDENE/TI, DE, ID (W) FLUORIDE/TI, DE, ID
             141 TEFZEL/TI, DE, ID
          121920 FLEXIBLE/TI, DE, ID
          365537 PIP?/TI, DE, ID
             900 FLEXIBLE/TI, DE, ID(W) PIP?/TI, DE, ID
          268842 DEGRADATION/TI, DE, ID
             272 ((PVDF OR COFLON OR POLYVINYLIDENE() FLUORIDE OR TEFZEL)
      S1
                  AND (FLEXIBLE()PIP? OR DEGRADATION))/TI,DE,ID
?rd s1
>>>Duplicate detection is not supported for File 94.
>>>Duplicate detection is not supported for File 322.
>>>Duplicate detection is not supported for File 340.
>>>Records from unsupported files will be retained in the RD set.
...examined 50 records (50)
...examined 50 records
                        (100)
...examined 50 records
                        (150)
...examined 50 records
...examined 50 records
                        (250)
...completed examining records
      S2
             253 RD S1 (unique items)
?s s2/eng
>>>Term "ENG" is not defined in one or more files
     S3
           183 S2/ENG
?s s3 and py=1992:8888
>>>One or more prefixes are unsupported
>>> or undefined in one or more files.
Processed 10 of 13 files ...
Completed processing all files
             183 S3
        13684327 PY=1992 : PY=8888
```

```
47 S3 AND PY=1992:8888
      S4
?t 4/6/all
 4/6/1
          (Item 1 from file: 8)
04187181
  Title: Piezo- and pyroelectric properties of dehydrofluorinated *PVDF*
 films
  Conference Title: Proceedings of the 8th International Symposium on
Electrets (ISE 8)
 4/6/2
           (Item 2 from file: 8)
03681363
  Title: Advanced *flexible* *pipe* materials for aggressive hydrocarbon
service
  Conference Title: Proceedings of the Third (1993) International Offshore
and Polar Engineering Conference
 4/6/3
           (Item 3 from file: 8)
03641106
  Title: Application of TGA/FTIR to the thermal *degradation* mechanism of
tetrafluoroethylene-propylene copolymers
 4/6/4
           (Item 4 from file: 8)
03623563
   Title:
           XPS
                 studies of radiation-induced structural changes in
*polyvinylidene* *fluoride*
 4/6/5
           (Item 5 from file: 8)
03423047
 Title: X-ray induced *degradation* of poly(vinylidene fluoride) films.
 4/6/6
           (Item 1 from file: 94)
03333595
          JICST ACCESSION NUMBER: 96A0668980 FILE SEGMENT: JICST-E
Systematic Peptide Fragmentation of Polyvinylidene Difluoride(*PVDF*
   )-Immobilizes Proteins Prior to Microsequencing.
4/6/7
           (Item 1 from file: 144)
 12383888
            PASCAL No.: 96-0030620
Thermal decomposition kinetics of a commercial fluoropolymer
4/6/8
           (Item 2 from file: 144)
 12251841
            PASCAL No.: 95-0476866
KeV ion beam irradiation of *polyvinylidene* *fluoride* (*PVDF*)
4/6/9
          (Item 3 from file: 144)
 12251412 PASCAL No.: 95-0476413
Temperature influence on the gas desorption from *polyvinylidene*
*fluoride* (*PVDF*) irradiated with helium beams
4/6/10
           (Item 4 from file: 144)
 11583591
            PASCAL No.: 94-0469531
A FTIR study of *PVDF* irradiated by means of swift heavy ions.
4/6/11
           (Item 5 from file: 144)
 11504125
            PASCAL No.: 94-0344727
Utilization of *PVDF* sensors to determine impact damage in
```

graphite/epoxy plates by acousto-ultrasonic technique

.

4/6/12 (Item 6 from file: 144)
11349383 PASCAL No.: 94-0171771
Monosaccharide and oligosaccharide analysis of glycoproteins electrotransferred onto *PVDF* membranes

4/6/13 (Item 7 from file: 144) 10964625 PASCAL No.: 93-0474091

Retention of beer spoilage microorganisms by *polyvinylidene* *fluoride* microporous membranes with various retention ratings

4/6/14 (Item 1 from file: 323)

00585817

TITLE: FAMILY FORTUNES

4/6/15 (Item 2 from file: 323)

00581044

TITLE: RADIATION EFFECTS ON FLUOROPOLYMERS: RADIATION-INDUCED STRUCTURAL AND CRYSTALLINITY CHANGES OF *TEFZEL*

4/6/16 (Item 3 from file: 323)

00577457

TITLE: COMPARISON OF HIGH PERFORMANCE, CLEAR FILMS USED FOR LONG TERM PRODUCTION

4/6/17 (Item 4 from file: 323)

00577237

TITLE: POLYMER COMPOSITIONS INTENDED FOR THE MANUFACTURE OF CABLES AND *FLEXIBLE* *PIPES* AND ARTICLES BASED ON THESE COMPOSITIONS

4/6/18 (Item 5 from file: 323)

00576607

TITLE: ACID-BASE INTERACTIONS AT POLYMER INTERFACES

4/6/19 (Item 6 from file: 323)

00575390

TITLE: CRYSTALLISATION BEHAVIOUR AND PHASE DIAGRAM OF EXTENDED-CHAIN CRYSTALS OF POLY(VINYLIDENE FLUORIDE) UNDER HIGH PRESSURE

4/6/20 (Item 7 from file: 323)

00564145

TITLE: RADIATION-INDUCED ENHANCEMENT OF CRYSTALLINITY IN POLYMERS

4/6/21 (Item 8 from file: 323)

00559287

TITLE: ELECTROCHEMISTRY AS THE WAY TO TRANSFORM POLYMERS

4/6/22 (Item 9 from file: 323)

00559286

TITLE: ELECTROCHEMICALLY INDUCED FUNCTIONALISATION OF FLUOROCONTAINING POLYOLEFINS

4/6/23 (Item 10 from file: 323)

00559052

TITLE: BLENDS OF GLYCIDYL METHACRYLATE (GMA) / METHYL METHACRYLATE (MMA)

4/6/24 (Item 11 from file: 323)

00554472

TITLE: POLYMER SUPPORTS IN SYNTHESIS

4/6/25 (Item 12 from file: 323)

00549288

TITLE: YIELD BEHAVIOUR OF *PVDF* AND THE DEFORMATION PROCESS AT HIGH TEMPERATURE

4/6/26 (Item 13 from file: 323)

00509576

TITLE: STUDY OF THE THERMAL *DEGRADATION* OF POLYCHLOROTRIFLUOROETHYLENE, *POLYVINYLIDENE* *FLUORIDE* AND COPOLYMERS OF CHLOROTRIFLUOROETHYLENE AND VINYLIDENE FLUORIDE

4/6/27 (Item 14 from file: 323)

00496424

TITLE: RADIATION EFFECTS ON *PVDF*

4/6/28 (Item 15 from file: 323)

00492182

TITLE: GRAFTING OF SILICON PHTHALOCYANINE DICHLORIDE ONTO *PVDF* FILM SURFACES BY ULTRASOUND

4/6/29 (Item 16 from file: 323)

00474223

TITLE: PLASTICS HEAT EXCHANGERS

4/6/30 (Item 17 from file: 323)

00472912

TITLE: SEEKING THE PERFECT BLEND

4/6/31 (Item 18 from file: 323)

00470711

TITLE: X-RAY PHOTOELECTRON SPECTROSCOPY(XPS) STUDIES OF RADIATION-INDUCED STRUCTURAL CHANGES IN *POLYVINYLIDENE* *FLUORIDE*

4/6/32 (Item 19 from file: 323)

00448524

TITLE: REACTIONS OF ATOMIC OXYGEN WITH POLYMER FILMS

4/6/33 (Item 1 from file: 340)

2620850 9516422

C/POLYMER COMPOSITIONS INTENDED FOR THE MANUFACTURE OF CABLES AND *FLEXIBLE* *PIPES* AND ARTICLES BASED ON THESE COMPOSITIONS;

POLYVINYLIDENE *FLUORIDE* BLEND

4/6/34 (Item 2 from file: 340)

2439164 9402057

C/METHOD FOR EXTRUDING POLYOLEFINS CONTAINING VINYLIDENE CHLORIDE POLYMERS; COATING EQUIPMENT WITH *POLYVINYLIDENE* *FLUORIDE* TO PREVENT *DEGRADATION* OF THE CHLOROPOLYMER

4/6/35 (Item 1 from file: 399) DIALOG(R) File 399: (c) 1996 American Chemical Society. All rts. reserv. KeV-MeV ion irradiation of polyvinylidene fluoride (PVDF) films (Item 2 from file: 399) 4/6/35 DIALOG(R) File 399:(c) 1996 American Chemical Society. All rts. reserv. Photodegradation of fluorocarbon resin films. A new evaluation method using an ESR spectrometer 4/6/37 (Item 3 from file: 399) DIALOG(R) File 399: (c) 1996 American Chemical Society. All rts. reserv. Testing the utility of EIS measurements to predict and monitor the behavior of organically coated aluminum during atmospheric exposure 4/6/38 (Item 4 from file: 399) DIALOG(R) File 399:(c) 1996 American Chemical Society. All rts. reserv. Swift heavy ion modification of polymers 4/6/39 (Item 5 from file: 399) DIALOG(R) File 399:(c) 1996 American Chemical Society. All rts. reserv. Radical in heavy ion-irradiated polyvinylidene fluoride 4/6/40 (Item 6 from file: 399) DIALOG(R) File 399:(c) 1996 American Chemical Society. All rts. reserv. Photoacoustic detection of the decomposition kinetics of polymers: interpretation of acoustic signals 4/6/41 (Item 7 from file: 399) DIALOG(R) File 399:(c) 1996 American Chemical Society. All rts. reserv. Crystallite damage studies on irradiated poly(vinylidene fluoride) 4/6/42 (Item 8 from file: 399) DIALOG(R) File 399: (c) 1996 American Chemical Society. All rts. reserv. Derivatization of polyvinylidene difluoride membranes for the solid-phase sequence analysis of a phosphorylated sea urchin embryo histone H1 peptide (Item 9 from file: 399) DIALOG(R) File 399:(c) 1996 American Chemical Society. All rts. reserv. Surface analysis of poly(vinylidene difluoride) membranes 4/6/44 (Item 10 from file: 399) DIALOG(R) File 399:(c) 1996 American Chemical Society. All rts. reserv. Low dose .gamma.-irradiation of some fluoropolymers: effect of polymer

Number of References: 15

chemical structure

(Item 1 from file: 434)

Genuine Article#: TF649

4/6/45

143495-54

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Abstract Available)
 4/6/46
            (Item 1 from file: 6)
1787467 NTIS Accession Number: PB94-886397/XAB
 Polyvinylidene Fluoride: Structure and Degradation. (Latest citations
from the Rubber and Plastics Research Association Database)
  (Published Search)
  NTIS Prices: PC N01/MF N01
 4/6/47
            (Item 1 from file: 293)
126857
Thermal Degradation of Commercial Fluoropolymers in Air.
### Status: Signing Off...
logoff
       22nov96 11:01:37 User036172 Session D803.3
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$3.50 47 Types
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    $1.00 TYMNET
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Title: THERMAL-DECOMPOSITION KINETICS OF A COMMERCIAL FLUOROPOLYMER (

Status: Signed Off. (7 minutes)

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4. Customer Services (telephone assistance, training, seminars, etc.)

5. Product Descriptions

Connections:

- 6. DIALOG Menus (SM)
- 7. DIALOG Business Connection(R) and DIALOG Headlines(SM)
- 8. KR SourceOne(SM) Document Delivery
- 9. Data Star(R)
- 10. Other Online Menu Services & Files (MoneyCenter(R), OAG, TNT, etc.)
 - (c) 1995 Knight-Ridder Information, Inc.

/H = Help /L = Logoff /NOMENU = Command Mode

Enter an option number to view information or to connect to an online service. Enter a BEGIN command plus a file number to search a database (e.g., B1 for ERIC). ?b8

22nov96 15:00:36 User036172 Session D804.1

Sub account: BULLUCK/N7402

\$0.00 0.001 Hrs FileHomeBase

- \$0.00 Estimated cost FileHomeBase
- \$0.01 TYMNET
- \$0.01 Estimated cost this search
- \$0.01 Estimated total session cost 0.001 Hrs.

File 8:Ei Compendex(R) 1970-1996/Dec W4

(c) 1996 Engineering Info. Inc.

Set Items Description

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so 2 03641106

?t 0/5/all

0/5/1

DIALOG(R) File 8:Ei Compendex(R)

(c) 1996 Engineering Info. Inc. All rts. reserv.

03681363 E.I. No: EIP93081046180

Title: Advanced flexible pipe materials for aggressive hydrocarbon service

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

Corporate Source: Wellstream Corp, Panama City, FL, USA

Conference Title: Proceedings of the Third (1993) International Offshore and Polar Engineering Conference

Conference Location: Singapore, Singapore Conference Date: 19930606-19930611

E.I. Conference No.: 18719

Source: 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 Engineerns (ISOPE), P.O.Box 1107, Golden, CO, USA. p 359-364

Publication Year: 1993

ISBN: 1-880653-09-5

Language: English

Document Type: CA; (Conference Article) Treatment: X; (Experimental)

Journal Announcement: 9309W4

Abstract: The increasing development of marginal offshore hydrocarbon deposits has resuld 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 H//2S and CO//2 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 PVDF homopolymer fluid
barrier material. (Author abstract) 7 Refs.
  Descriptors: *Offshore pipelines; Submarine pipelines; Corrosion
 resistance; Seawater effects; Plastic pipe
   Identifiers: Flexible pipes; Plasticized homopolymer; Corrosive fluids;
PVDF homopolymer, Carcass layer
  Classification Codes:
   619.1.2 (Pipe Materials)
   619.1 (Pipe, Piping & Pipelines); 539.2 (Corrosion Protection); 511.2
 (Oil Field Equipment)
  619 (Pipes, Tanks & Accessories); 539 (Metals Corrosion & Protection);
511 (Oil Field Equipment & Production Operations)
  61 (PLANT & POWER ENGINEERING); 53 (METALLURGICAL ENGINEERING); 51
 (PETROLEUM ENGINEERING)
 0/5/2
DIALOG(R) File 8: Ei Compendex(R)
(c) 1996 Engineering Info. Inc. All rts. reserv.
03641106
          E.I. No: EIP93050995198
  Title: Application of TGA/FTIR to the thermal degradation mechanism of
tetrafluoroethylene-propylene copolymers
  Author: Schild, H.G.
  Source: Journal of Polymer Science, Part A: Polymer Chemistry v 31 n 6
May 1993. p 1629-1632
  Publication Year: 1993
  CODEN: JPACEC ISSN: 0887-624X
  Language: English
  Document Type: JA; (Journal Article) Treatment: A; (Applications); G;
(General Review); X; (Experimental)
  Journal Announcement: 9307W4
  Abstract: Aflas, produced by Asahi Glass Co. Ltd., Tokyo, Japan and
marketed in the United States by 3M, is a high-performance elastomeric
tetrafluoroethylene (TFE)/propylene copolymer with superior high
temperature stability, electrical, and chemical resistance properties.
(Edited author abstract) 14 Refs.
  Descriptors: *Copolymers; Polypropylenes; Polytetrafluoroethylenes;
Pyrolysis; Thermoanalysis; Infrared spectroscopy
  Identifiers: Aflas; Tefzel; Thermogravimetric analysis; Fourier transform
infrared spectroscopy; Tetrafluoroethylene; Reaction mechanism
  Classification Codes:
  815.1.1 (Organic Polymers)
  815.1 (Polymeric Materials); 802.2 (Chemical Reactions); 801.1
(Chemistry, General)
  815 (Plastics & Polymeric Materials); 802 (Chemical Apparatus & Plants)
; 801 (Chemical Analysis & Physical Chemistry)
  81 (CHEMICAL PROCESS INDUSTRIES); 80 (CHEMICAL ENGINEERING)
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     $4.63 Estimated cost this search
     $4.64 Estimated total session cost 0.018 Hrs.
File 144: Pascal 1973-1996/Oct
       (c) 1996 INIST/CNRS
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?t 0/5

0/5/1

DIALOG(R) File 144: Pascal

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12383888 PASCAL No.: 96-0030620

Thermal decomposition kinetics of a commercial fluoropolymer

VISHWA PRASAD A; SINGH R P

National chemical lab., polymer chemistry div., Pune 411 008, India

Journal: Materials research bulletin, 1995, 30 (11) 1407-1412

ISSN: 0025-5408 CODEN: MRBUAC Availability: INIST-13343;

354000059050140120

No. of Refs.: 15 ref.

Document Type: P (Serial) ; A (Analytic)

Country of Publication: USA

Language: English

Thermal decomposition kinetics of a fluoropolymer (Tefzel) in the temperature range 359-550 Degree C by thermogravimetry (TG) and 235-270 Degree C by differential scanning calorimetry (DSC) was investigated under nitrogen flux. The temperature range studied (225-550 Degree C) includes the range of recommended continuous use and processing temperature of these commercial resins. The activation energy (DELTA E) for decomposition was 53.33 k.cal mole SUP - SUP 1 in TG at 10 Degree C/min rate, whereas it was 9.49 k.cal mole SUP - SUP 1 in DSC. The effect of heating rate on TG and DSC thermograms was studied.

English Descriptors: Fluoroelastomer; Ethylene copolymer;

Tetrafluoroethylene copolymer; Commercial form; Thermal degradation; Kinetics; Rate constant; Activation energy; Heating rate; Experimental study

French Descriptors: Caoutchouc fluor; Ethylene copolymere; Ethylene(tetrafluoro) copolymere; Forme commerciale; Degradation thermique; Cinetique; Constante vitesse; Energie activation; Vitesse chauffage; Etude experimentale; Tefzel

Classification Codes: 001D09D03D ?b323

22nov96 15:01:51 User036172 Session D804.3

Sub account: BULLUCK/N7402

\$0.96 0.016 Hrs File144

\$1.15 1 Types

\$2.11 Estimated cost File144

\$0.19 TYMNET
\$2.30 Estimated cost this search

\$6.94 Estimated total session cost 0.034 Hrs.

File 323: RAPRA Rubber & Plastics 1972-1996/Dec B2 (c) 1996 RAPRA Technol Ltd

Set Items Description

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?k 00549288;k 00509576

SO 1 00549288

S0 2 00509576

?t 0/5/all

DIALOG(R)File 323:RAPRA Rubber & Plastics (c) 1996 RAPRA Technol Ltd. All rts. reserv.

00549288

TITLE: YIELD BEHAVIOUR OF PVDF AND THE DEFORMATION PROCESS AT HIGH

TEMPERATURE

AUTHOR(S): Hellinckx S; Bauwens J C

CORPORATE SOURCE: Brussels, Free University

SOURCE: Coll.Polym.Sci.; 273, No.3, March 1995, p.219-26

CODEN: CPMSB6 JOURNAL ANNOUNCEMENT: 9507 RAPRA UPDATE: 9513

DOCUMENT TYPE: Journal LANGUAGE: English SUBFILE: (R) RAPRA

ABSTRACT: Tensile yield stress measurements on PVDF were carried out, covering as many as seven decades of strain rates at temps. from -50 to 150C. The data were analysed on the basis of four Ree-Eyring processes acting in parallel, three of which were identified. At high temps. and low strain rates, a threshold yield stress was observed. A model was proposed, permitting description of the yield behaviour both at the threshold and in its adjacent strain rate dependent range. It consisted of a modification of the Ree-Eyring theory using an asymmetrical free energy barrier for the rate process. The data were consistent with the above model, which implied that yielding occurred only when the stress was higher than a threshold. In this case, a rejuvenation (or de-ageing) effect should be assumed in the deformation process which could consist of a melting of a chain segment located between two folds. 21 refs.

SUBJECT HEADING (RAPRA): TENSILE PROPERTIES, PVDF; VINYLIDENE FLUORIDE POLYMERS, tensile properties

GEOGRAPHIC LOCATION: BELGIUM; EUROPEAN COMMUNITY; EUROPEAN UNION; WESTERN EUROPE

DESCRIPTORS: DATA; DEFORMATION; FLUOROPOLYMER; FREE ENERGY; GRAPH; HIGH TEMPERATURE; LOW TEMPERATURE; MECHANICAL PROPERTIES; MELTING; MODEL; PLASTIC; POLYVINYLIDENE FLUORIDE; PROPERTIES; PVDF; STRAIN RATE; TABLES; TECHNICAL; TEMPERATURE; TENSILE PROPERTIES; THEORY; THERMAL DEGRADATION; THERMOPLASTIC; YIELD; YIELD STRESS

RAPRA CLASSIFICATION CODE: 42C386; 95111

CATEGORY CODES: UG; KM

0/5/2

DIALOG(R) File 323: RAPRA Rubber & Plastics (c) 1996 RAPRA Technol Ltd. All rts. reserv.

00509576

TITLE: STUDY OF THE THERMAL DEGRADATION OF POLYCHLOROTRIFLUOROETHYLENE, POLYVINYLIDENE FLUORIDE AND COPOLYMERS OF CHLOROTRIFLUOROETHYLENE AND VINYLIDENE FLUORIDE

AUTHOR(S): Zulfiqar S; Zulfiqar M; Rizvi M; Munir A; McNeill I C CORPORATE SOURCE: Quaid-i-Azam, University; Glasgow, University

SOURCE: Polym.Degradat.Stabil.; 43, No.3, 1994, p.423-30

CODEN: PDSTDW JOURNAL ANNOUNCEMENT: 9406 RAPRA UPDATE: 9410

DOCUMENT TYPE: Journal

LANGUAGE: English SUBFILE: (R) RAPRA

ABSTRACT: A systematic study of the thermal degradation of a series of homopolymers and copolymers of chlorotrifluoroethylene and vinylidene fluoride was carried out using TGA and thermal volatilisation analysis (TVA). Volatile products were separated by sub-ambient TVA and characterised by means of IR spectroscopy and mass spectrometry. On degradation, polychlorotrifluoroethylene gave the monomer as the major product and carbon dioxide, C2F2Cl2, C3F5Cl and C2F3Cl3 in traces. Polyvinylidene fluoride formed hydrogen fluoride in appreciable amount along with the monomer and C4H3F3. The copolymers showed a similar type of degradation pattern. The structural changes which took place during degradation were also studied and mechanisms of formation of the various products are discussed. 12 refs.

SUBJECT HEADING (RAPRA): DEGRADATION, thermal, PCTFE, PVDF; CHLOROTRIFLUOROETHYLENE POLYMERS, thermal degradation; VINYLIDENE FLUORIDE POLYMERS, thermal degradation

IDENTIFIERS (Non-Polymer Terms): CARBON DIOXIDE; CHLORINE COMPOUND; CHLOROPENTA; LUOROPENE; DICHLORODIFLUOROPROPENE; FLUORINE COMPOUND; HYDROFLUORIC; ACID; HYDROGEN FLUORIDE; TRICHLOROTRIFLUOROPROPENE; TRIFLUOROBUTADIENE

GEOGRAPHIC LOCATION: EUROPEAN COMMUNITY; PAKISTAN; UK; WESTERN EUROPE DESCRIPTORS: CHARACTERISATION; CHLOROTRIFLUOROETHYLENE COPOLYMER; DATA; DEGRADATION; DEGRADATION PRODUCT; FLUOROPOLYMER; GRAPH; IR SPECTROSCOPY ; MASS SPECTROMETRY; MECHANISM; PCTFE; PLASTIC; POLYTRIFLUOROCHLOROETHYLENE; POLYVINYLIDENE FLUORIDE; PROPERTIES; PVDF; TABLES; TECHNICAL; TGA; THERMAL DEGRADATION; THERMAL VOLATILISATION ANALYSIS; THERMOGRAVIMETRIC ANALYSIS; THERMOPLASTIC; TRIFLUOROCHLOROETHYLENE COPOLYMER; VINYLIDENE FLUORIDE COPOLYMER; VOLATILE

RAPRA CLASSIFICATION CODE: 42C38; 932 CATEGORY CODES: UE; KM

?b340

22nov96 15:02:34 User036172 Session D804.4

Sub account: BULLUCK/N7402

0.016 Hrs File323 \$0.96

\$3.80 2 Types

\$4.76 Estimated cost File323 \$0.19 TYMNET

\$4.95 Estimated cost this search \$11.89 Estimated total session cost 0.051 Hrs.

File 340:CLAIMS(R)/US PATENTS ABS 1950-1996/SEP (c) 1996 IFI/Plenum Data Corp.

Set Items Description ?k 2620850 S0 1 2620850 ?t 0/5

0/5/1

DIALOG(R) File 340: CLAIMS(R) /US PATENTS ABS (c) 1996 IFI/Plenum Data Corp. All rts. reserv.

2620850 9516422

C/POLYMER COMPOSITIONS INTENDED FOR THE MANUFACTURE OF CABLES AND FLEXIBLE PIPES AND ARTICLES BASED ON THESE COMPOSITIONS; POLYVINYLIDENE FLUORIDE BLEND

Document Type: UTILITY

Inventors: Lambert Yves-Julian (BE); Lasson Pierre (BE); Maquet Nestor (BE) ; Thulliez Vincent (BE); Vanderveken Yves (BE)

Assignee: Solvay S A BE Assignee Code: 78176

	Patent Number	Issue Date	Applic Number	Applic Date
Patent:	US 5429849	950704	US 188418	940124
Priority Applic:			BE 9300070	930125

Polymer compositions based on fluoropolymers intended especially for the manufacture of electrical cables and of flexible pipes, comprising, by weight, (A) from 25 to 75% of PVDF homopolymer; (B) from 25 to 75% of a thermoplastic copolymer of VF2 and of at least one other fluoromonomer, exhibiting a content of 5 to 25% of this other monomer. This other fluorocomonomer may be in particular CTFE (chlorotrifluoroethylene), HFP (hexafluoropropylene) or TrFE (trifluoroethylene). Articles produced from these compositions have good mechanical properties at low temperature.

Exemplary Claim:

1. A polymer composition based on polyvinylidene fluoride (PVDF) homopolymer and at least one fluorocopolymer, comprising: a mixture of fluoropolymers comprised of, by weight: (A) from about 25 to about 75% of at least one PVDF homopolymer; and (B) from about 25 to about 75% of at least one thermoplastic copolymer comprising vinylidene fluoride (VF2) and from about 5 to about 25% by weight of at least one other fluoromonomer selected from the group consisting of hexafluoropropylene and chlorotrifluoroethylene.

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Class: 428036900
Class Cross Ref: 428379000; 525199000
IPC: C08L-027/16
IPC Cross Ref: B29D-022/00; B29D-023/22
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            Estimated cost this search
    $15.10 Estimated total session cost
                                           0.068 Hrs.
File 399:CA SEARCH(R) 1967-1996/UD=12522
       (c) 1996 American Chemical Society
*File 399: Use is subject to the terms of your user/customer agreement.
For format prices, including formats 6 & 8, see HELP RATES 399.
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           43550 DECOMPOSITION/TI
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          110434 KINETICS/TI
          125120 POLYMERS/TI
                               (SEE ?CLASS)
      S1
                  (PHOTOACOUSTIC AND DECOMPOSITION AND KINETICS AND
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?t 1/9
 1/9/1
DIALOG(R) File 399:CA SEARCH(R)
(c) 1996 American Chemical Society. All rts. reserv.
 121058553
              CA: 121(6)58553r
                                  JOURNAL
 Photoacoustic detection of the decomposition kinetics of polymers:
interpretation of acoustic signals
  AUTHOR(S): Kukreja, L. M.; Hess, P.
  LOCATION: Institute of Physical Chemistry, University of Heidelberg, Im
Neuenheimer Feld 253, Heidelberg, Germany, 69120
  JOURNAL: Appl. Surf. Sci. DATE: 1994 VOLUME: 79-80 NUMBER: 1-4
  PAGES: 399-402 CODEN: ASUSEE ISSN: 0169-4332 LANGUAGE: English
  SECTION:
CA237004 Plastics Manufacture and Processing
CA273XXX Optical, Electron, and Mass Spectroscopy, and Other Related
  IDENTIFIERS: polyimide degrdn kinetics photoacoustic detection, PVDF
piezoelec transducer polyimide ablation
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Photoacoustic effect...
    in laser-induced ablation of polyimides
Polyethers, polyimide-, properties... Polyimides, polyether-, properties...
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Ablation, laser-induced... Polymer degradation, laser-induced...
    of polyimides, photoacoustic detection of
Kinetics of polymer degradation, photochem....
    with laser source, of polyimides, photoacoustic detection of
  CAS REGISTRY NUMBERS:
25036-53-7 25038-81-7 laser-induced degrdn. of, PVDF for photoacoustic
    detection of
24937-79-9 piezoelec. foil transducer, for photoacoustic detection of
    polyimide degrdn.
?s (crystallite and irradiated and poly(vinylidene()fluoride))/ti
             783 CRYSTALLITE/TI
           24726 IRRADIATED/TI
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 DIALOG(R) File 399:CA SEARCH(R)
 (c) 1996 American Chemical Society. All rts. reserv.
  120219059
               CA: 120(18)219059f
                                      JOURNAL
  Crystallite damage studies on irradiated poly(vinylidene fluoride)
   AUTHOR(S): Zhao Zhudi; Chu Jin; Chen Xinfang
   LOCATION: Inst. Mater. Sci., Jilin Univ., Changchun, Peop. Rep. China,
 130023
   JOURNAL: Radiat. Phys. Chem. DATE: 1994 VOLUME: 43 NUMBER: 6 PAGES:
 523-6 CODEN: RPCHDM ISSN: 0146-5724 LANGUAGE: English
   SECTION:
 CA236005 Physical Properties of Synthetic High Polymers
   IDENTIFIERS: PVDF crystallite radiation damage, polyvinylidene fluoride
 crystallite irradn
   DESCRIPTORS:
 Crystallites...
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 Polymer degradation, radiochem....
     of poly(vinylidene fluoride), crystallite damage in
 Crystallinity... Heat of fusion and Heat of freezing... Polymer
 morphology, cryst., spherulitic...
     of poly(vinylidene fluoride), radiation effect on
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 24937-79-9 crystallite damage of irradiated
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                              (SEE ?IGNOTE)
           306491 ANALYSIS/TI (SEE ?IGNOTE)
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 6/9/1
DIALOG(R) File 399:CA SEARCH(R)
(c) 1996 American Chemical Society. All rts. reserv.
 117050189
              CA: 117(6)50189y
                                  JOURNAL
 Surface analysis of poly(vinylidene difluoride) membranes
  AUTHOR(S): Floesch, D.; Lehmann, H. D.; Reichl, R.; Inacker, O.; Goepel,
  LOCATION: Inst. Phys. Theor. Chem., Univ. Tuebingen, Tuebingen, Germany,
  JOURNAL: J. Membr. Sci. DATE: 1992 VOLUME: 70 NUMBER: 1 PAGES: 53-63
  CODEN: JMESDO ISSN: 0376-7388 LANGUAGE: English
  SECTION:
CA238003 Plastics Fabrication and Uses
  IDENTIFIERS: PVDF membrane surface analysis spectroscopy
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DESCRIPTORS:
Membranes, microporous...
    hydrophilic and hydrophobic PVDF, surface anal. of, by x-ray, IR and
    mass spectroscopy
Fluoropolymers...
    membranes, hydrophilic and hydrophobic, surface anal. of, by x-ray, IR
    and mass spectroscopy
Surface analysis...
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Polymer degradation, radiochem....
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Surface structure...
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Mass spectra, secondary-ion...
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                    0.100 Hrs File399
            $5.85 3 Types
    $17.85 Estimated cost File399
     $1.20 TYMNET
    $19.05 Estimated cost this search
$34.15 Estimated total session cost 0.168 Hrs.
```

Status: Signed Off. (9 minutes)