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(NASA-CR-148567) STUDY OF THERMAL STABILITY
AND DEGRADATION OF FIRE RESISTANT CANDIDATE
POLYMERS FOR AIRCRAFT INTERIORS Final
Report, Aug. 1975 - Jul. 1976 (San Jose
State Univ., Calif.) 33 p HC \$4.00 CSCL 11D G3/27

N76-29419
HC \$4.00

Unclas
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Study of Thermal Stability and Degradation of
Fire Resistant Candidate Polymers for Aircraft Interiors

Final Report (August 1975 - July 1976)

by

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NASA Grant No. NSG-2093



prepared for

National Aeronautics and Space Administration
Ames Research Center
Moffett Field, California 94035

Study of Thermal Stability and Degradation of Fire Resistant Candidate Polymers for Aircraft Interiors

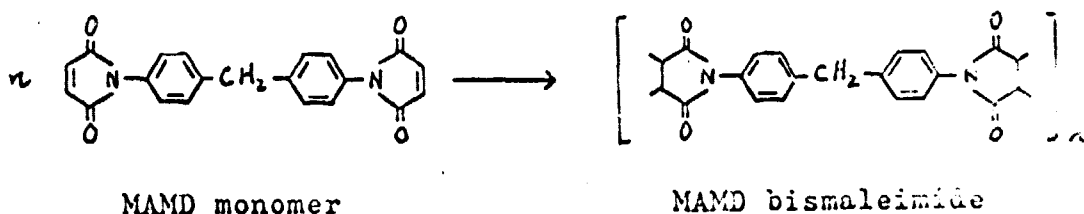
I. Introduction

The thermochemistry of bismaleimide resins and phenolphthalein polycarbonate were studied. Both types of materials are fire-resistant polymers and may be suitable for aircraft interiors. The chemical composition of the polymers have been determined by nuclear magnetic resonance and infrared spectroscopy and by elemental analysis. Thermal properties of these polymers have been characterized by thermogravimetric analyses. Qualitative evaluation of the volatile products formed in pyrolysis under oxidative and non-oxidative conditions have been made using infrared spectrometry. The residues after pyrolysis were analyzed by elemental analysis. Thermal stability of composite panel and thermoplastic materials for aircraft interiors were studied by thermogravimetric analyses.

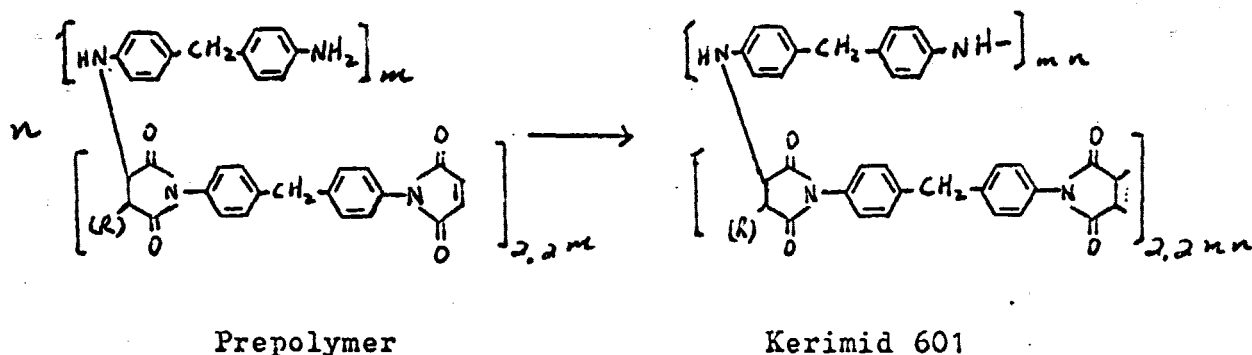
II. Bismaleimide resins.

A. Chemical composition

Two types of bismaleimide resins have been studied: MAMD and Kerimid 601. The MAMD bismaleimide resin was prepared by molding the monomer at high temperature (240°C) and high pressure (5000 psi) for two hours and then post curing at 240°C for 24 hours to give a highly crosslinked material.



The chemical structure of the MAMD monomer was confirmed by nuclear magnetic resonance and infrared spectroscopy. Elemental analysis data of the MAMD bismaleimide were compared with the theoretical value shown in Table I. Kerimid 601 was prepared by molding the prepolymer in the same manner as MAMD bismaleimide. Nuclear magnetic resonance spectrum of the Kerimid 601 prepolymer showed that it was a mixture of p-methylene dianiline and bismaleimide monomer in 1:2.2 ratio, and that it also contained about 5% aliphatic groups.



Elemental analysis of the prepolymer and Kerimid 601 were compared with their theoretical composition without counting the aliphatic groups shown in Table I. Kerimid 601 was not as brittle as MAMD since Kerimid 601 was not a highly crosslinked polymer compared with MAMD. Both bismaleimide resins were solvent resistant and stable below 300°C.

B. Thermal analysis

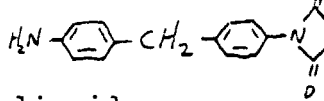
Thermal analyses of MAMD bismaleimide and Kerimid 601 were conducted on a Du Pont 950 Thermogravimetric Analyzer using both air and nitrogen atmospheres. Figs. 1 and 2 are the thermograms for MAMD

and Kerimid 601 in air and in nitrogen at various heating rates. These polymers were powdered prior to analysis. The 2% weight loss at about 100°C was the loss of absorbed moisture. The char yield for both polymers were about 60% in nitrogen. The decomposition temperature of Kerimid 601 was lower than that of MAMD bismaleimide. From the dynamic TGA curves,¹ the activation energy of decomposition of MAMD was found to be 45 and 67 Kcal/mole in air and in nitrogen, respectively. The activation energy of decomposition of Kerimid 601 was found to be 48.7 and 56.8 Kcal/mole in air and in nitrogen, respectively.

Isothermal analysis curves for MAMD bismaleimide and Kerimid 601 at different temperatures¹ in air and in nitrogen are shown in Figs. 3 to 6. Based on the isothermal analysis curves, the activation energy² of initial decomposition for MAMD bismaleimide was found to be 52.7 and 61.6 Kcal/mole in air and in nitrogen, respectively; the activation energy for Kerimid 601 was found to be 45.7 and 53.9 Kcal/mole in air and in nitrogen, respectively. These data are in good agreement with the data derived from the dynamic TGA method.

C. Thermal Degradation

Thermal degradation of both bismaleimides have been studied in air, nitrogen and in vacuum. The results are shown in Table II. The percentage of evolved gases was obtained by difference. Qualitative analyses of the gases were made using infrared spectroscopy³. The evolved gases from pyrolysis of both polymers in vacuum consisted of carbon dioxide, carbon monoxide, some methane, ammonia, water and a trace of hydrogen cyanide. The volatile materials from

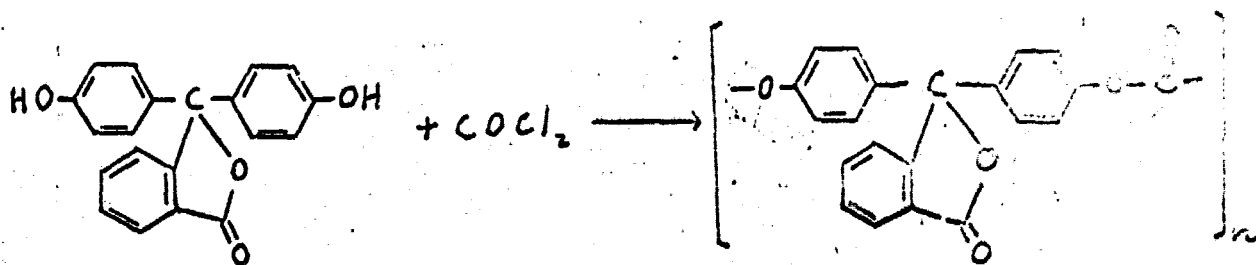
pyrolysis of both polymers in air contained mainly carbon dioxide and water. The residues after pyrolysis in nitrogen have been analyzed by elemental analysis. The residues showed increases in carbon content and decreases in hydrogen and oxygen content (Table I). Two major products shown by gas and liquid chromatography were present in the clear volatile liquid from pyrolysis of both polymers in vacuum. The two major products in the clear liquid were analyzed by gas chromatography-mass spectrometer (GCMS) and found they are aniline and methylaniline in the ratio of 3:2. The mass spectra of aniline and methylaniline from pyrolysis of bismaleimide polymers are shown in Fig 7 and 8. There are trace amount of benzene and toluene also present in the clear liquid. The brown liquid is not very volatile and would not pass through the gas chromatograph column. 100 ng of this brown liquid was introduced to the ion source of the mass spectrometer using direct inlet probe (DIP). The DIP was gradually heated from room temperature to 430°C. Start from 230°C some of the materials are volatile enough to give mass spectra. All the mass spectra above 230°C contained a peak of m/e 280 indicated that  or its dimer, trimer may be present in the brown liquid.

The mechanisms of thermal degradation of bismaleimide needs more study.

III. Phenolphthalein Polycarbonate

A. Chemical composition

Phenolphthalein polycarbonate was a white powder prepared by Dow Chemical Company from interaction of phenolphthalein and phosgene.



The phenolphthalein polycarbonate studied had an intrinsic viscosity of 0.342 in dichloromethane at 25°C. The molecular weight was around 1×10^4 . Infrared and nuclear magnetic resonance spectra and elemental analysis confirmed the chemical structure.

B. Thermal analyses

Thermogravimetric curves in air and in nitrogen were shown in Fig. 9. The polymer contained 2% absorbed moisture and decomposed at 390°C in both air and nitrogen. The char yield in nitrogen at 800°C was 48%. The activation energy of the initial decomposition determined from TGA curves was 41 Kcal/mole in air and in nitrogen. Isothermal thermogravimetric analyses of phenolphthalein polycarbonate in air have been studied (Fig. 10). The estimated activation energy for initial decomposition was 43.6 Kcal/mole which agrees with the dynamic TGA data. The activation energy of decomposition of phenolphthalein polycarbonate is similar to the value reported for bisphenol A polycarbonate⁴, since the initial decomposition for both polycarbonates is the loss of carbon dioxide.

C. Thermal degradation

Although the decomposition temperature of phenolphthalein polycarbonate was 390°C in air and in nitrogen, as shown by thermograms, the polymer decomposed isothermally below 390°C without showing much weight loss and gave yellow to brown colored material depending on the temperature and duration. Unlike bisphenol A polycarbonate which underwent chain scission only at temperatures below 380°C in a sealed vacuum tube⁵, phenolphthalein polycarbonate underwent both chain scission and crosslinking below 300°C in a sealed vacuum tube. The results are shown in Table III. When phenolphthalein polycarbonate was heated at 254°C in a sealed vacuum tube, the polymer showed slightly yellow color after 2.5 hours of heating. The yellow phenolphthalein polycarbonate was soluble in dichloromethane and had less than 0.1% gel. After 30 hours of heating at 254°C , 97% of the polymer was soluble in dichloromethane with less than 0.5% weight loss. The sol was bright yellow in color with an absorption at 370 nm and had an intrinsic viscosity of 0.28 which was smaller than the original polymer. Infrared and proton nuclear magnetic resonance spectra were identical to those for phenolphthalein polycarbonate, indicating little microstructural changes other than chain scission. The yellow color of the sol may be due to low percentage quinone of structural formation in the chain which was not detectable by infrared and nuclear magnetic resonance techniques. For prolonged heating the sol showed some decrease in intrinsic viscosity, but the formation of gel increased substantially with longer heating

loss. Gel formation may be associated with weight loss (loss of carbon dioxide and phenol). The formation of gel was much faster at higher temperature. A thin film of phenolphthalein polycarbonate was casted on a sodium chloride salt plate and then heated at 300-420°C in vacuum. After 1-2 hours the film turned to brown color, the infrared spectrum of the brown film showed some change in carbonate absorption due to loss of carbon dioxide and also showed several new bands at 1240, 1735 and 1665 cm^{-1} corresponding to aromatic ether, ester and diaromatic ketone absorptions.

Phenolphthalein polycarbonate was heated at 420°C under vacuum for 1 hour and gave 59.6% black residue, 24.7% gases, and 15.6% volatile solids. The gases consisted of mainly carbon dioxide, carbon monoxide and a small amount methane. The volatile solids contained mainly phenol which was identified by infrared. After removal of the phenol by sublimation, the rest of the volatile solids were analyzed by mass spectrometer using direct inlet probe for identification. When the DIP was heated to 190°C some solid is volatile. The mass spectrum of this solid is shown in Fig. 11. The mass spectrum indicated this solid might be 2-hydroxy-anthraquinone. Phenolphthalein polycarbonate was stable at 210°C in vacuum for one week. If phenolphthalein polycarbonate was heated in air at 210°C, the polymer gradually changed to yellow color after two days.

A proposed mechanisms of thermal degradation of phenolphthalein polycarbonate is shown in Fig. 12.

loss. Gel formation may be associated with weight loss (loss of carbon dioxide and phenol). The formation of gel was much faster at higher temperature. A thin film of phenolphthalein polycarbonate was casted on a sodium chloride salt plate and then heated at 365-420°C in vacuum. After 1-2 hours the film turned to brown color, the infrared spectrum of the brown film showed some change in carbonate absorption due to loss of carbon dioxide and also showed several new bands at 1240, 1735 and 1665 cm^{-1} corresponding to aromatic ether, ester and diaromatic ketone absorptions.

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A proposed mechanisms of thermal degradation of phenolphthalein polycarbonate is shown in Fig. 12.

IV. Composite and Thermoplastic materials

In the later part of my grant period I was involved in the thermal stability study of composite panel and thermoplastic materials used in aircraft. Seven Boeing composites and two NASA-Ames developed composite panel materials and 25 thermoplastic materials were studied by thermogravimetric analyses both in air and in nitrogen. The results are summarized in Table IV-VII.

0.3255 g. of Boeing standard panel was pyrolyzed in air at 700°C for 30 min. in a 5 liter chamber. After pyrolysis it gave 0.1323 g. residue (40.64%), 0.05 g. volatile solid and gases. The gases was analyzed by infrared and specific ion electrode and found 289.5 mg carbon dioxide (3.2% by volume), 40.32 mg carbon monoxide (7000 ppm), moisture, 0.24 mg hydrogen cyanide (42 ppm) and trace of methane.

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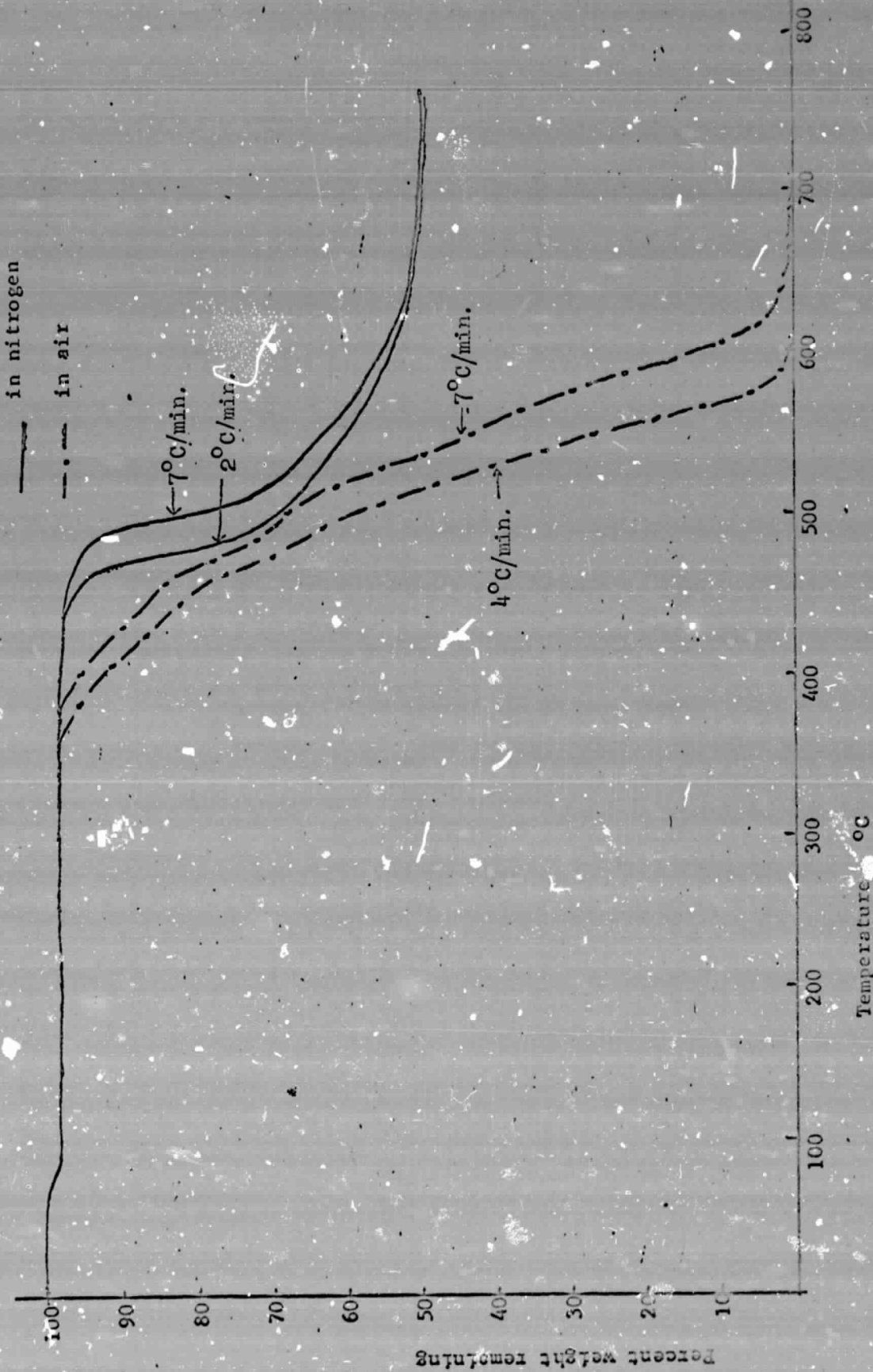


FIG. 1. Thermograms of MMD bismaleimide in air and in nitrogen at various heating rates.

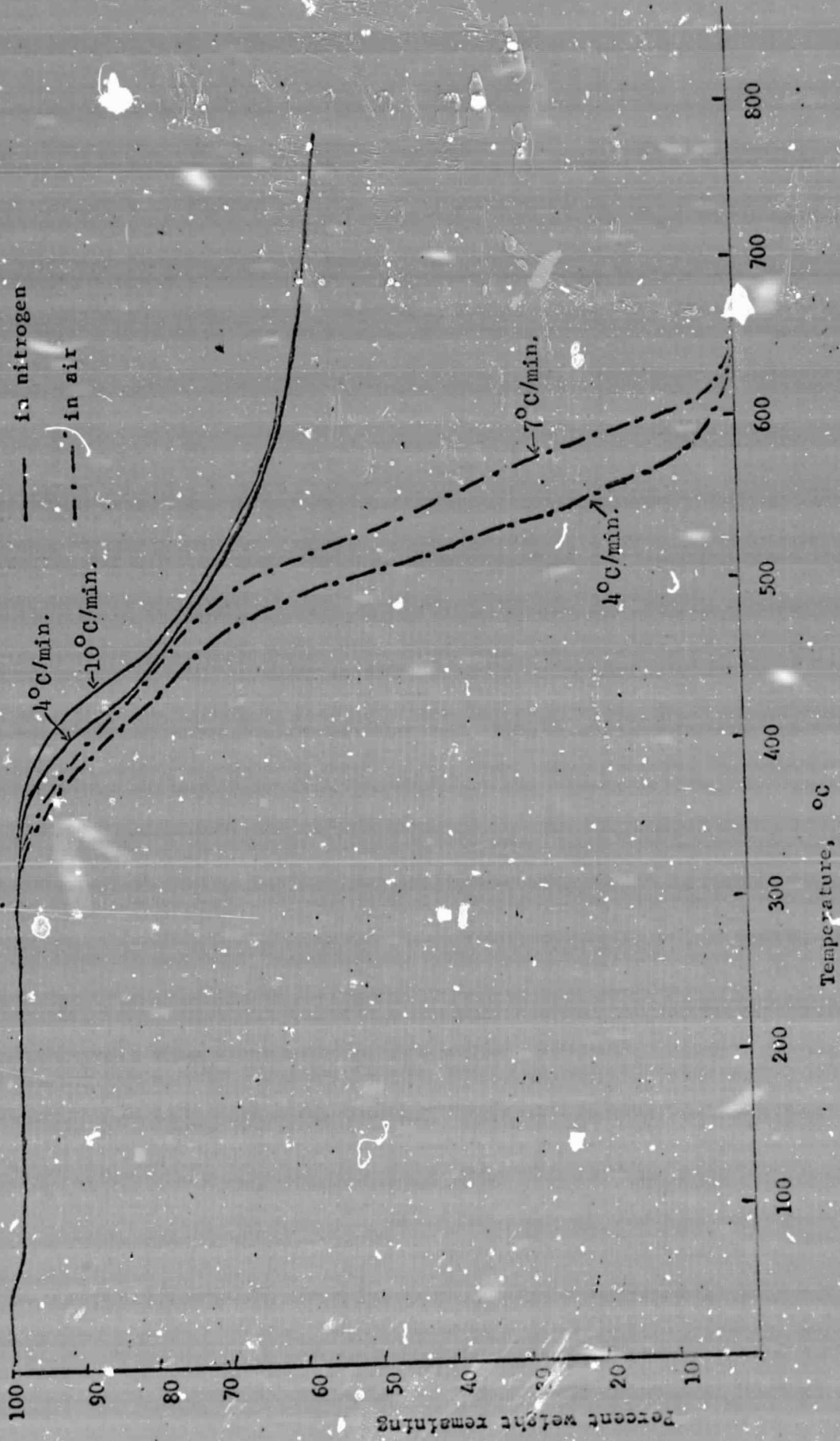


FIG. 2. Thermograms of Kerimid 601 in air and in nitrogen at various heating rates.

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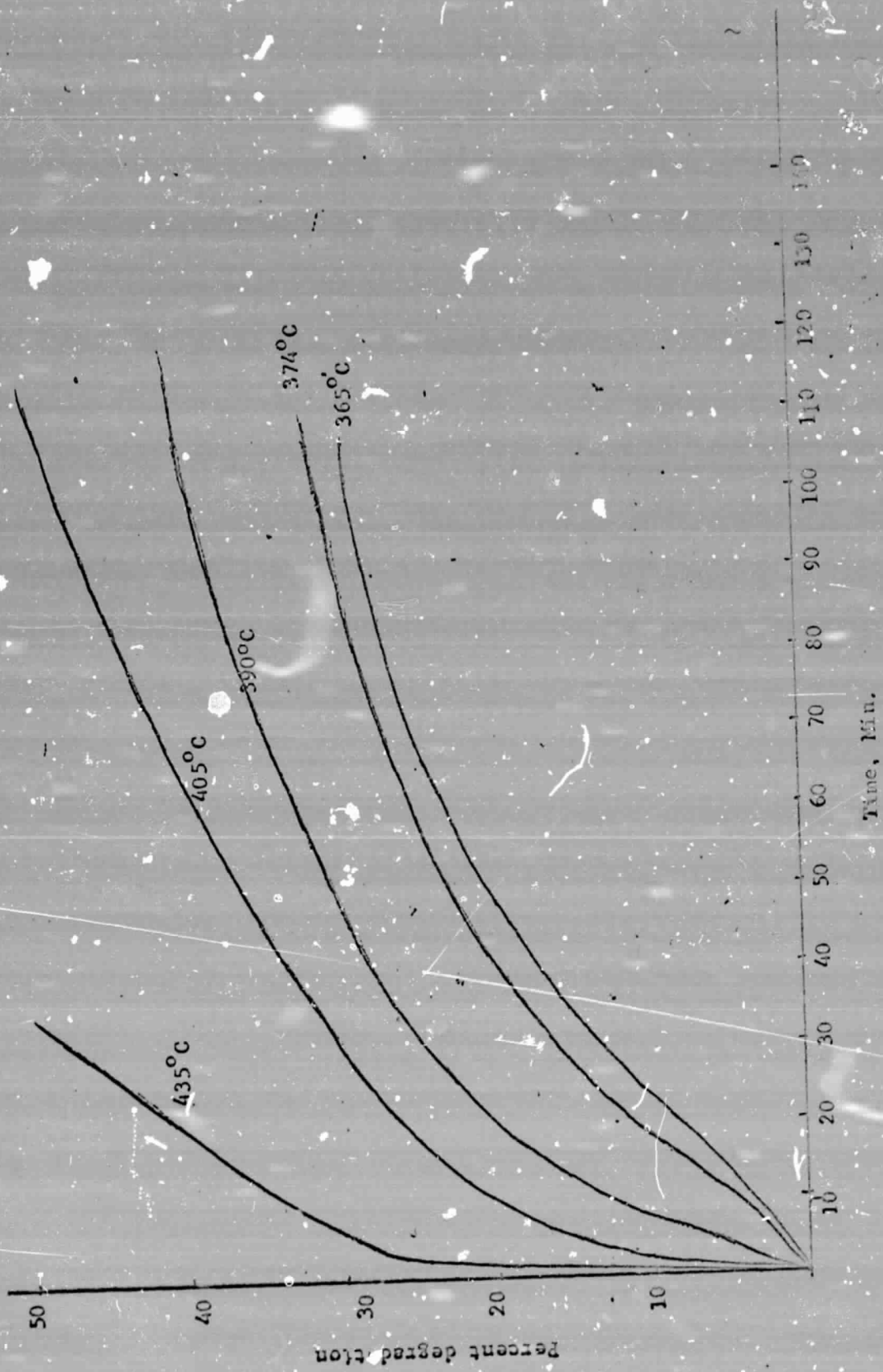


Fig. 3. Isothermal TGA of MAMD bismaleimide in air

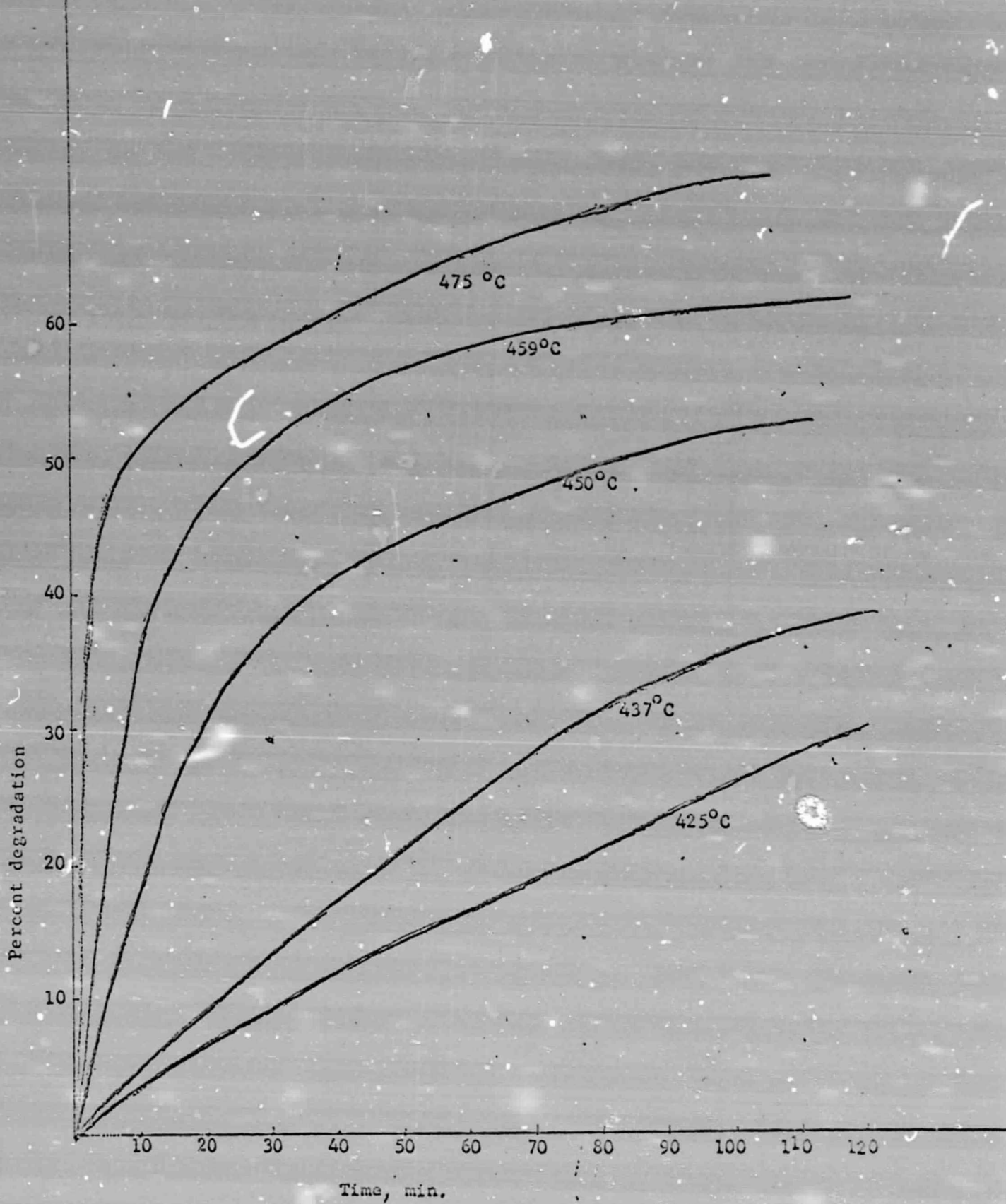


Fig. 4. Isothermal TGA of MAMD bismaleimide in nitrogen

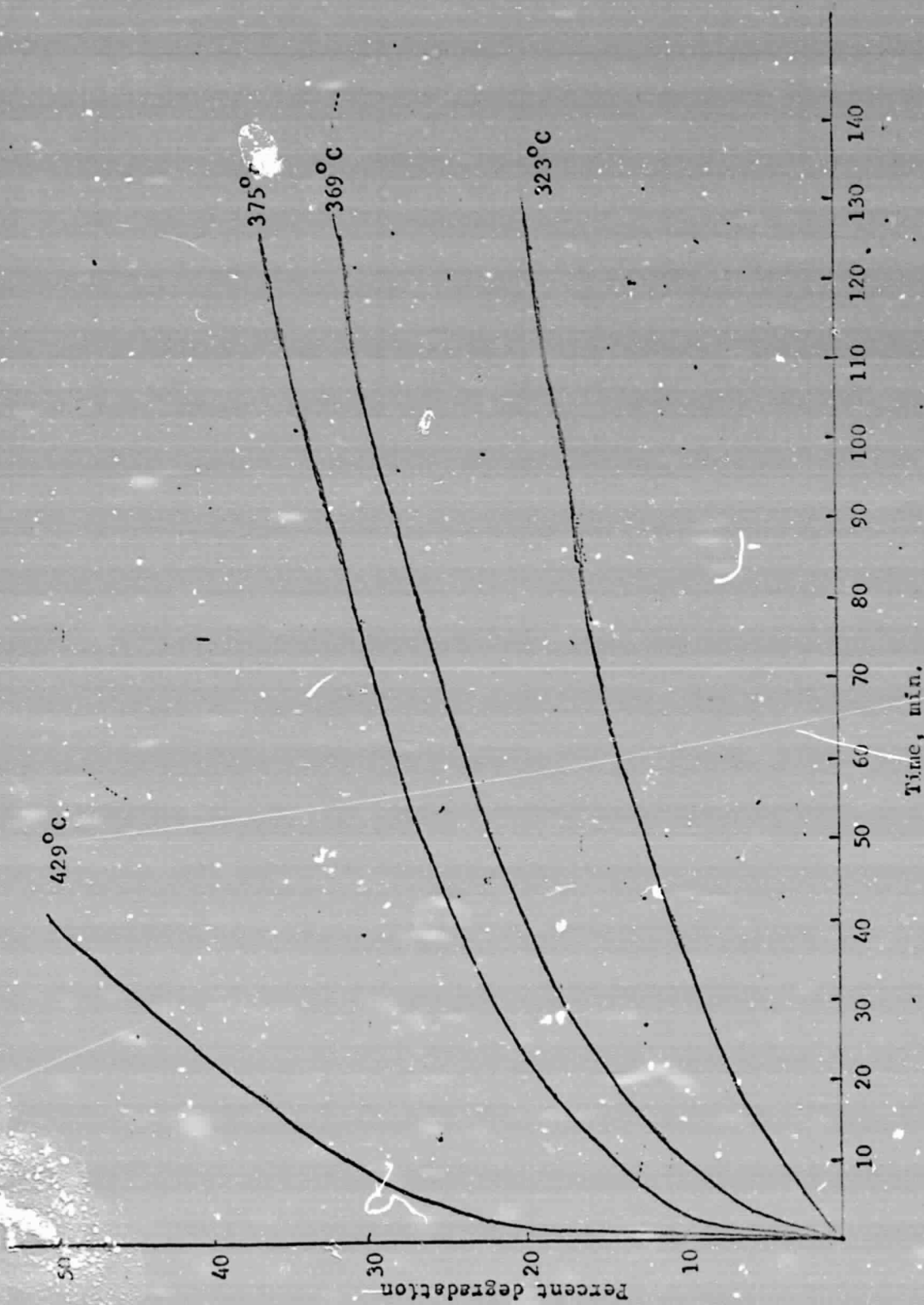


Fig. 5. Isothermal TGA of Kerimid 601 in air

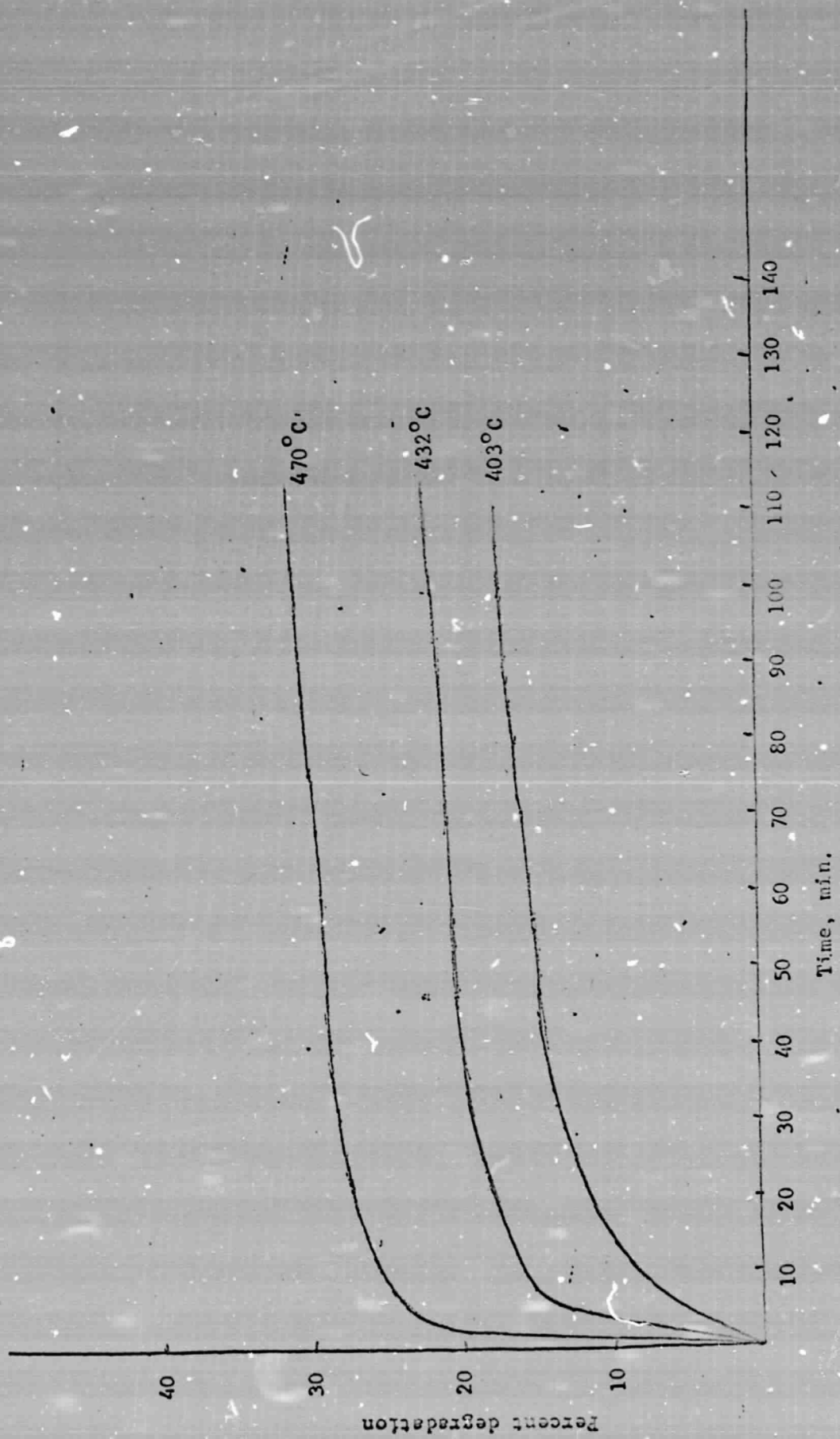


FIG. 6. Isothermal TGA of Kerimid 601 in nitrogen

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SAMPLE	S103	SPECTRUM	31	RETENTION TIME	2.4
LARGST 4:	106.0	100.0	107.1	8.8	108.1
LAST 4:	280.9	.6	341.0	1.7	355.0
			.3		

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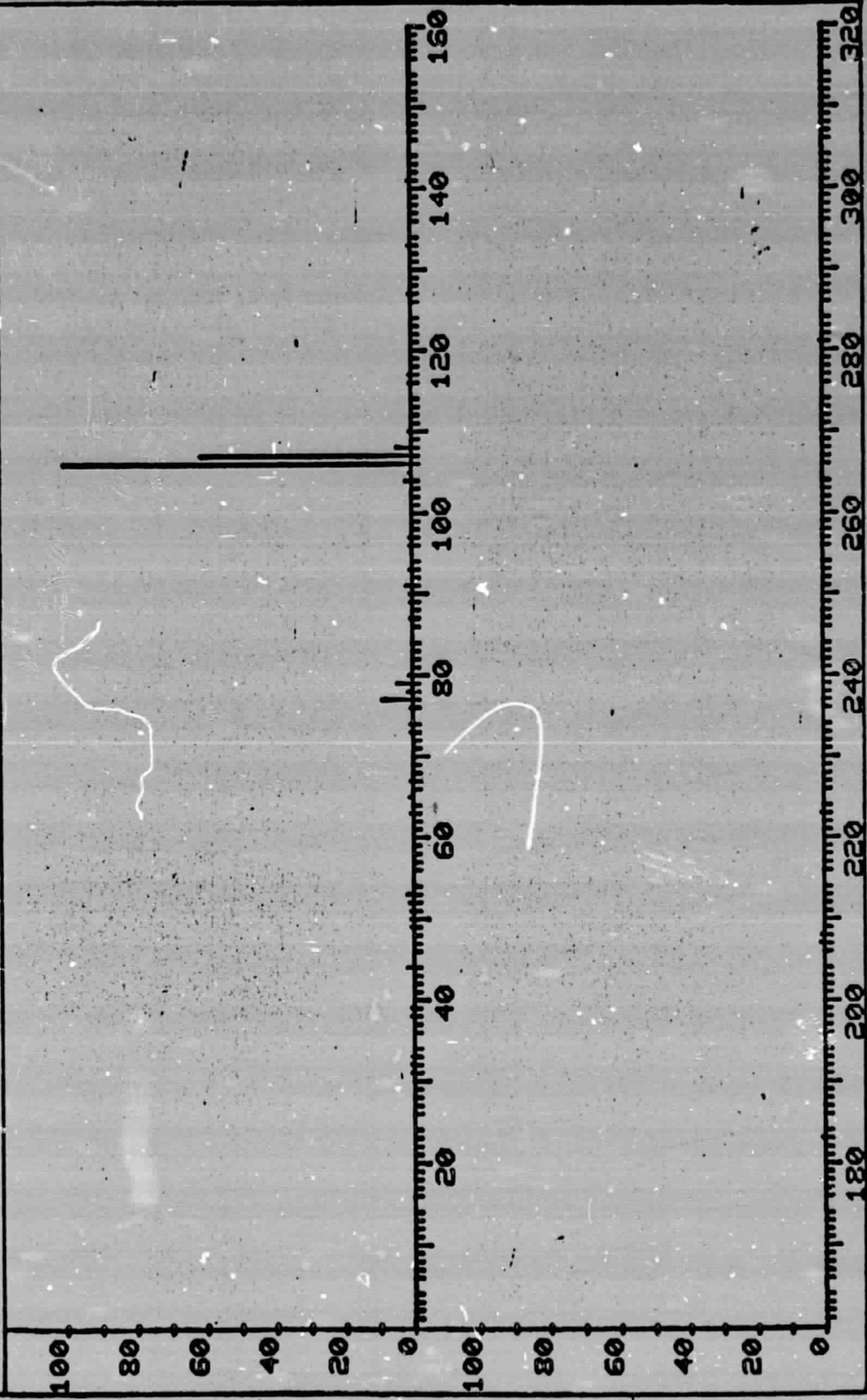


Fig. 8 Mass spectrum of methyl aniline which is one of the liquid products from pyrolysis of MAMD and Kerimid 601 bismaleimide in vacuum.

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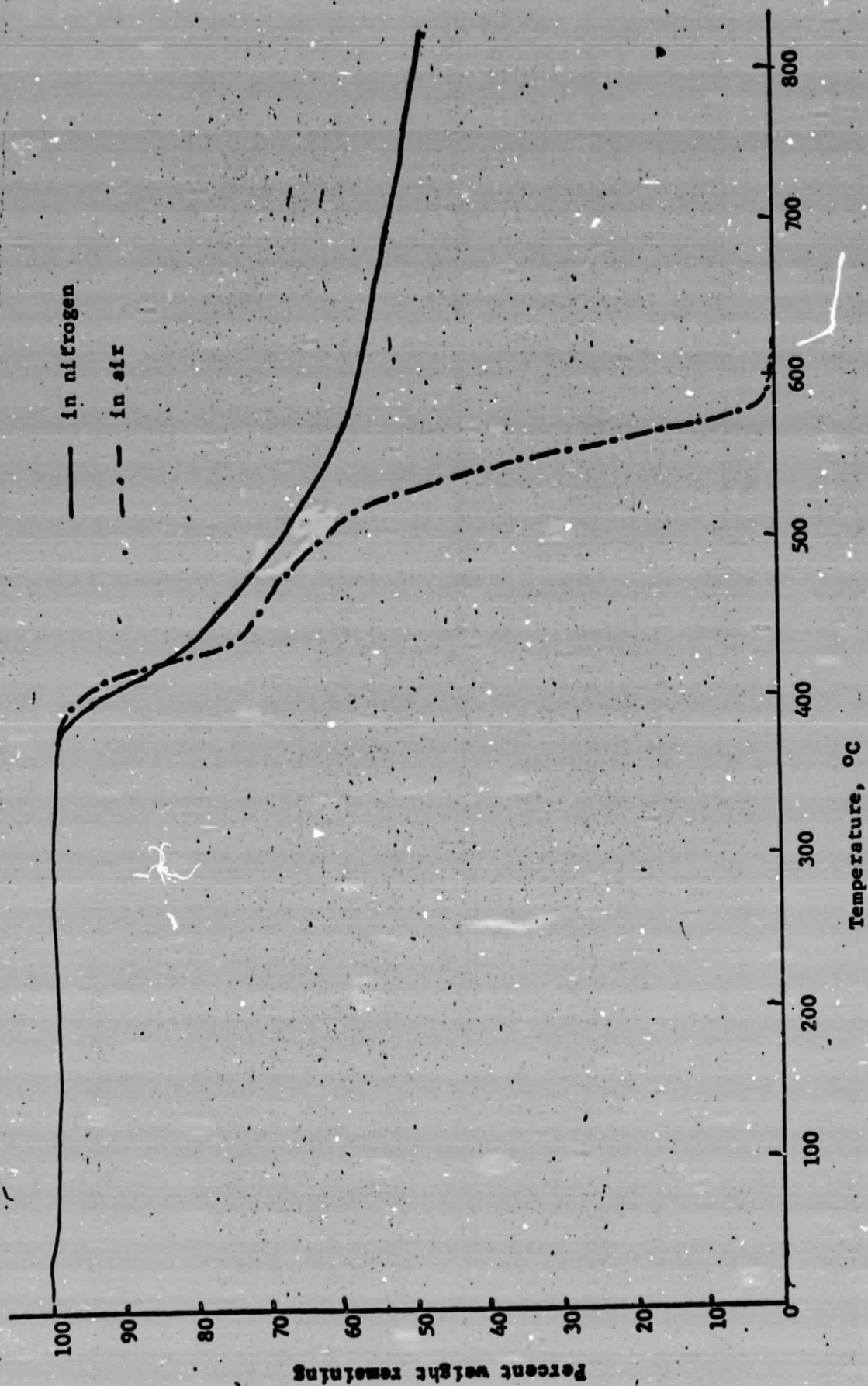


Fig. 9. Thermograms of phenolphthalein polycarbonate (heating rate of 15°C/min.)

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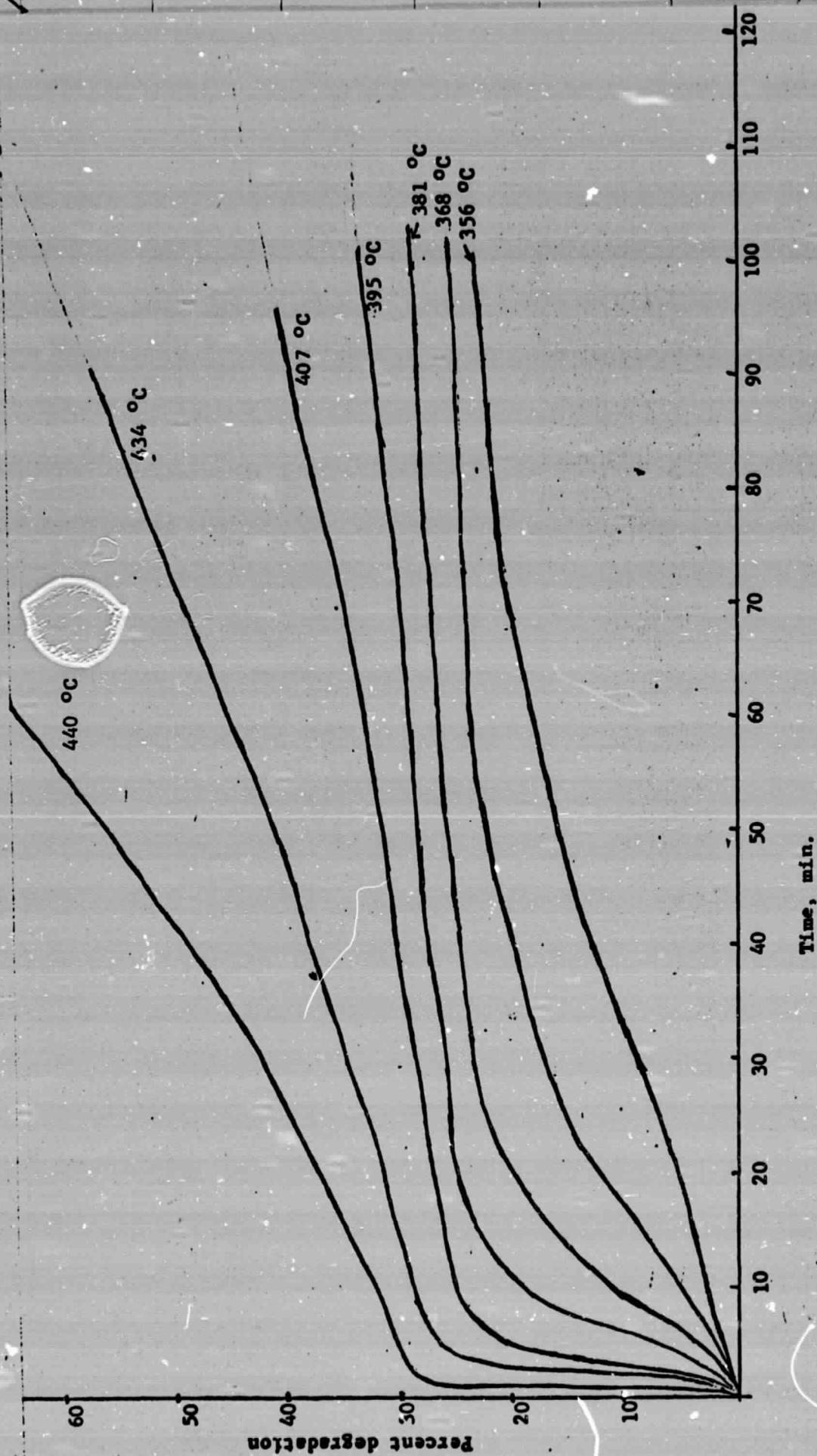


Fig. 10. Isothermal TGA of phenolphthalein polycarbonate in air

SAMPLE 5101
 LARGST 4: 223.9, 100.0
 LAST 4: 354.0, 8

SPECTRUM 144
 100.1, 26.9
 354.9, 8

RETENTION TIME 5.9
 168.0, 22.8
 406.1, 1.5

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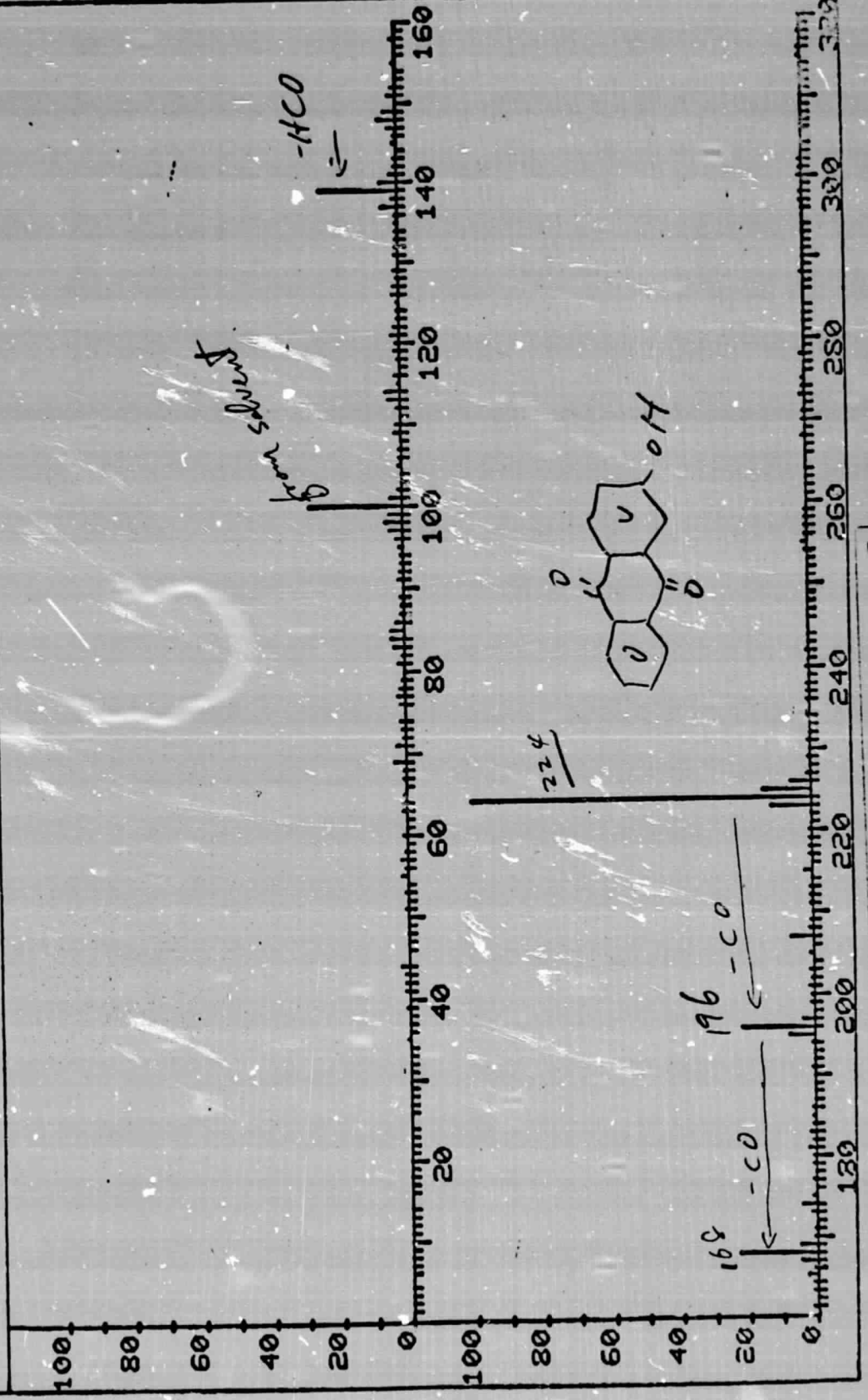


Fig. 11 Mass spectrum of 2-hydroxyanthraquinone which is the minor volatile solid product from pyrolysis of phenolphthalein polycarbonate in vacuum.

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Table 1. Elemental composition of MAMd and Kerimid 601 Bismaleimide resins and pyrolysis residues

Element	Percent composition							
	MAMd bismaleimide				Kerimid 601 bismaleimide			
	Theoretical	Found	Residue after 500°C pyrolysis in nitrogen	Theoretical (without R)	Prepolymer	Polymer	Residue after 550°C pyrolysis in nitrogen	Residue after 388°C pyrolysis in air
Carbon	70.4	70.16	83.17	72.08	71.73	70.58	86.75	69.97
Hydrogen	3.9	4.12	3.29	4.55	4.72	4.65	3.00	2.56
Nitrogen	7.8	7.67	7.76	9.09	8.76	8.79	8.03	11.80
Oxygen	17.9	18.05	5.78	14.28	14.79	15.98	2.22	15.67

Table II. Thermal degradation of bismaleimide resins

Polymer	Environment	Temp °C	Duration	Percent gases	Percent volatiles other than gases	Percent residue
Kerimid 601	Vacuum	460	30 min.	16 (CO ₂ , CO, CH ₄ , NH ₃ and trace HCN)	36 (11% clear liq. 25% brown materials)	48
Kerimid 601	Nitrogen	460	2 hr.	12.44	30.88	56.67
Kerimid 601	Nitrogen	550	30 min.	14	33	53
Kerimid 601	Air	388	5 hr.	-- (CO ₂ , CO, CH ₄)	48	52
MAMD	Vacuum	435	1.5 hr.	15.3	13 (7% clear liq. 6% brown oil)	71.7
MAMD	Vacuum	485	2 hr.	25 (CO ₂ , CO, CH ₄ , NH ₃ and trace HCN)	22	53
MAMD	Nitrogen	500	2 hr.	10	36.64	46.64
MAMD	Air	425	2.5 hr.	--	45.84	21.65

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Table III. Thermal Degradation of Phenolphthalein Polycarbonate in Vacuum

Temp °C	Duration	Intrinsic viscosity of sol.*	% gel.	% Weight loss
254	30 hr	0.28	3	<1
254	50 hr	0.308	24	2.5
274	3 hr	0.285	2.7	<1
274	9.5 hr	0.30	8	1
274	28 hr	0.205	36	2.9
274	50 hr	0.277	40	3.1
274	122 hr	0.213	41	3.1
283	4.5 hr	0.277	4.9	1.2
283	7.5 hr	0.269	34.8	3.6
420	1 hr	59.69% residue; 24.74% gases (CO ₂ , CO, CH ₄) and 15.57% volatile solids (most are phenol).		
210	1 wk	stable		

* intrinsic viscosity of phenolphthalein polycarbonate before pyrolysis is 0.342

Table IV

Thermogravimetric analysis of composites in nitrogen (heating rate = 10°C/min.)

Panel Identification	Percent weight remaining at various temperature									
	100°C	200°C	300°C	400°C	500°C	600°C	700°C	800°C	900°C	
Boeing No. 1 (1B)	98.7	98.2	96	86.5	71.8	65.5	59	54.5	52	
Boeing No. 2 (2B)	98.5	97.9	94.3	90.4	80.3	70.7	63	56.5	52	
Boeing No. 3 (3B)	98.5	97.6	96	93.3	83.8	75	68.5	64.2	62	
Boeing No. 4 (4B)	98.6	98	95.5	94.4	83.8	72.8	64.2	56.8	50.8	
Boeing No. 5 (5B)	98.5	97.8	94.7	91.3	81.5	71.5	63	55.5	49.3	
Boeing No. 6 (6B)	98.6	97.8	93.6	89.5	78	70.5	65.5	61.3	58	
#7 NASA-Ames Panel	95.5	93	87	83.5	79	73	66	58	47	
Boeing standard (#8)	98	97.8	96.5	89.5	77.5	69	61.5	56.5	53	
9B NASA-Ames Panel	98	97.5	97	96	91.5	82.2	73.5	68.5	65	

Table V
 Thermogravimetric analysis of composites in air (heating rate = 10°C/min.)

Panel Identification	Percent weight remaining at various temperature (°C)									
	100 C	250 C	350°C	400°C	450°C	500°C	550°C	600°C	700-900°C	
Boeing No. 1 (1B)	98	97.4	92	87.2	80	67.2	40	22.3	21.8	
Boeing No. 2 (2B)	98.3	97	93.5	89.7	81.5	70	47.4	22.5	20.5	
Boeing No. 3 (3B)	98.5	97.6	94	90.3	82.2	70.5	48.8	23.5	23	
Boeing No. 4 (4B)	98.5	97.6	94.5	89.5	79	63.5	43.8	22	21.5	
Boeing No. 5 (5B)	98.5	97.7	94	90.3	82	70	47	24	23	
Boeing NO. 6 (6B)	98.3	96.7	90.5	87	80	69.5	47	31	29	
#7 NASA-Ames Panel	95.5	89.7	86.3	84	80	74.5	68.5	59.2	39-25	
Boeing standard (#8)	98.5	98	92.5	88	80.3	66.2	45	32.4	31.5	
9B NASA-Ames Panel	96.5	96	95.7	94	86	53	25	25	24.3	

Table VI Thermogravimetric Analyses of Thermoplastic Materials in nitrogen (heating rate = 40°C/Min.)

Material	Percent weight remaining at various temperature (°C)									
	100	200	300	400	500	600	700	800	900	
#10 Carborundum Lac Code 30991-7T	100	99	99	99	98	62	49	47	46	
#11 Phillips Pet Lac Code 30991-6T	100	100	100	100	99	71	65	64	62	
#12 Polyether Sulfone 212-P	100	100	99	99	98	73	44	42	40	
#13 Polyether Sulfone 200-P	100	100	99	99	99	72	43	40	38	
#14 Polycarbonate	100	100	100	100	97	30	28	26	24	
#15 Modified Polysulfone	100	100	100	100	96	48	43	39	36	
#16 Modified Poly- phenylene Oxide (Noryl)	100	100	94	52	20	16	15	13	12	
#17 CPVC	100	100	94	37	33	29	28	26	24	
#18 Acrylonitrile Buta- diene Styrene (ABS)	100	100		51	21	17	16	14	13	

Table VI Cont.

Material	Percent weight remaining at various temperature (°C)									
	100	200	300	400	500	600	700	800	900	
#19 New L.S. Poly-carbonate-F-6000	100	100	100	100	99	25	23	21	21	
#20 Polyphenylene Sulphide	100	100	100	99	98	72	69	68	66	
#22 Polyethersulfone (ICI)	100	100	100	89	11	9	8	7	6	
#23 RPF Polycarbonate-Silicone block Copoly-er	99	99	99	99	81	54	40	16	16	
#24 Ryton PPS R-4 1/8	100	100	100	100	99	76	72	71	70	
#25 Chlorinated PVC	100	100	90	39	32	29	26	23	20	
#27 Fluorine bisphenol Polycarbonate (uncured)	100	100	100	100	86	68	58	56	55	
#30 Phenolphthalein 80% bisphenol A Copolymer	99	99	94	92	61	54	50	46	44	
#31 Phenolphthalein 70% bisphenol A Copolymer	100	100	97	95	63	48	43	40	38	

Table VI Cont.

Material	Percent weight remaining at various temperature (°C)									
	100 -	200	300	400	500	600	700	800	900	
#32 Polyvinyl fluoride film	100	100	100	91	12	10	9	7	5	
#33 2787-145B 5mil. PES 300-P, 181-38 fabric	99	99	99	99	99	85	72	70	68	
#34 Lockheed Kynol	97	96	94	92	87	76	69	65	63	
#35 Lockheed Lreuted Wool	97	94	84	46	37	34	31	27	21	
#36 Lockheed 90% Wool 10% Nylon	99	99	96	94	10	8	5	4	3	
#37 Lockheed Nomex	96	93	92	89	78	65	60	54	50	
#38 Lockheed Fire Retardant Nylon	98	98	95	92	11	5	3	2	2	

Table VII Thermogravimetric Analyses of Thermoplastic Materials in air (heating rate = 40°C/Min.)

Material	Percent weight remaining at various temperature (°C)									
	100	200	300	400	500	600	700	800	900	
#10 Carborundum Lac Code 30991-7T	100	99	99	99	98	62	29	4	3	
#11 Philips Pet Lac Code 30991-6T	100	100	100	100	99	76	65	38	26	
#12 Polyether Sulfone 212-P	100	100	99	99	98	71	34	6	3	
#13 Polyether Sulfone 200-P	100	100	99	99	99	64	33	7	2	
#14 Polycarbonate	100	100	100	100	94	31	3	3	3	
#15 Modified Polysulfone	100	100	100	100	94	50	9	8	8	
#16 Modified Poly- phenylene Oxide (Noryl)	100	100	94	55	33	22	12	12	12	
#17 CPVC	100	100	94	37	35	25	6	6	6	
#18 Acrylonitrile Buta- diene Styrene (ABS)	100	100	92	51	27	11	6	6	6	

Table VII Cont.

Material	Percent weight remaining at various temperature (°C)									
	100	200	300	400	500	600	700	800	900	
#19 New L.S. Polycarbonate-F-6000	100	100	100	100	92	17	5	5	5	900
#20 Polyphenylene Sulphide	100	100	100	99	98	83	63	42	42	
#22 Polyethersulfone (ICI)	100	100	100	89	29	0	0	0	0	
#23 BPF Polycarbonate-Silicone block Copolymer	99	99	99	99	81	74	41	16	16	
#24 Ryton PPS R-4 1/8	100	100	100	100	99	79	70	43	42	
#25 Chlorinated PVC	100	100	90	90	37	31	10	7	7	
#27 Fluorine bisphenol Polycarbonate (uncured)	100	100	100	100	84	76	54	19	18	
#30 Phenolphthalein 80% bisphenol A Copolymer	100	99	94	88	58	46	4	0	0	
#31 Phenolphthalein 70% bisphenol A Copolymer	100	99	96	95	53	40	10	1	1	

Table VII Cont.

Material	Percent weight remaining at various temperature (°C)									
	100	200	300	400	500	600	700	800	900	
#32 Polyvinyl fluoride film	100	99	99	88	22	7	3	3	3	
#33 2787-145B 5mil PES 300-P, 181-58 fabric	100	100	100	100	99	81	66	50	50	
#34 Lockheed Kynol	97	96	95	85	26	6	2	2	2	
#35 Lockheed Larcuted Wool	96	93	79	49	39	16	9	6	5	
#36 Lockheed 90% Wool 10% Nylon	99	99	96	93	16	3	3	3	3	
#37 Lockheed Nomex	96	93	92	90	78	47	16	1	1	
#38 Lockheed Fire Retardant Nylon	99	99	96	96	11	4	1	1	1	