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Rock Island Arsenal
Laboratory

TECHNICAL REPORT

POLYMERIZATION OF ALLYL MONOMERS
BY MEANS OF GAMMA RADIATION

By

W. Dennis McHenry

Department of the Army Project No. 599-25-001

Ordnance Management Structure Code No. 5010.11.838

Report No. 62-4163

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Approved by:

A. C. Hanson

A. C. HANSON
Laboratory Director

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Rock Island Arsenal
Rock Island, Illinois

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ABSTRACT

Poly (allyl alcohol), poly (allyl acetate), poly (allyl phenyl ether), and poly (allyl isothiocyanate) were produced from their monomers by the action of gamma radiation from Cobalt-60. Polymerization rates and solubilities of the polymers are given. Molecular weights were determined for poly (allyl acetate) and poly (allyl phenyl ether). Infrared spectra for poly (allyl alcohol) and poly (allyl phenyl ether) are presented.

POLYMERIZATION OF ALLYL MONOMERS BY MEANS OF GAMMA RADIATION

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POLYMERIZATION OF ALLYL MONOMERS BY MEANS OF GAMMA RADIATION

OBJECT

To study the effect of gamma radiation on allyl monomers in regards to polymerization and to determine polymerization rates and molecular weights of the resulting polymers.

INTRODUCTION

There is an urgent and continuing need for new polymers and copolymers which have improved solvent resistance, age resistant properties, low temperature properties, heat resistance and resistance to ionizing radiation. A promising technique for the preparation of these new materials lies in the exposure of a monomer or mixture of monomers to radiation. However, for the most efficient use of this technique, the polymerization rates and molecular weights of the resultant polymers should be known. A search of the literature shows that considerable effort has been expended in the field of radiation polymerization of vinyl compounds. Eisler(1), in his work in this field at this laboratory, studied the polymerization rates and resultant molecular weights of polymers produced from butadiene and styrene.

With the great amount of data on vinyl polymerization available, it is surprising that so little work has been done on the radiation polymerization of allyl compounds. Laible(2) in his literature review cites 158 publications and 243 patents pertaining to allyl polymerization, but no mention is made of the use of radiation. Scheiber(3) in a later review also makes no mention of the technique. Horan(4) states that radiation polymerization of triallylcyanurate produces a hard, brittle polymer. Everard, et al(5) have recently reported that phenol and polymer are the main products in the radiolysis of allyl phenyl ether.

Since there was this dearth of data on the radiation polymerization of allyl compounds, this project was initiated for the purpose of studying polymerization rates and the molecular weights of polymers obtained by the irradiation of allyl monomers.

MATERIALS AND EQUIPMENT EMPLOYED

Monomers

Allyl isothiocyanate (practical) (Eastman P288), allyl alcohol (Eastman 518), allyl acetate (Eastman 5096) and allyl phenyl ether (Eastman 6464) were the monomers chosen for this investigation. Each was purified by distillation prior to use.

Distillation Apparatus

Distillation was accomplished in a system consisting of a 500 ml, three neck boiling flask with ground joints, a variable reflux ratio distilling head (Ace Glass Company #6632) and a 250 ml single neck receiving flask with a ground joint. The distilling head was inserted in the center neck of the boiling flask. A thermometer was inserted in one side neck and a capillary tube in the other. The capillary allowed the admission of argon to the system for flushing prior to distillation and to prevent violent boiling during distillation. When vacuum techniques were used, the single neck receiving flask was replaced by an udder type receiver.

The freshly distilled monomers were refrigerated under argon until needed for the irradiation studies.

Irradiation Facilities

Samples were irradiated in one of the two Cobalt-60 facilities available in this laboratory. One, of Brookhaven design(6), can be heated for studies at elevated temperatures. The strength of this source is approximately 200 curies and the mean dose rate during this investigation was 7.2 X 10^4 rad/hr. The second source is an underwater, kilocurie source having a much larger capacity and higher dose rate than the Brookhaven source. The strength is approximately 4600 curies, which yielded mean dose rates of 2.5 X 10^5 rad/hr. in the main irradiation chamber and 3.2 X 10^4 rad/hr. in supplementary chambers outside of the main chamber.

Dose rates in the Brookhaven source were determined by the ferrous sulfate oxidation method(7). Silver-phosphate glass dosimetry(8) was used for the kilocurie source. The average dose rate during the period of each irradiation is given in the tables.

PROCEDURE AND RESULTS

Allyl Alcohol

The monomer was dried by refluxing over fused potassium carbonate(9) prior to distillation. Only the portion of the distillate which carried over at a constant temperature was used in the irradiation studies.

Samples were prepared for irradiation by sealing the allyl alcohol, under argon, in 10 or 20 milliliter ampoules. The weight of the monomer was determined after sealing. All of the allyl alcohol samples were irradiated at ambient temperatures except numbers 10-5 and 10-6 which were maintained at 70 C during irradiation.

After irradiation, the unreacted monomer was separated from the polymer by distillation. The contents of the ampoule and three ethanol rinsings were transferred to a tared wide mouth flask (Ace Glass Company #9025). The flask was then attached to a vacuum system by means of a rotary film evaporator (Labline #5100). After most of the monomer had been removed, the flask was detached and placed in a vacuum oven (60 C) until a constant weight was obtained. Percent yield was calculated by dividing the weight of the residue by the original weight of monomer and multiplying by 100. In all cases the residue had the consistancy of a heavy oil.

Table I summarizes the initial results on the irradiation of allyl alcohol.

TABLE I

ALLYL ALCOHOL

Sample No.	Dose Rate (Rad/hr.)	Total Dose (Megarad)	Yield (%)	Yield/Megarad (%/Megarad)
20-2	2.80×10^5	6.70	0.504	0.075
20-3	2.80×10^5	20.0	1.62	0.081
20-4	2.79×10^5	46.9	3.64	0.078
20-5	2.79×10^5	147.0	12.4	0.084
10-1	3.60×10^4	4.23	0.368	0.087
10-4	3.53×10^4	126.0	11.9	0.094
10-5*	7.54×10^4	3.32	0.653	0.197
10-6*	7.54×10^4	39.6	10.0	0.252

*Irradiated at 70 C.

In order to develop a procedure for the direct precipitation of the polymer, aliquots of the irradiated monomer were added to several solvents and observations were made as to whether the two were immiscible, a solution formed or a precipitate formed. The results of this study and others similar to it for the other monomers are shown in Table II.

Four additional 10 milliliter ampoules of allyl alcohol were prepared and placed in the kilocurie source for an indefinite period of time to determine the dose required to produce a solid sample, if possible. One of the ampoules exploded in the source after a dose of approximately 325 megarads. Another exploded after approximately 360 megarads. The other two ampoules, with their contents still in the liquid state, were removed from the source with a total dose of 378 megarads.

One of these two remaining ampoules exploded while being opened and the irradiated material was lost. The fourth was opened successfully. The polymer was precipitated in dioxane and this material was used to determine several properties of poly (allyl alcohol).

Five-hundred milligram portions of the polymer were weighed into each of several 100 milliliter volumetric flasks. Different solvents were added to the flasks and after shaking were allowed to stand. After forty-eight hours a 10 milliliter aliquot was taken from each flask and evaporated to dryness. These dry weights were used to calculate the results shown in Table II.

A portion of the polymer was sent to an independent laboratory for a molecular weight determination. Unfortunately, the other laboratory was unable to make the determination and the sample was not returned.

The remainder of the polymer was used in obtaining its infrared spectrum. This spectrum along with one of the monomer is presented in Figure 1.

Allyl Acetate

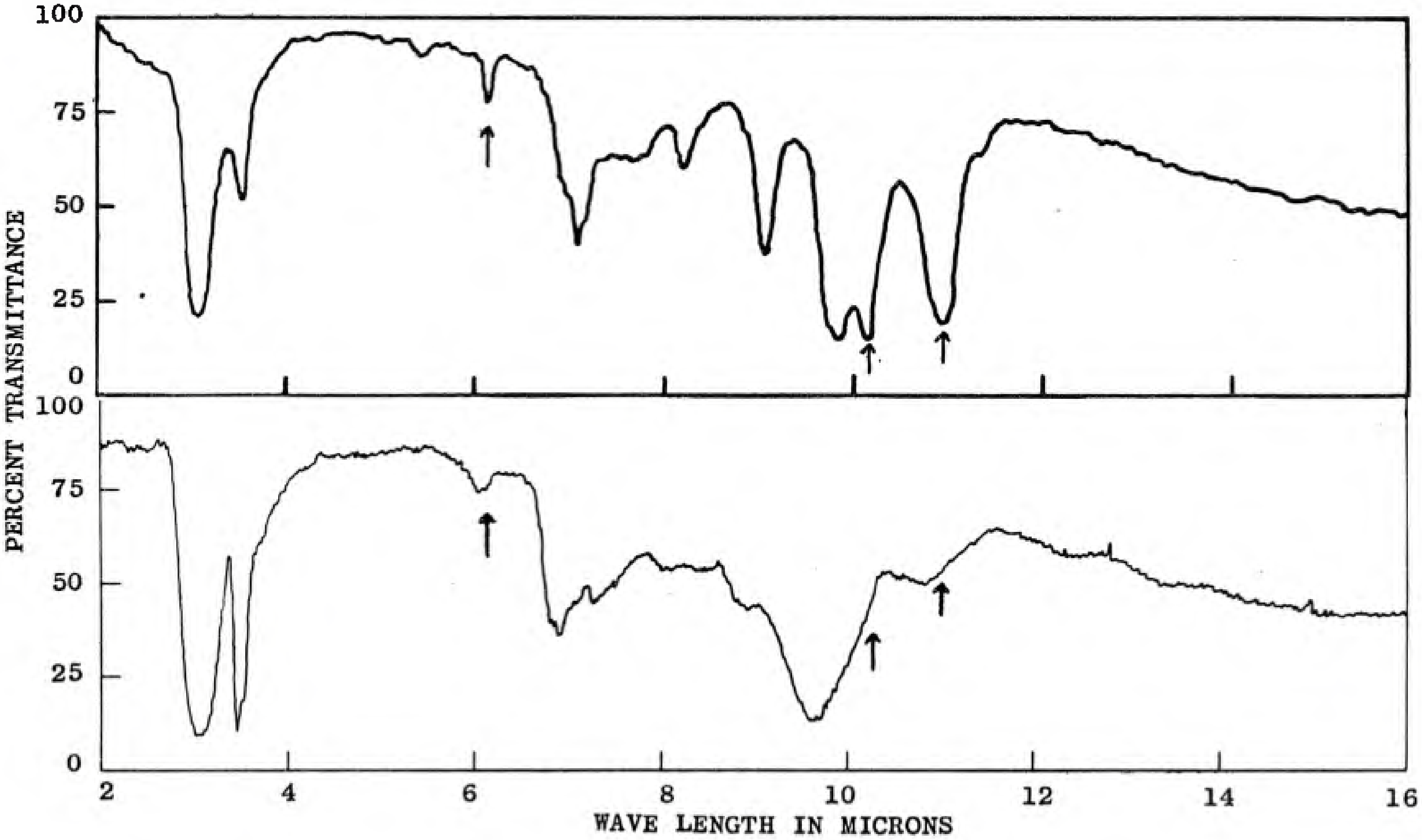
All of the initial phases of the work on allyl acetate, except sample preparation, i.e., distillation, irradiation and monomer-polymer separation, were conducted in the same manner as for allyl alcohol. Sample preparation was the same as allyl alcohol except for those with the prefix B. These samples were not sealed in ampoules, but were transferred to Pyrex culture tubes which were closed with aluminum lined screw caps. All irradiations were at ambient temperatures. The results of this work are given in Table III.

TABLE II

SOLUBILITIES OF MONOMERS AND POLYMERS

MATERIAL	SOLVENTS Acetone	Benzene	Chloroform	Dioxane	Ethanol	Hexane	Toluene	Water	Ethylene Glycol	Carbon Disulphide	1,2-Dichloroethane	Dimethylformamide
Irradiated allyl alcohol	s	P		P	s			s				
Poly (allyl alcohol)	I	I	I	I	S			s	S			
Irradiated allyl acetate		s		s	P	P	s	Im				
Poly (allyl acetate)	S	S	S	S	Im	I	S	Im				
Irradiated allyl isothiocyanate	s	s	s	s	s		s	Im				
Poly (allyl isothiocyanate)	I	I	I	I	I		I	Im		I	I	s
Irradiated allyl phenylether	s	s		s	P	P	s	s				
Poly (allyl phenylether)		S										

S - soluble (at least 0.5 g/100 ml.)
s - soluble, but exact quantities unknown
I - insoluble
P - precipitate forms
Im - immiscible



Top: Spectrum of Allyl Alcohol
Bottom: Spectrum of Poly (Allyl Alcohol)

INFRARED SPECTRA

FIGURE 1

TABLE III

ALLYL ACETATE

Sample No.	Dose Rate (Rad/hr.)	Total Dose (Megarad)	Yield (%)	Yield/Megarad (%/MR)
A5	2.60×10^5	6.24	3.26	0.52
A6	2.60×10^5	12.5	6.00	0.48
A7	2.60×10^5	18.7	8.94	0.48
B1	6.93×10^4	4.90	3.19	0.65
B2	6.93×10^4	8.32	5.59	0.67
B3	6.93×10^4	16.6	11.6	0.70
B4	6.93×10^4	33.3	23.2	0.70
B6	3.30×10^4	7.92	5.31	0.67
B7	3.30×10^4	15.8	9.51	0.60

All of the polymers produced were clear, colorless, and quite viscous.

Four additional culture tubes were filled with allyl acetate and placed in the kilocurie source for an indefinite period in order to investigate the effect of longer irradiations. Yields were not calculated for these samples since they were determined in the preliminary studies. Also, it is known that the conversion rate changes as the upper limit of conversion is reached(10). The results of this work are summarized in Table IV.

TABLE IV

ALLYL ACETATE EXTENDED IRRADIATION

Sample No.	Total Dose	Description of Material
1	186×10^6 rad	Quite viscous
2	245×10^6 rad	Very viscous (required 4 hrs. to drain from tube)
3	269×10^6 rad	Almost solid (required 3 days to drain from tube)
4	282×10^6 rad	Solid, very tacky, quite elastic

Sample 1 was transferred to a wide mouth flask and placed in the vacuum oven. The sample was contaminated by material from another experiment in the oven and was discarded.

Sample 2 was used to determine the solubility of the polymer after the monomer had been removed in the vacuum oven. The results are given in Table II.

Sample 3 was used in an attempt to obtain a solid polymer, as in the case of allyl alcohol, by a fractionation of the polymer using the technique of Moore and Murphy(11). Acetone was used as the solvent and hexane as the nonsolvent. The first and what was assumed to be the highest molecular weight fraction was not solid, but was very viscous. Subsequent factions were not solid nor did a refractionation of the first produce a solid.

Approximately one gram of very sticky, elastic polymer was pulled with great difficulty from sample 4 and taken up in benzene. This solution appeared to be clear and no check for the presence of gel was made. After removal of the benzene in the vacuum oven, the polymer was still very tacky. In an attempt to reduce this tackiness, the remainder of sample 4 was replaced in the kilocurie source for an additional 23×10^6 rads. The tackiness was reduced and in addition the material was made so much more cohesive that the tube had to be broken in order to recover the material.

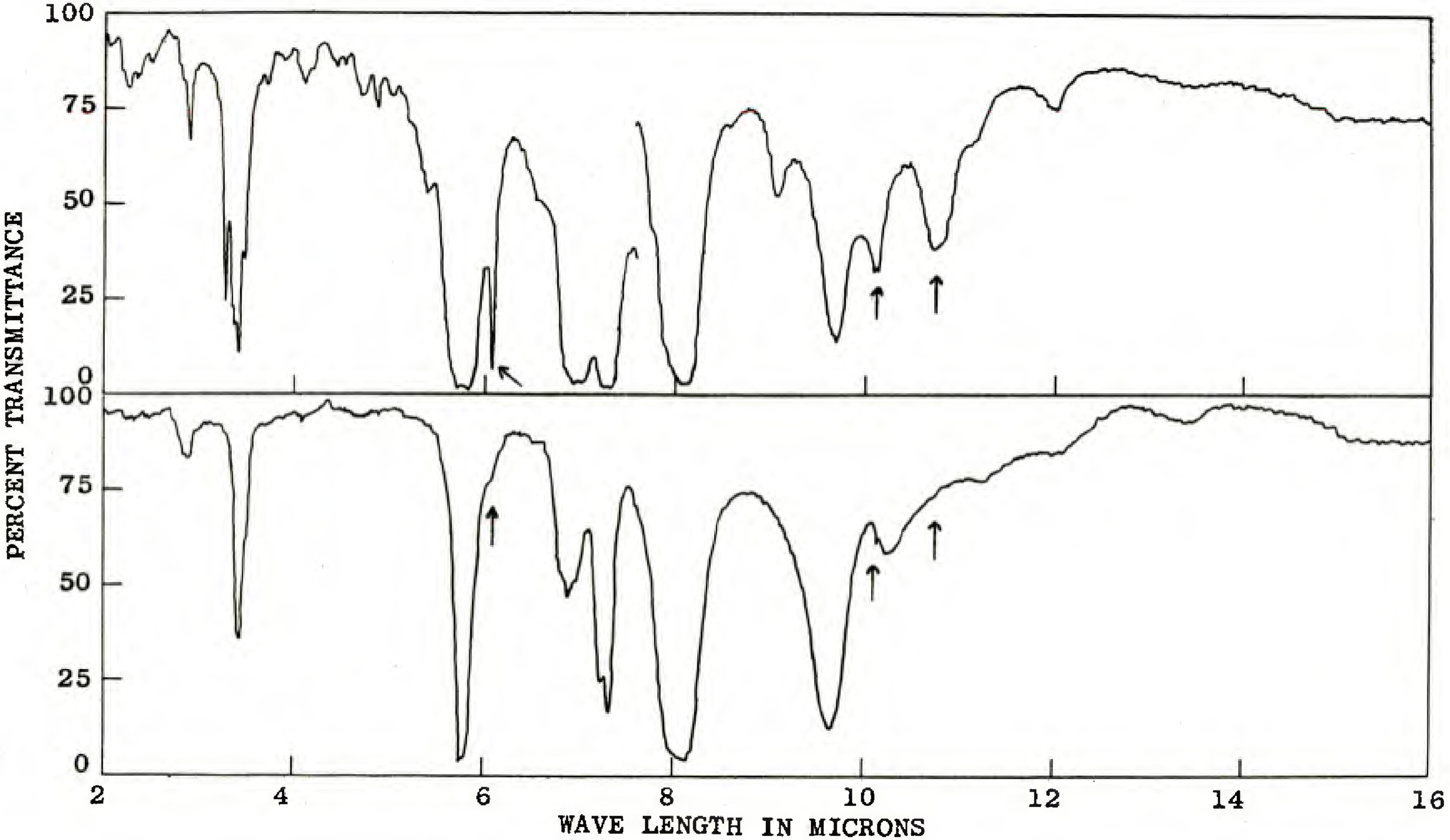
A portion of the re-irradiated material was examined for gel by extraction of the polymer from the solid material with acetone. Thirty-seven percent was not soluble.

The remainder of the re-irradiated material was extracted with benzene in order to obtain samples of the polymer for molecular weight and infrared spectra determinations. The ebullioscopic method of Barr and Anhorn(12) was used for the molecular weight determinations. This gave a number average molecular weight (M_n) for poly (allyl acetate) of 1280. The infrared spectra of the monomer and the polymer are presented in Figure 2.

Allyl Isothiocyanate

The preparation of allyl isothiocyanate differed from that of the previous monomers only in distillation which was under reduced pressure. Only two yield determinations were made. These results are given in Table V.

The polymer produced was red in color, and hard and brittle in structure. Small amounts of this material were



Top: Spectrum of Allyl Acetate
Bottom: Spectrum of Poly (Allyl Acetate)

<u>INFRARED SPECTRA</u>

FIGURE 2

TABLE V

ALLYL ISOTHIOCYANATE

Sample No.	Dose Rate (Rad/hr.)	Total Dose (Megarad)	Yield (%)	Yield/Megarad (%/MR)
1	3.17×10^4	3.71	0.246	0.066
3	2.43×10^5	166	8.37	0.050

chipped from the evaporation flask and placed in the various solvents shown in Table II. As can be seen in the table, only dimethylformamide would dissolve the polymer, and no nonsolvent for reprecipitation of the polymer was found. Due to the lack of suitable solvents, neither the molecular weight nor the infrared spectrum were obtained for this polymer.

Allyl Phenyl Ether

Preparation of this monomer was the same as for allyl isothiocyanate. The first two tubes removed after irradiation (3.71 and 6.38 X 10^6 rad) were used in attempts to separate monomers and polymer by vacuum techniques, but both were unsuccessful due to the low conversion rate and consequent lack of polymer. The third sample (247 X 10^6 rad) was used for solution studies wherein it was found, as shown in Table II, that the polymer will precipitate from the irradiated solution with the addition of hexane or ethanol.

The poly (allyl phenylether) was precipitated from the fourth sample and the following information obtained:

Dose rate = 2.41 X 10^5 rad/hr
Total dose = 269 X 10^6 rad
Yield = 1.81%
Yield/MR = 0.0068 %/MR
Color of irradiated material = canary yellow
Color of polymer = brown

The polymer was taken up in benzene and used for the determination of molecular weight - $\bar{M}_n = 1380$.

DISCUSSION

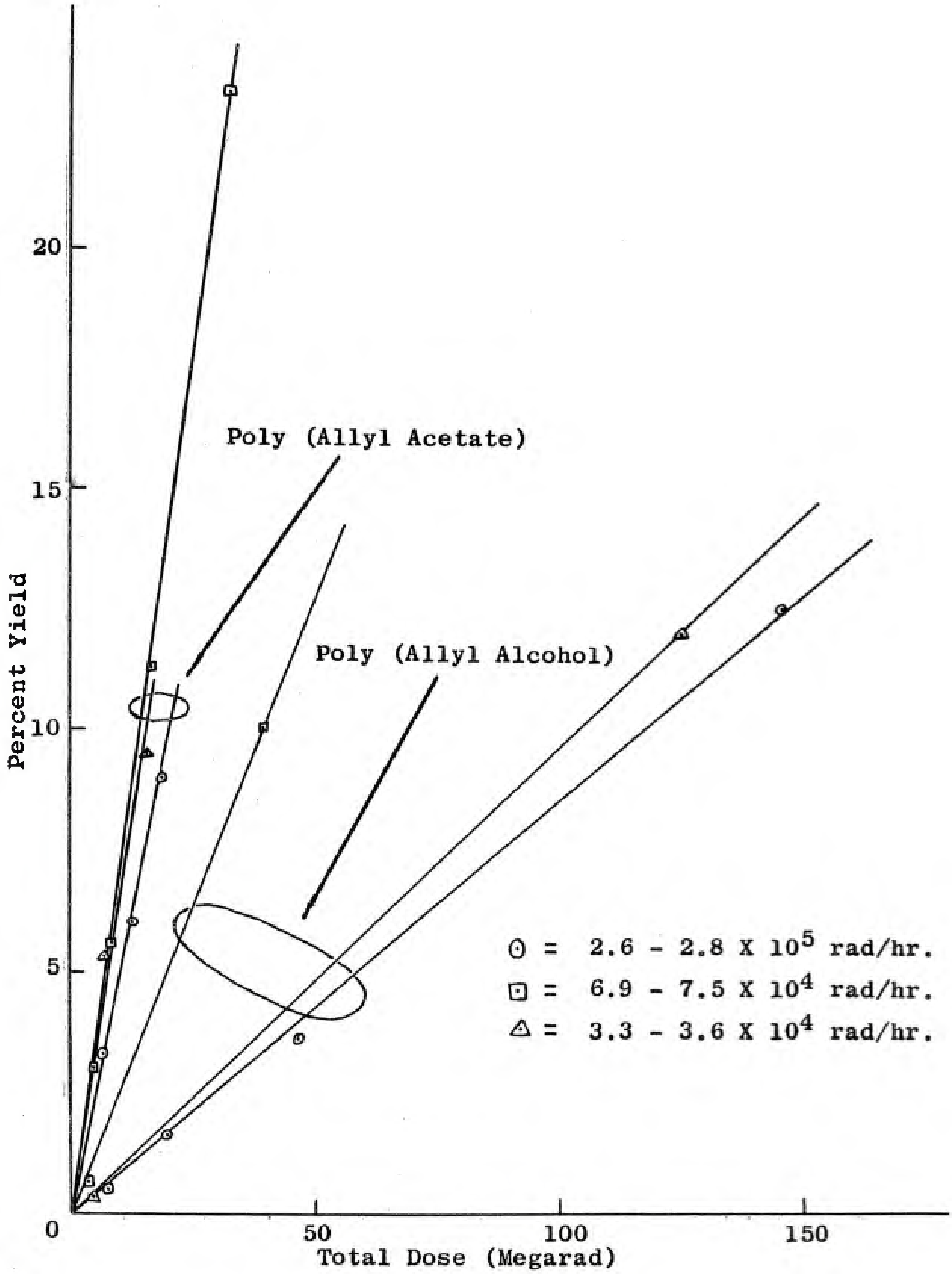
The infrared spectra of the monomers and what were assumed to be polymers were obtained in an attempt to determine whether or not polymers actually had been produced.

There is complete agreement between the spectrum of poly (allyl alcohol) produced by radiation in this laboratory and that chemically produced by Schulz and Elzer(13). A comparison of the monomer-polymer spectra shows the disappearance of absorption bands at 6.1, 10.2 and 10.9 microns for poly (allyl alcohol), Figure 1, and at 6.1, 10.1 and 10.8 microns for poly (allyl acetate), Figure 2. The disappearance of these bands, which are characteristic of the double band in allyl monomers(14), also indicates that polymers were formed. (The discontinuity at 7.5 microns in the allyl acetate spectrum was due to a change in sample thickness required to resolve the longer wave lengths).

Figure 3 is a plot of the yield data given in Tables I and II. It should be noted that irradiation at dose rates of 3.3 - 3.6 X 10^4 (△) and 2.6 - 2.8 X 10^5 rad/hr (⊙) were carried out in the kilocurie source at its ambient temperature. A comparison of the curves at these dose rates indicates that yield varies inversely with dose rate. The curve for allyl alcohol which was irradiated at 70 C at a dose rate of 7.5 X 10^4 rad/hr. in the multicurie source (⊡) clearly shows that yield varies directly with temperature. This temperature dependence could explain the anomalous behavior of allyl acetate where the conversion rate is higher at 6.9 X 10^4 rad/hr. (⊡) than at 3.3 X 10^4 rad/hr. since the ambient temperature of the multicurie source was higher than that of the kilocurie source.

Two of the four polymers produced, poly (allyl acetate) and poly (allyl phenyl ether), exhibited elastic properties. The poly (allyl acetate), which was crosslinked by excess radiation was very tough and difficult to separate from the glass tube in which it was irradiated. The two polymers, when precipitated from solution, were quite viscous liquids. When "strings" were pulled horizontally from the polymer surface and allowed to go slack, they pulled back up to a horizontal position indicating some degree of elasticity. No attempt was made to crosslink the poly (allyl phenyl ether).

The molecular weight of 1280 for poly (allyl acetate) agrees fairly well with the dgree of polymerization of 13.7 ± 0.4 reported by Bartlett and Altschul(15) for this polymer. Everard(5) did not report a molecular weight for poly (allyl phenylether), but did state that the polymer was brown which is in agreement with the results obtained in this laboratory. The molecular weights of 1380 determined for poly (allyl phenyl ether) and 1280 for poly (allyl acetate) are low as compared with most elastomers, but irradiation at higher temperatures and/or lower dose rates



CONVERSION RATES OF MONOMERS

FIGURE 3

might produce higher molecular weight and more elastic polymers.

The objective of this investigation was not attained in its entirety due to the fact that molecular weights were not determined for two of the four polymers produced. However, polymerization rates were determined as planned and, in addition, data on solubilities and infrared spectra were obtained.

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1. Polymerization
2. Allyl Monomers
3. Gamma Irradiation

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