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Hot-Atom Chemistry of Bromine. III.[1] The Use of Bromides and Bromate in an Attempt at the Recoil-Synthesis of Two Organic Radiobromides in Solid Mixture Systems

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Since the early study of recoil syntheses with organic bromides in organic solutions,[2] many experiments on the recoil syntheses of ^{82}Br-labeled organic bromides have been undertaken in the liquid phase. A few, however, have been done in solid phase It has previously been reported that the radiative neutron capture and the isomeric transition produce bond disruption in irradiated aqueous bromate[3] and that the radiobromine-82 so produced in a pyrenebromanil-molecular compound could combine with the pyrene ring to produce ^{82}Br-labeled pyrene.[1] The present note describes an attempt at the recoil labeling of the ^{82}Br atoms in pyrene or *p*-benzoquinone, with potassium bromate, bromide or ammonium bromide used as the bromine source.

Experimental

The samples were composed of the organic target and the bromine source (in a molar ratio of 1 : 4[4]); the ingredients were mixed by pestling them together

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[1] Part II: C. Shinomiya *et al.*, This Bulletin, **42** (8), (1969), in press.

[2] E. Glückauf and J. W. J. Fay, *J. Chem. Soc.*, **1936**, 390.

[3] C. Shinomiya, A. Oyoshi and T. Kishikawa, This Bulletin, **42**, 1437 (1969).

[4] The equal ratio of the quinone ring: bromine in bromanil reported in the previous paper.[1]

on an agate motar. They were then irradiated at the reactor temperature (*ca.* 40°C) in a pneumatic tube of KUR[5] for 1—80 min. The thermal neutron flux and the accompanying gamma-dose rate at 1 MW operation were 5×10^{12} n/cm²/sec and 2.7×10^{7} R/hr respectively. After standing for more than 2 hr[6] at room temperature, the samples were extracted into a two-phase mixture of a carbon disulfide solution of bromine and an aqueous mixture of potassium bromide and bromate; radioactivity measurements were then made with respect to the ^{82}Br activities. The organic yield was calculated in the manner described in a previous paper.[1]

Results

Table 1 shows the organic yields in the irradiated pyrene or *p*-benzoquinone and bromine compound mixtures at various irradiation times. No definite dependency of the irradiation time on the organic yield was established. In these heterogeneous mixtures a chance for a recoiled atom to escape from the bromide or bromate crystal may be small, and only the atoms recoiled at or near the surface of the crystal may be stabilized in both the organic crystal and the parent one. No dependency of the irradiation time can be supported by this explanation.

TABLE 1. ORGANIC YIELDS IN IRRADIATED MIXTURES

Bromine source	Irrad time min	Organic yield, % in organic targets	
		Pyrene	*p*-Benzoquinone
NH_4Br	20	4.4	2.1
KBr	1	0.7	1.3
	5	0.4	0.4
	10	2.7	3.3
	20	3.2	1.9
	30	3.6	4.3
$KBrO_3$	1	1.0	3.3
	5	2.4	4.4
	10	1.8	2.0
	30	3.7	2.2
	80	1.6	3.9

Ammonium bromide was considered to be more effective than potassium bromide on the organic yields in both pyrene and *p*-benzoquinone. Bromate was effective for a short period of irradiation, and bromide, for a long period.

The authors wish to express their appreciation to Dr. Shiro Iwata, Dr. Yoshiyuki Kiso, and the personnel of the Research Reactor Institute, Kyoto University, for their assistance in the irradiation.

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[5] Kyoto University Reactor.

[6] The isomeric transition reaction of ^{82m}Br (half-life *ca.* 6 min) was completed during the time; O. U. Anders, *Phys. Rev.*, **138**, B1 (1965); J. F. Emery, *J. Inorg. Nucl. Chem.*, **27**, 903 (1965).