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AMRL-69-71

IDENTIFICATION OF VOLATILE CONTAMINANTS OF SPACE CABIN MATERIALS

J. V. PUSINGER, JR.

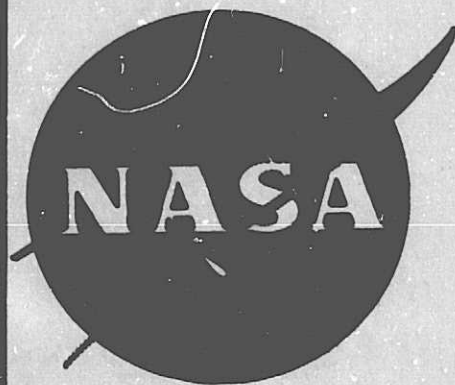
F. N. HODGSON

J. E. STROBEL

R. L. EVERS

Monsanto Research Corporation

JOINT NASA/USAF STUDY



OCTOBER 1969

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13. ABSTRACT Nineteen candidate space cabin materials were tested to determine weight-loss characteristics and to identify their gas-off products. The materials were screened initially, using thermogravimetric techniques for measuring weight loss at moderate temperatures (ambient to 68 ± 2°C) for 22.5 hours in 5 psia nitrogen, to select those candidate materials that lose from 0.001 to 1.0% of their weight, excluding water. The selected materials were then stored in 9-liter chambers at 68 ± 2°C for 72 hours and at 25 ± 2°C for 30 days. Atmosphere in the chambers was oxygen at a pressure of 5 psia. The gaseous contaminants evolved from the test materials were identified by combinations of gas chromatography and mass spectrometry.		
<u>Key Words</u>		
Space cabin candidate materials Volatile contaminant analyses Mass spectrometry Gas chromatography		

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FOREWORD

The study was conducted at the Dayton Laboratory of Monsanto Research Corporation, Dayton, Ohio, under Contract No. F33615-67-C-1357. The principal investigator was Mr. F. Neil Hodgson for Monsanto Research Corporation. The study was started in January 1969 and was completed in June 1969. The entire study was under the project leadership of Mr. John V. Pustinger, Jr. of Monsanto Research Corporation.

This research was initiated by the Chemical Hazards Branch, Toxic Hazards Division in support of Project 6302, "Toxic Hazards of Propellants and Materials," Task 630204, "Environmental Pollution," Work Unit 008, "Identification of Volatile Contaminants from Space Cabin Materials." Dr. Gerd A. Kleineberg of the Chemical Hazards Branch was the contract monitor for the Aerospace Medical Research Laboratory.

This is the fifth of a series of reports on the identification of volatile contaminants of space cabin materials. Previous reports were AMRL-TR-66-53, AMRL-TR-67-58, AMRL-TR-68-27, and AMRL-TR-69-18.

This technical report has been reviewed and is approved.

CLYDE H. KRATOCHVIL, Colonel, USAF, MC
Commander
Aerospace Medical Research Laboratory

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SECTION I

INTRODUCTION

As a continuation of a series of material evaluation studies (refs. 1,2,3,4), 19 candidate space cabin materials were tested to determine weight-loss characteristics and to identify their gas-off products. The materials were screened initially, using thermogravimetric techniques for measuring weight loss at moderate temperatures (ambient to $68 \pm 1^\circ\text{C}$) for 22.5 hours in 5 psia nitrogen, to select those candidate materials that lose from 0.001 to 1.0% of their weight, excluding water. The selected materials were then stored in 9-liter chambers at $68 \pm 2^\circ\text{C}$ for 72 hours and at $25 \pm 2^\circ\text{C}$ for 30 days. Atmosphere in the chambers was oxygen at a pressure of 5 psia. The gaseous contaminants evolved from the test materials were identified by combinations of gas chromatography and mass spectrometry.

SECTION II

GAS-OFF EXPERIMENTS

A. EXPERIMENTAL METHOD

1. Types of Candidate Materials and Sample Preparation

Table I lists the candidate materials for cabin construction used in these experiments; all materials tested are commercial products provided by the Government. In some cases, the materials were prepared by the Air Force prior to testing. Whenever sample preparation was required, as in the case of some paints or two-part resins, mixing or curing was accomplished according to procedures provided by the manufacturers or the Air Force.

Materials such as paints and inks were applied to an aluminum foil substrate and subsequently tested.

Specimens used for thermogravimetric analysis (TGA) were conditioned at 23°C in a desiccator over phosphorus pentoxide for 24 hours prior to testing. For storage tests at 72 hours and 30 days, no pretreatment of samples was performed beyond the curing procedures cited by the manufacturers or the Air Force. The procedure for preconditioning the TGA specimens was devised to minimize adsorbed water and to put all samples on the same basis for comparing relative weight loss.

For storage tests of 72 hours and 30 days, a weighed portion of each sample was placed into a 9-liter chamber in such a manner as to expose the largest possible surface area. Generally, approximately 10-gram specimens were used; however, in cases where less sample was available, or when the bulk volume of the sample was excessively large, smaller specimens were used. When the bulk volume was too large and subdividing was necessary, freshly exposed surfaces were further cured at ambient conditions, i.e., 23°C and atmospheric air pressure, for 30 days or a minimum of 14 days.

Individual specimens of each candidate material were contained in 9-liter, borosilicate glass chambers for 30 days at $25 \pm 2^\circ\text{C}$ and for 72 hours at $68 \pm 2^\circ\text{C}$ under an oxygen atmosphere at 5 psia and 20-40% relative humidity. The chamber design and pretreatment of the chambers were the same as reported earlier (ref. 1). Control chambers (containing only aluminum foil) were processed concurrently with those chambers containing the test materials. No contamination was detected from the control chambers.

Table I

SPACE CABIN TEST MATERIALS

<u>DAC No.</u>	<u>Material</u>
004	Adhesive, Epon 919
009	Cat-L-Ink W/20 cat. (Yellow)
010	Tefglas Tape, DMS 1603A (Size 2, Lot 80367)
020	Boltaron (6200) PVC Type I, (Grey)
023	Selectron (5016)
024	Nextel Velvet Coating 401-C10-Black
038	Silicone Rubber RTV-1016 W/Cat. RTV 9910
041	Rod PO#RI45074 (4 ft. x 1-3/4 in.)
047	Scotchply Reinforced Plastic (Type 1100)
048	Pyralin, Polyimide (35-502-38)
049	Royalite (Imitation Leather) (R-56-8163)
050	Polasheet 11[73-3062(07-0701-3006)]
051	Zytel Nylon Resin, Type 103 (Color NC10)
052	Durez Molding Compound 1900 (Black Phenolic)
053	Nopcofoam G-302
054	Electrical Tape, TFE Fluorocarbon Film (Silicone) (No. 60)
055	Pressure Sensitive Adhesive Tape, E-284-6-ERH, Run 2355
056	Printed Circuit Board
057	Scotch Tape, #4116 YAK 41171T

2. Preparation of Chamber Atmospheres

After insertion of each specimen, the 9-liter test chamber was filled to a pressure of one atmosphere with oxygen saturated with water vapor. The gas was saturated with water by bubbling 99.5% oxygen (conforming to Type I of MIL-O-27210) through triple distilled water at 23°C. Test conditions were attained by subsequently reducing the pressure in the chamber to 5 psia, resulting in a test atmosphere of oxygen at 5 psia with a relative humidity of approximately 33%.

Test atmospheres were maintained at $25 \pm 2^\circ\text{C}$ by storing chambers in a temperature-controlled room for 30 days. The chambers tested at $68 \pm 2^\circ\text{C}$ were stored in a constant-temperature cabinet (Blue M Electric Co., Stabil-Therm DL132C).

3. Analytical Methods

Methods of analyses used in this program have been described elsewhere (ref. 1,2,3) and are summarized below.

a. Weight Loss Measurements

Conditional screening of candidate materials was performed by measuring the weight loss of the material, using thermogravimetric measurements (TGA). Weight loss from approximately 10 g of a material was recorded continuously as the temperature of its environment was raised from ambient (approximately 23°C) to $68 \pm 1^\circ\text{C}$ in 90 minutes and then maintained at $68 \pm 1^\circ\text{C}$ for 21 hours or until the weight remained constant for 2 hours. All TGA measurements were made in dried, prepurified nitrogen at 5 psia.

Thermogravimetric measurements were made with a Cahn RH Electrobalance equipped with a modified F&M Model 240-00 Power Proportioning Temperature Programmer, Flo-Thru tube, a temperature-programmed oil bath, and a 1 mv recorder (ref. 3).

Water evolving from the sample was monitored continuously with a Panametrics Hygrometer, Model 1000. The probe of the hygrometer was located at the sample site. Probe response under test conditions was calibrated against weight loss measurement for known amounts of water by using the Cahn electrobalance.

b. Gas Chromatographic Analysis of Gas-Off Products

Carbon monoxide, methane, and gas chromatographic analyses were performed by techniques reported earlier (refs. 1,2,3). All atmospheres in the test chamber were sampled for analysis at the temperature of the test, i.e., 25°C or 68°C.

The general analyses of the gas-off products by gas chromatography were performed on an F&M Model 810 Research Gas Chromatograph equipped with dual flame ionization detectors and a general purpose column, 20-ft x 0.25-in. ss., 20% Triton X-305 on 60/80 mesh Gas Chrom Z.

Quantitative gas chromatography data were obtained by comparing the peak heights with those of a standard mixture. Gas chromatographic instrument conditions are presented in Appendix III, Table XXII.

Identification of gas chromatographic components were made by mass spectrometric analysis of the gas chromatographic effluent. In most cases, component identification was accomplished by the direct, tandem coupling of a fast scan mass spectrometer, CEC 21-104, to the gas chromatograph. By splitting the effluent a portion was directed to the flame ionization detector and a second portion was introduced directly into the mass spectrometer. With some samples, a concentration step requiring the cryogenic trapping of the major portion of the total 9-liter volume was necessary. This condensate was subsequently separated into its components and characterized by the coupled gas chromatograph-mass spectrometer system.

c. Mass Spectrometric Analysis of Gas-Off Products

Two types of mass spectrometric analyses were performed for each sample. A composite analysis (ref. 1) of the atmosphere of each 9-liter bottle was made on an aliquot (125 cc) of the atmosphere with a Consolidated Electrodynamics Corporation Model 21-103C Mass Spectrometer. As indicated in Section II-A-3-b, a fast scan Consolidated Electrodynamics Corporation Model 21-104 Mass Spectrometer was used in a direct couple with a gas chromatograph. Both approaches are necessary to insure complete characterization of the chamber atmospheres.

Identification of individual components was made by mass spectrometry, supported by infrared absorption and gas chromatographic data as needed. Most of the mass spectra obtained were compared to the American Petroleum Institute (API) reference spectra. In cases where the required mass spectrum did not appear in the API collection, comparison was made with spectra from our laboratory files or from the literature.

B. RESULTS AND DISCUSSION

Weight loss data, obtained from thermogravimetric measurements, are reported in Table II for 19 materials.

Table III lists the types of compounds detected in the chamber atmospheres. These data represent compounds exclusive of H₂O, CO₂, O₂ and N₂. The off-gas products are mostly entrapped solvents or low molecular weight polymers, e.g., siloxanes.

Analytical data are presented in Appendix I, Figures 1 to 19 (TGA and Water Loss Curves); Appendix II, Tables IV-XXI (Analytical Results for Gas-Off Experiments); and Appendix III, Figures 20 to 32 (Gas Chromatograms for Gas-Off Experiments).

All values appearing in the tables of Appendix II are calculated on the basis of the dried or cured samples (this is important in the case of paints and coatings where the weight of the material is substantially reduced by drying).

No gas-off products, other than H₂O, were detected from DAC 010 (Tefglas Tape, DMS1603A, Size 2).

Some gas-off products are identified by compound type only, e.g., alkylbenzene(s), C₄ alkylbenzene(s), or C₄ hydrocarbons. In these cases, several homologues or isomers may be present; however, they have not been identified individually.

Some of the gas-off products from silicone-base materials were also calculated collectively. These were the volatile linear and cyclic siloxane polymers (having dimethyl siloxy groups as monomer units) which had been observed in previous gas-off studies (refs. 1,2,3,4). Although separate peaks are noted in the gas chromatograms (Appendix III), these volatile silicones are listed collectively in the tables of gas-off data (Appendix II) as silicone oil.

C. CONCLUSIONS AND RECOMMENDATIONS

The gas-off products, which are similar to those observed in previous studies in this series (refs. 1,2,3,4), are mostly entrapped solvents. Only in materials DAC 009 and DAC 048 do the quantities of emitted products reach excessively high values, i.e., >1.0%.

Table II

WEIGHT LOSS DATA FOR CANDIDATE SPACE CABIN MATERIALS

(Obtained from Thermogravimetric Measurements)

<u>DAC No.</u>	<u>Wt. of Sample (g)</u>	<u>Total Wt. Loss (mg)</u>	<u>Wt. Loss Due to H₂O (mg)</u>	<u>Wt. Loss Exclusive of H₂O (mg)</u>	<u>Wt. Loss Exclusive of H₂O (%)</u>
004	9.6729	10.8	10.0	0.8	0.008
009	9.7863	250.0	98.5	151.5	1.54
010	9.4899	6.6	5.9	0.7	0.007
020	10.0704	1.0	0.5	0.5	0.005
023	10.0785	4.5	3.8	0.7	0.007
024	9.9021	210.2	156.0	54.2	0.55
038	10.0386	7.6	4.5	3.1	0.031
041	8.5239	0.5	0.3	0.2	0.002
047	2.3489	2.4	2.0	0.4	0.02
048	6.9444	130.5	11.0	119.5	1.72
049	9.9363	8.4	7.2	1.2	0.012
050	10.1792	52.2	25.0	27.2	0.27
051	9.2670	10.0	9.9	0.1	0.001
052	5.2663	34.5	33.2	1.3	0.025
053	1.1902	6.0	4.3	1.7	0.14
054	11.6018	6.5	4.5	2.0	0.017
055	10.0494	0.6	0.4	0.2	0.002
056	9.3736	1.6	1.2	0.4	0.004
057	7.9346	10.8	9.5	1.3	0.016

Table III

TYPES OF COMPOUNDS DETECTED

<p><u>I. Inorganics</u> Carbon Monoxide</p> <p><u>II. Alkanes</u> Methane C₄ Sat. Hydrocarbon C₅ Sat. Hydrocarbon C₆ Sat. Hydrocarbon</p> <p><u>III. Alcohols</u> n-Butanol 2-n-Butoxyethanol 2(2-Butoxyethoxy)ethanol 2(2-Dieethoxy)ethanol Ethanol 2-Ethoxyethanol Methoxyethanol 6-Methylheptanol 2-Phenyl-2-propanol n-Propanol</p> <p><u>IV. Ethers</u> Dimethylether</p>	<p><u>V. Ketones</u> Acetone Acetophenone Diisobutylketone</p> <p><u>VI. Esters</u> 2-Ethoxyethylacetate Ethyl Esters of C₄ or C₅ Acids</p> <p><u>VII. Amides</u> 2-Methylpyrrolidone</p> <p><u>VIII. Aromatic Hydrocarbons</u> Benzene C₃ Alkylbenzene C₄ Alkylbenzene Styrene Toluene Xylenes</p> <p><u>IX. Silicon Compounds</u> Various Cyclic and Linear Methylsiloxane Polymers</p>
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The thermogravimetric procedure for screening candidate materials provides a good measure of the rate of volatile emission. However, the use of this approach as a "rapid pre-screening" technique under present test conditions requires excessive time. Although the programming rate was increased from 23 to 68°C in 4 hours (ref. 4) to 23 to 68°C in 90 minutes, the total time of 22.5 hours plus set-up and clean-up time limits the number of samples that can be evaluated on one TGA system. Consideration should be given to a shorter test period (6 hours or less).

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APPENDIX I

THERMOGRAVIMETRIC PATTERNS OF
CANDIDATE SPACE CABIN MATERIALS

The thermogravimetric analysis (TGA) patterns shown in this appendix were obtained on a Cahn RH Electrobalance. Comparison of the weight loss patterns should be made with care since varying amounts of sample were used to obtain the TGA patterns. The quantity of material used for each TGA measurement is shown on the reproduced pattern.

Water loss curves were computed from data obtained from a Panametrics Model 1000 Hygrometer which was used to monitor continuously the evolution of water.

TGA curves appear in order of their Air Force serial numbers. Names of materials are those submitted by the Air Force.

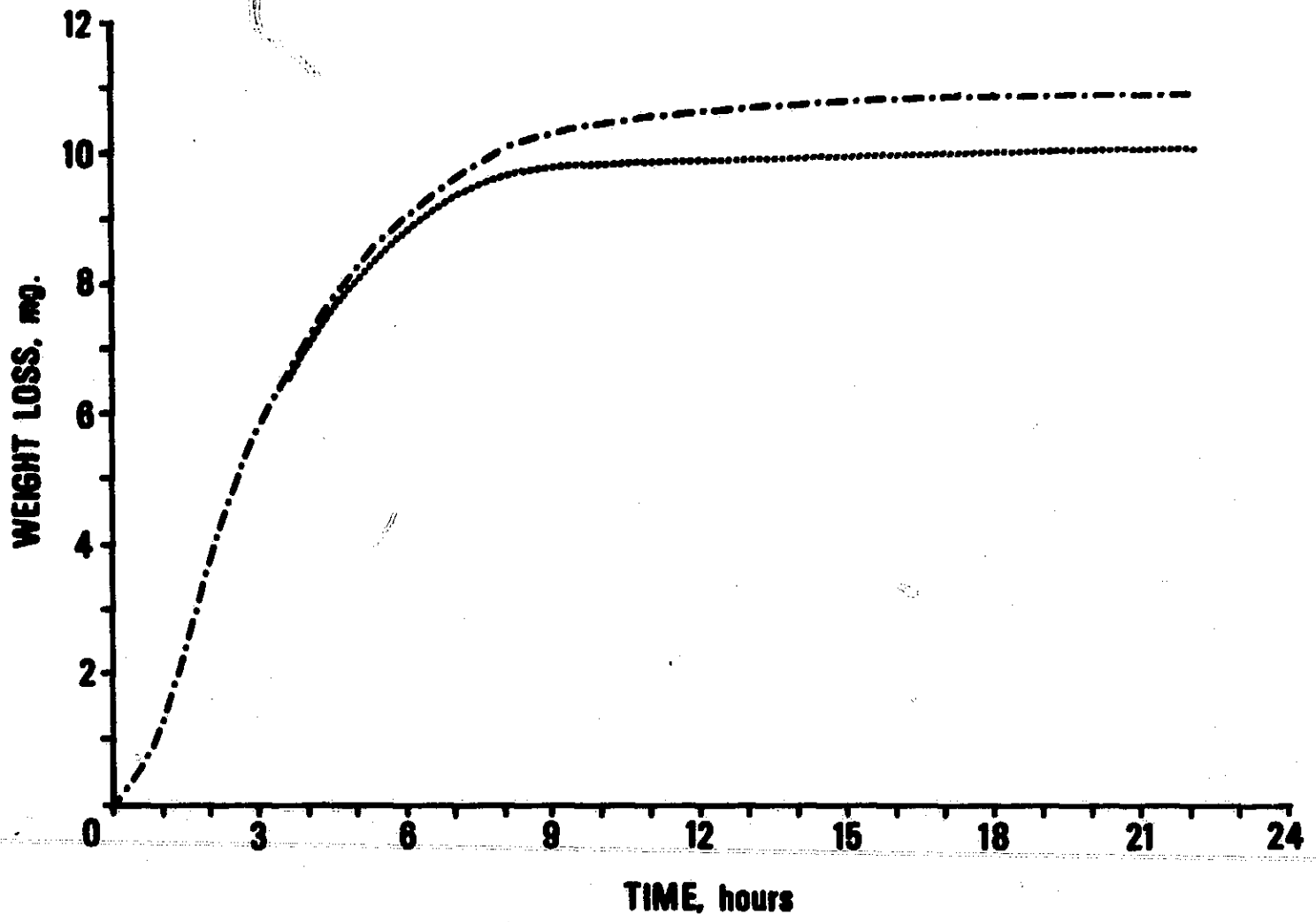


Figure 1. TGA (Upper) and Water Loss (Lower) Curves of Adhesive Epon 919 (DAC 004).

Specimen Weight - 9.6729 grams

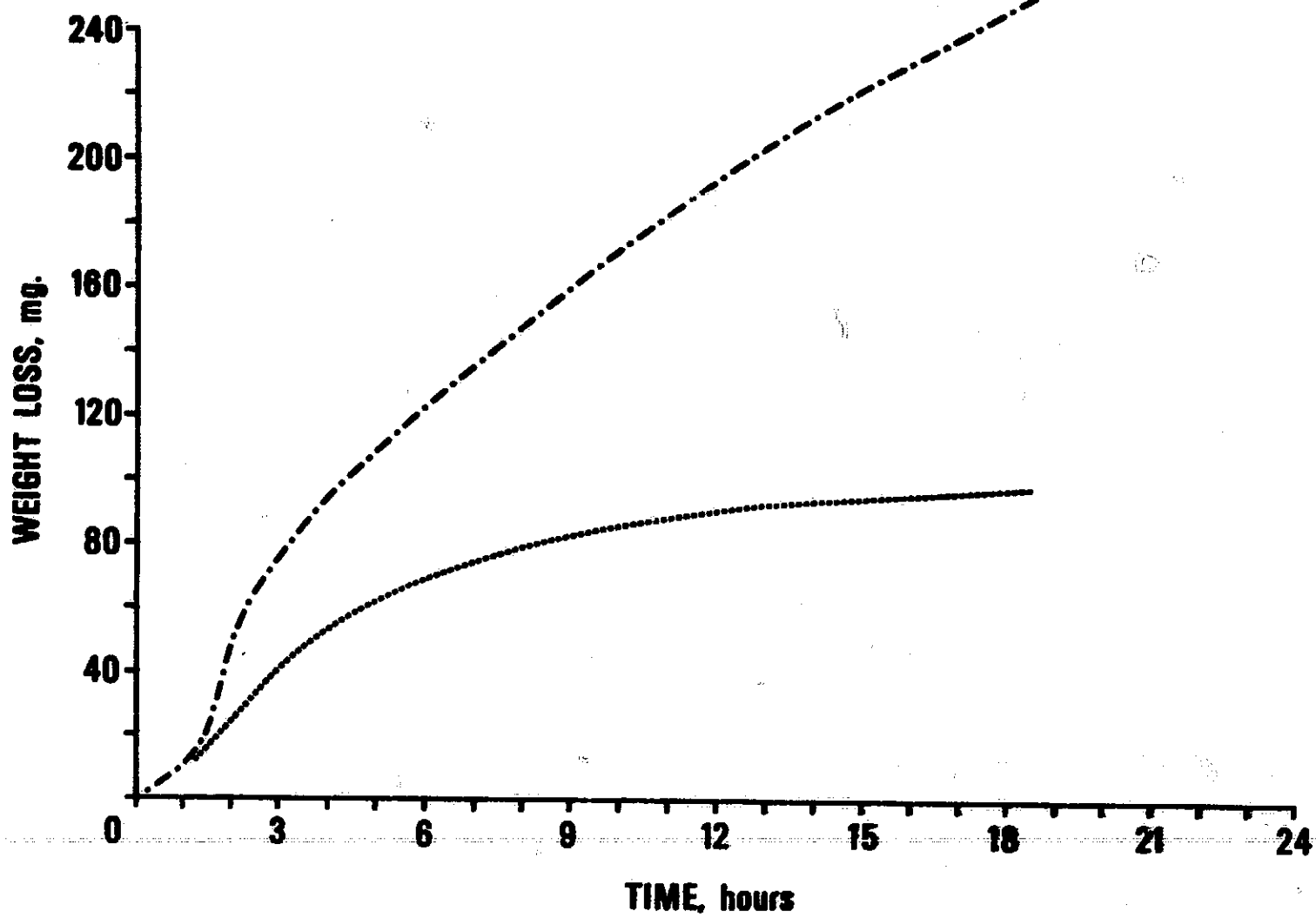


Figure 2. TGA (Upper) and Water Loss (Lower) Curves of Cat-L-Ink W/20 Cat. (Yellow) (DAC 009).
Specimen Weight - 9.7863 grams

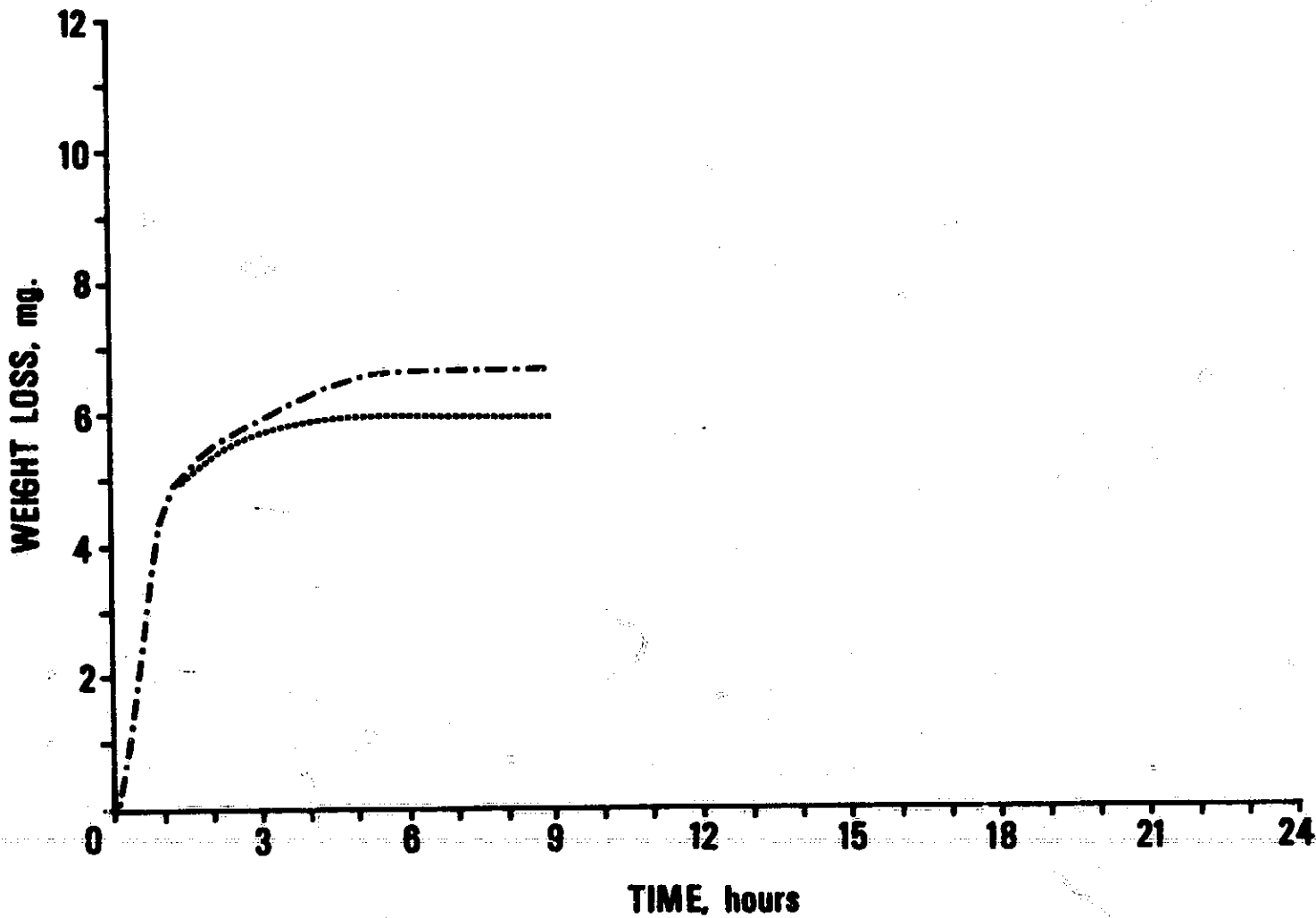


Figure 3. TGA (Upper) and Water Loss (Lower) Curves of Tefglas Tape, DMS 1603A (Size 2 , Lot 80367) (DAC 010).

Specimen Weight - 9.4899 grams

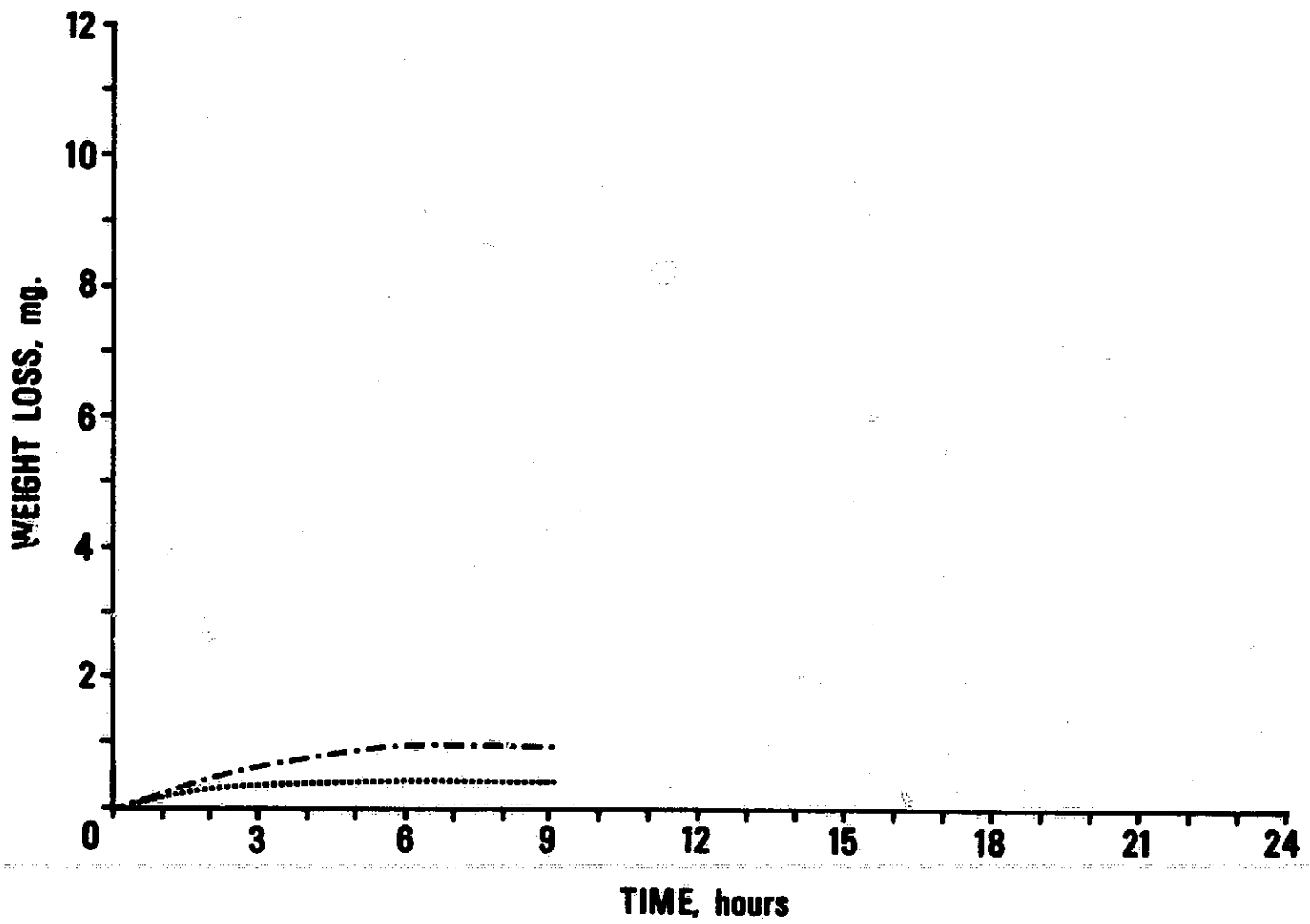


Figure 4. TGA (Upper) and Water Loss (Lower) Curves of Boltaron (6200) PVC Type I, (Grey) (DAC 020).
Specimen Weight - 10.0704 grams

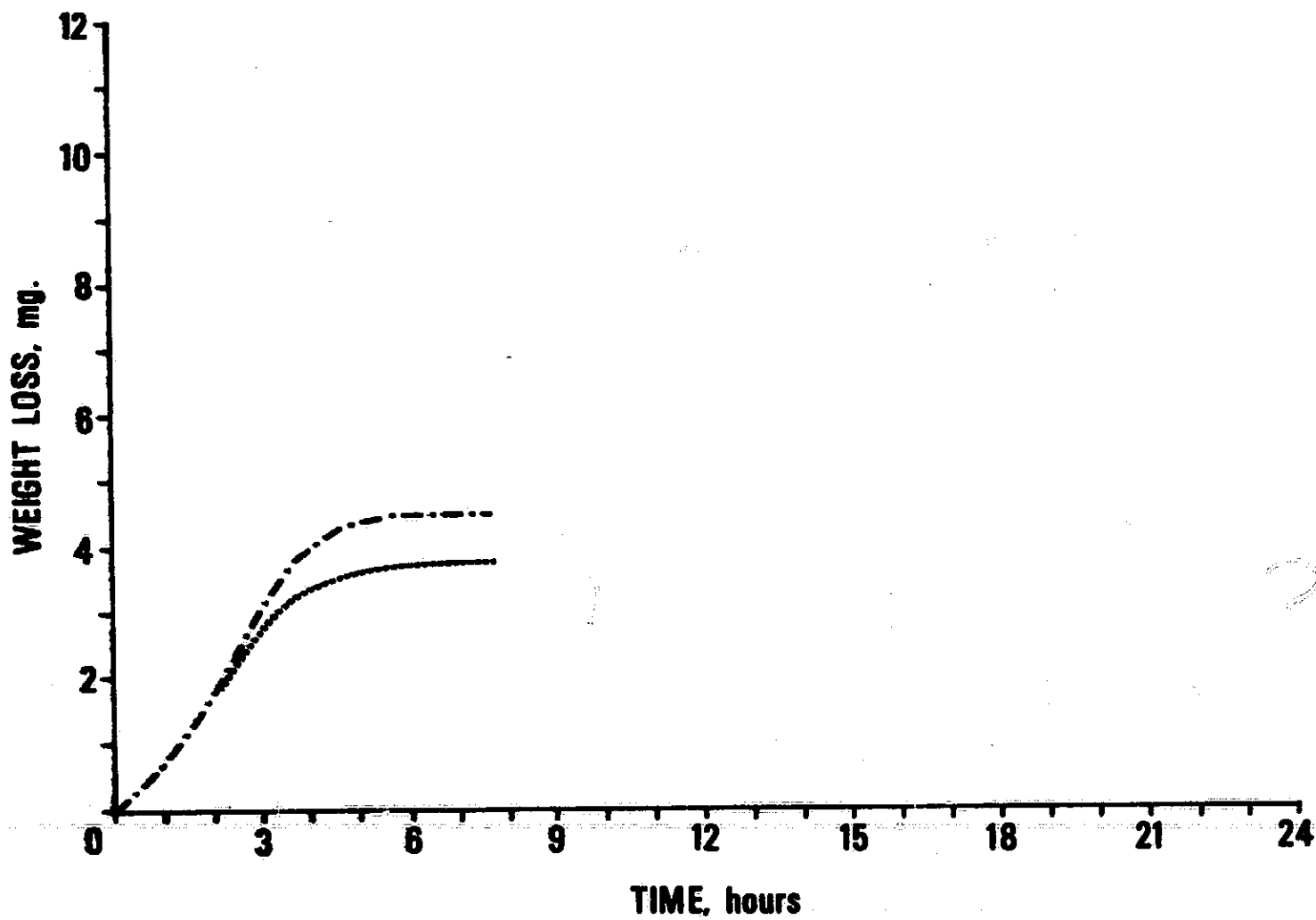


Figure 5. TGA (Upper) and Water Loss (Lower) Curves of Selectron 5016 (DAC 023).

Specimen Weight - 10.0785 grams

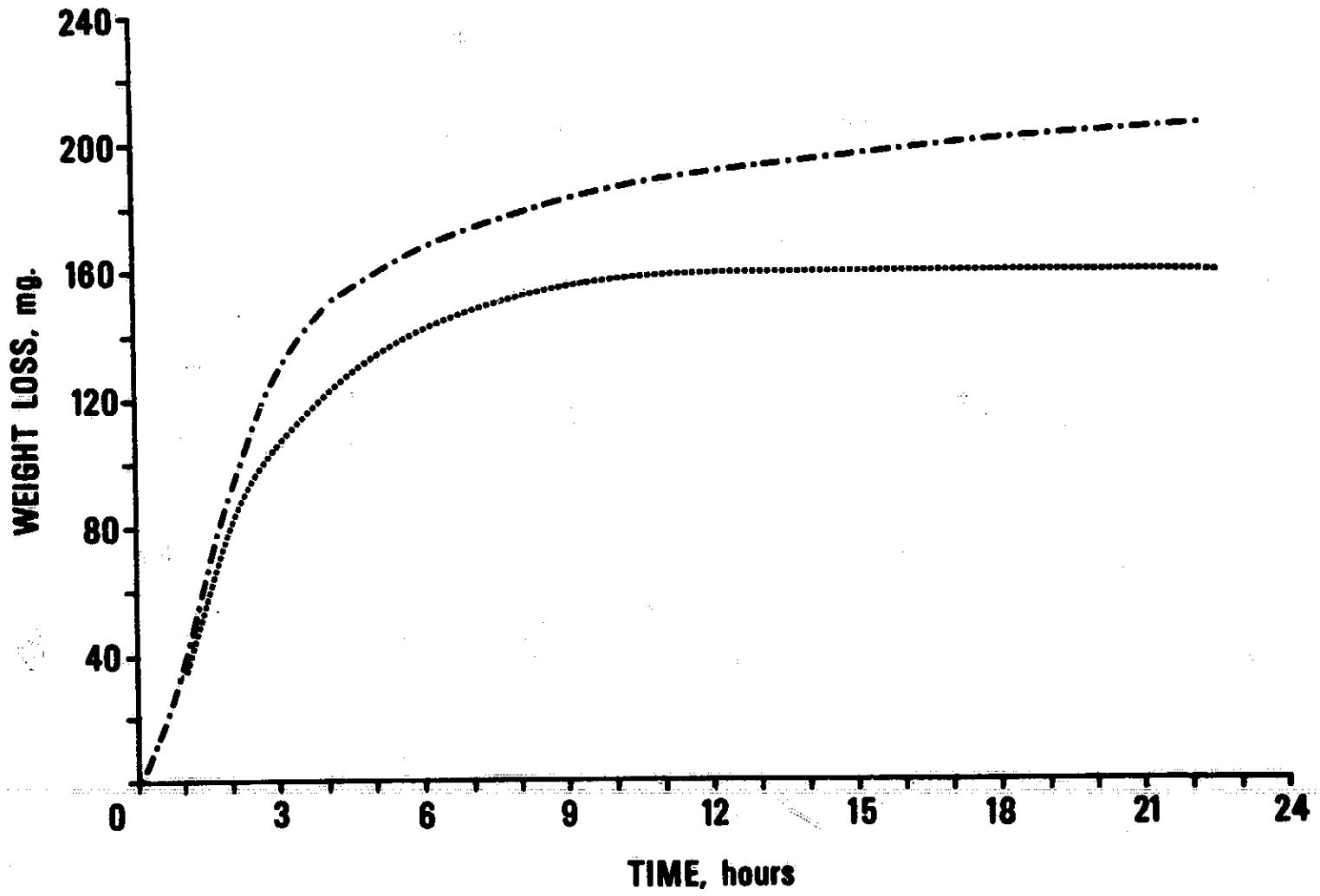


Figure 6. TGA (Upper) and Water Loss (Lower) Curves of Nextel Velvet Coating 401-C10-Black (DAC 024).
Specimen Weight - 9.9021 grams

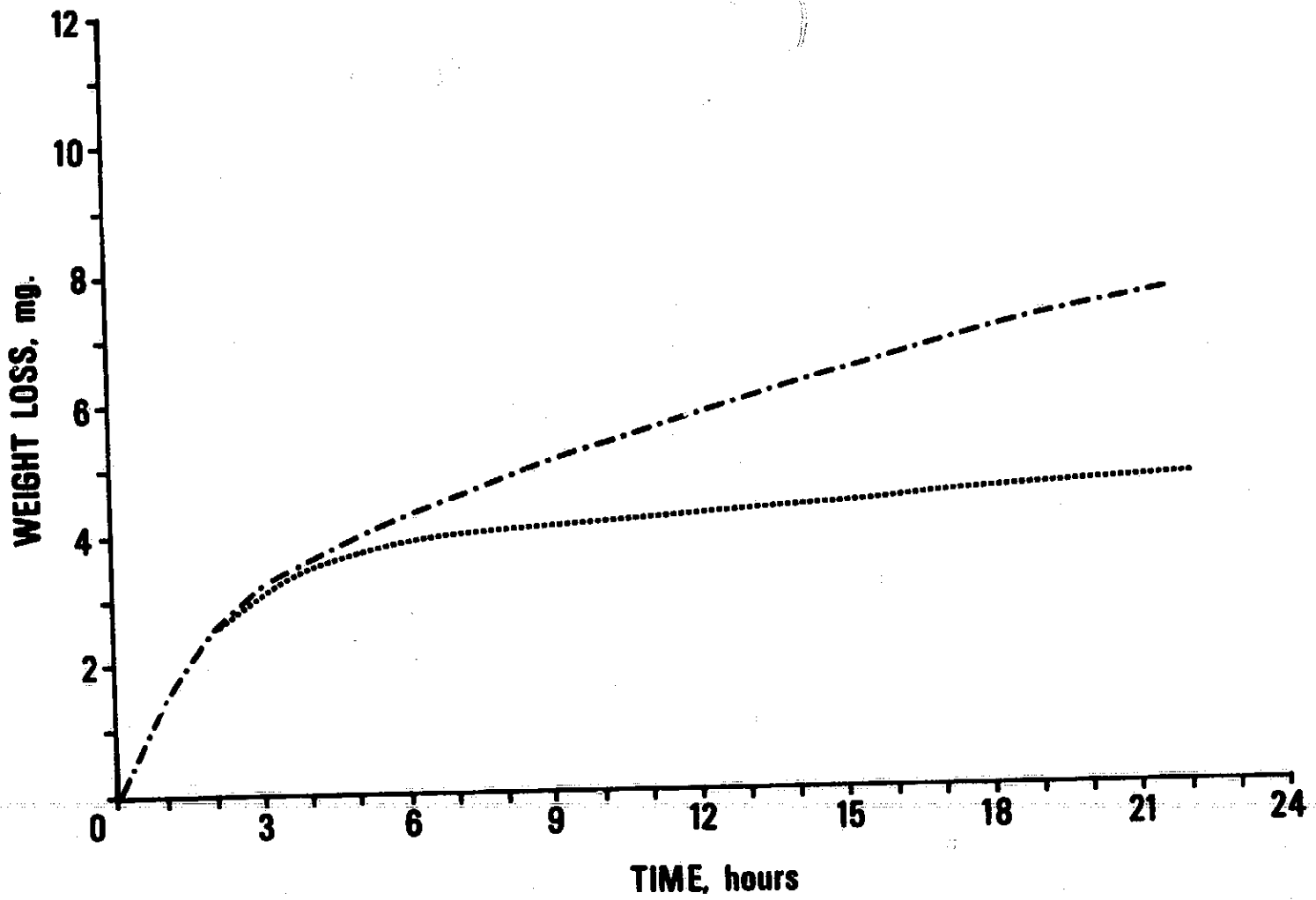


Figure 7. TGA (Upper) and Water Loss (Lower) Curves of Silicone Rubber RTV-1016 W/Cat. RTV 9910 (DAC 038).

Specimen Weight - 10.0386 grams

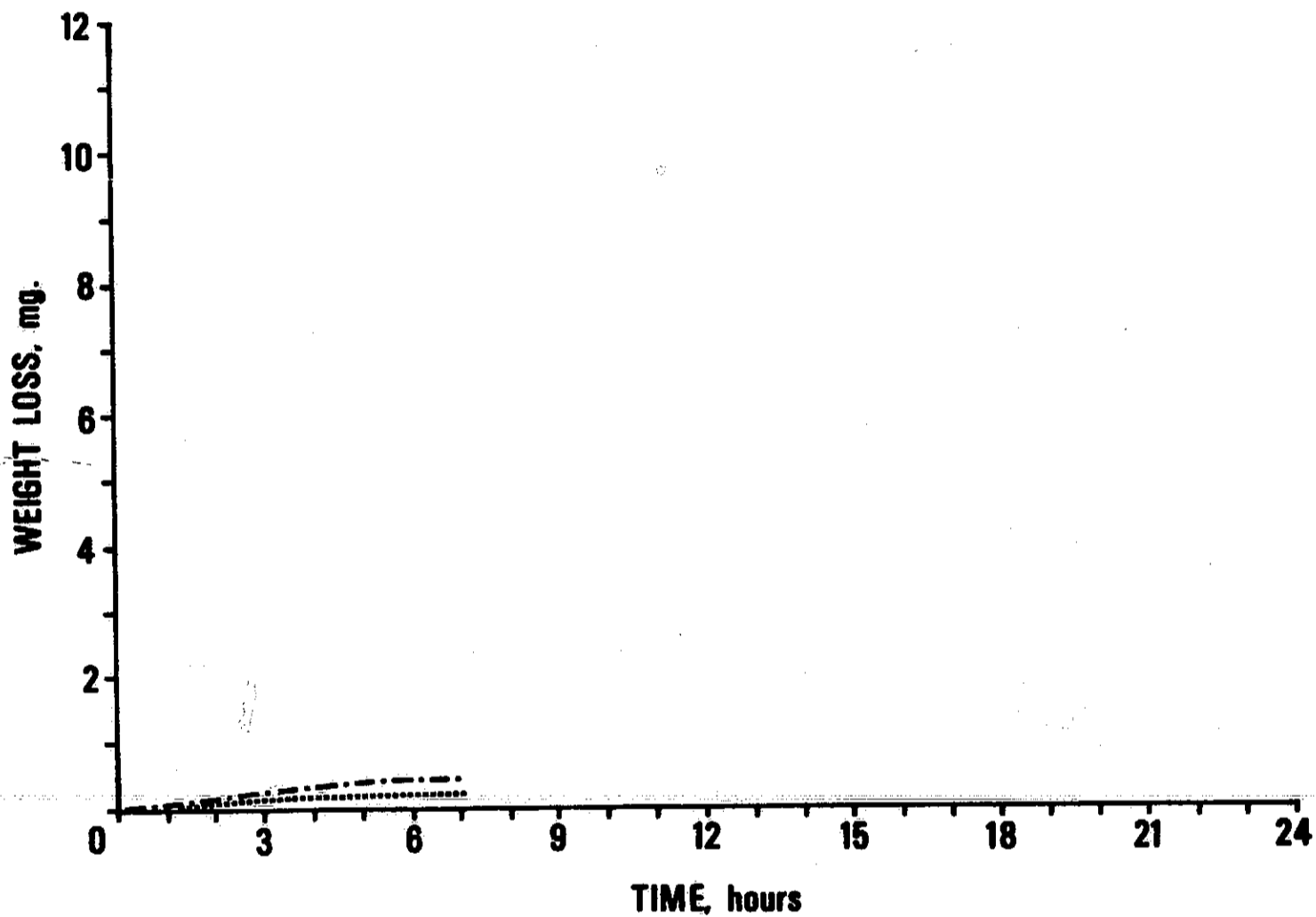


Figure 8. TGA (Upper) and Water Loss (Lower) Curves of Rod PO#RI45074 (4 ft x 1-3/4 in.) (DAC 041).
Specimen Weight - 8.5239 grams

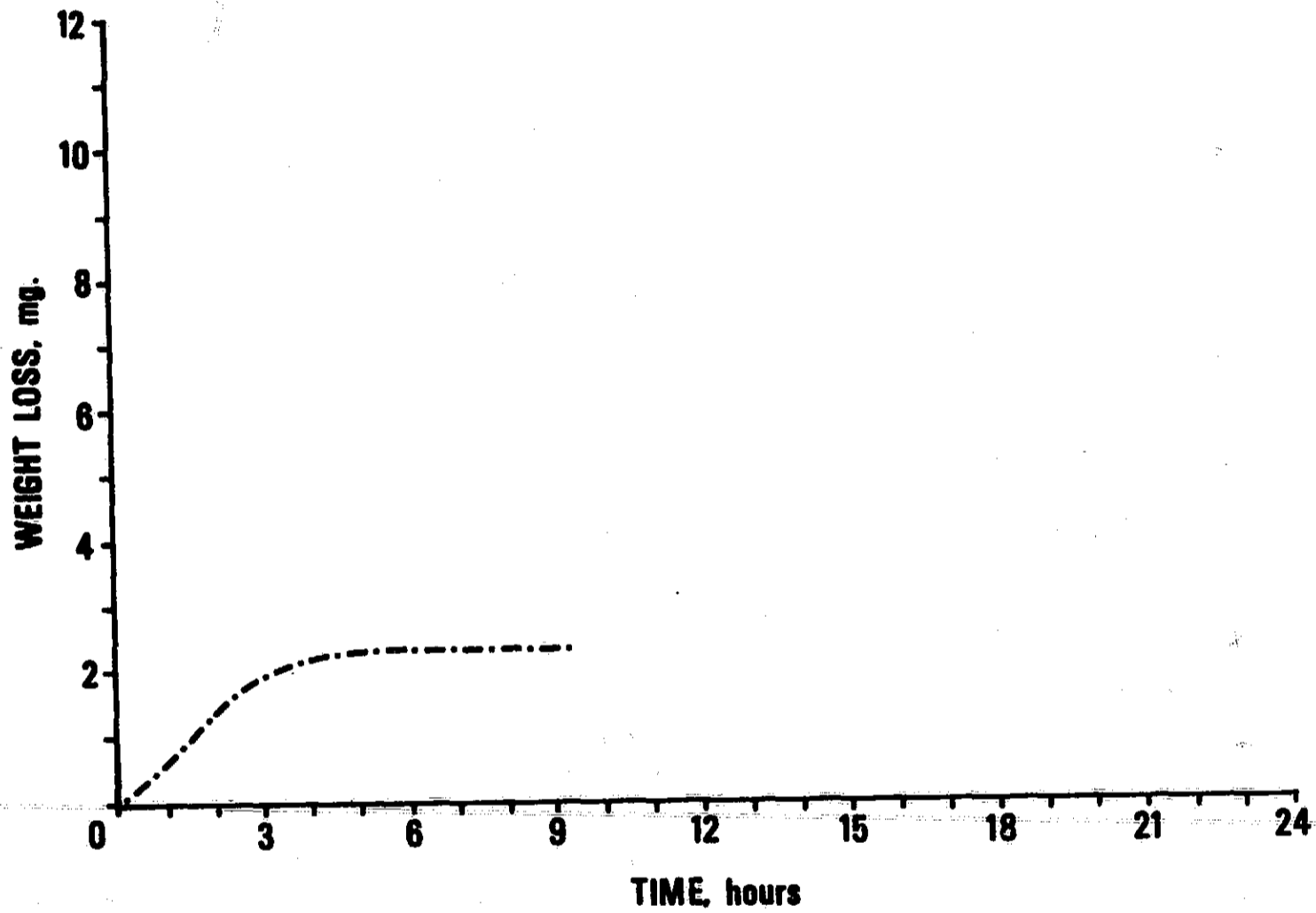


Figure 9. TGA Curve of Scotchply Reinforced Plastic (Type 1100) (DAC 047).

Specimen Weight - 2.3489 grams

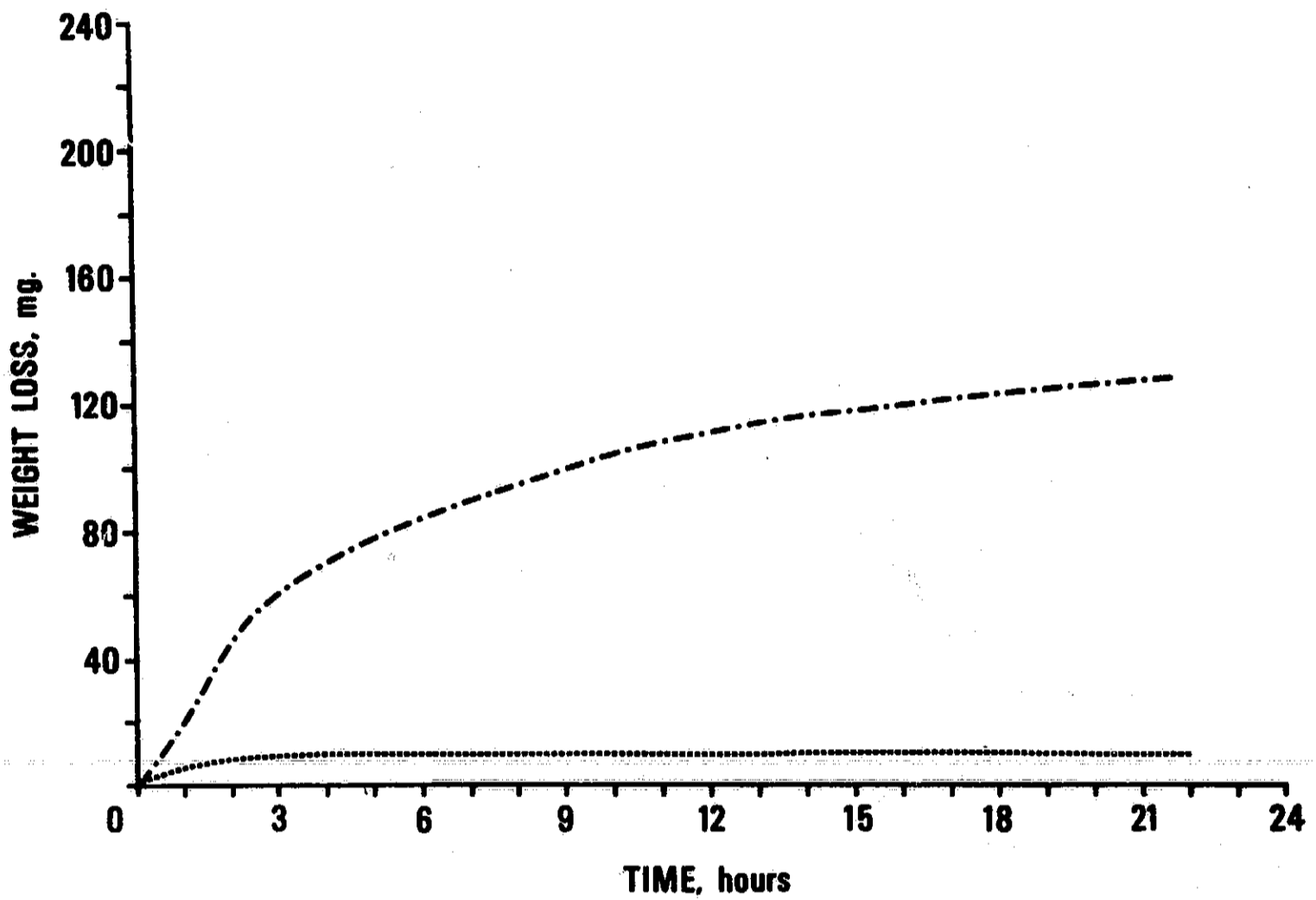


Figure 10. TGA (Upper) and Water Loss (Lower) Curves of Pyralin, Polyimide (35-502-38) (DAC 048).

Specimen Weight - 6.9444 grams

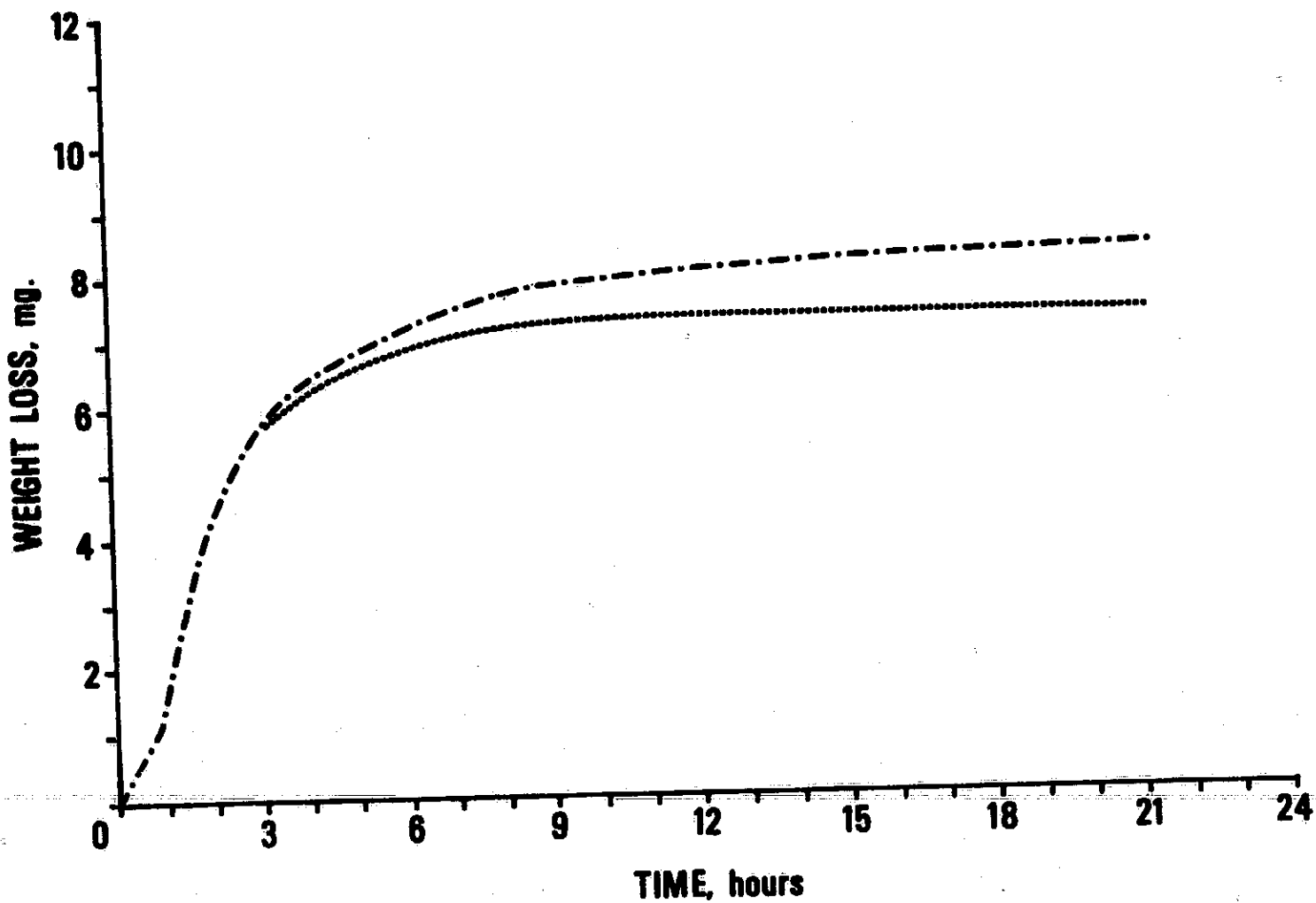


Figure 11. TGA (Upper) and Water Loss (Lower) Curves of Royalite (Imitation Leather) (R-56-8163) (DAC 049).

Specimen Weight - 9.9363 grams

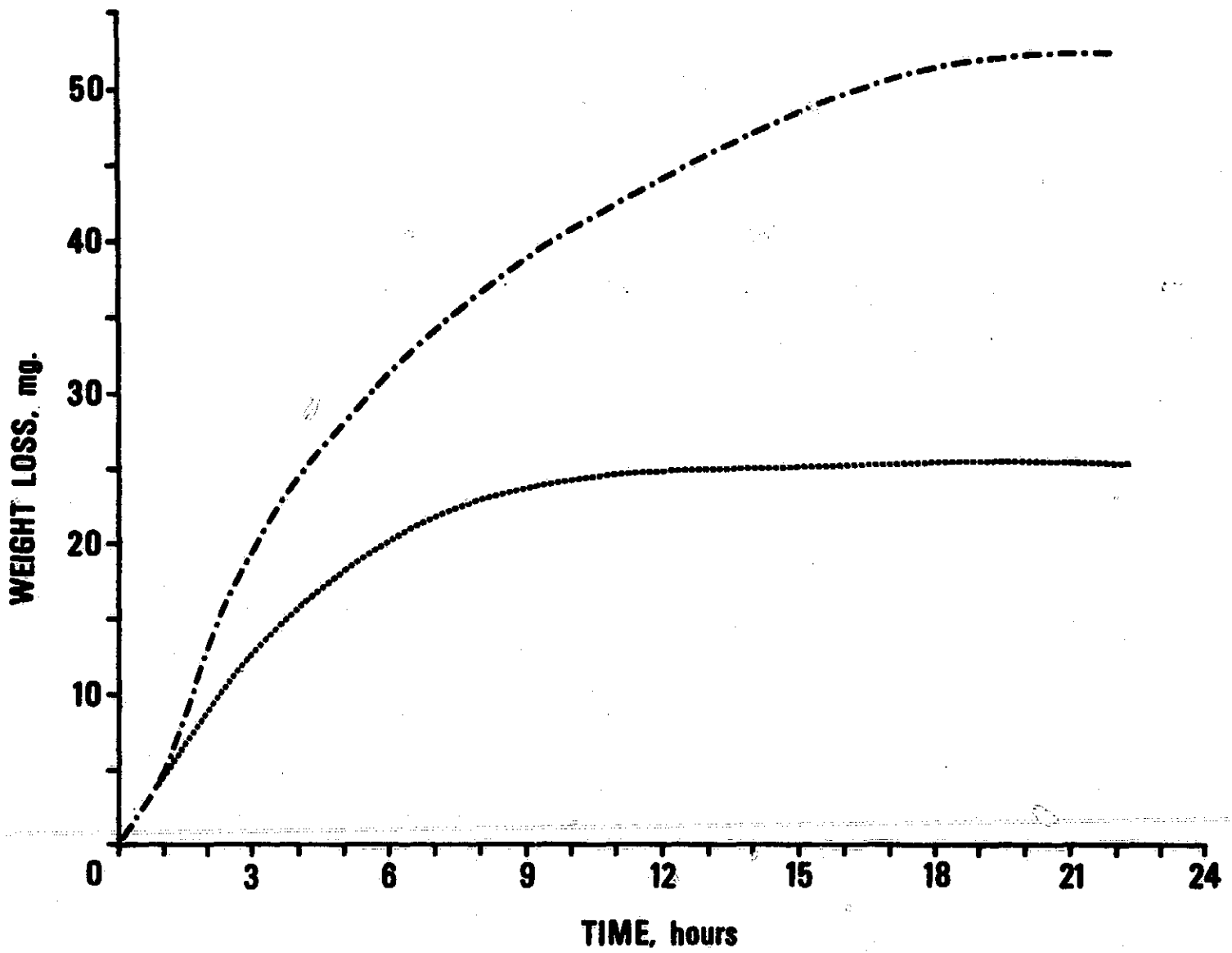


Figure 12. TGA (Upper) and Water Loss (Lower) Curves of Polasheet 11[73-3062(07-0701-3006)] (DAC 050).
Specimen Weight - 10.1792 grams

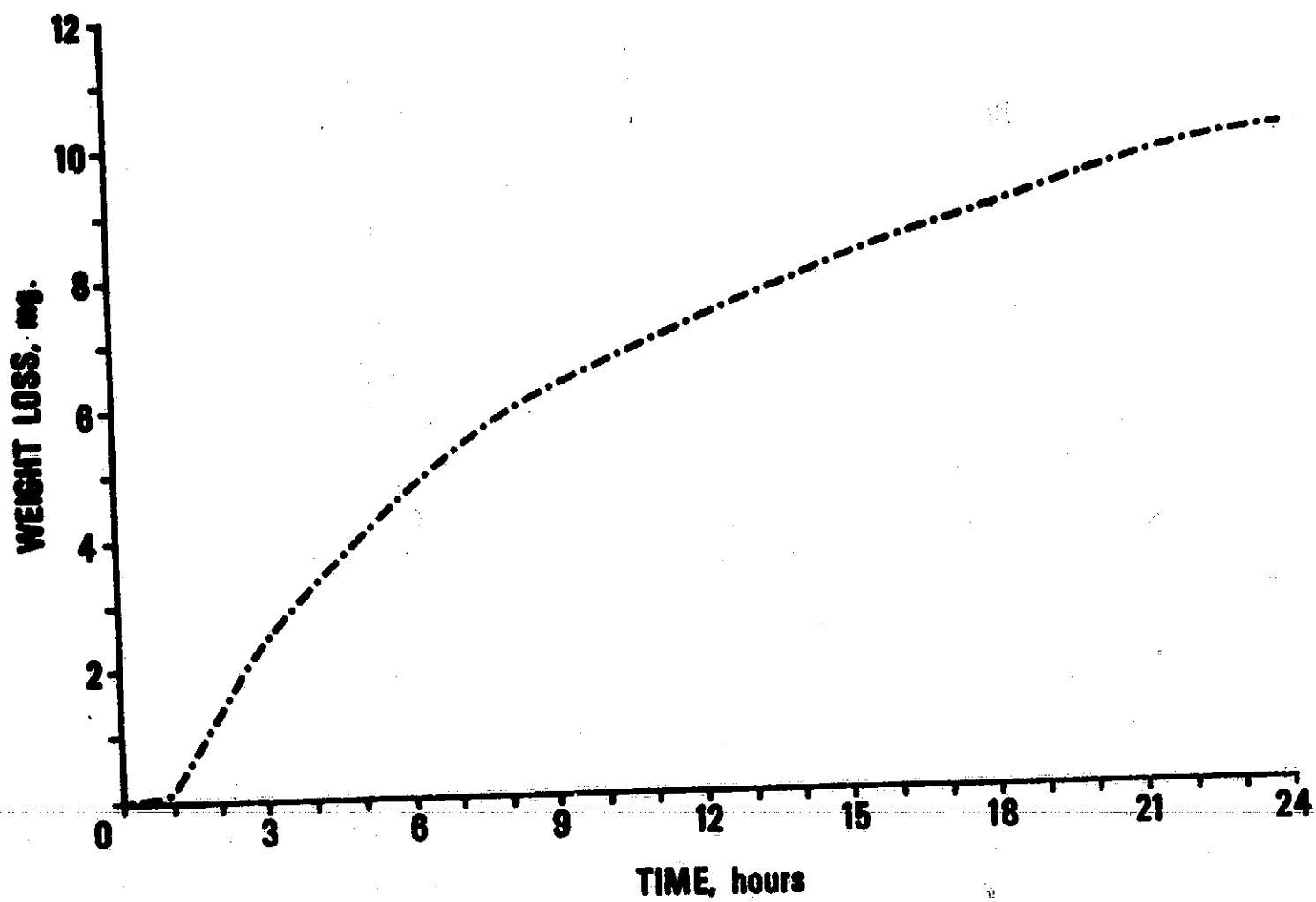


Figure 13. TGA Curve of Zytel Nylon Resin, Type 103
(Color NC10) (DAC 051).
Specimen Weight - 9.2670 grams

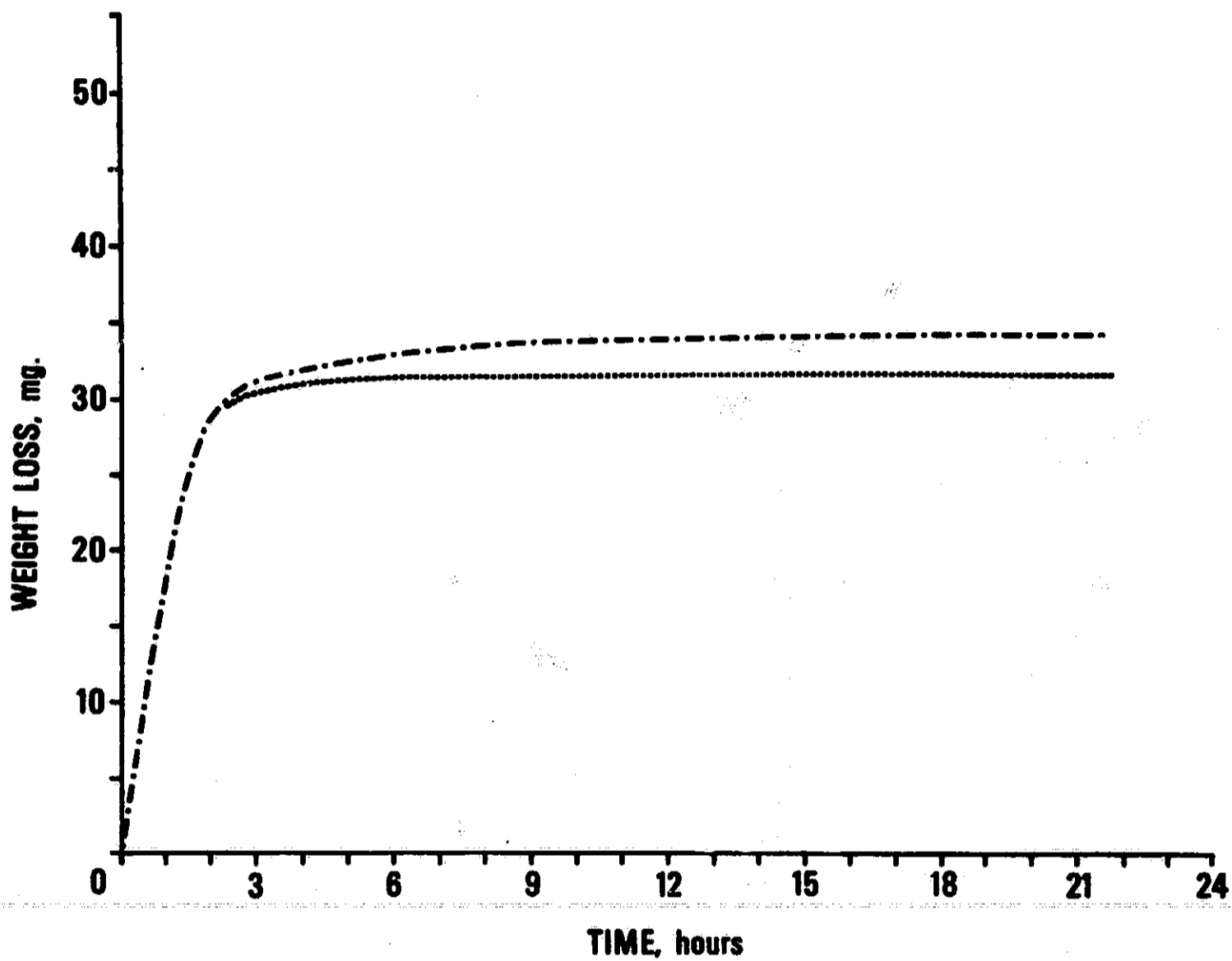


Figure 14. TGA (Upper) and Water Loss (Lower) Curves of Durez Molding Compound 1900 (Black Phenolic) (DAC 052).

Specimen Weight - 5.2663 grams

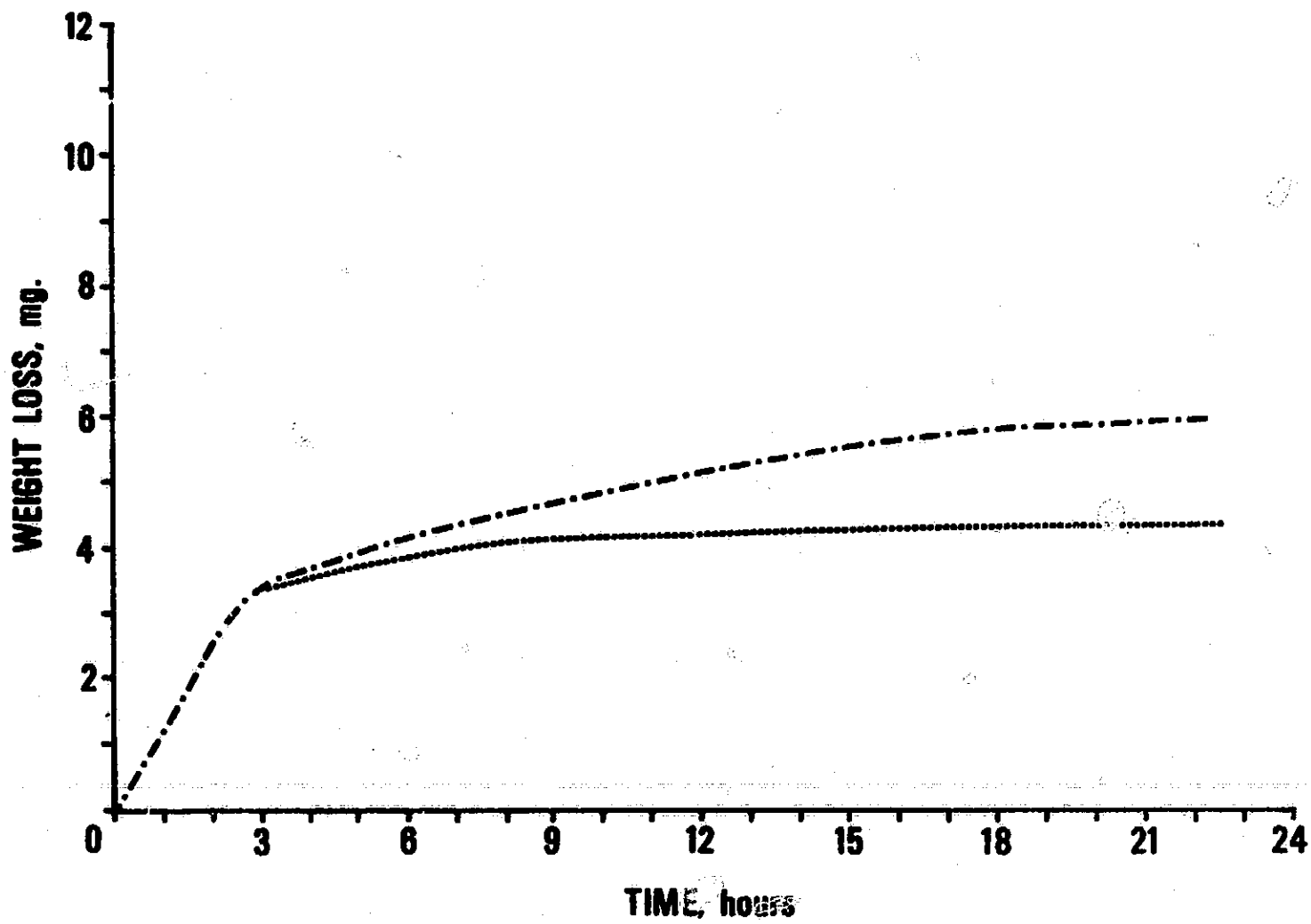


Figure J5. TGA (Upper) and Water Loss (Lower) Curves of Nopcofoam G-302 (DAC 053).

Specimen Weight - 1.1902 grams

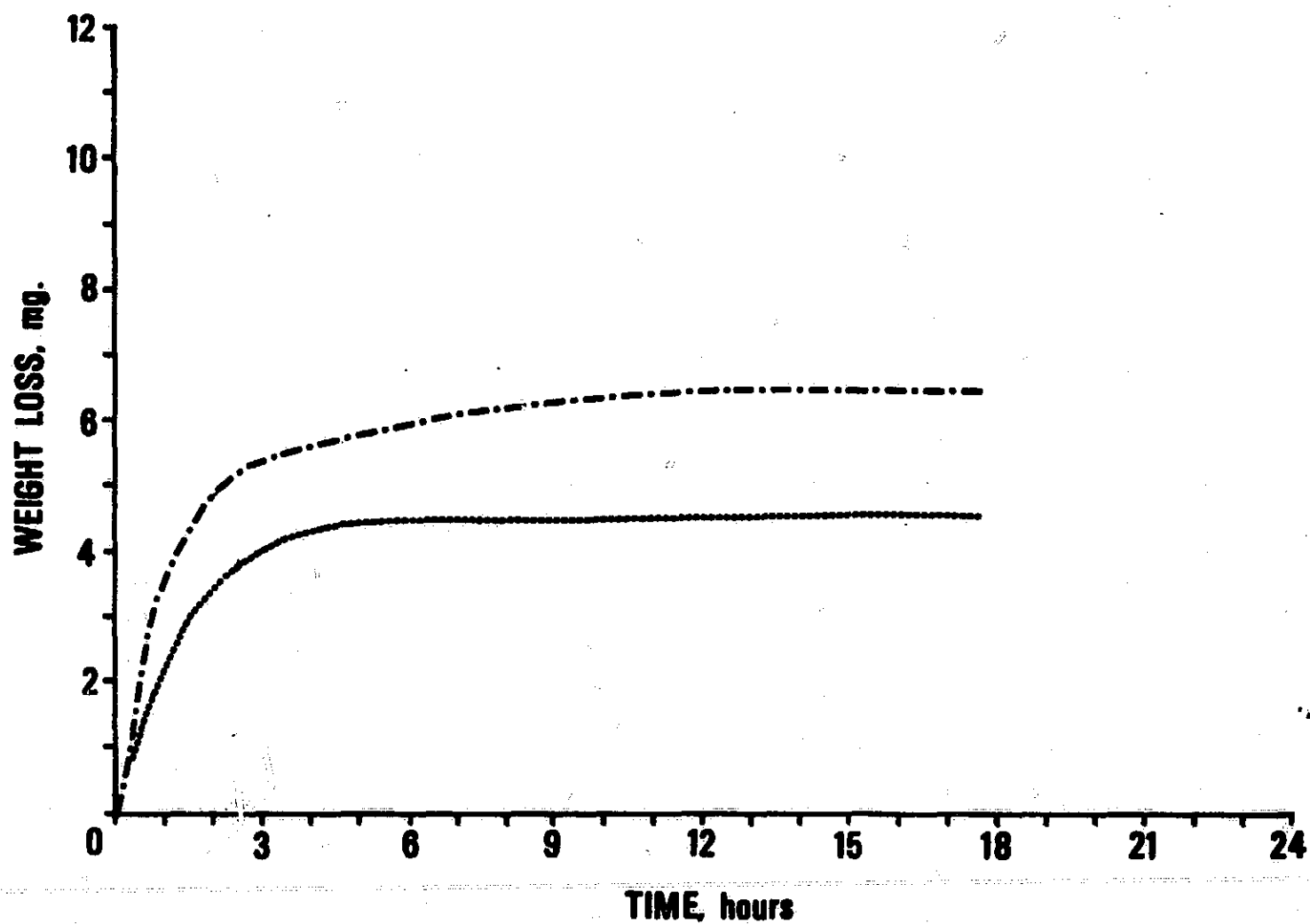


Figure 16. TGA (Upper) and Water Loss (Lower) Curves of Electrical Tape, TFE Fluorocarbon Film (Silicone) (No. 60) (DAC 054).

Specimen Weight - 11.6018 grams

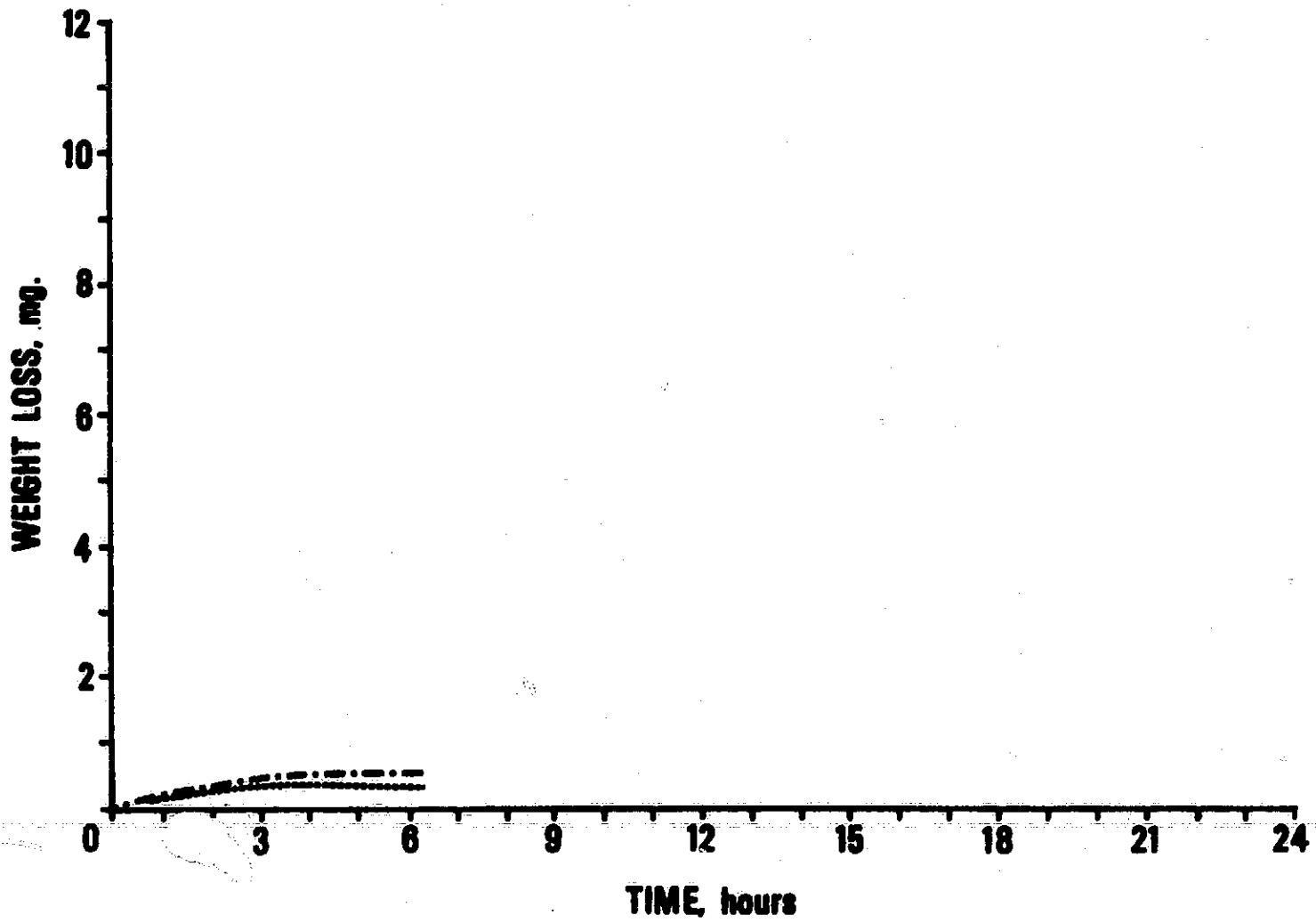


Figure 17. TGA (Upper) and Water Loss (Lower) Curves of Pressure Sensitive Adhesive Tape, E-284-6-ERH, Run 2355 (DAC 055).

Specimen Weight - 10.0494 grams

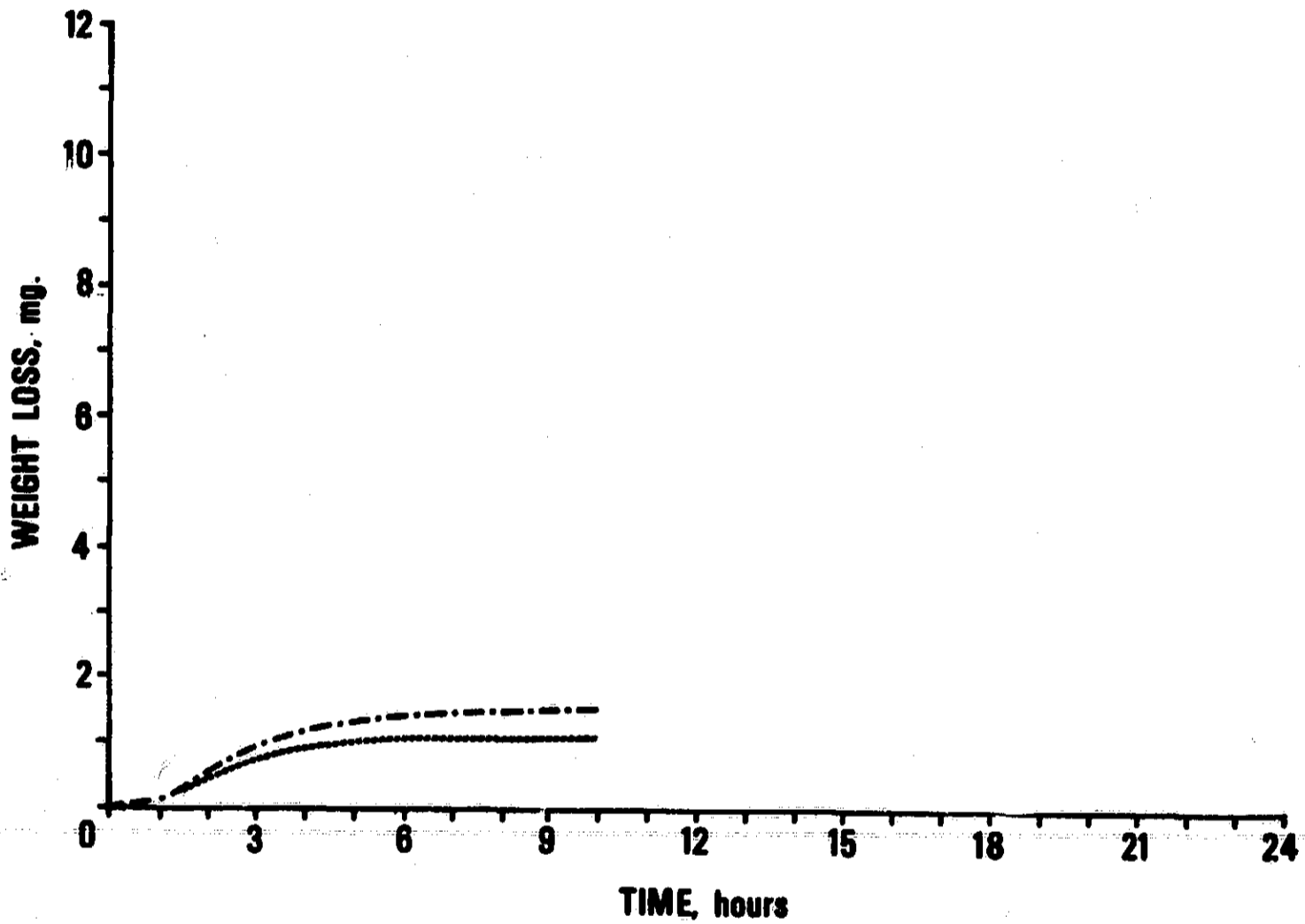


Figure 18. TGA (Upper) and Water Loss (Lower) Curves of Printed Circuit Board (DAC 056).

Specimen Weight - 9.3736 grams

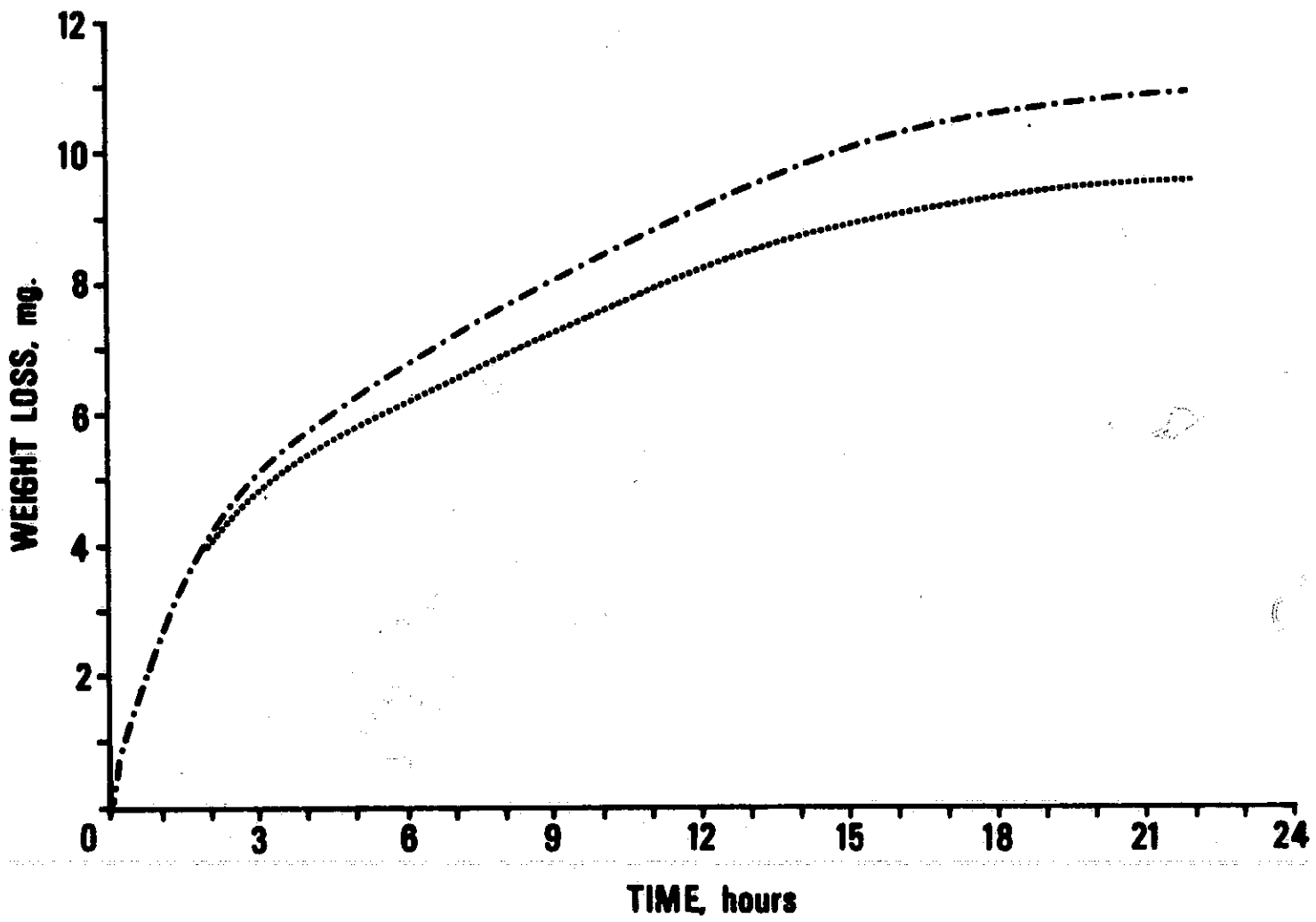


Figure 19. TGA (Upper) and Water Loss (Lower) Curves of Scotch Tape, #4116 YAK 41171T (DAC 057).

Specimen Weight - 7.9346 grams

APPENDIX II

ANALYTICAL RESULTS FOR GAS-OFF EXPERIMENTS

Compounds found as gas-off products from candidate space cabin materials are listed in the following tables. Values for the gas-off product levels are given as: milligrams per 10 grams (mg/10 gms) of the cured candidate material. In some cases, either more or less than 10 grams of material was used, but each yield of gas-off products was normalized to that of a 10-gram sample.

The order of the tables in this appendix is by Air Force serial number. Names of materials are those submitted by the Air Force.

Table IV

GAS-OFF PRODUCTS FROM
DAC 004 (ADHESIVE, EPON-919)

<u>Component</u>	<u>Weight of Component (mg/10 gms Candidate Material)</u>	
	<u>72 Hours (68°C)</u>	<u>30 Days (25°C)</u>
C ₆ Sat. Hydrocarbon	0.011	0.009
Toluene	0.002	ND
Xylenes	0.014	ND
Carbon Monoxide	0.05	0.002
Methane	0.002	ND

ND - Not detected

Table V

GAS-OFF PRODUCTS FROM
DAC 009 [CAT-L-INK W/CAT. 20 (YELLOW)]

Component	Weight of Component (mg/10 gms Candidate Material)	
	72 Hours (68°C)	30 Days (25°C)
Acetone	0.058	0.080
Ethanol	0.046	ND
Benzene	0.010	ND
n-Propanol	0.20	0.039
Toluene	0.55	0.19
n-Butanol	0.079	0.007
Methoxyethanol	1.0	0.13
Xylenes	33.6	5.7
2-Ethoxyethanol	46.7	4.2
C ₃ Alkylbenzene	4.3	0.61
2-n-Butoxyethanol	0.61	0.037
2(2-Diethoxy)ethanol	11.4	0.60
Diisobutylketone	74.3	25.1
2(2-Butoxyethoxy)ethanol	0.36	ND
Carbon Monoxide	0.14	0.02
Methane	0.009	0.001

ND - Not detected

Table VI

GAS-OFF PRODUCTS FROM
 DAC 020 (BOLTARON 6200,
RIGID PVC TYPE I, COLOR INDUSTRIAL GREY)

<u>Component</u>	<u>Weight of Component</u> <u>(mg/10 gms Candidate Material)</u>	
	<u>72 Hours</u> <u>(68°C)</u>	<u>30 Days</u> <u>(25°C)</u>
C ₄ or C ₅ Hydrocarbon	0.10	ND
Toluene	0.10	ND
Carbon Monoxide	0.004	ND
Methane	0.018	ND

ND - Not detected

Table VII

GAS-OFF PRODUCTS FROM
DAC 023 (SELECTRON 5016)

<u>Component</u>	<u>Weight of Component (mg/10 gms Candidate Material)</u>	
	<u>72 Hours (68°C)</u>	<u>30 Days (25°C)</u>
Benzene	0.002	ND
Toluene	0.012	ND
Carbon Monoxide	ND	ND
Methane	ND	ND

ND - Not detected

Table VIII

GAS-OFF PRODUCTS FROM
DAC 024 (NEXTEL VELVET COATING 401-C10-BLACK)

<u>Component</u>	<u>Weight of Component (mg/10 gms Candidate Material)</u>	
	<u>72 Hours (68°C)</u>	<u>30 Days (25°C)</u>
Ethanol	0.084	0.080
Benzene	0.014	0.014
Toluene	6.3	3.5
Xylenes	23.8	14.3
2-Ethoxyethylacetate	7.9	2.0
Carbon Monoxide	0.07	0.003
Methane	0.002	ND

ND - Not detected

Table IX

GAS-OFF PRODUCTS FROM
DAC 038 (SILICONE RUBBER RTV-1016 W/CATALYST RTV 9910)

<u>Component</u>	<u>Weight of Component (mg/10 gms Candidate Material)</u>	
	<u>72 Hours (68°C)</u>	<u>30 Days (25°C)</u>
Ethanol	2.1	3.6
Toluene	0.009	0.006
Carbon Monoxide	ND	ND
Methane	ND	ND

ND - Not detected

Table X

GAS-OFF PRODUCTS FROM
DAC 041 (Rod PO#RI45074)

<u>Component</u>	<u>Weight of Component (mg/10 gms Candidate Material)</u>	
	<u>72 Hours (68°C)</u>	<u>30 Days (25°C)</u>
Xylenes	0.036	ND
Carbon Monoxide	0.03	0.002
Methane	0.005	ND

ND - Not detected

Table XI

GAS-OFF PRODUCTS FROM
DAC 047 (SCOTCHPLY REINFORCED PLASTIC, TYPE 1100)

<u>Component</u>	<u>Weight of Component (mg/10 gms Candidate Material)</u>	
	<u>72 Hours (68°C)</u>	<u>30 Days (25°C)</u>
Carbon Monoxide	0.007	0.001
Methane	0.01	0.002

Table XII

GAS-OFF PRODUCTS FROM
DAC 048 (PYRALIN, POLYIMIDE, 35-502-38)

<u>Component</u>	<u>Weight of Component (mg/10 gms Candidate Material)</u>	
	<u>72 Hours (68°C)</u>	<u>30 Days (25°C)</u>
Ethanol	245	24.6
Toluene	0.38	0.070
Xylenes	40	7.3
2-Methylpyrrolidone	6.9	0.060
Carbon Monoxide	0.006	0.002
Methane	0.009	ND

ND - Not detected

Table XIII

GAS-OFF PRODUCTS FROM
DAC 049 (ROYALITE, R-56-8163)

<u>Component</u>	<u>Weight of Component (mg/10 gms Candidate Material)</u>	
	<u>72 Hours (68°C)</u>	<u>30 Days (25°C)</u>
Toluene	0.007	ND
Styrene	0.058	ND
Carbon Monoxide	ND	ND
Methane	ND	ND

ND - Not detected

Table XIV

GAS-OFF PRODUCTS FROM
DAC 050 [POLASHEET 11, 73-3062(07-0701-3006)]

<u>Component</u>	<u>Weight of Component</u> <u>(mg/10 gms Candidate Material)</u>	
	<u>72 Hours</u> <u>(68°C)</u>	<u>30 Days</u> <u>(25°C)</u>
Silicone Oil	7.4 (4 types)	0.56 (2 types)
Toluene	2.9	0.054
Acetophenone	4.8	0.29
2-Phenyl-2-propanol	1.4	0.015
Carbon Monoxide	0.003	0.004
Methane	0.007	ND

ND - Not detected

Table XV

GAS-OFF PRODUCTS FROM
DAC 051 (ZYTEL NYLON RESIN TYPE 103 COLOR NC10)

<u>Component</u>	<u>Weight of Component (mg/10 gms Candidate Material)</u>	
	<u>72 Hours (68°C)</u>	<u>30 Days (25°C)</u>
Carbon Monoxide	0.007	0.001
Methane	0.007	ND

ND - Not detected

Table XVI

GAS-OFF PRODUCTS FROM
DAC 052 (DUREZ MOLDING COMPOUND, 1900 BLACK PHENOLIC)

<u>Component</u>	<u>Weight of Component (mg/10 gms Candidate Material)</u>	
	<u>72 Hours (68°C)</u>	<u>30 Days (25°C)</u>
Carbon Monoxide	0.08	0.004
Methane	0.007	ND

ND - Not detected

Table XVII

GAS-OFF PRODUCTS FROM
DAC 053 (NOPCOFOAM G-302)

<u>Component</u>	<u>Weight of Component (mg/10 gms Candidate Material)</u>	
	<u>72 Hours (68°C)</u>	<u>30 Days (25°C)</u>
Carbon Monoxide	0.007	0.001
Methane	0.002	ND

ND - Not detected

Table XVIII

GAS-OFF PRODUCTS FROM
DAC 054 (ELECTRICAL TAPE NO. 60,
TFE-FLUOROCARBON FILM, THERMOSETTING SILICONE)

<u>Component</u>	<u>Weight of Component</u> <u>(mg/10 gms Candidate Material)</u>	
	<u>72 Hours</u> <u>(68°C)</u>	<u>30 Days</u> <u>(25°C)</u>
Dimethyl Ether	0.12	0.049
Acetone	0.031	0.008
Ethanol	2.1	0.69
Carbon Monoxide	0.03	0.006
Methane	0.009	ND

ND - Not detected

Table XIX

GAS-OFF PRODUCTS FROM
DAC 055 (PRESSURE SENSITIVE ADHESIVE TAPE,
#E-284-6-ERH, RUN #2355)

<u>Component</u>	<u>Weight of Component</u> <u>(mg/10 gms Candidate Material)</u>	
	<u>72 Hours</u> <u>(68°C)</u>	<u>30 Days</u> <u>(25°C)</u>
Ethanol	0.15	0.090
Carbon Monoxide	0.002	ND
Methane	0.01	0.002

ND - Not detected

Table XX

GAS-OFF PRODUCTS FROM
DAC 056 (PRINTED CIRCUIT BOARD)

<u>Component</u>	<u>Weight of Component (mg/10 gms Candidate Material)</u>	
	<u>72 Hours (68°C)</u>	<u>30 Days (25°C)</u>
Carbon Monoxide	0.006	ND
Methane	0.007	ND

ND - Not detected

Table XXI

GAS-OFF PRODUCTS FROM
DAC 057 (TAPE, SCOTCH BRAND #4116 YAK 41171T)

Component	Weight of Component (mg/10 gms Candidate Material)	
	72 Hours (68°C)	30 Days (25°C)
Acetone	0.004	ND
n-Propanol	0.004	ND
Toluene	0.002	ND
n-Butanol	0.014	ND
Xylenes	0.010	ND
C ₄ Alkylbenzene	0.019	ND
Ethyl Esters of C ₄ or C ₅ Acids (2 Types)	0.27	ND
6-Methylheptanol	0.23	ND
Carbon Monoxide	0.02	0.006
Methane	0.007	ND

ND - Not detected

APPENDIX III

REPRESENTATIVE GAS CHROMATOGRAMS FOR GAS-OFF EXPERIMENTS

The gas chromatograms shown in this appendix were obtained on an F&M Scientific Corporation Model 810 Research Gas Chromatograph. Instrument conditions and column specifications are listed in Table XXII. Since retention times tended to shift somewhat due to column aging, a standard mixture was used as a day-to-day reference. The first peak appearing in each chromatogram is air.

The gas chromatograms are representative of a particular candidate material. Comparison of peak intensities in chromatograms for different candidate materials should be made with care, since sensitivity factors and atmosphere sample sizes vary.

Chromatograms appear in order of assigned Air Force serial numbers. Names of materials are those submitted by the Air Force.

Table XXII

GAS CHROMATOGRAPHIC INSTRUMENT CONDITIONS

All samples were analyzed using a flame ionization detector and an F&M Model 810 Research Gas Chromatograph in a single column and single detector mode.

Instrument Conditions

Column: 20-ft x 1/4-in. O.D. Stainless Steel, 20% Triton X-305 on 60/80 mesh Gas Chrom Z.

Column Temperature: programmed 50°-170°C @ 8°C/min.

Detector Temperature: 300°C

Injection Port Temperature: 250°C

Flow Split: none

Flow Rate: 60 ml/min. He

Range: 10

Attenuation: X8, or as noted

Sample Size: 50 cc of gas

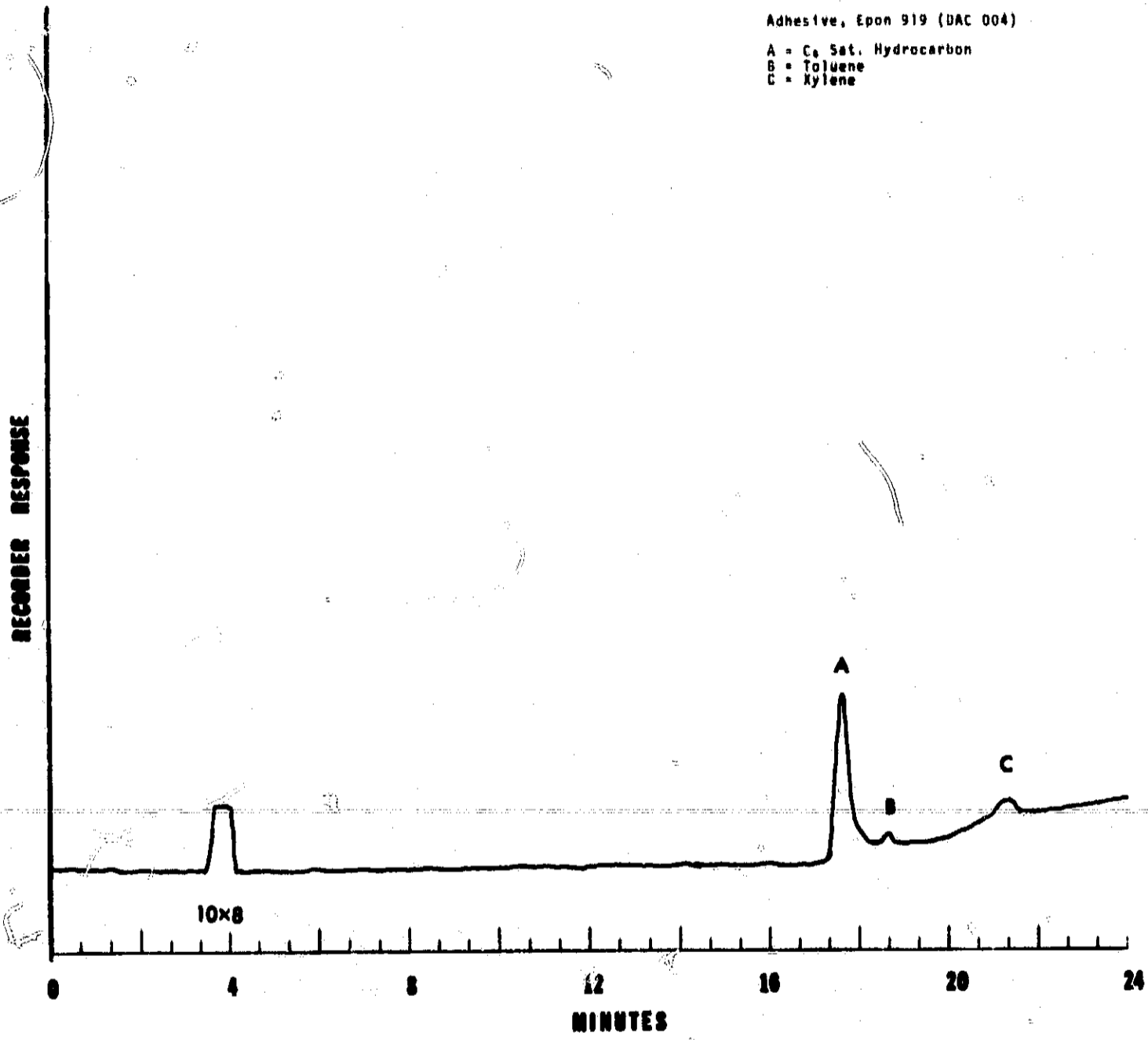


Figure 20. Gas Chromatogram of Gas-Off Products From Adhesive, Epon 919 (DAC 004) (72 hours @ 68°C).

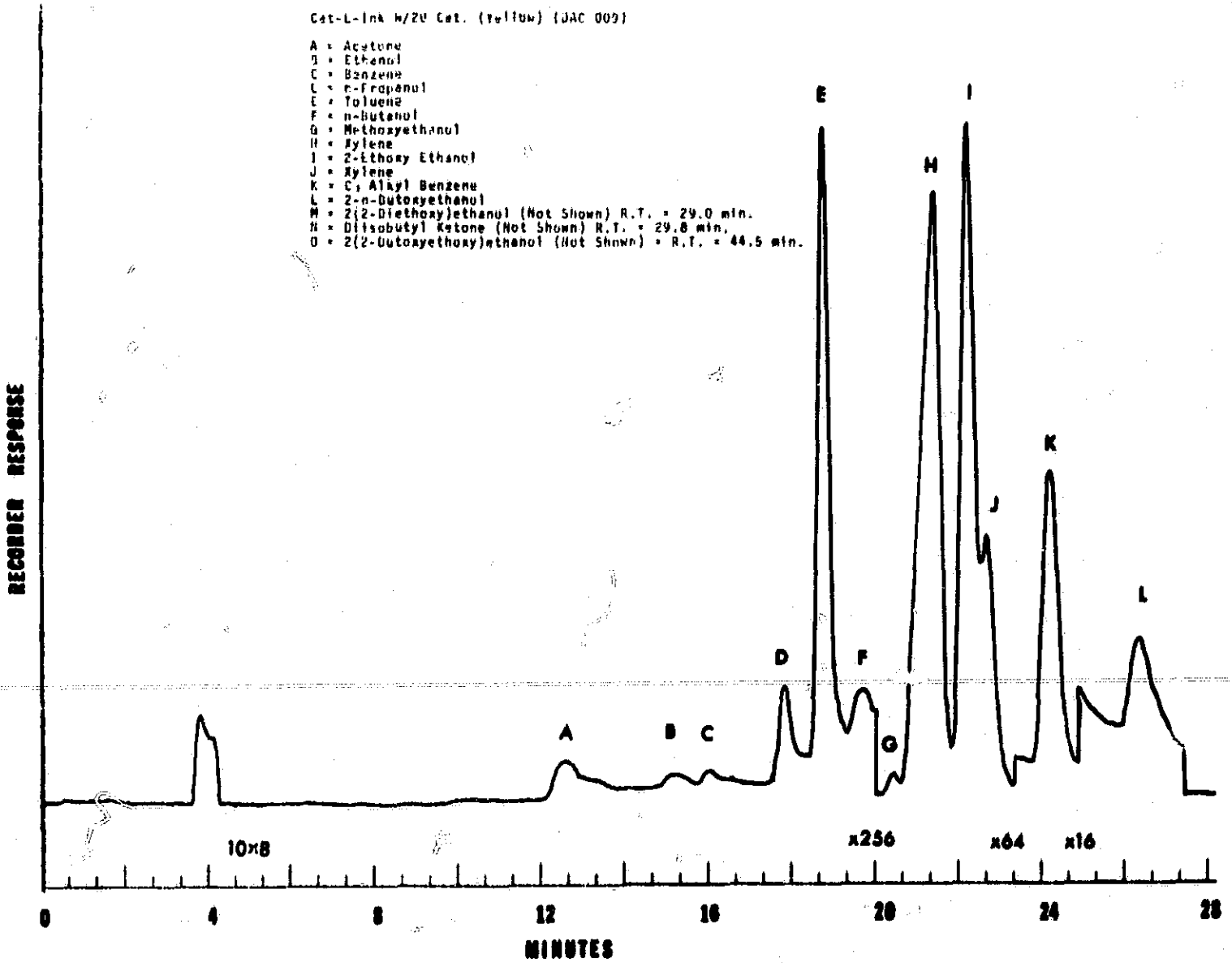


Figure 21. Gas Chromatogram of Gas-Off Products From Cat-L-Ink W/20 Cat. (Yellow) (DAC 009) (72 hours @ 68°C).

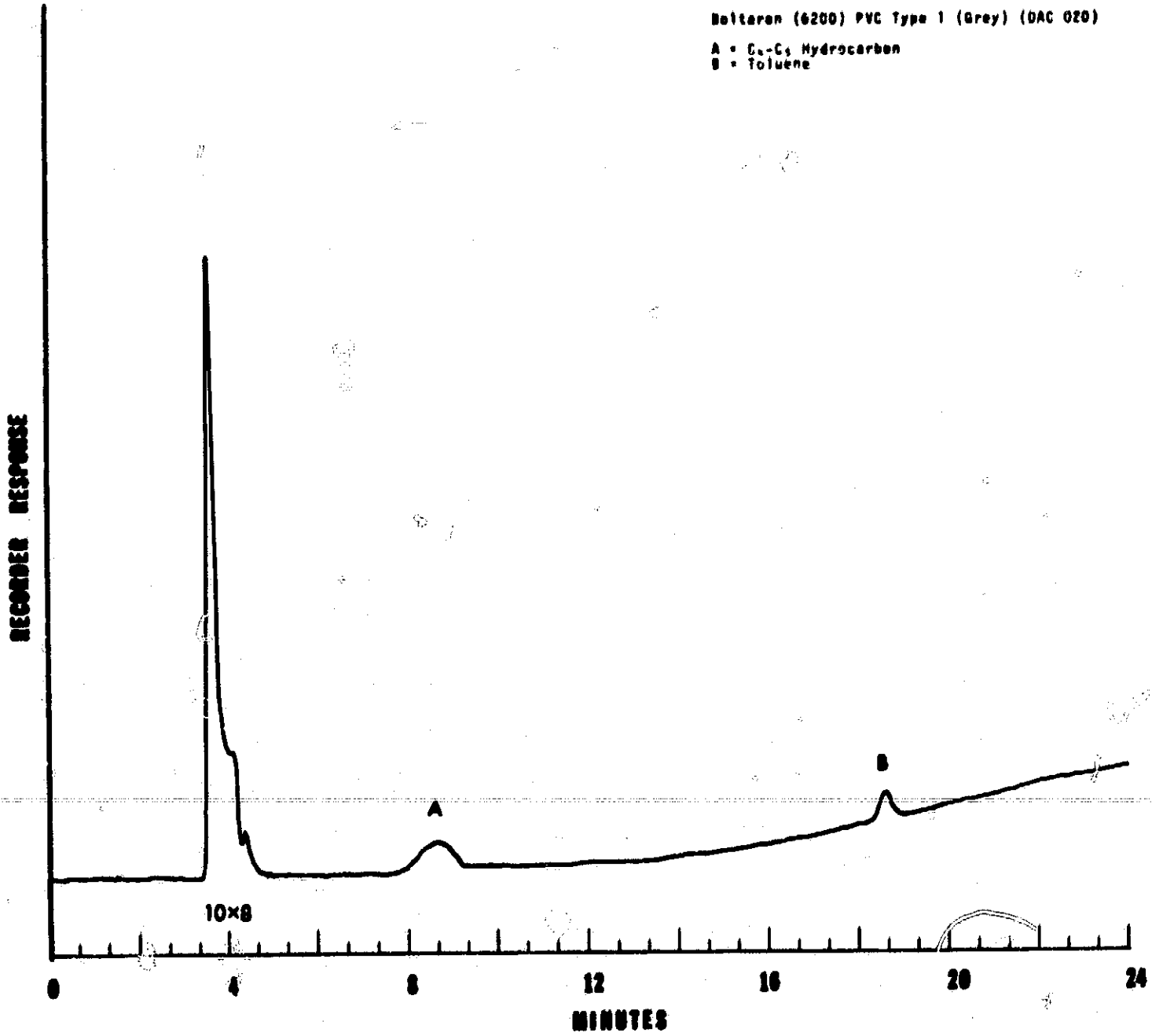


Figure 22. Gas Chromatogram of Gas-Off Products From Boltaron (6200) PVC Type 1 (Grey) (DAC 020) (72 hours @ 68°C).

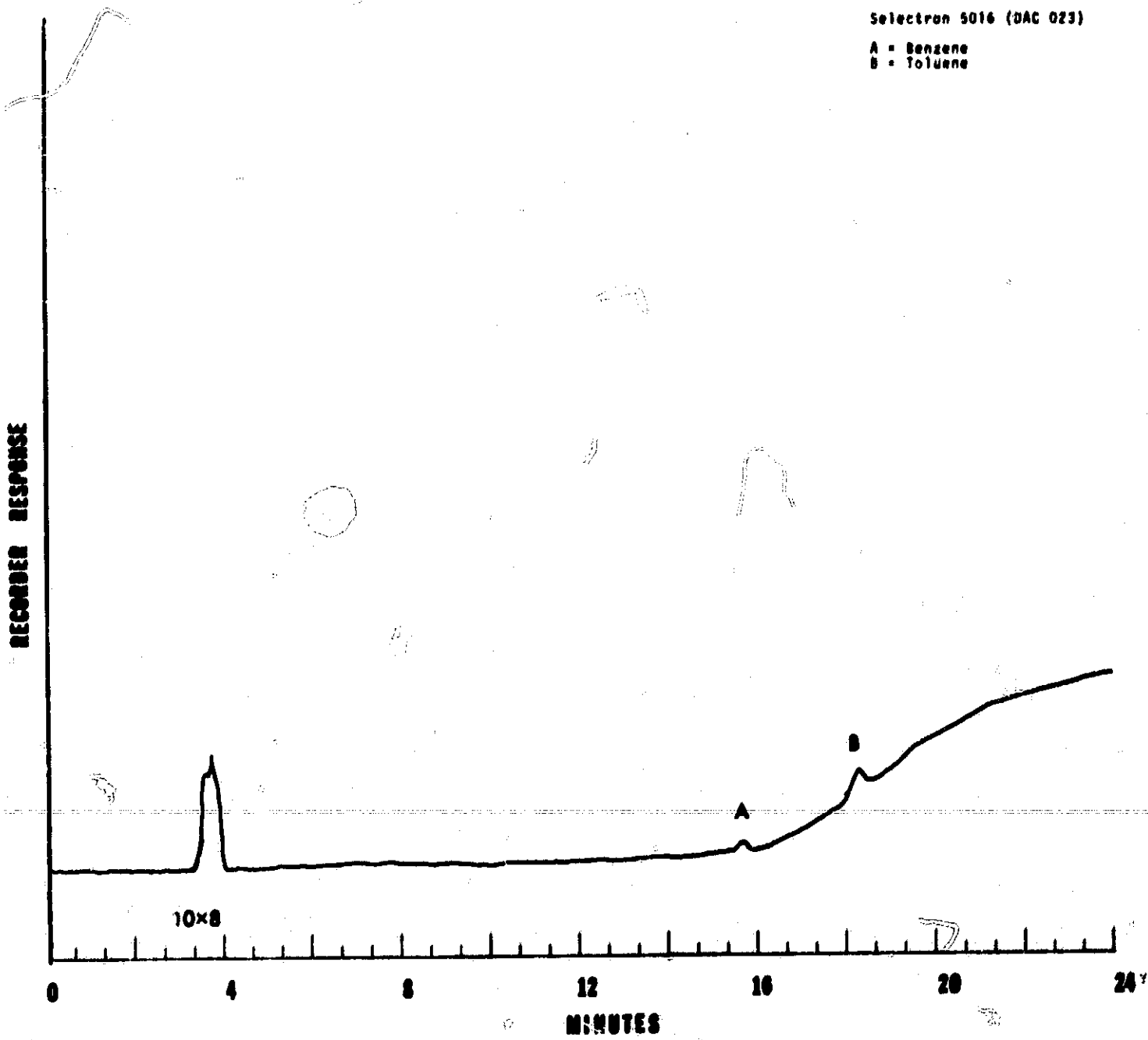


Figure 23. Gas Chromatogram of Gas-Off Products From Selectron 5016 (DAC 023) (72 hours @ 68°C).

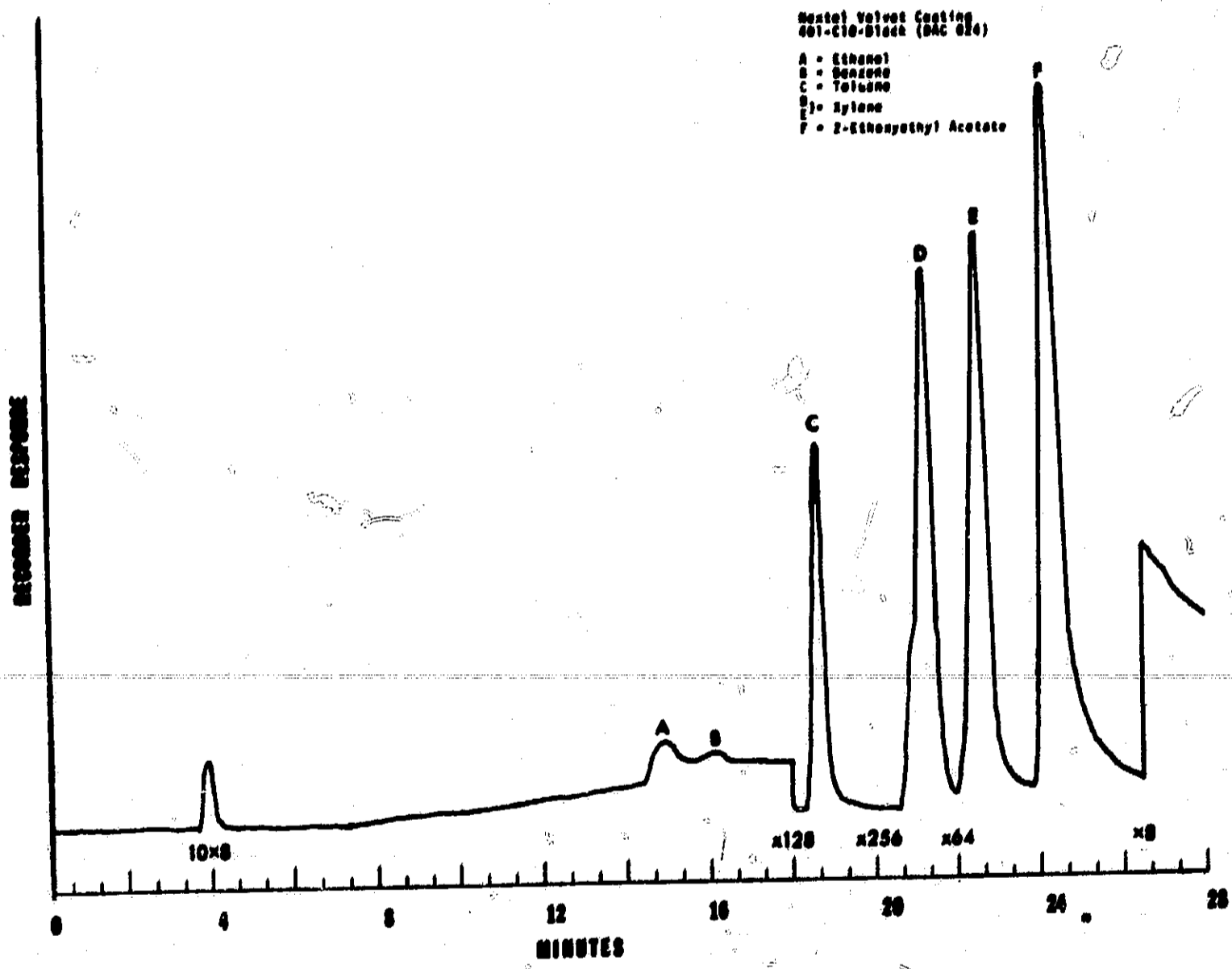


Figure 24. Gas Chromatogram of Gas-Off Products From Nextel Velvet Coating 401-C10-Black (DAC 024) (72 hours @ 68°C).

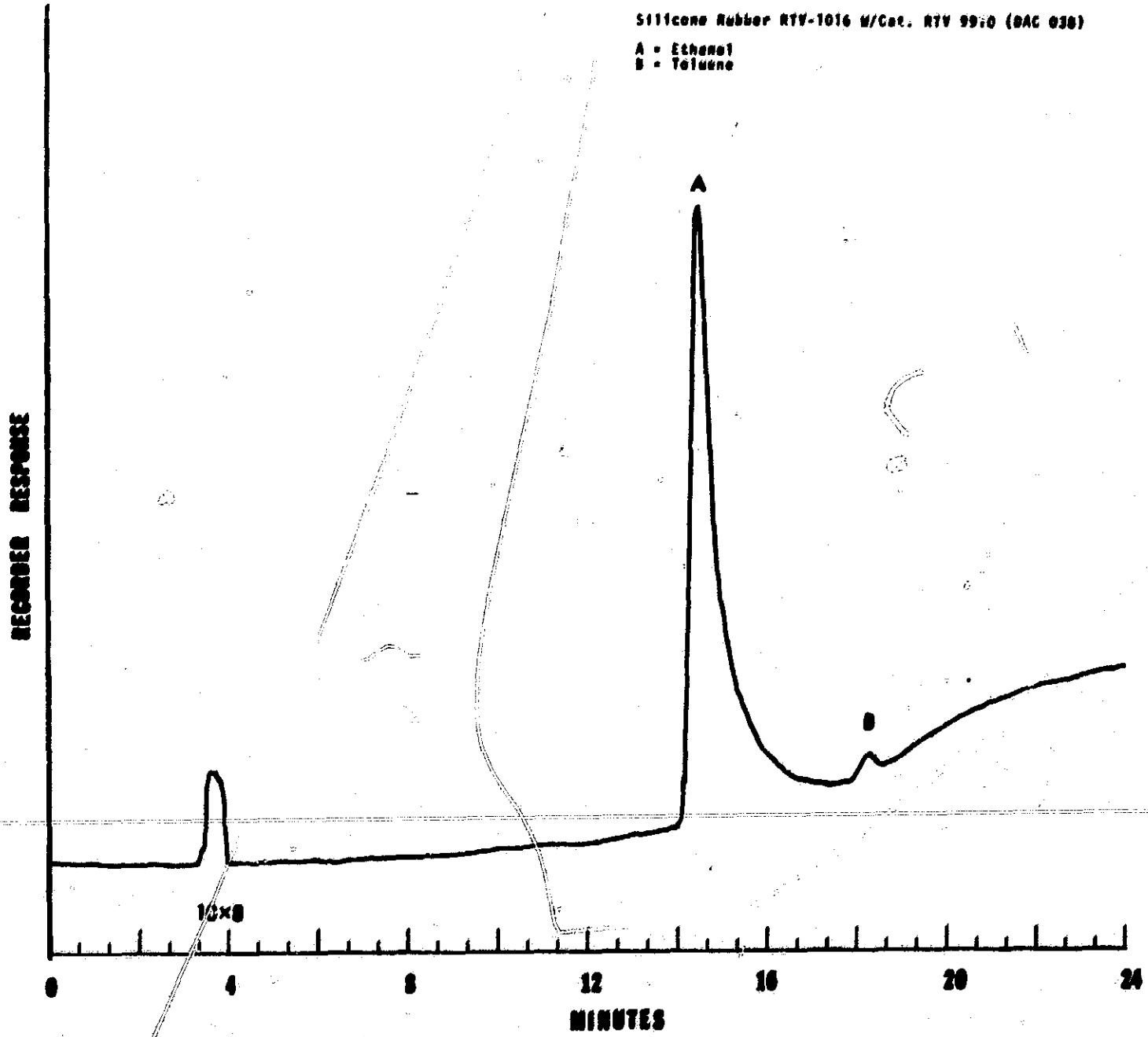


Figure 25. Gas Chromatogram of Gas-Off Products From Silicone Rubber RTV-1016 W/Cat. RTV 9910 (DAC 038) (72 hours @ 68°C).

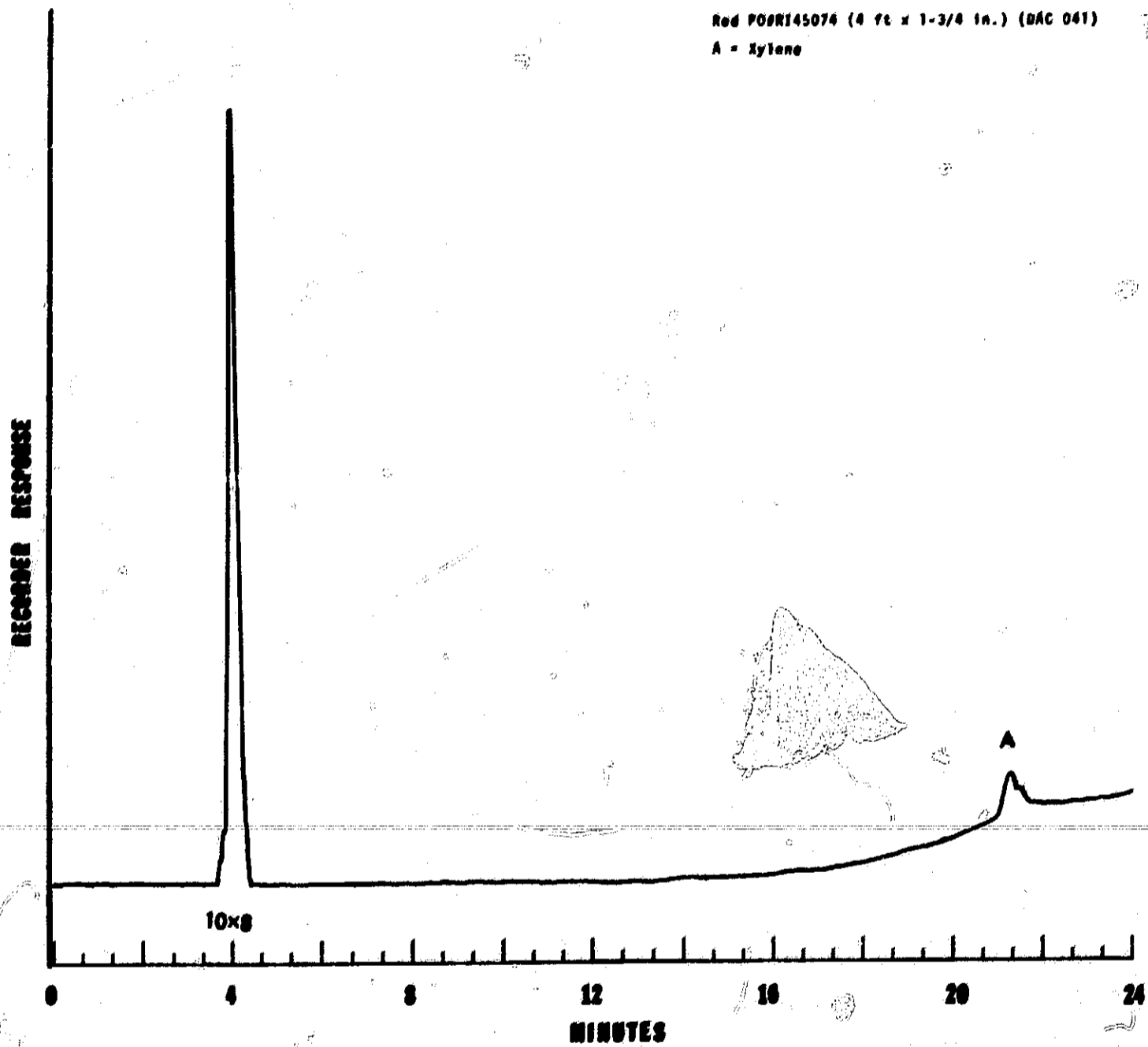


Figure 26. Gas Chromatogram of Gas-Off Products From Rod PO#RI45074 (4 ft x 1-3/4 in.) (DAC 041) (72 hours @ 68°C).

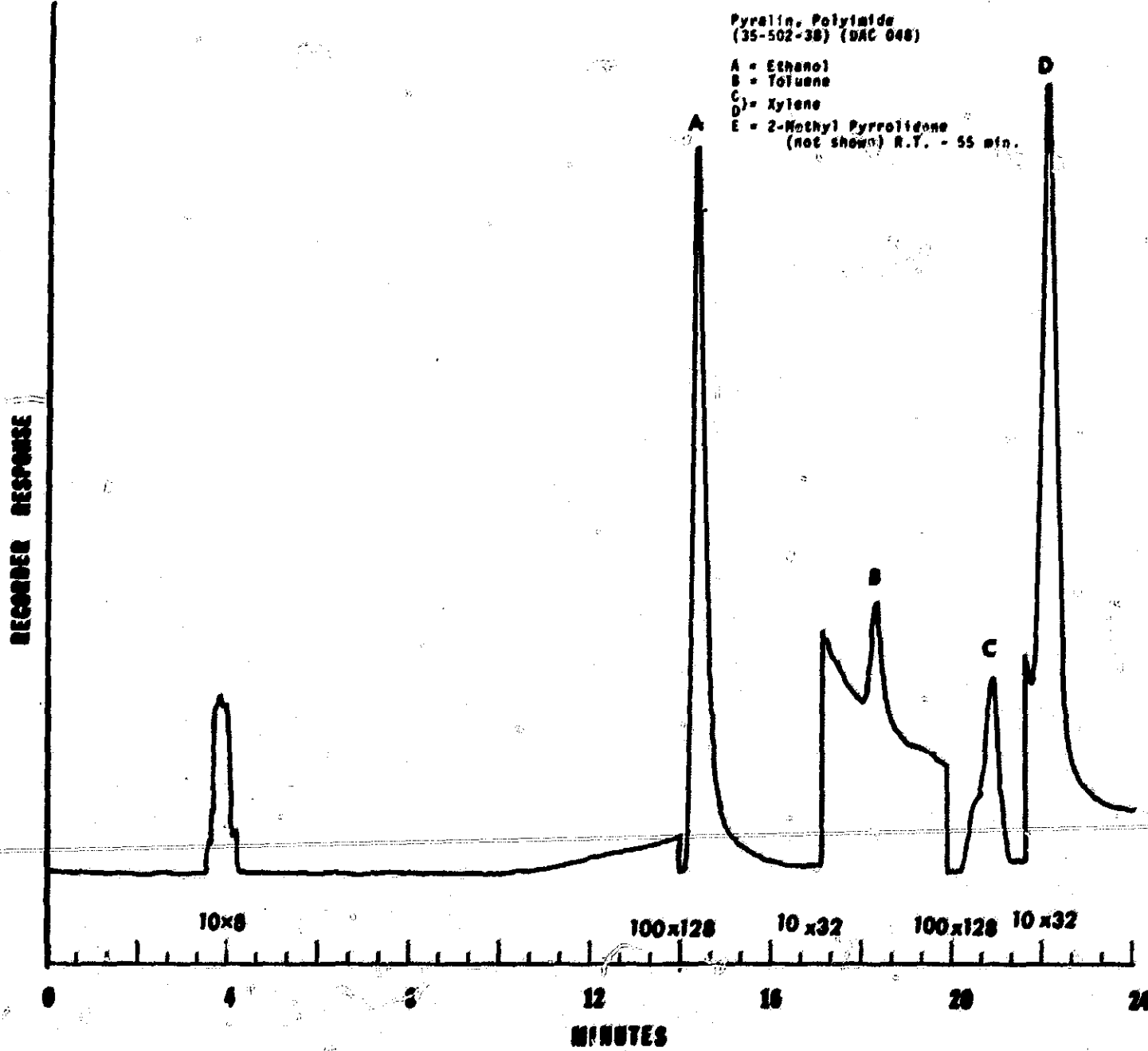


Figure 27. Gas Chromatogram of Gas-Off Products From Pyralin, Polyimide (35-502-38) (DAC 048) (72 hours @ 68°C).

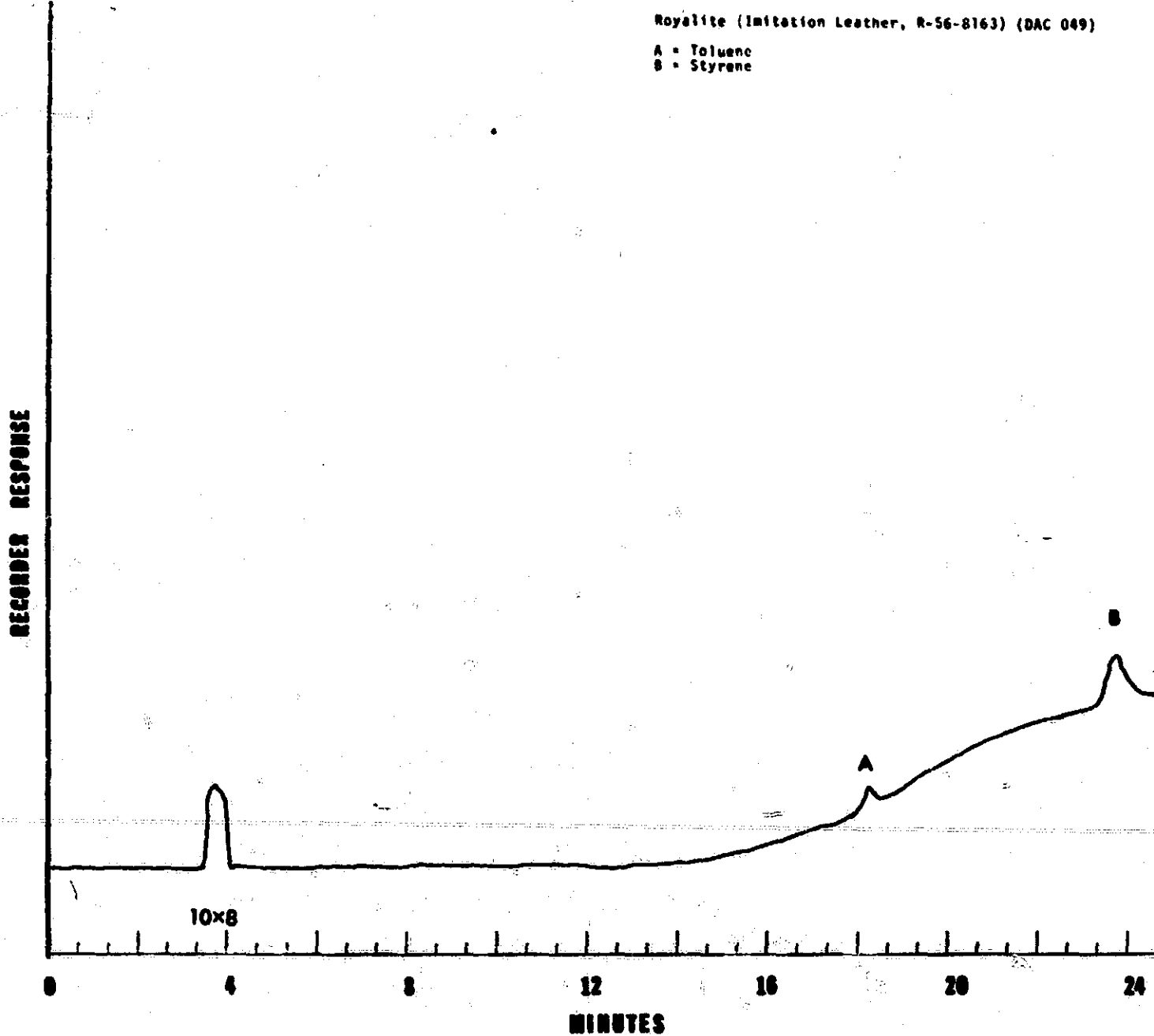


Figure 28. Gas Chromatogram of Gas-Off Products From Royalite (Imitation Leather, R-56-8163) (DAC 049) (72 hours @ 68°C).

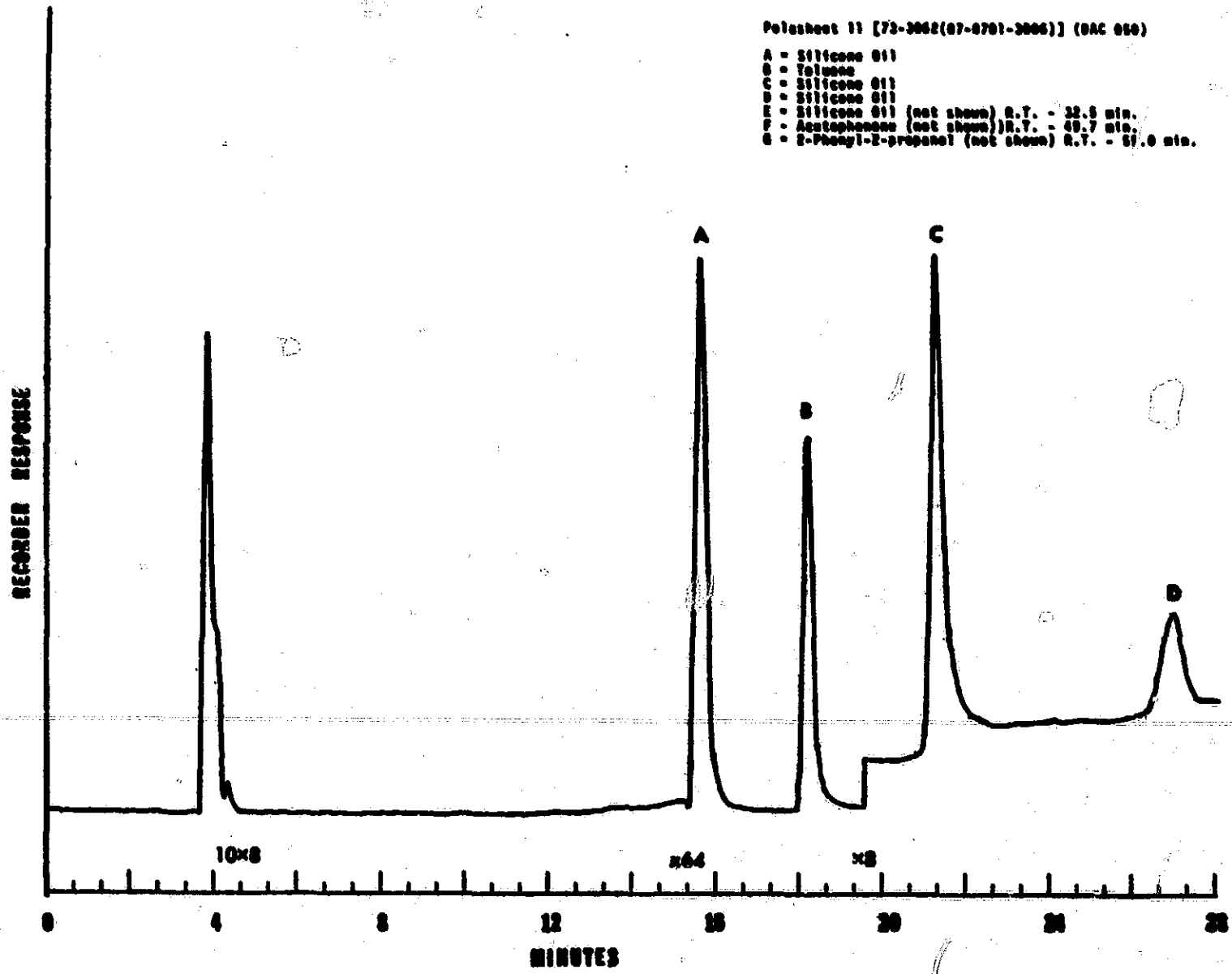


Figure 29. Gas Chromatogram of Gas-Off Products From Polasheet 11[73-3062(07-0701-3006)] (DAC 050) (72 hours @ 68°C).

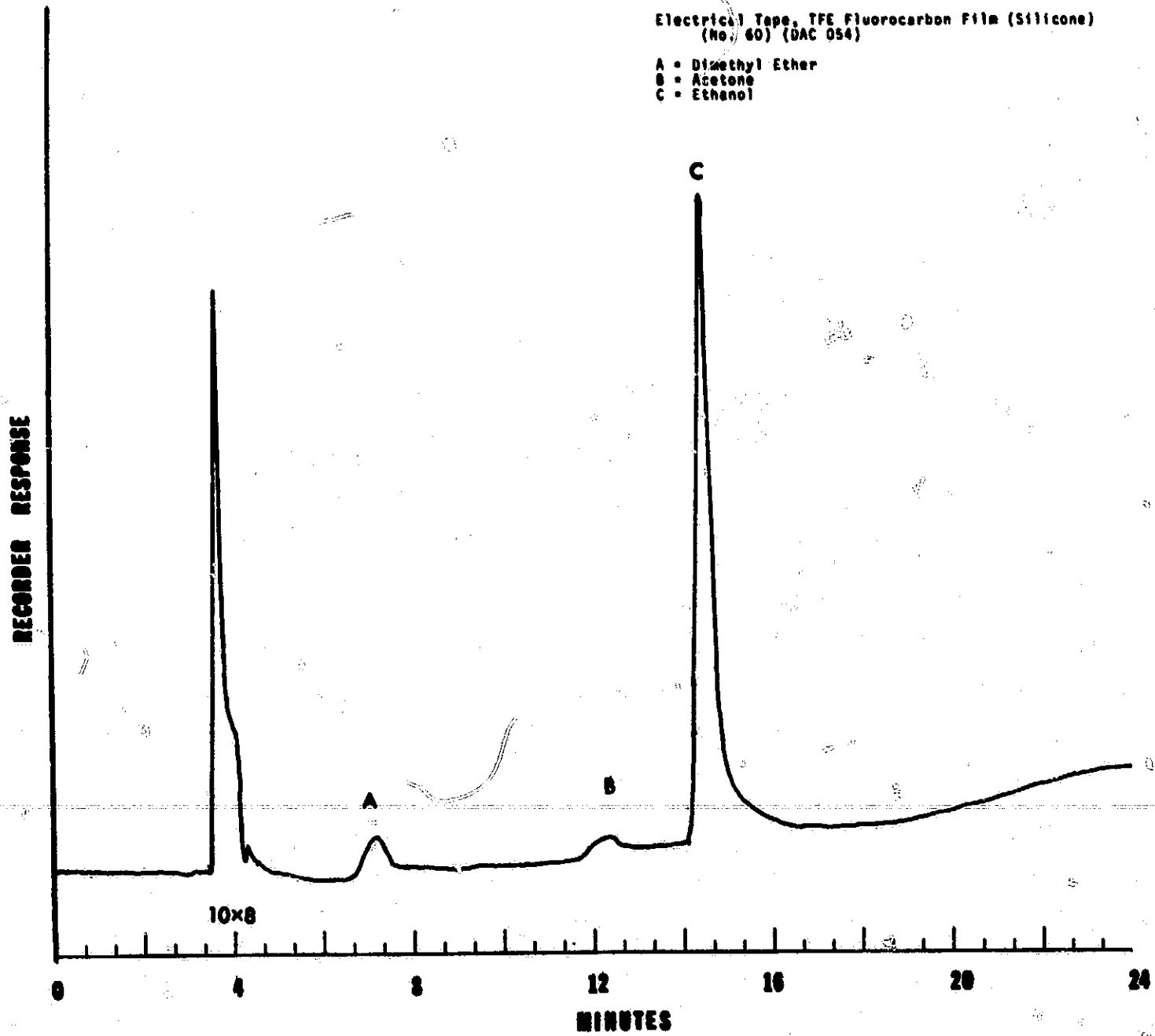


Figure 30. Gas Chromatogram of Gas-Off Products From Electrical Tape, TFE Fluorocarbon Film (Silicone) (No. 60) (DAC 054) (72 hours @ 68°C).

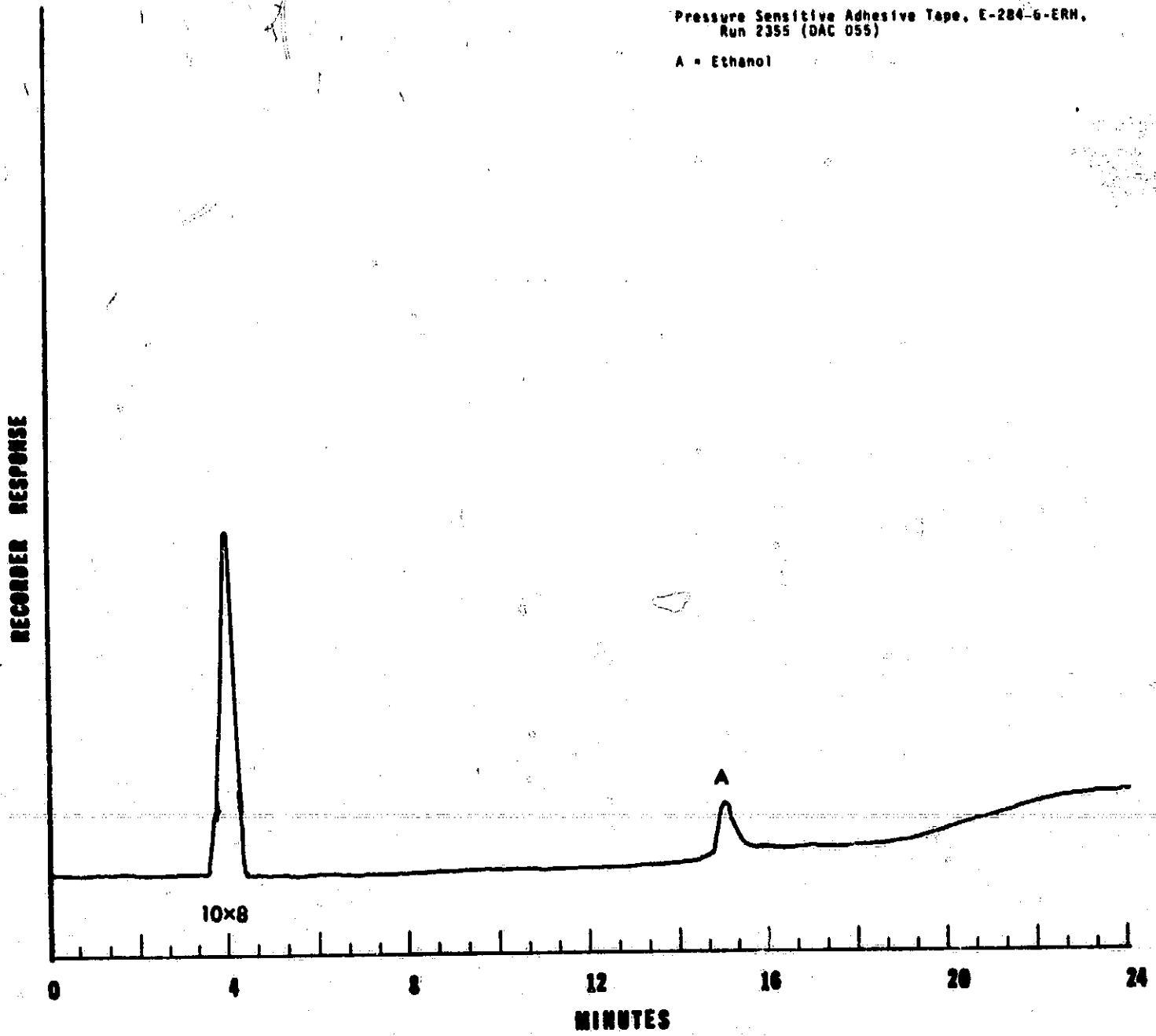


Figure 31. Gas Chromatogram of Gas-Off Products From Pressure Sensitive Adhesive Tape, E-284-6-ERH, Run 2355 (DAC 055) (72 hours @ 68°C).

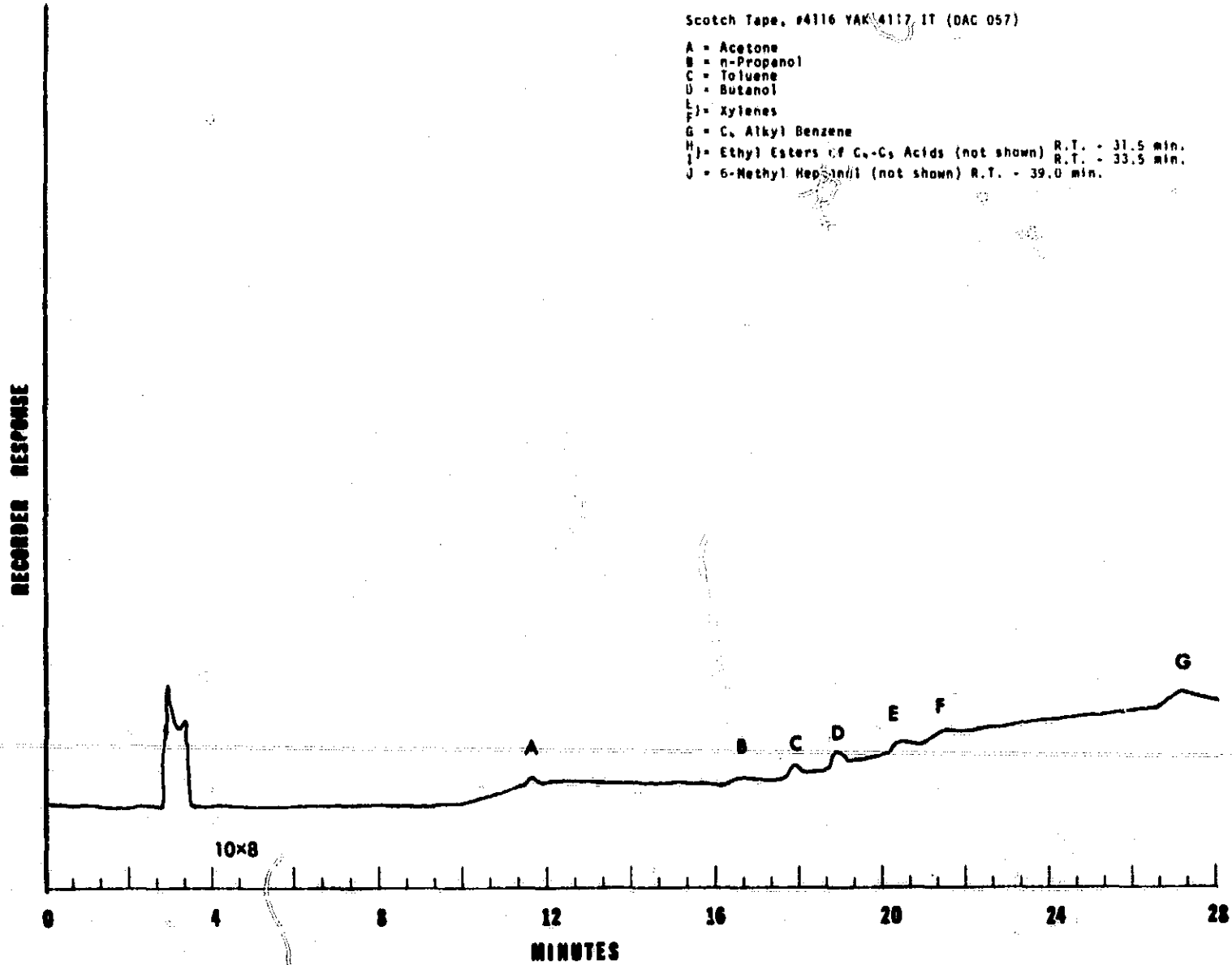


Figure 32. Gas Chromatogram of Gas-Off Products From Scotch Tape, #4116 YAK 4117IT (DAC 057) (72 hours @ 68°C).

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