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Technique for Increasing Yield of Trifluoronitrosomethane — Tetrafluoroethylene Copolymer

It has been demonstrated that an aqueous suspension polymerization technique using equimolar amounts of trifluoronitrosomethane (CF_3NO) and tetrafluoroethylene (C_2F_4) will provide a 68-percent yield of a 1 : 1 copolymer ($\text{CF}_3\text{NO}:\text{C}_2\text{F}_4$).

A persulfate-bisulfite redox system was investigated for polymerization of equimolar amounts of CF_3NO and C_2F_4 in aqueous lithium bromide in an attempt to incorporate more than an equimolar amount of C_2F_4 into the copolymer. The reaction was allowed to proceed for 48 hours at -35°C , and then for 72 hours at 10°C . The gum copolymer of $\text{CF}_3\text{NO}-\text{C}_2\text{F}_4$ which was isolated was found to represent only a 49 percent conversion. In subsequent experiments, a 50-percent excess of C_2F_4 was used. Yields were markedly increased, and as much as 90 percent of the copolymer was formed; moreover, the products displayed less tack than those produced by equimolar ratios of principal reactants. Details of the procedure are given in the following paragraph.

A 250-ml heavy-walled glass reactor was charged with 70 ml of 35 percent (by weight) aqueous lithium bromide, 2.0 g magnesium carbonate, 0.005 g sodium persulfate, and 0.005 g sodium bisulfite. Ten-gram portions of CF_3NO (0.10 mole) and 15.0-g portions of C_2F_4 (0.15 mole) were added successively to the polymerization vessel; the vessel was kept cool at liquid nitrogen temperature. Transfers of the monomers were made by expanding the gases into a calibrated volume at a measured pressure, followed by condensation at liquid nitrogen temperature. The

sealed reaction vessel was placed in a refrigerated bath (-35°C) for 48 hours; then the temperature of the vessel and contents was allowed to rise to 10°C , kept at 10°C for 72 hours, and finally cooled to -35°C . During the reaction period, the mixture was agitated by shaking the vessel. The vessel was opened cautiously and the suspension of the copolymer was allowed to settle. Then the supernatant liquid was decanted; the precipitate was washed with water and treated with 6N hydrochloric acid until no further reaction was noted. The isolated gum was washed free of acid and then dried under vacuum at 50°C .

Notes:

1. No homopolymerization occurs when C_2F_4 is subjected to the reaction conditions described above.
2. Requests for further information may be directed to:

Technology Utilization Officer
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Reference: TSP 72-10418

Patent status:

NASA has decided not to apply for a patent.

Source: Samuel A. Glickman of
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