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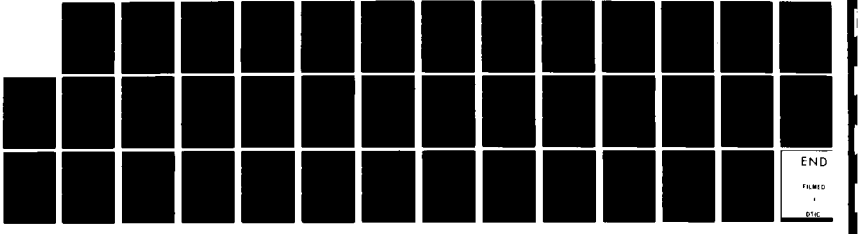

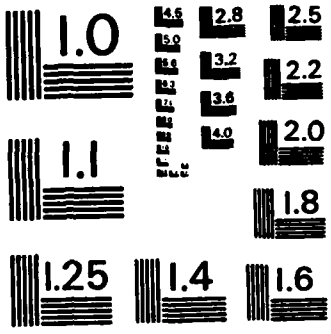

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MASS SPECTROMETRIC THERMAL DECOMPOSITION AND ULTRAVIOLET IRRADIATION STUDIES OF SOME AZIDO AND NITRATO POLYMERIC BINDERS

Milton Farber, S. P. Harris and R. S. Srivastava

ANNUAL SUMMARY REPORT

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azido oxetane (AZOX)
bis nitrato methyl oxetane (DNAO)
copolymers
thermal decomposition
activation energies
mass spectrometry
ultraviolet irradiation

20. ABSTRACT *(Continue on reverse side if necessary and identify by block number)*

Thermal decomposition studies were conducted on AZOX and AMMO homopolymers and on 50-50% BAMO-AZOX and 50-50% BAMO-AMMO copolymers. Decomposition was initiated at approximately 120 C, with activation energies for the materials approximately 170 kJ mol^{-1}. The primary mechanism for the decomposition is the release of molecular N_2. Backbone decomposition occurs at temperatures above 150 C

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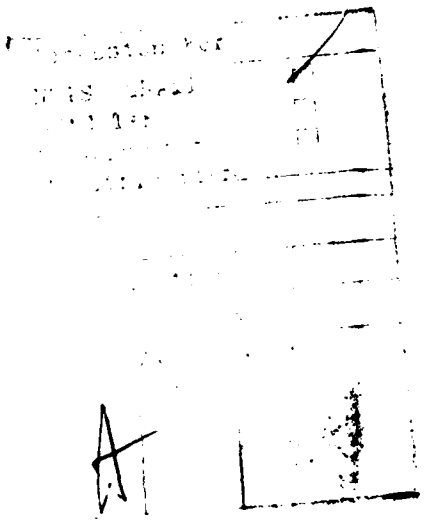
with thermal destruction of the three-carbon chain. A thermal decomposition study of a new copolymer consisting of an azido (BAMO) and nitrato ester, bis nitrato methyl oxetane (DNAO) showed both azide and nitrato group decomposition at differing rates. Preliminary mass spectrometric studies of the decomposition of a new amino nitro ring compound, $C_6H_8N_8O_{12}$, indicated NO_2 release with ring stability. Qualitative UV irradiation experiments at 2540 Å and 3660 Å wave lengths then were conducted on AZOX and AMMO homopolymers and 50-50% BAMO-AMMO and 50-50% BAMO-AZOX copolymers. Upwards of 25% of the polymeric N_2 was released with considerable cross-linking. The samples changed from viscous liquids to gummy, rubbery semi-solids. The irradiated samples were maintained for a number of weeks under ordinary atmospheric conditions as well as under vacuum without any indication of autocatalysis.



I. INTRODUCTION

Thermal decomposition studies on azido polymers were continued during the past twelve months. Also, the thermal decomposition of a newly prepared energetic nitro amine ring compound from the Naval Surface Weapons Center, White Oak, was investigated. The azido materials studied included BAMO, AMMO, AZOX and DNAO polymers and copolymers supplied by SRI International.

The experimental program consisted of two phases: the kinetic effusion-mass spectrometric studies on the polymeric materials and the new amino nitro ring compound, and qualitative ultraviolet decomposition measurements on the azido polymers.

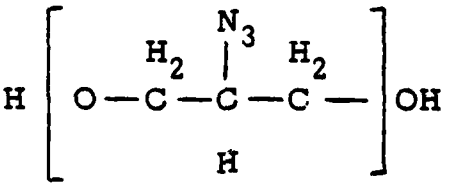
The Annual Summary Report dated September 1981 presented the results of thermal decomposition studies on the homopolymers of BAMO and GAP and monomeric AZOX.

II. MASS SPECTROMETER STUDIES

A. Thermal Decomposition of Azido Polymers

1. AZOX

In order to obtain reliable thermal decomposition kinetics it was imperative that the polymeric samples be completely pure or that impurities such as residual solvent components be removed by low temperature heating in vacuum prior to the kinetic studies. Upon the onset of effusion-mass spectrometric investigation of the AZOX homopolymer



at low temperature a large concentration of ion peaks were observed in the mass spectra. For example, Figure 1 shows these solvent products in the 27 to 32 amu range at 60 C, which nearly disappear at 95 C. Similarly, Figure 2 shows these products in the 38 to 45 amu range at 80 C, with their virtual disappearance when the temperature has reached 100 C. These

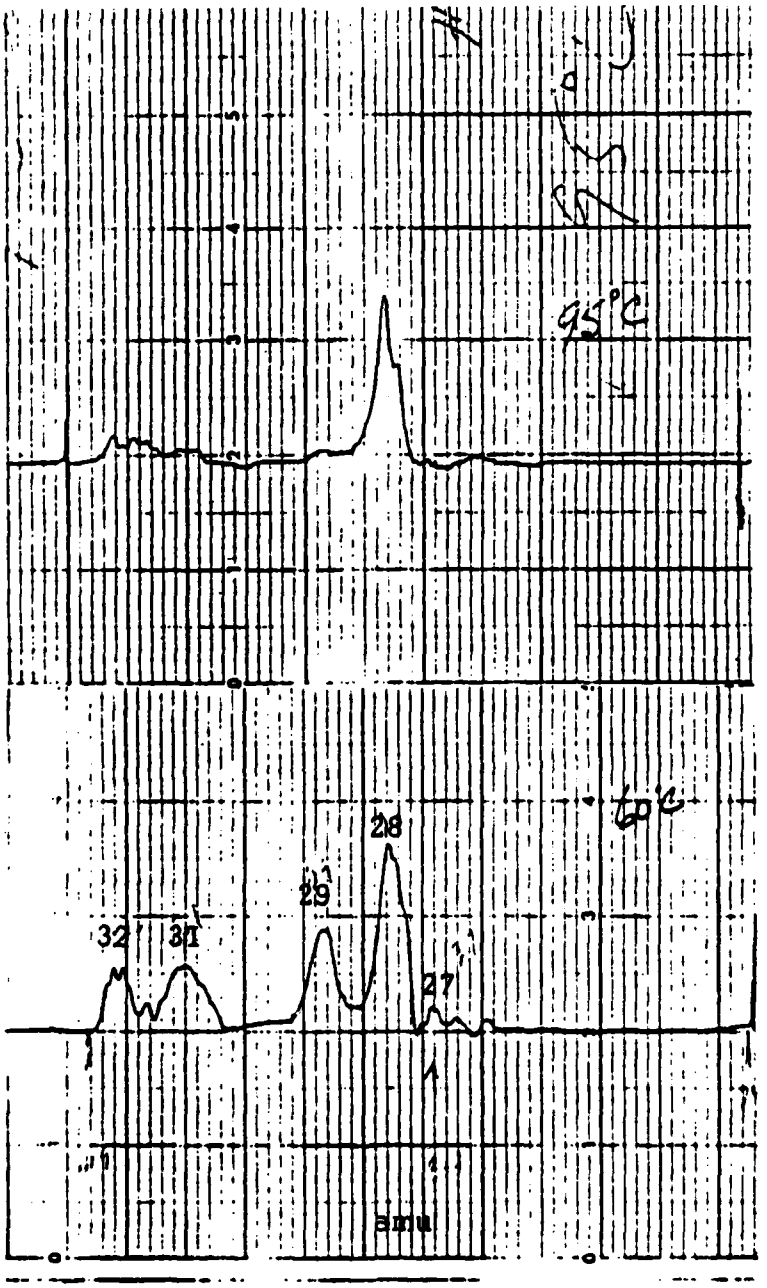


Fig. 1. Solvent products recorded by the mass spectrometer while studying the thermal decomposition of AZOX homopolymer. These products are seen at the lower temperatures, approximately 60 C, and disappear or are greatly reduced as the temperature reaches 95 C.

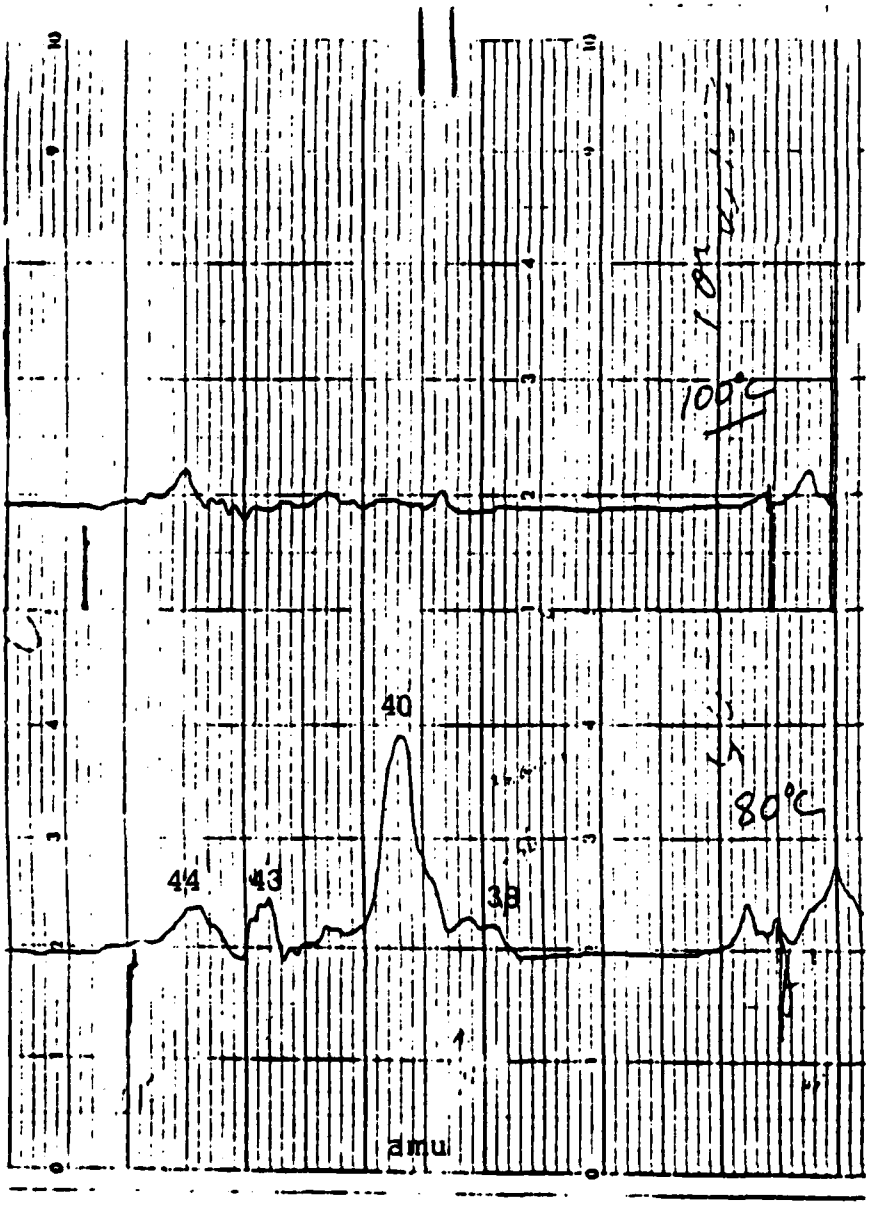


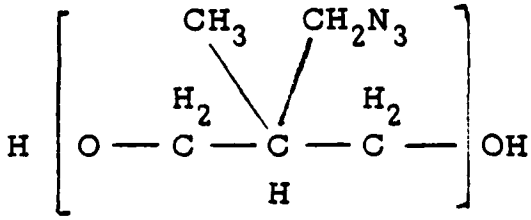
Fig. 2. Solvent products recorded by the mass spectrometer while studying the thermal decomposition of AZOX homopolymer. Although the products appear at the lower temperatures, approximately 80 C, they have disappeared at 100 C.

temperatures are below the temperature of the onset of thermal decomposition of the AZOX polymer.

After heating the AZOX homopolymer samples until the mass spectra indicated no further impurities the kinetic studies were commenced. As in previous azido polymers, molecular N_2 was the first thermal decomposition product occurring at approximately 120 C. Figure 3 shows the mass spectra of the thermal decomposition from 160 to 195 C. In this 35-degree range the N_2 intensity increased by a factor of 20. In Figure 3 the amu range is 24 to 32. The central peaks are molecular nitrogen, with smaller peaks on each side of HCN at 27 amu and HCO at 29 amu. These graphs are composites of the individual peak heights and are not continuous as a function of temperature. By plotting the log of the N_2 intensity against the reciprocal of the absolute temperature (Fig. 4) an activation energy, E_a, of 167.8 kJ mol^{-1} (40.1 kcal/mole) is obtained. As the temperature is raised the three-membered carbon backbone of AZOX begins to disintegrate with the ion spectra showing the species CH_2, CH_3, OH, and H_2O in the low mass range. The higher amu range shows peaks attributed to CO, CH_2OH, C_2OH, and CO_2 at 230 C (Fig. 5). A small ion intensity has been attributed previously to HN_3 at amu 43.

2. AMMO

The thermal decomposition of a relatively new azide polymer, AMMO, (azido methyl methyl oxetane)



was initiated. This polymer is similar to BAMO (the bis azido methyl oxetane) except that it has one azido methyl group on the center carbon of the three-carbon backbone, whereas BAMO has two.

AMMO was found to have a somewhat higher stability than the other azide polymers. A study of N_2 evolution, which is the

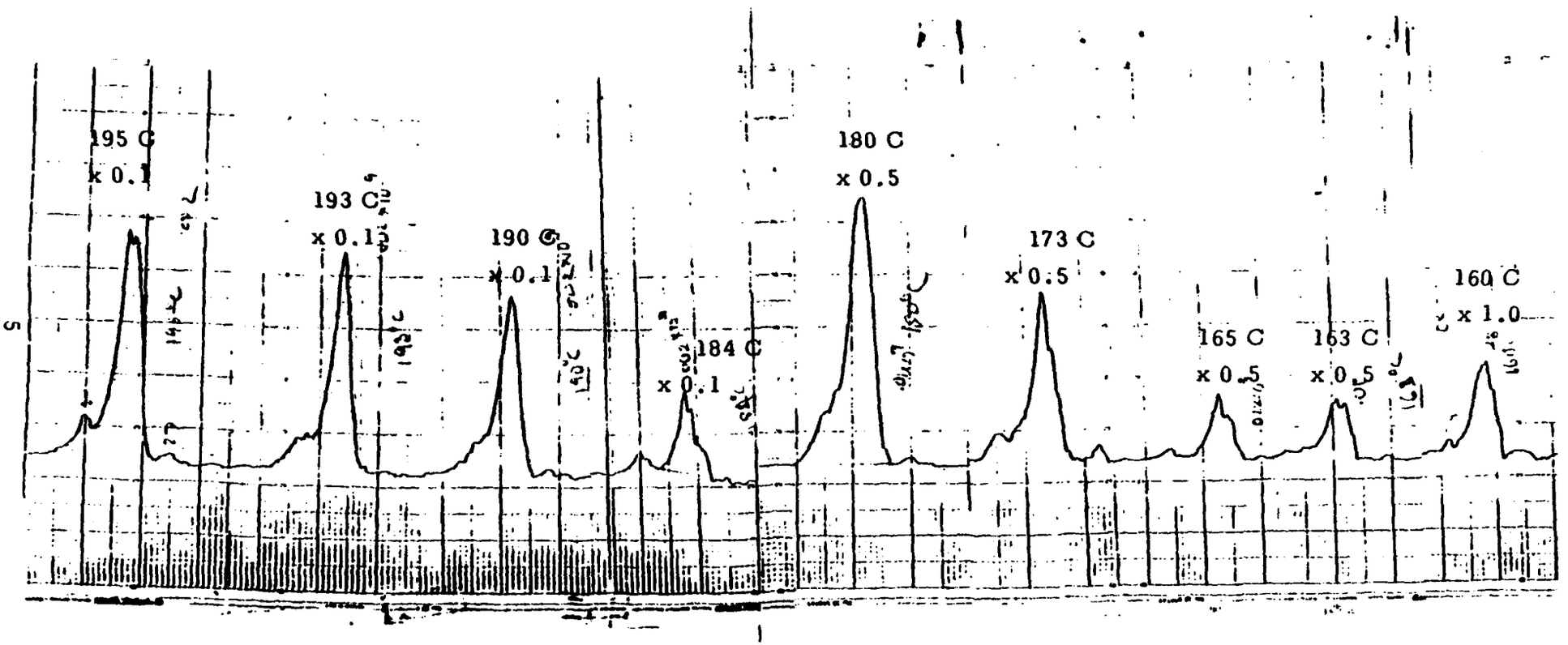


Fig. 3. Decomposition of AZOX monomer as a function of temperature.

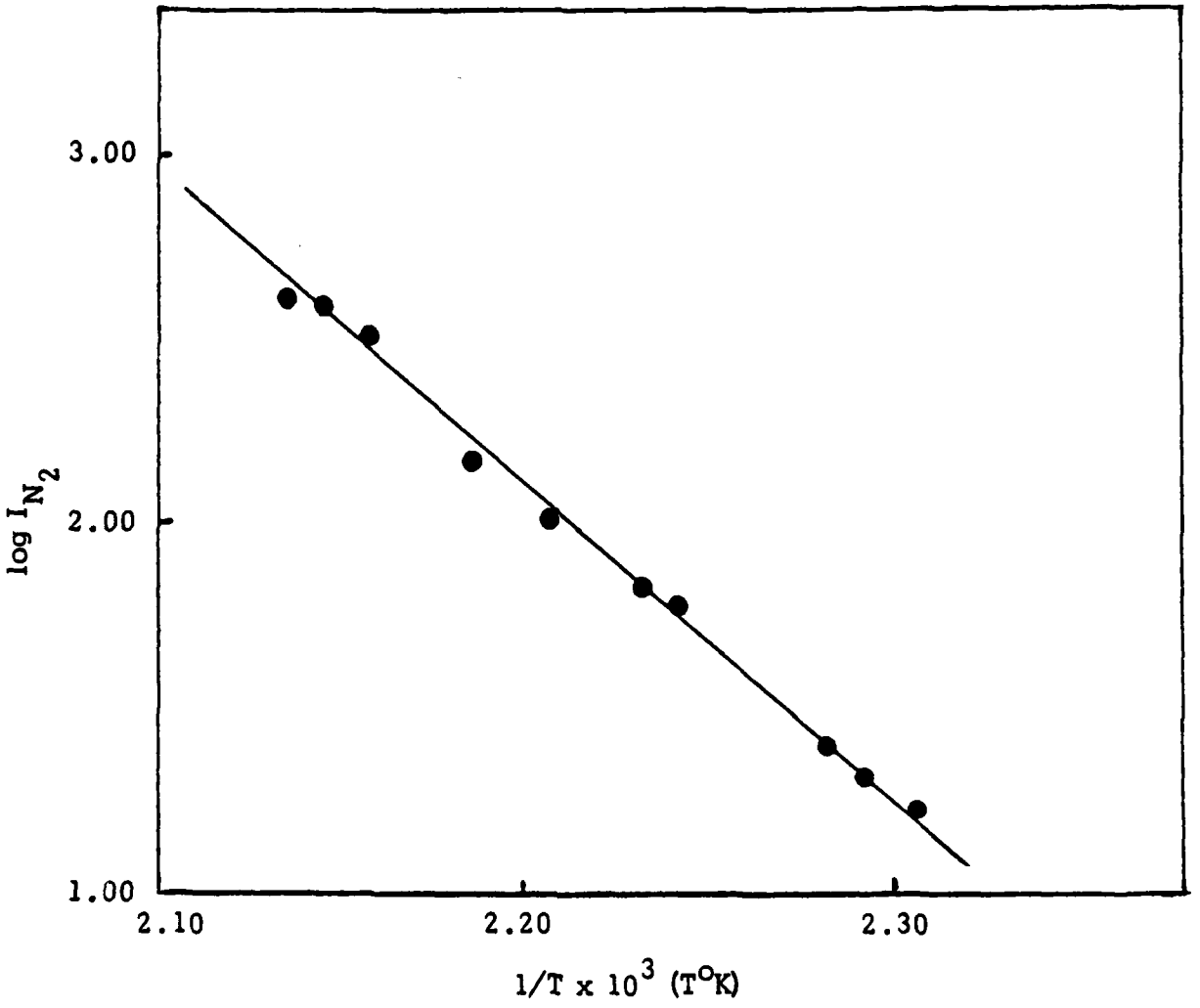


Fig. 4. Log of the relative intensity of N_2 from the thermal decomposition of AZOX plotted as a function of the reciprocal of the absolute temperature ($1/T^oK$). Activation energy = 167.8 kJ mol^{-1} (40.1 kcal/mole)

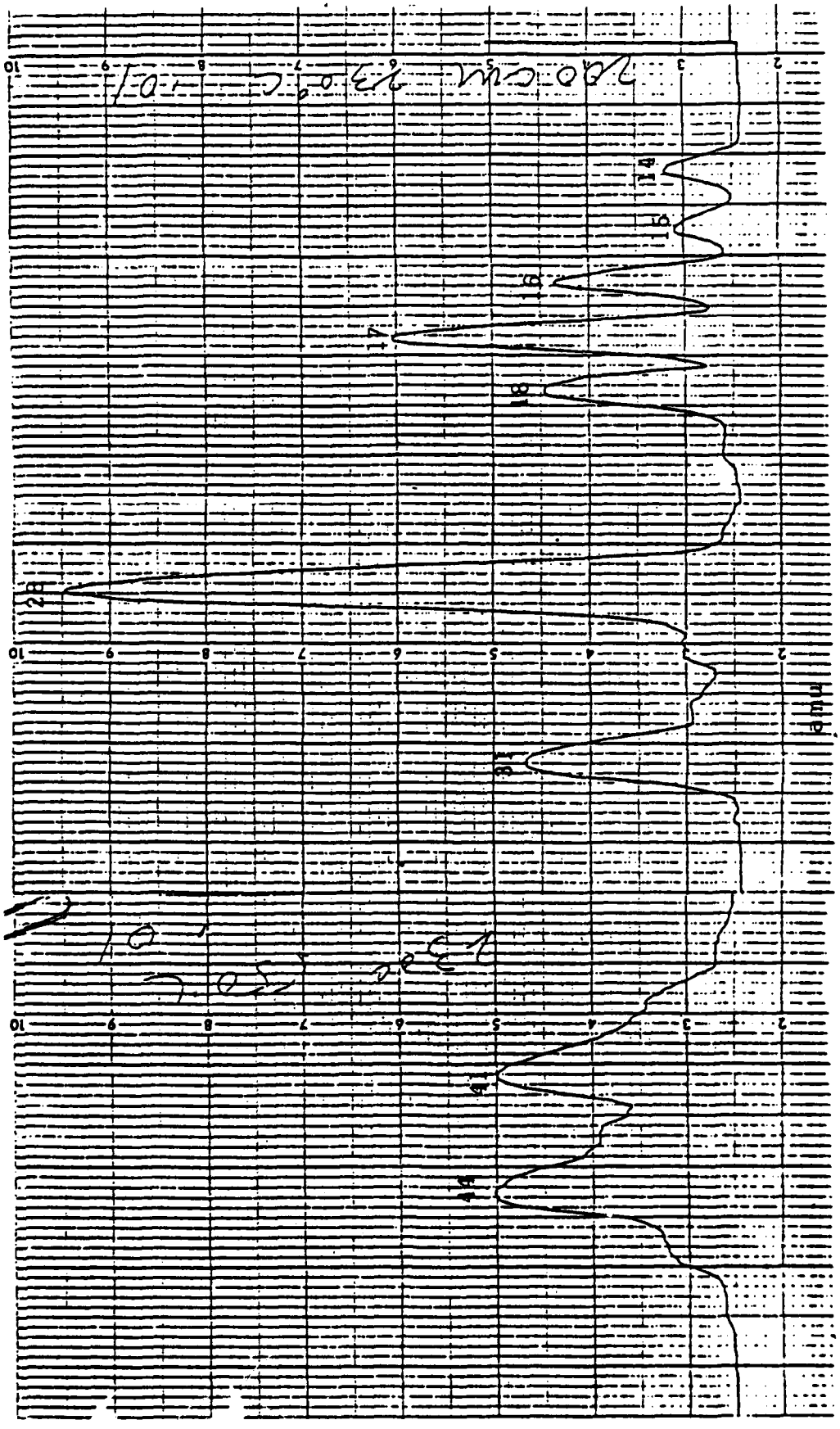


Fig. 5. Backbone decomposition of AZOX at 230 C

onset of thermal decomposition, shows that it continues until the destruction of the azide group is completed. An activation energy of 182 kJ mol^{-1} (43.6 kcal/mole) was calculated from these data. All the azido polymers, including their monomers, homopolymers and copolymers, were found to have activation energies in the range of 165 to 182 kJ mol^{-1} (40 to 43 kcal/mole). These results, based on the number of azide materials investigated, should be conclusive evidence that the polymers, copolymers, or monomers decompose primarily by the fracturing of the azide N—N bond, yielding an activation energy of approximately 164 kJ mol^{-1} (40 kcal/mole).

The thermal decomposition of AMMO was observed from 120 to 300 C. Its degree of stability can be seen in Figure 6, which shows very little backbone decomposition at 210 C. At 290 C (Fig. 7) a 30 amu peak corresponding to CH_2O indicates backbone decomposition. The relative OH and H_2O concentrations increase significantly at 290 C, as seen in Figure 7. Also, some methyl radicals at amu 15 were observed.

A qualitative indication of the relative thermal stability of BAMO and AMMO at 235 can be seen from Fig. 8.a. Considerably more H_2O is seen from the BAMO decomposition (Part B, Fig. 8.a.), indicating the release of greater quantities of OH recombining within the effusion cell to form water. Also, the relative amount of CH_3 radicals is larger from BAMO decomposition than from AMMO. As the temperature increased from 215 to 235 C (Fig. 8.b.) the thermal decomposition rate of BAMO increased rapidly, creating a fairly high cell pressure causing OH recombination to form H_2O. This does not occur with AMMO; even at temperatures as high as 290 C the OH/H_2O ratio of AMMO appears fairly constant.

High temperature decomposition products of BAMO (Fig. 9) include mass peaks at 40, 42, 43 and 44 amu. Within the effusion cell, where numerous collisions between gaseous species and the cell walls and also with the condensed polymeric materials can occur, the probability is high that some of the products observed mass spectroscopically are produced within the effusion cell itself and are not original decomposition products from the condensed phase. The three-membered carbon backbone of BAMO is definitely beginning to disintegrate at 230 C, as seen in Figure 10, with the production of CH_2O and CH_2OH at amu values of 30 and 31. The

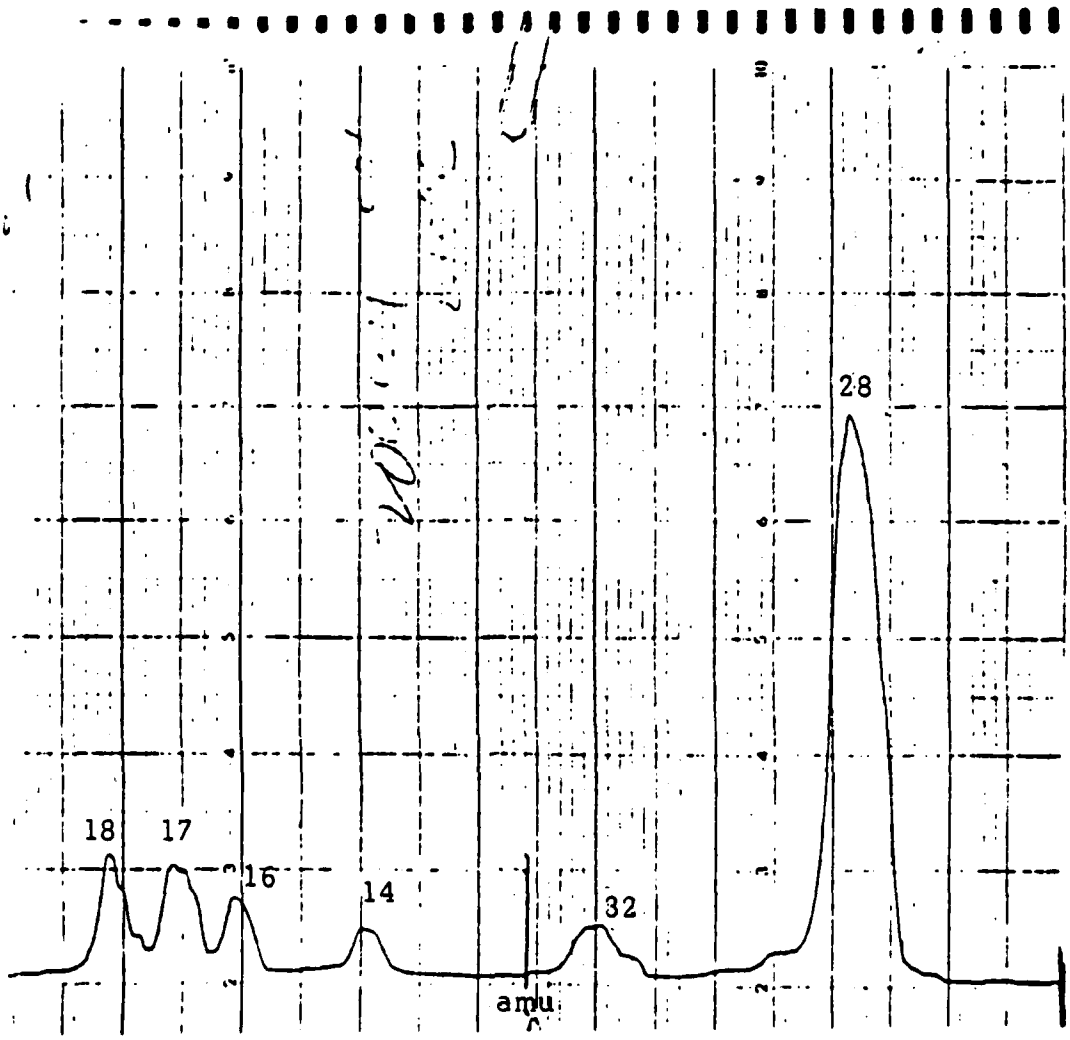


Fig. 6. Thermal decomposition of AMMO at 210 C. The azide group is decomposing, with the release of N_2 and some end group decomposition taking place; OH and H_2O peaks are observed at amu 17 and 18.

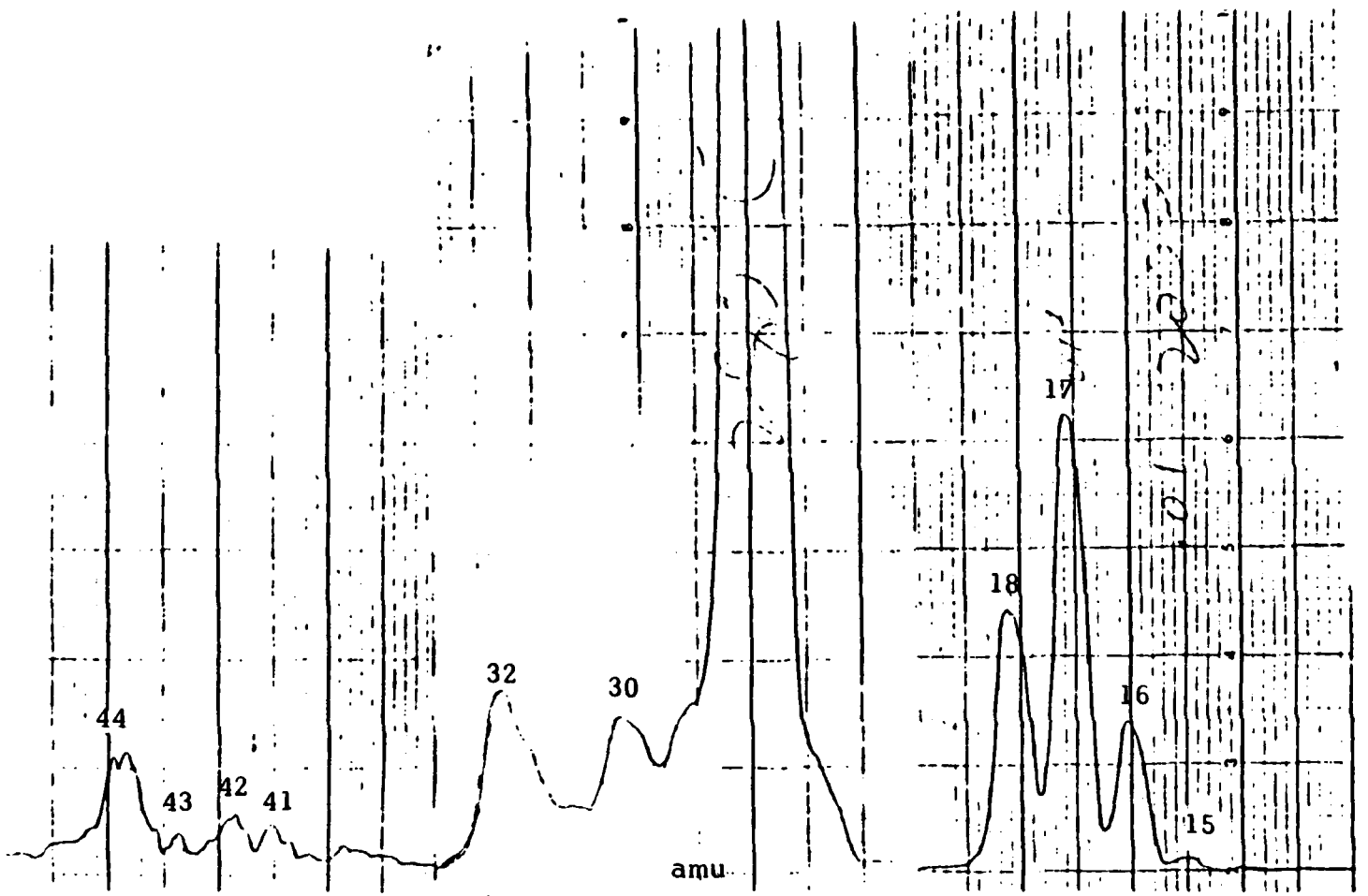
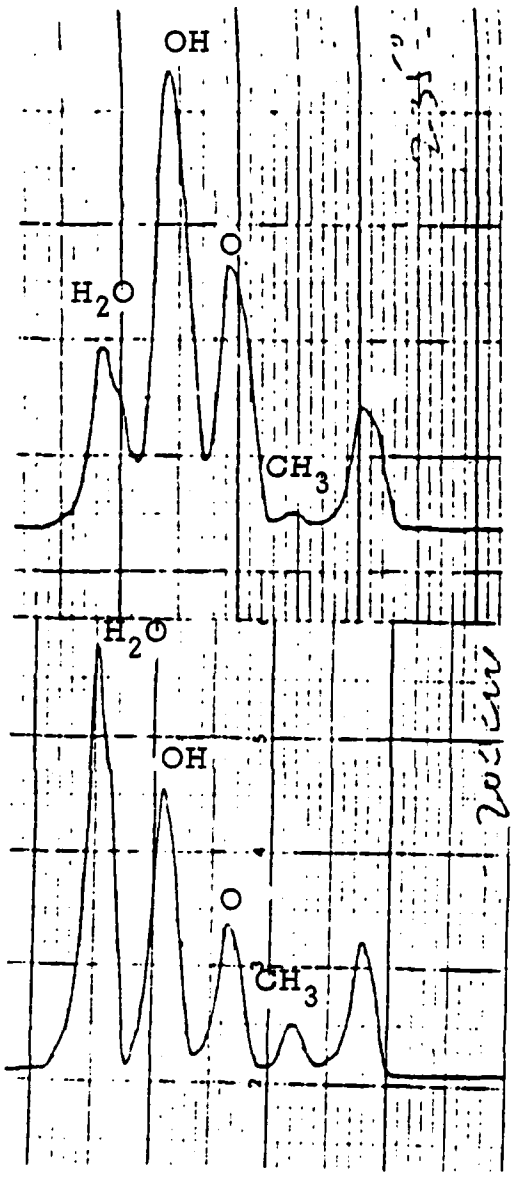


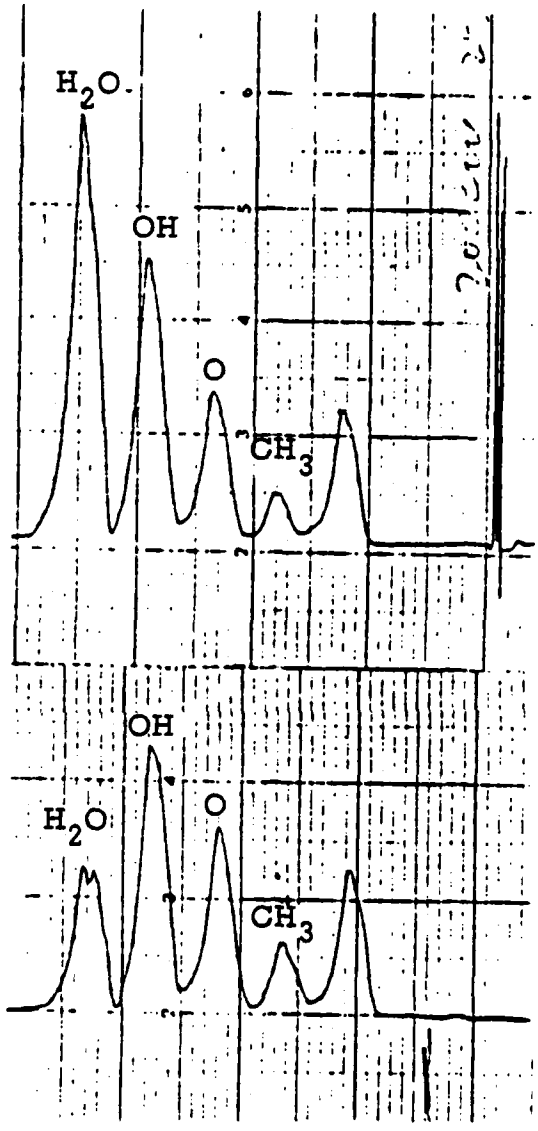
Fig. 7. AMMO products at 290 C in three amu ranges showing disintegration of the backbone



Part A

Part B

Fig. 8.a. Thermal decomposition comparison of AMMO (Part A) and BAMO (Part B) showing relative peaks of OH, H_2O and CH_3



Part A

Part B

Fig. 8.b. Thermal decomposition comparison of BAMO at 235 C (Part A) and 215 C (Part B) showing the reversal of the relative concentration of OH and H_2O at 215 and 235 C

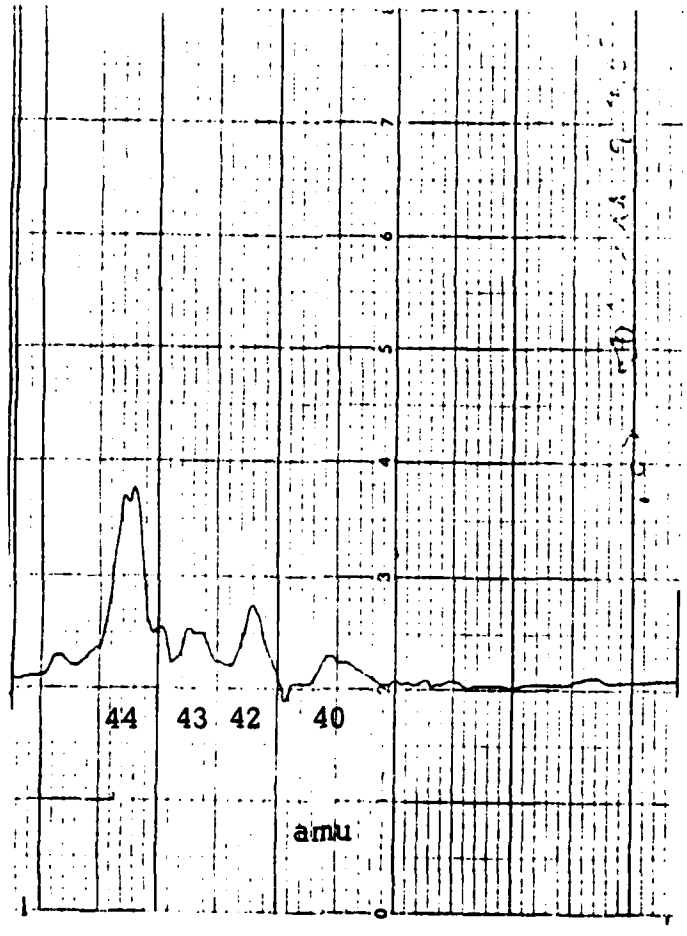


Fig. 9. BAMO products at 230 C in the 40 amu range

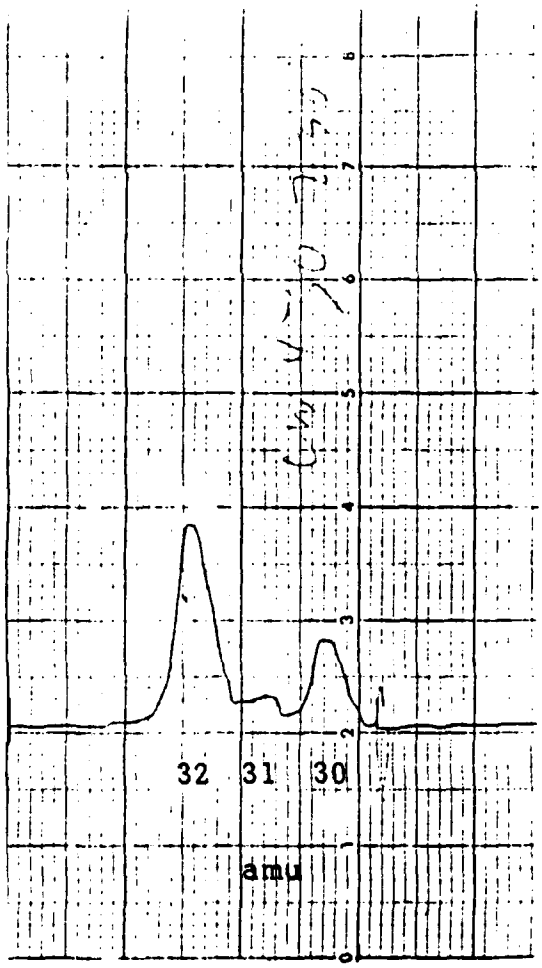


Fig. 10. Backbone disintegration of BAMO at 230 C, with production of CH_2O and CH_2OH at amu 30 and 31

relative concentrations of these species at 235 C compared with the azide N_2 evolution are shown in Figure 11.

B. Thermal Decomposition of Azido Copolymers

The thermal decomposition of several copolymers was investigated, including BAMO-AZOX, BAMO-AMMO, and BAMO-DNAO.

1. 50-50% BAMO-AZOX

Figure 12 shows the temperature dependence as measured by the N_2^+ intensity of the rate of nitrogen evolution resulting from the splitting of the $N-N_2$ bond of the azide group in the 50-50% BAMO-AZOX copolymer. The recording of these intensities (Fig. 12) shows a smooth increase in the rate of N_2 evolution as a function of temperature from 185 to 205 C. A semilog plot of these corrected nitrogen intensities versus the reciprocal of the absolute temperature yielded an activation energy of 176 kJ mol^{-1} (42.1 kcal/mole) for the copolymer (Fig. 13).

In addition to the release of nitrogen from the decomposition of the azide group, the 50-50% BAMO-AZOX copolymer shows decomposition of the polymeric backbone, as can be seen in Figure 14. This figure shows decomposition products with amu values of 27, 29, 30, 31, and 32 at 150 and 160 C. The probable species having these amu values are HCN, HCO, H_2CO and H_3CO, as well as molecular oxygen.

2. 50-50% BAMO-AMMO

The 50-50% BAMO-AMMO copolymer yielded a smooth nitrogen rate variation as a function of temperature. Figure 15 depicts the relative nitrogen intensities from 175 to 210 C. The corrected N_2 values when plotted against the reciprocal of the absolute temperature (Fig. 16) yielded an activation energy of 178 kJ mol^{-1} (42.5 kcal/mole) for the BAMO-AMMO copolymer. These results indicate that the thermal decomposition of the copolymers follows the same general mechanistic pattern as that of the homopolymers; that is, the splitting of the azide bond is the key factor in the mechanism for the thermal decomposition of the copolymers and the hompolymers.

3. 50-50% BAMO-DNAO

A new copolymer was recently prepared at SRI International involving BAMO with a nitrato ester, bis (nitrato methyl) oxetane, DNAO,

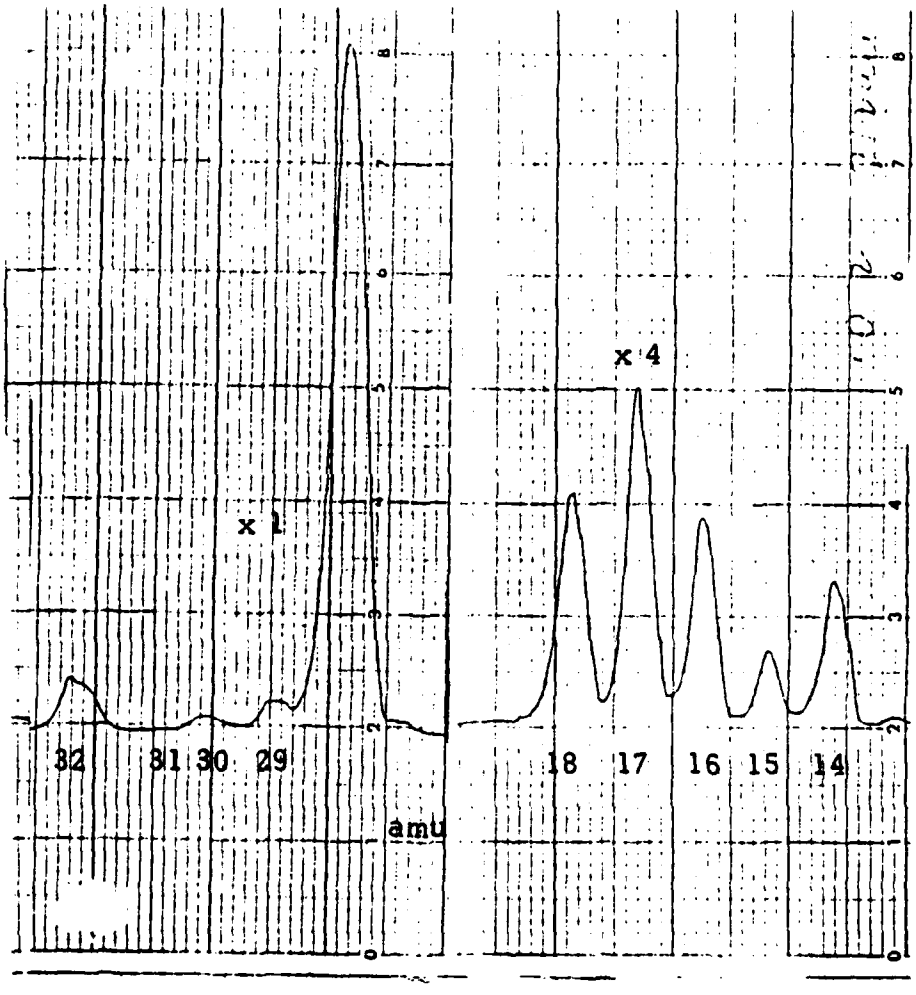


Fig. 11. Relative intensities of BAMO products at 235 C. Species 14 - 18 intensities are magnified by a factor of 4 over that of the N_2 intensities.

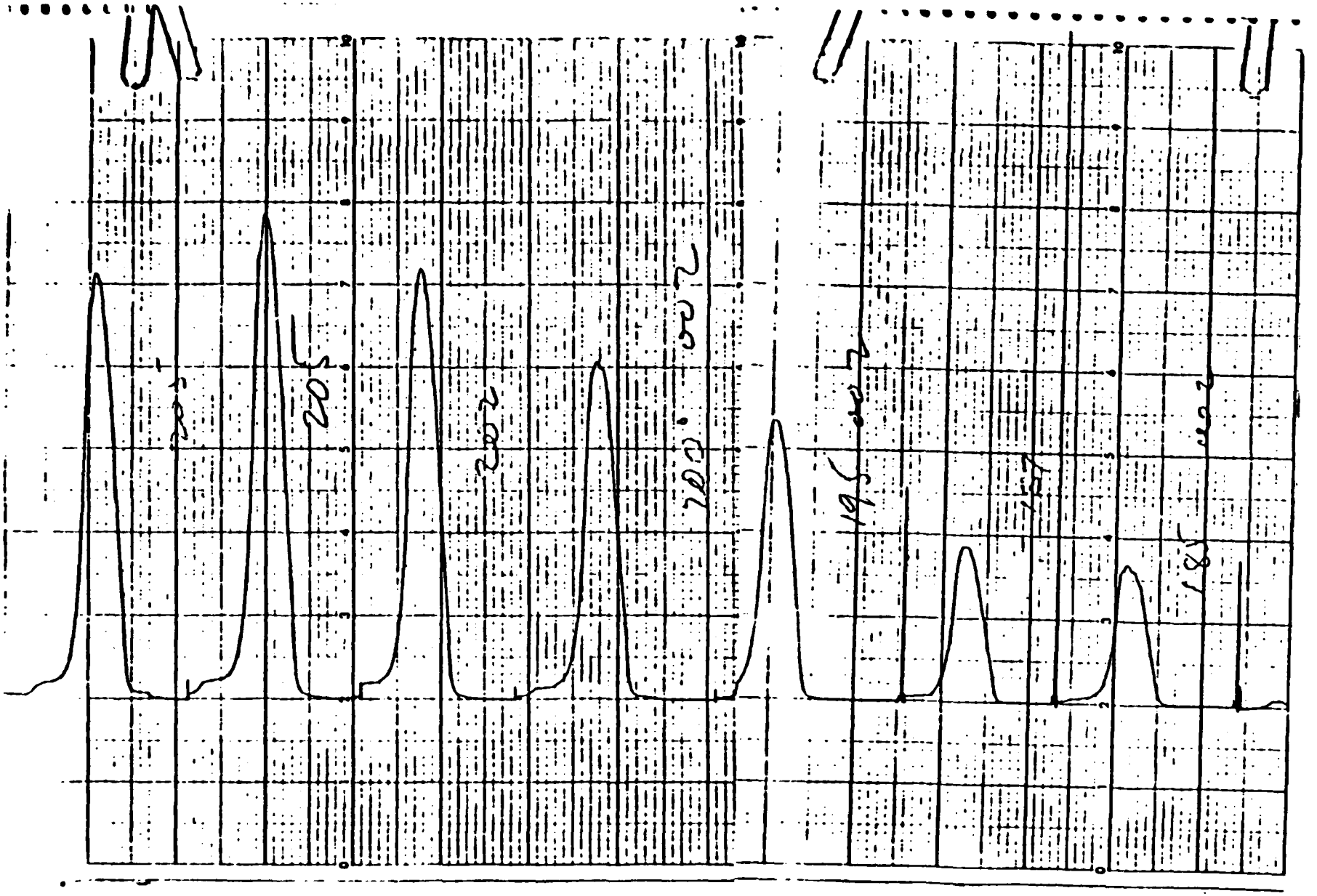


Fig. 12. Temperature dependence of the N_2 intensity (uncorrected) occurring from the thermal decomposition of 50%-50% BAMO-AZOX

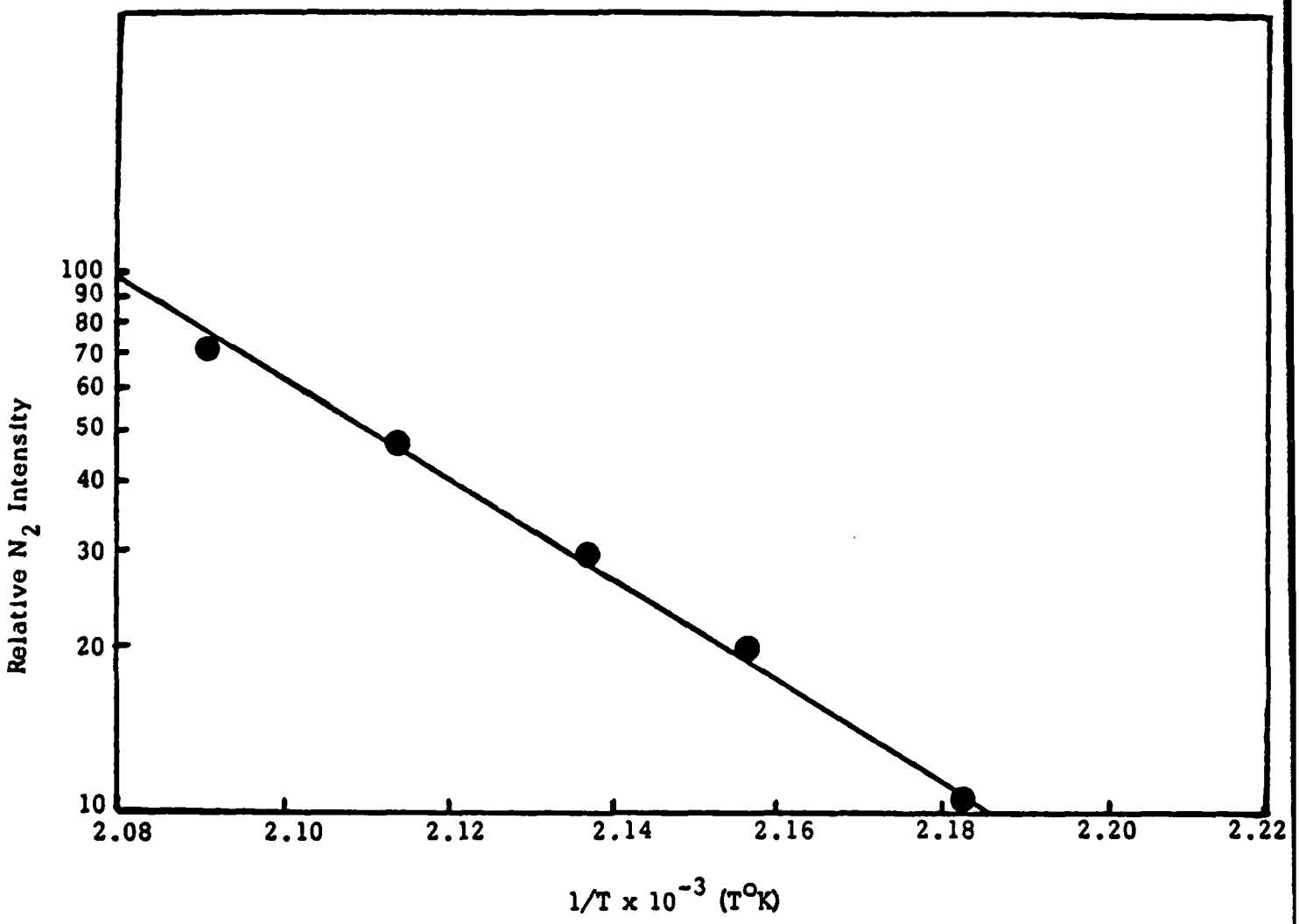


Fig. 13. Semilog plot of the N_2 intensity as a function of the reciprocal of the absolute temperature for the 50%-50% BAMO-AZOX copolymer. The E_a is 176 kJ/mole (42.1 kcal/mole)

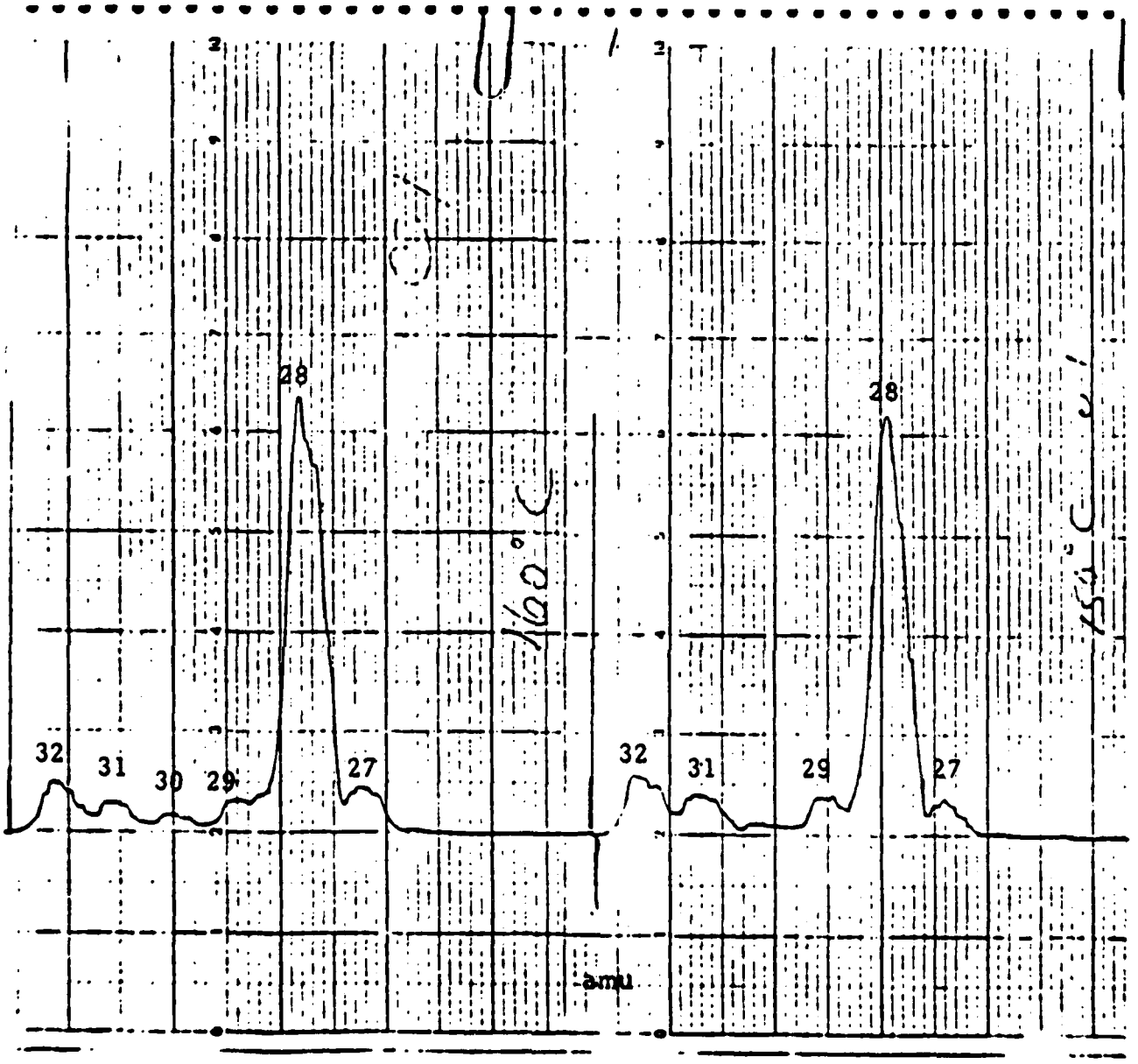


Fig. 14. Thermal decomposition of the 50%-50% BAMO-AZOX copolymer at 150 and 160 C

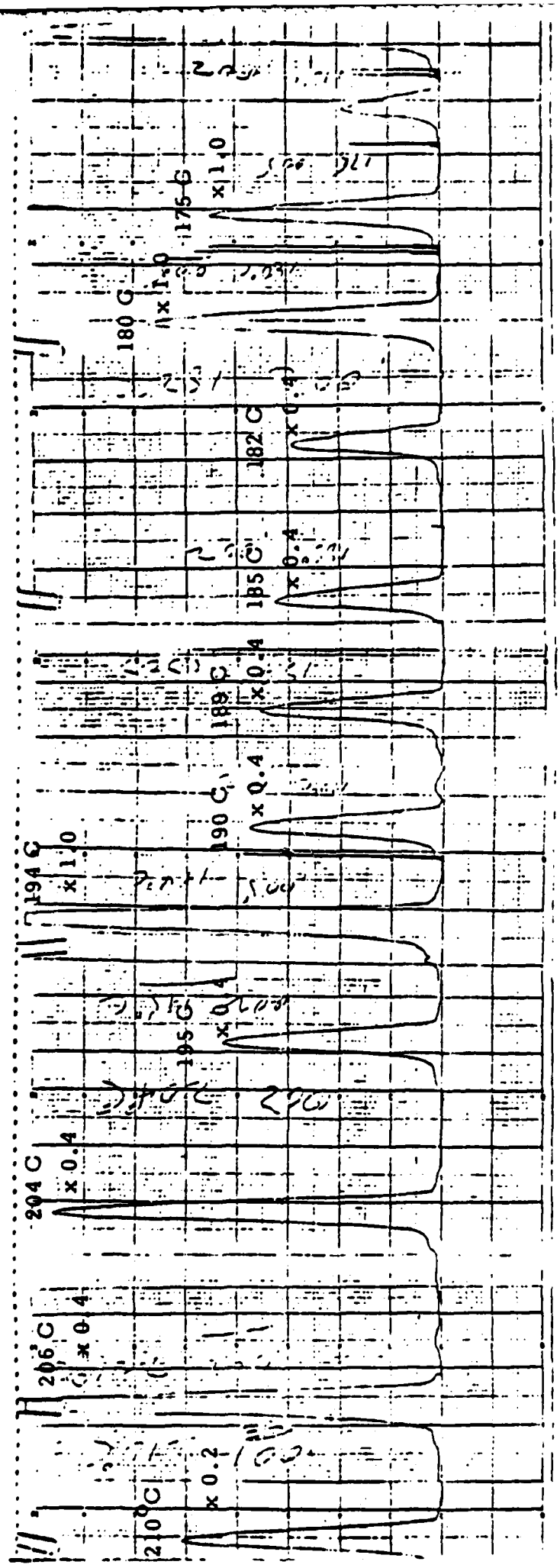


Fig. 15. Temperature dependence of the N_2 intensity (uncorrected) occurring from the thermal decomposition of 50%-50% BAMO-AMMO

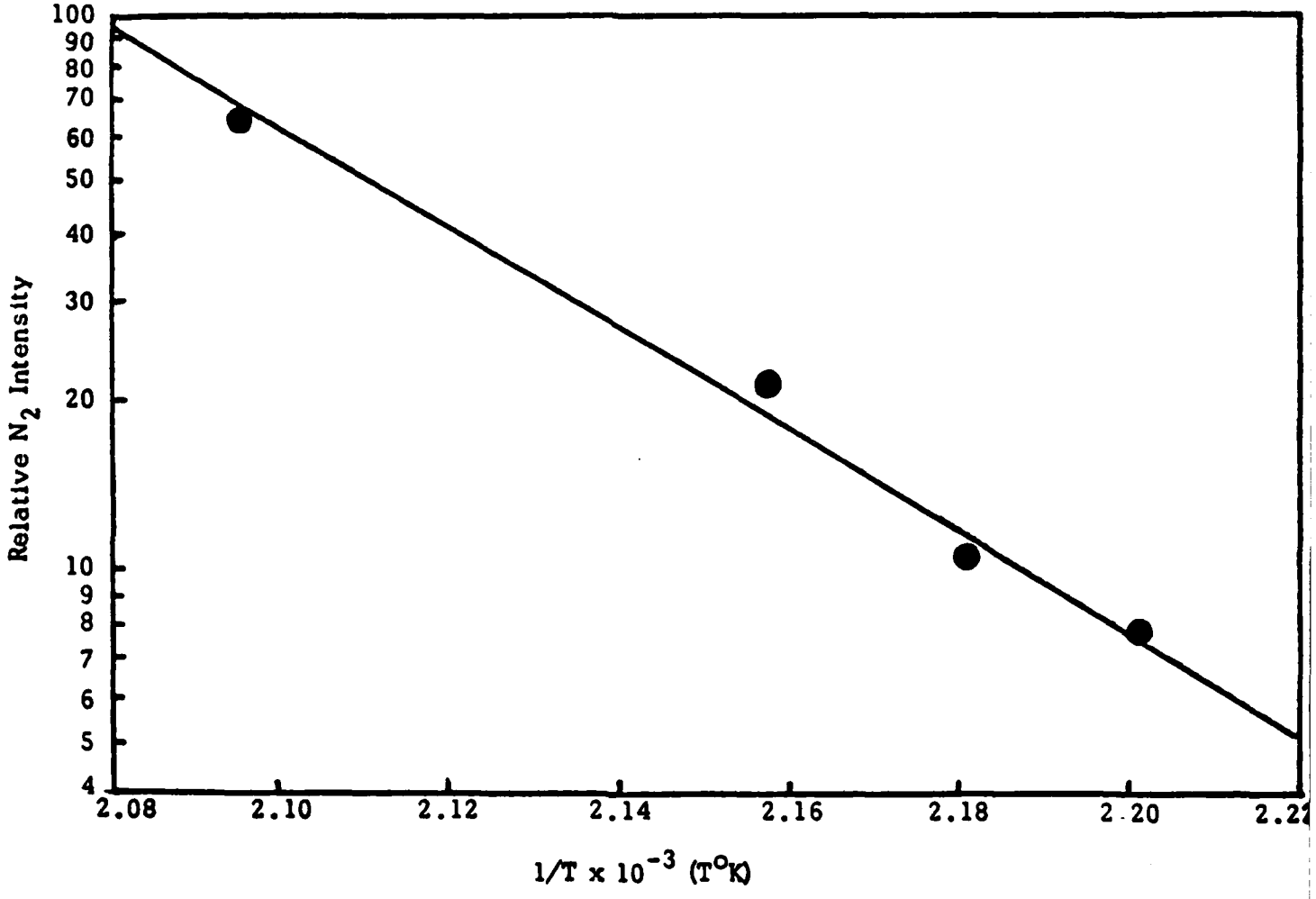
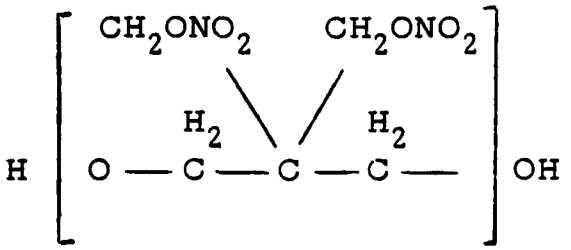


Fig. 16. Semilog plot of the N_2 intensity as a function of the reciprocal of the absolute temperature for the 50%-50% BAMO-AMMO copolymer. The E_a is 178 kJ/mole (42.5 kcal/mole).



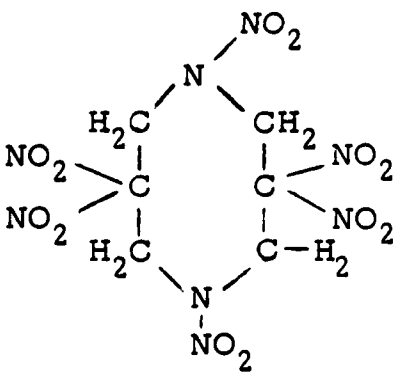
Unlike the copolymers of two azido compounds, this copolymer has two different reactant groups, an azide and nitrato.

The thermal decomposition mechanism of this copolymer is quite complicated in that both the azide and nitrato groups are initially decomposing at different rates. Figure 17 shows the decomposition products of this copolymer in the temperature range 110 to 208 C. Nitrato decomposition occurs well below that of the azido polymers, as shown by the prominent intensity peaks of 29, 30 and 31 amu arising from the CH_2ONO_2 group. As the temperature increases the rate of N_2 evolution from the BAMO decomposition is enhanced and agrees with the E_a for azide decomposition. Figure 18 shows the decomposition of 50-50% BAMO-DNAO as a function of three temperatures in three atomic mass ranges. At the lower temperature of 140 C the NO_2 peak is very pronounced due to the CH_2ONO_2 decomposition. At 200 C it has nearly disappeared. This can also be seen clearly from Figure 19, which shows the decreasing NO_2 concentration from 140 to 170 C.

Thermal decomposition studies on the homopolymer, as well as the copolymer of DNAO are continuing.

C. Thermal Decomposition of a New Energetic Amino-Nitro Ring Compound

A preliminary mass spectrometer study was made on a sample of the eight-membered C, N ring with NO_2 groups, having a molecular weight of 384, prepared at NSWC, White Oak



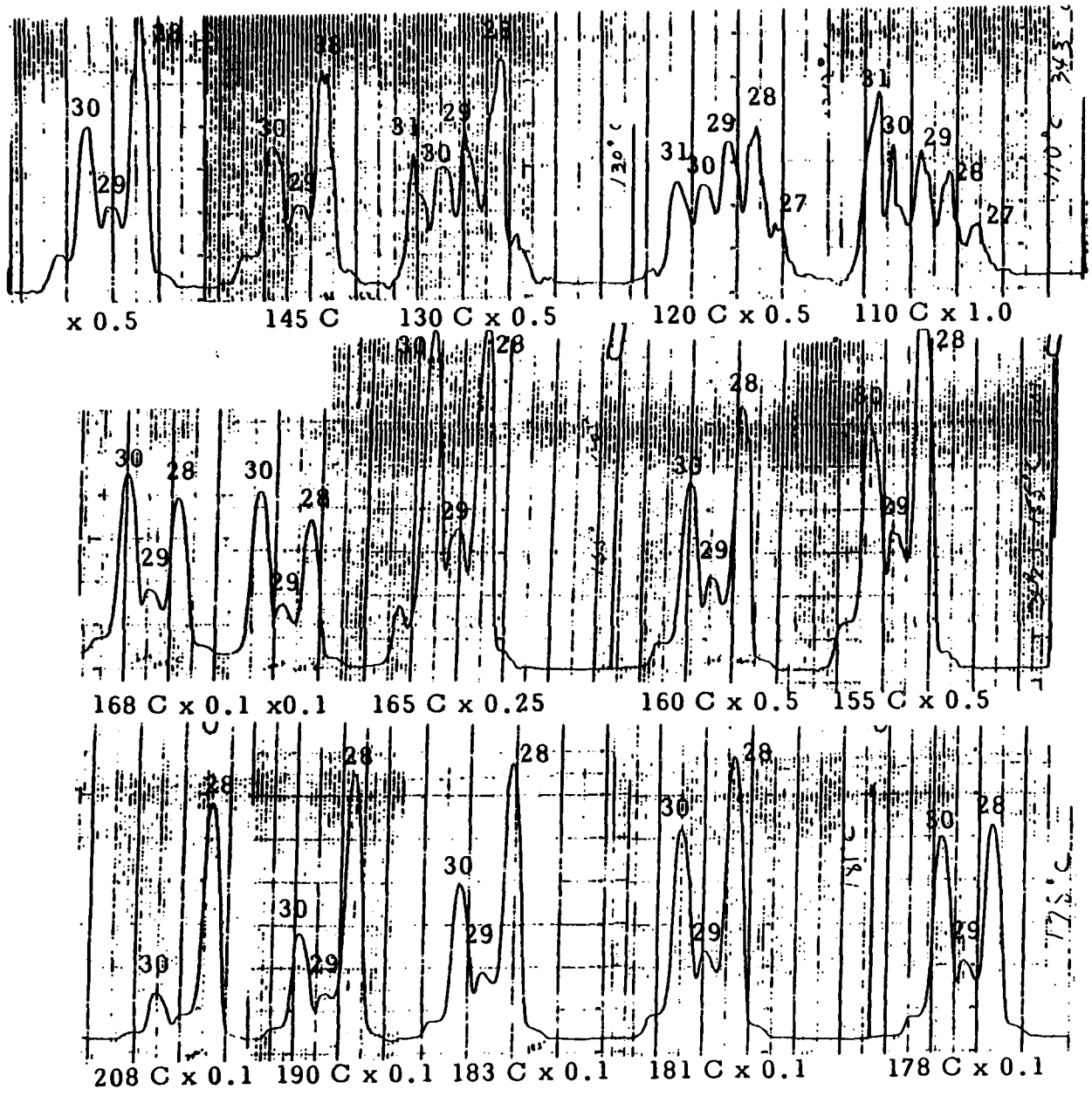


Fig. 17. Thermal decomposition of 50-50% BAMO-DNAO as a function of temperature

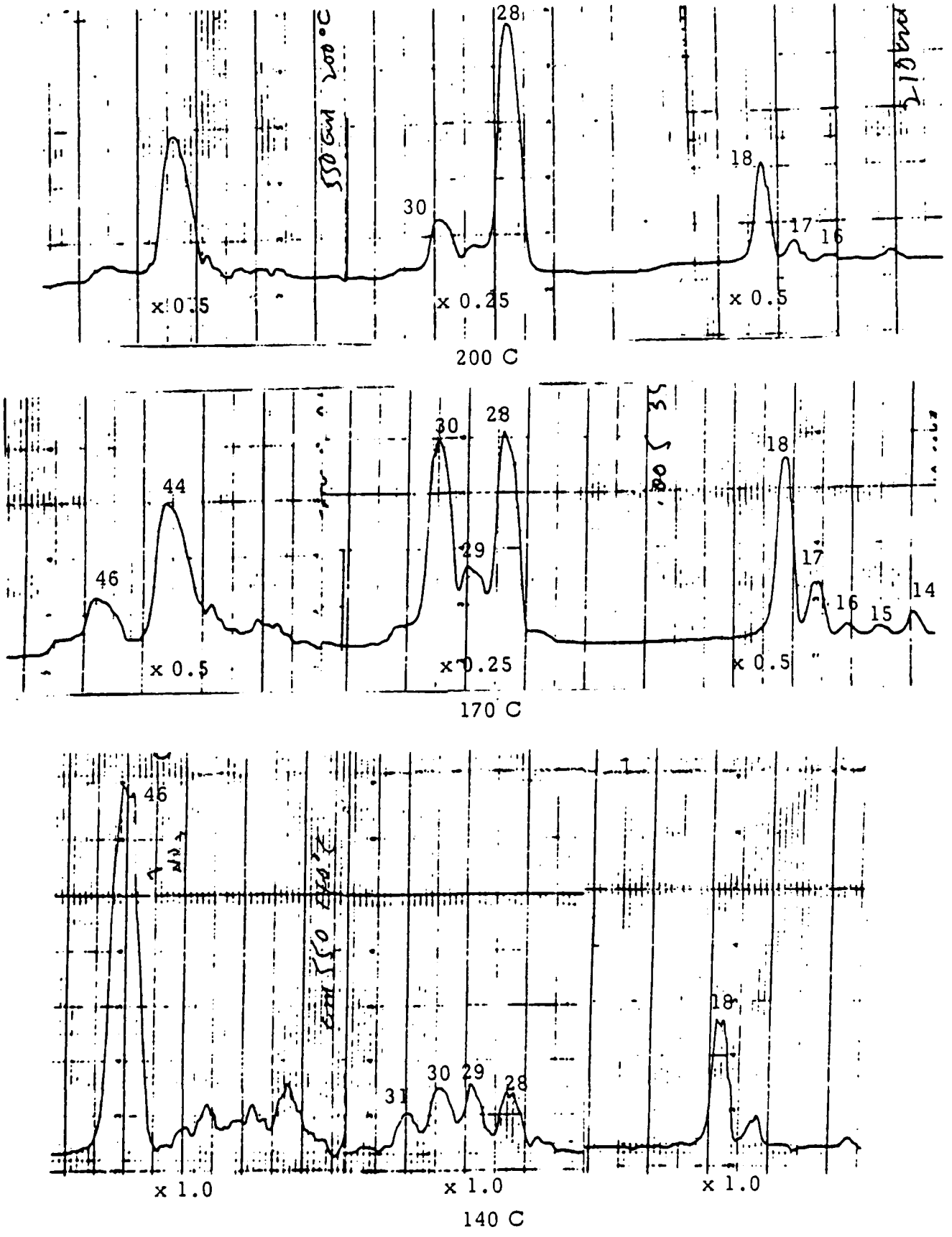


Fig. 18. Thermal decomposition of 50-50% BAMO-DNAO as a function of three temperatures in three amu ranges

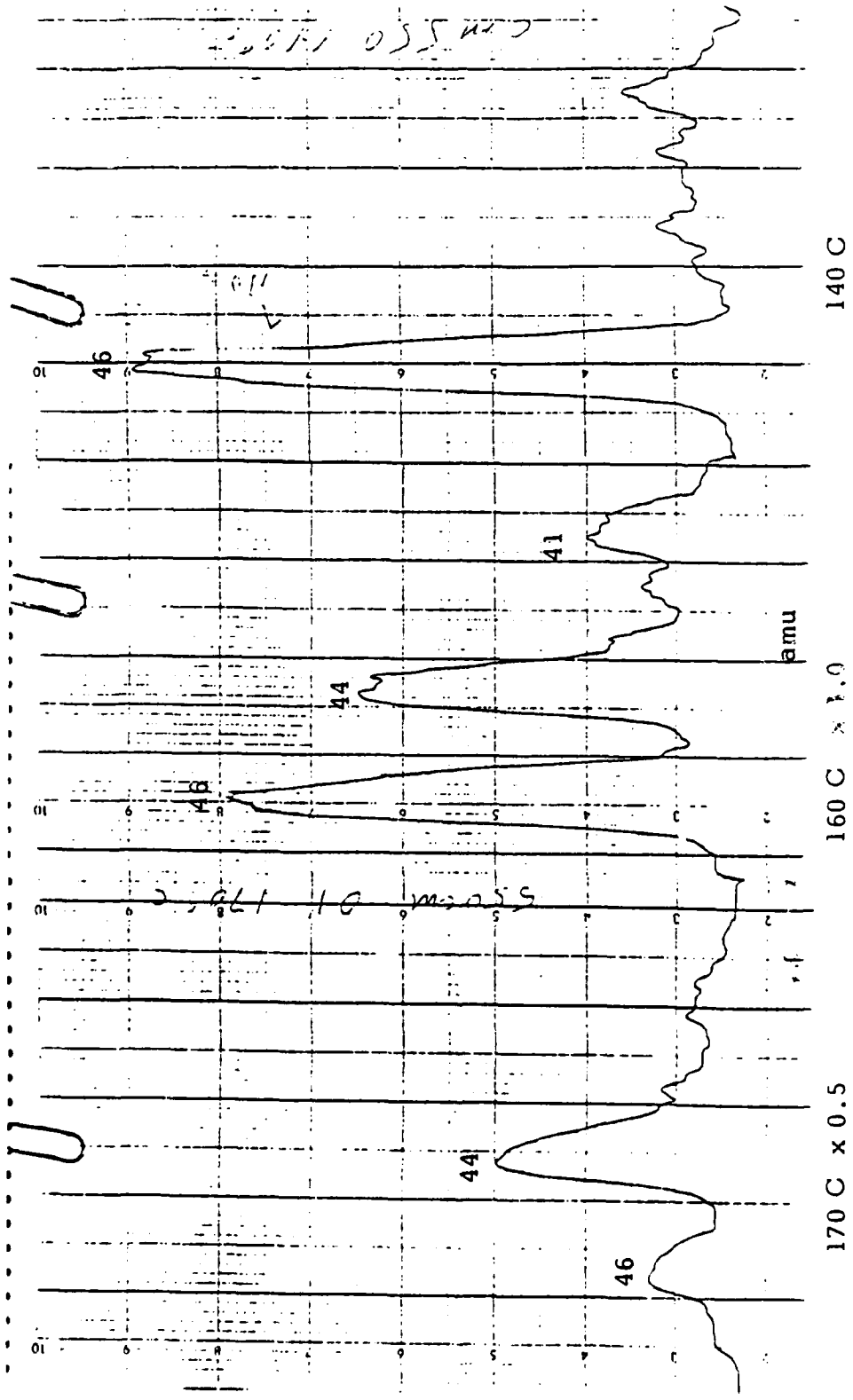


Fig. 19. Thermal decomposition of 50-50% BAMO-DNAO showing variation of species in the 40 - 46 amu range

The initial decomposition product of this compound is NO_2, which appears at approximately 150 C. A semilog plot of the NO_2 intensity (Fig. 20) yields an approximate activation energy of 165 kJ mol^{-1} (40 kcal/mole). The ring itself appears to be stable, with only NO_2 release. For example, at 260 C (Fig. 21) the most prominent peak observed is at 292 amu, which corresponds to the molecule (amu 384) less two NO_2 groups. The next highest intensity peak is observed at 200 amu, corresponding to the molecule less four NO_2 groups. It is possible that the molecule is evaporating as well as decomposing, since Figure 21 also shows a very low concentration ion intensity of 384 amu which corresponds to the compound itself. However, since this peak is barely discernible above the noise level, it should not be definitely construed that the molecules does undergo evaporation. More experiments will be conducted on this molecule.

III. ULTRAVIOLET STUDIES

There has been some concern in the propellant community regarding the sensitivity of the new azido polymers to sunlight degradation, especially the ultraviolet, and also, if decomposition were initiated, whether it would continue as a result of autocatalysis. A series of qualitative ultraviolet decomposition studies was conducted in an attempt to answer these questions. The materials investigated were the homopolymers of AZOX and AMMO and the copolymers 50-50% BAMO-AZOX and 50-50% BAMO-AMMO.

Two types of UV lamps were employed, a short wave of 2540 Å and a long wave of 3660 Å. The radiation obtained from these lamps is 7.83×10^{-19} joules/photon, equivalent to 4.88 eV, for the short wave, and 5.43×10^{-19} joules/photon, equivalent to 3.39 eV, for the long wave. These energies correspond to 112 kcal/mole and 78 kcal/mole, respectively, for the short and long wave UV lamps. The deposition energy at a distance of 1 inch from the surface to be irradiated is 7200 μwatts/cm^2 and 10,000 μwatts/cm^2 for the short and long wave lamps.

The first material studied was the AZOX homopolymer employing both the short and long wave UV radiation. Samples of the polymer of

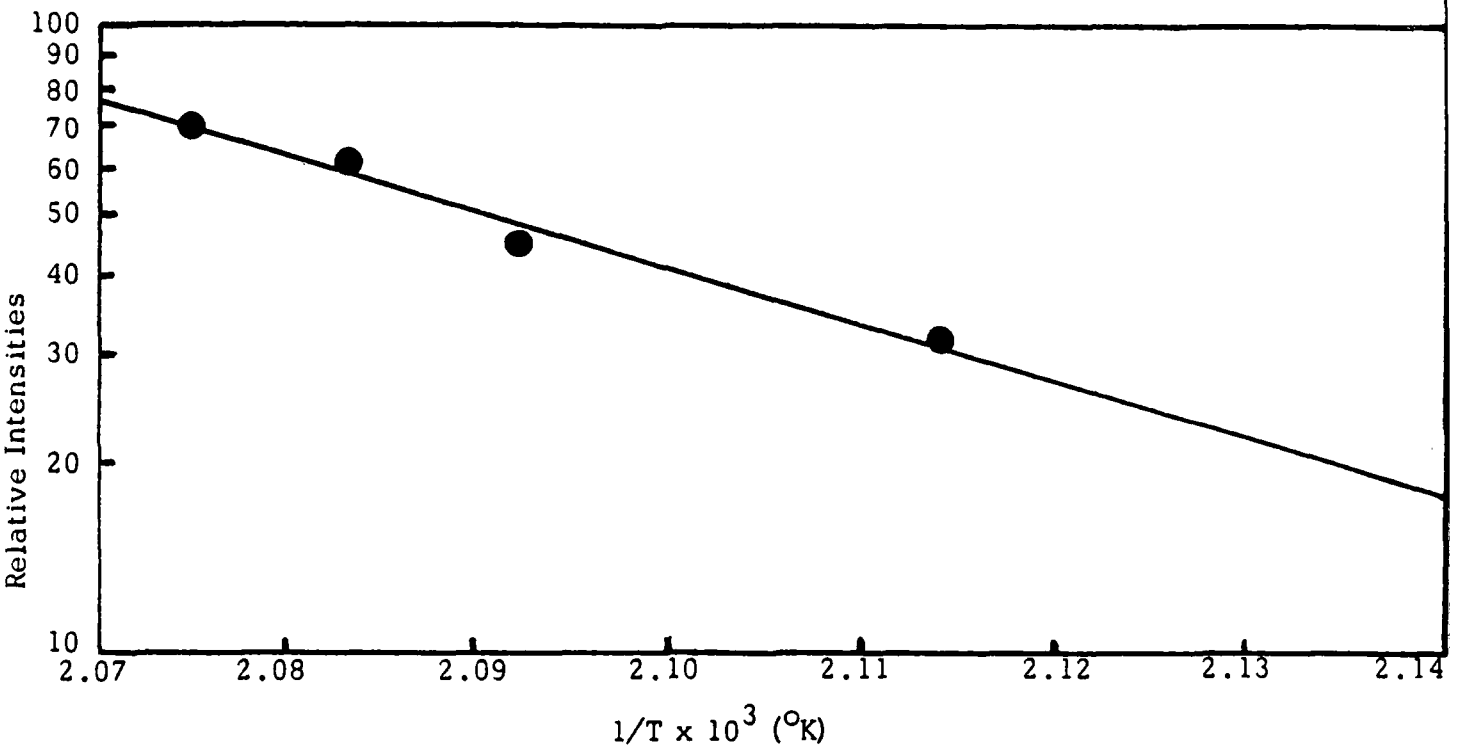


Fig. 20. Activation energy based on NO_2 ion intensities for the new NSWC compound

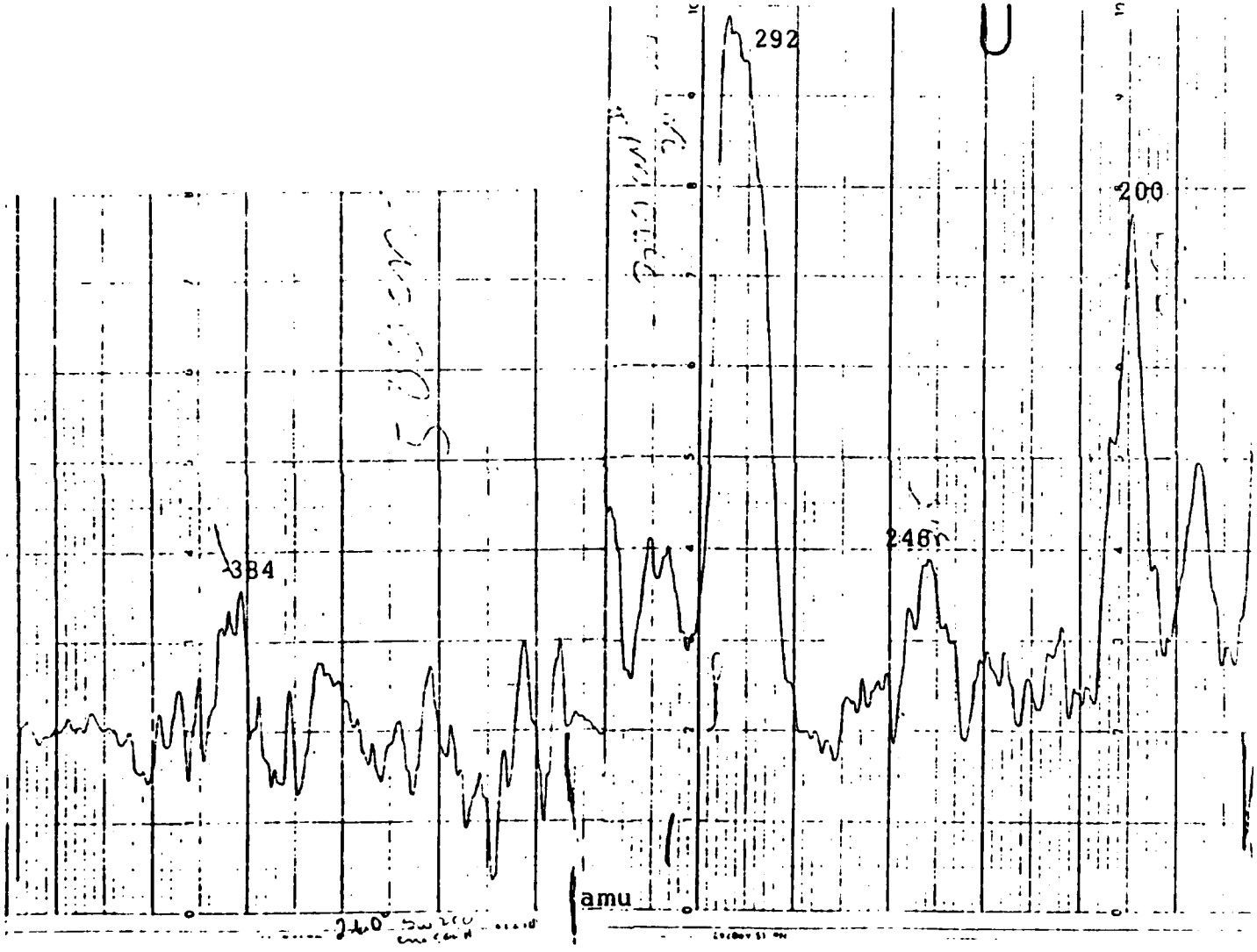


Fig. 21. Principal decomposition fragments observed from the thermal decomposition of the NSWC compound at 260 C

approximately 25 mg in weight were placed in a 1 cm^2 area on aluminum foil at a distance of 1 inch from the UV lamp.

After several preliminary experiments with the short wave 2540 Å ultraviolet they were discontinued due to considerable adsorption of the short wave radiation caused by its path through the air. Short wave UV irradiation studies would thus best be accomplished in a vacuum enclosure.

The results for both the long and short wave radiation involving the AZOX homopolymer are shown in Figure 22. Considerable decomposition was observed in relatively short times. As can be seen approximately 4% and 8% reduction in the sample weight was accomplished in a one-hour irradiation. The nitrogen content of AZOX is 42%, whereas the N_2 of the azide group is only 28%. Thus in a five-hour exposure to UV 3660 Å radiation between one-third and one-half of the N_2 content of the polymer was released. Visual observation of the polymer after irradiation revealed that the material changed from viscous liquid to a gummy, or rubbery, semi-solid. In order to determine whether ordinary light would cause decomposition, a sample of the AZOX polymer was exposed to fluorescent light for five days; there was no apparent weight loss.

Experiments on the AMMO homopolymer, the 50-50% BAMO-AZOX, and the 50-50% BAMO-AMMO copolymers were performed with the 3660 Å long wave lamp. The samples were irradiated at distances of 1" (10,000 $\mu watts/cm^2$), 3" (1120 $\mu watts/cm^2$), and 6" (380 $\mu watts/cm^2$) for periods up to five hours. The radiation intensity, for example, at a distance of 1" from the sample surface provides 36 J/cm^2 for each hour of exposure. The decomposition rate of the AMMO homopolymer (Fig. 23) appears to be similar to that of the AZOX homopolymer as shown in Figure 22.

Qualitative results for the copolymers are shown in Figure 24. In all cases the irradiated samples show considerable gas release with polymeric cross-linking producing rubbery solids from the initial viscous liquids. The irradiated samples (homopolymers and copolymers) were maintained for a number of weeks under ordinary atmospheric conditions as well as under vacuum. No autocatalysis was observed.

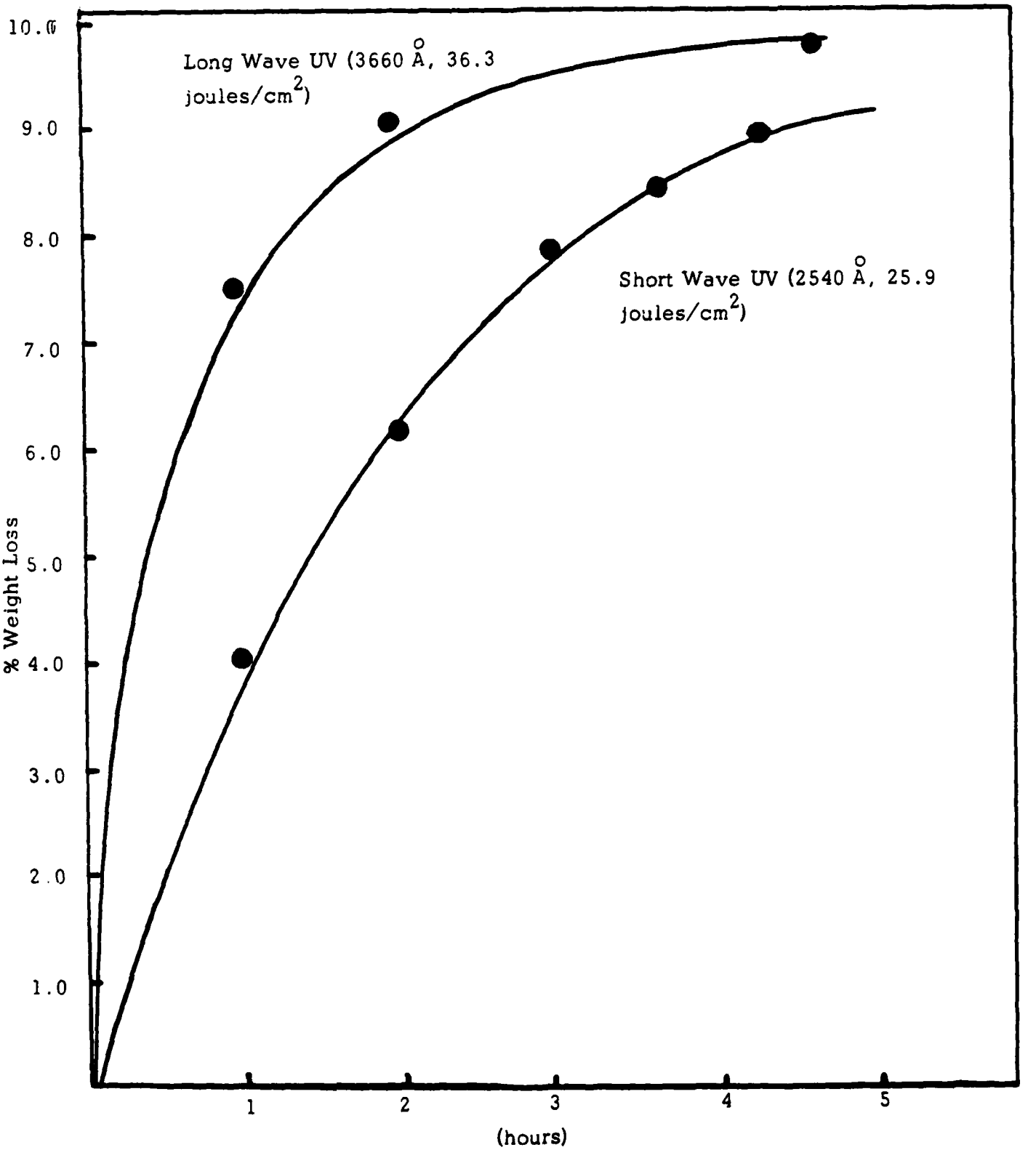


Fig. 22. Ultraviolet irradiation of AZOX polymer sample size 20 mg on 1 cm^2 area

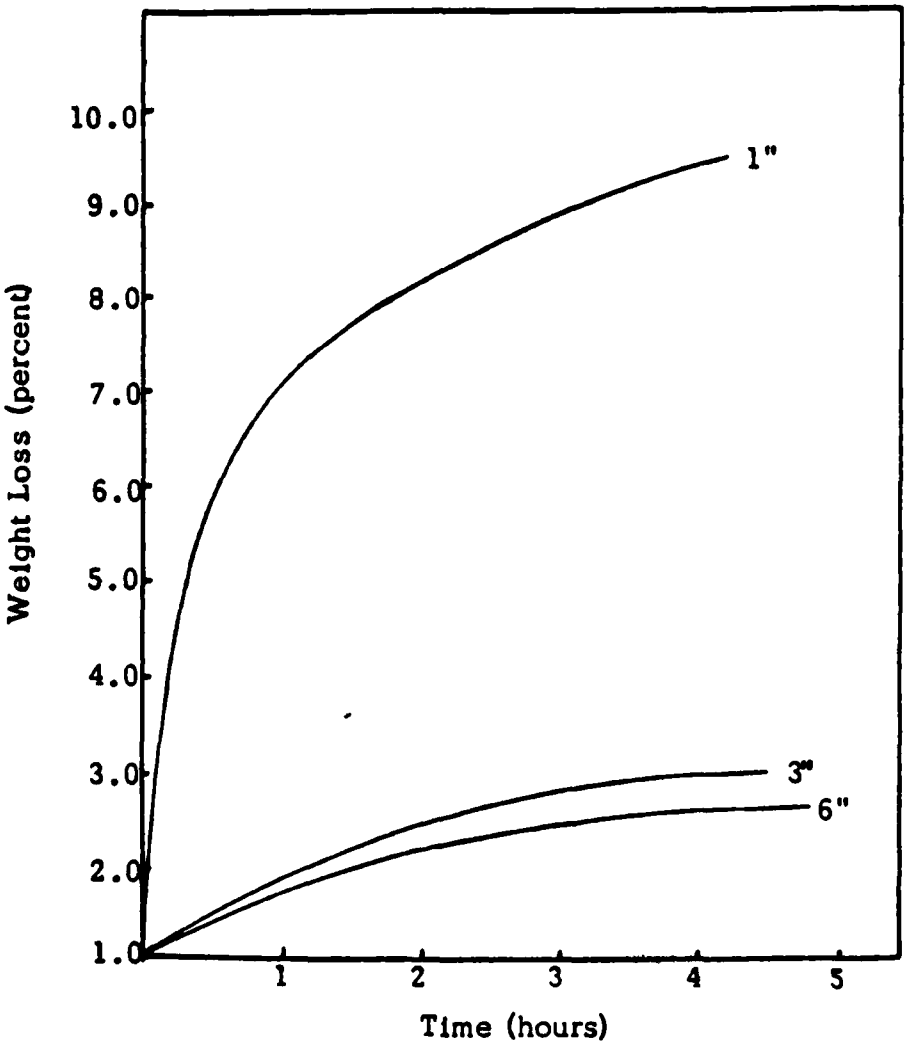


Fig. 23. Weight loss of the AMMO homopolymer as a function of long wave (3660Å) ultraviolet radiation at distances of 1" (10,000 μwatts/cm^2), 3" (1120 μwatts/cm^2), and 6" (380 μwatts/cm^2) from the sample surface. The intensity of the radiation is 3.39 eV (78 kcal/mole).

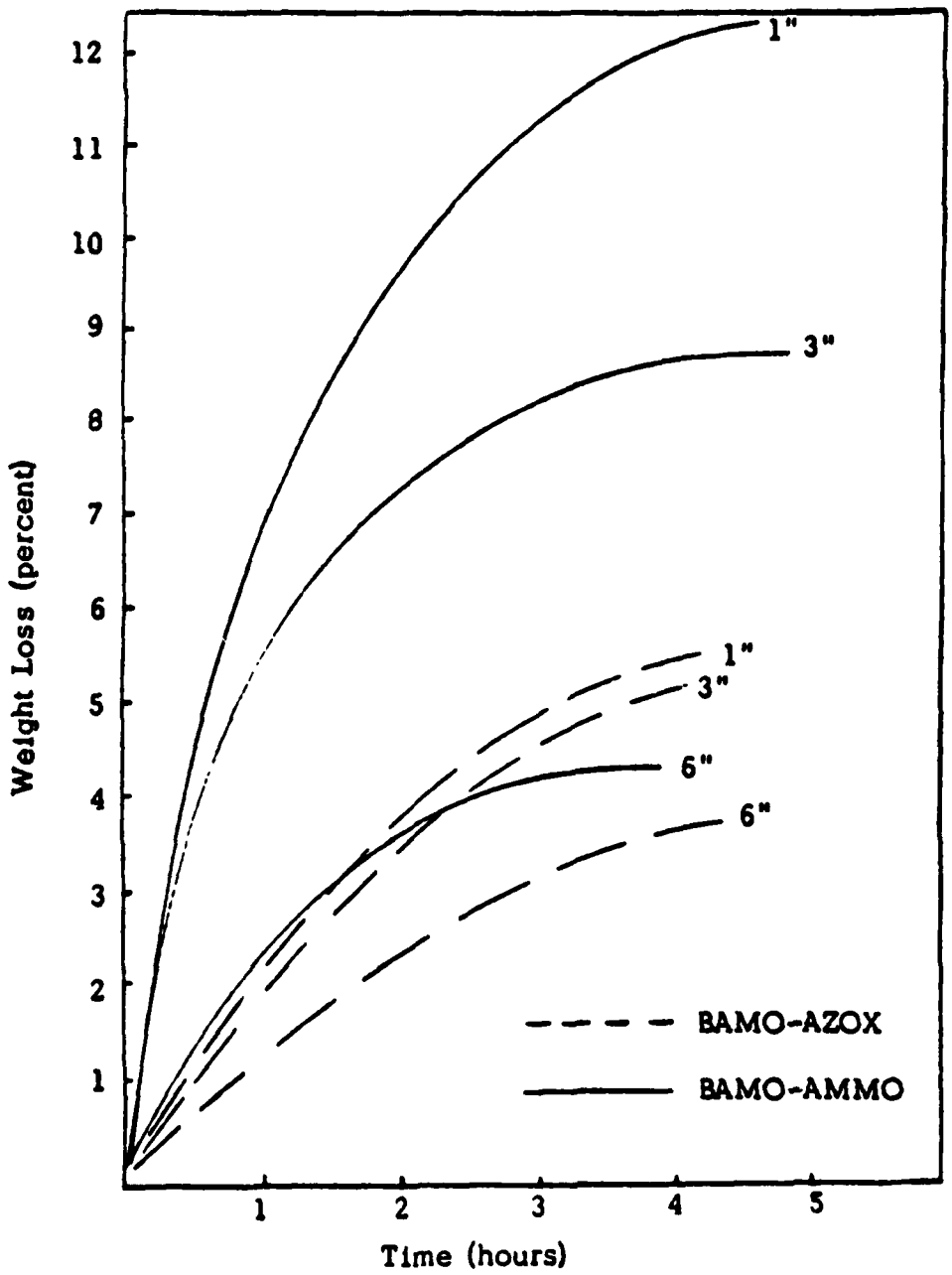


Fig. 24. Weight loss of the BAMO-AZOX and BAMO-AMMO copolymers as a function of long wave (3660Å) ultraviolet radiation at distances of 1" (10,000 μwatts/cm^2), 3" (1120 μwatts/cm^2), and 6" (380 μwatts/cm^2) from the sample surface. The intensity of the radiation is 3.39 eV (78 kcal/mole).

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