silicate. The soluble silver bromide complex solution was made by dissolving 10 g silver bromide (*CP* grade) in 50 ml. hydrobromic acid (6 normal). Crystals of silver bromide belonging to the cubic system became visible after about an hour. Their maximum size was in the order of 0·8 to 1·0 mm and was attained within 8 to 10 days.

As noted, for the growth of silver chloride crystals, best results were obtained by decomposing silver chloride ammonia complexes. Gels were prepared by mixing equal volumes of sodium-silicate solution (specific gravity 1·06) and 2·9 normal hydrochloric acid. After gelation they were covered with a top solution prepared by dissolving 10 g silver chloride in 50 ml. ammonium hydroxide (8.33 normal). After 10 to 12 days crystals of up to 5–8 mm in edge were obtained. Most of the crystals were octahedral or truncated tetrahedral forms.

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Structural Changes of Barium Methacrylate Dihydrate under X-radiation

Schneller and Flanagan[1] have discussed the solid state decomposition of silver nitrite after irradiation with gamma rays. They noted that similar results were obtained by Boldyrev and Eroshkin[2], who irradiated the compound with X-rays. We report similar work on the solid state polymerization of barium methacrylate dihydrate.

We have examined the structural decomposition of a sample of barium methacrylate dihydrate continuously irradiated with X-radiation. The experiment was performed on a finely powdered sample of barium methacrylate dihydrate left open to the atmosphere and mounted in a standard aluminium flat sample holder in a Philips *PW* 1051 wide angle powder diffractometer. A standard size collimator with anti-scatter slits of 1° diversion and an 0·1 mm receiving slit was used. Throughout the experiment the temperature remained constant at 25° C.

The kinetics were followed by measuring the decrease in the intensity of three powder diffraction peaks (100, 200, 002). The peaks were scanned at a speed of 0·125°/min at regular intervals of time and between scans the machine was left oscillating through the 2θ range covering all three peaks. Intensities were estimated by weighing cut-out tracings of the diffracted peaks. The investigation was carried out at three different dose rates achieved by using: (*a*) nickel filtered copper radiation ($\lambda = 1{\cdot}5418$ Å) at 40 kV and 6 m.amp; (*b*) nickel filtered copper radiation at 40 kV and 20 m.amp; and (*c*) unfiltered copper radiation at 40 kV and 20 m.amp with a nickel filter placed between the sample and counter. The estimated dose rate (private communication from the Australian AEC Dosimetry Service) for each of these conditions is 69,500 rad/h, 205,000 rad/h and 556,000 rad/h, respectively. The results are shown in Fig. 1.

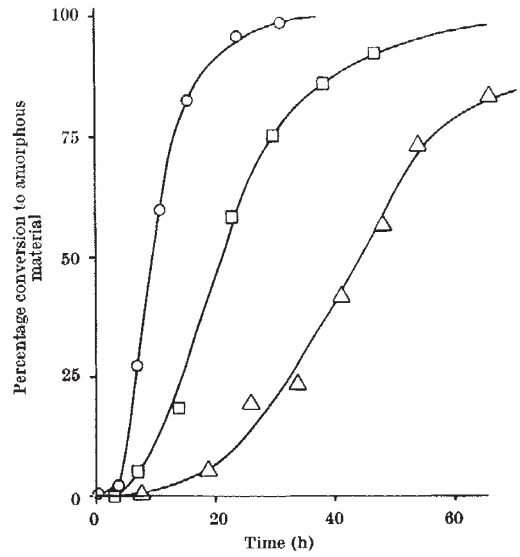

Fig. 1. Dose rate dependence of conversion of crystalline barium methacrylate dihydrate to amorphous material during continuous X-irradiation at 25° C. Mean lines for 200 and 002 reflexions. Dose rates: ○, 556,000 rad/h; □, 205,000 rad/h; △, 69,500 rad/h.

O'Donnell, McGarvey and Morawetz[3] have postulated, from electron spin resonance measurements, a free radical mechanism for the polymerization of barium methacrylate dihydrate induced by gamma radiation. Subsequent gravimetric determinations (results by J. H. O'Donnell and M. J. Bowden, to be published) of the kinetics of this polymerization show an expected increase in rate with an increase in the intensity of the irradiating beam. Our results show a marked similarity to those obtained by the gravimetric separation of polymer formed during the irradiation barium methacrylate dihydrate with gamma rays. As the large absorption coefficient of the compound results in a half-thickness of 20μ for the X-ray beam, the observed effect is a surface one and the comparison is only qualitative.

In view of the results of Schneller and Flanagan[1] and Boldyrev and Eroshkin[2], we feel that the comparison of our results with those found in the gamma ray polymerization of barium methacrylate dihydrate is justified, and that the observed structural decomposition is caused by polymer being formed. Further developments in the method we have outlined could provide a means of studying the kinetics of solid state polymerization *in situ*, thus overcoming a number of errors inherent in the normal gravimetric analysis techniques.

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