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## Photochemical Reactions of Acyl Iodides with Aryl Halides

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**Abstract**—Photochemical reactions of acyl iodides RC(O)I (R = Me, Ph) with aryl halides, fluoro-, chloro-, and bromobenzenes, 1,4-dibromobenzene, 2- and 3-bromotoluenes, and 4-bromo-1,2-dimethylbenzene, were studied. Acetyl iodide reacted with chloro- and bromobenzenes and 1,4-dibromobenzene according to the exchange pattern to give iodobenzene and 1,4-diiodobenzene, respectively. No halogen exchange was observed in the reactions of acetyl iodide with fluorobenzene and hexafluorobenzene. Benzoyl iodide failed to react with chloro- and brombenzene under UV irradiation but underwent polycondensation with formation of black nonfusible oligomers which were found to possess paramagnetic and semiconducting properties. Ultraviolet irradiation of a mixture of MeCOI with 2- or 3-bromotoluene, as well as with 4-bromo-1,2-dimethylbenzene, also led to the formation of polymeric products as a result of polycondensation of aryl iodides formed initially via replacement of bromine by iodine. Irradiation of benzoyl iodide in 2- or 3-bromotoluene involved recombination of benzoyl radicals to give benzil as the only product.

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We previously found that irradiation of acyl iodides in arenes induces photochemical Friedel–Crafts reaction [1]. For example, *o*- and *m*-xylenes with enhanced polarity of the aromatic ring are smoothly acylated with acetyl iodide under photochemical conditions. Acylation of the aromatic ring was the only reaction path in the photolysis of trimethylacetyl and valeryl iodides in toluene. We anticipated analogous process to occur under UV irradiation of mixtures of acyl iodides RC(O)I (R = Me, Ph) with aryl halides, fluoro-, chloro-, and bromobenzenes, 1,4-dibromobenzene, 2- and 3-bromotoluenes, and 4-bromo-1,2-dimethylbenzene.

However, the photolysis of acetyl iodide in chlorobenzene gave no expected acylation product, 4-chloroacetophenone  $MeCOC_6H_4Cl-4$ . After UV irradiation of an equimolar mixture of acetyl iodide and chlorobenzene over a period of 29 h, we isolated acetyl chloride, biacetyl, and iodobenzene (Scheme 1). In addition, a black powder-like nonfusible oligomer 1 with a layered structure was isolated. Oligomer 1 was insoluble in water and common organic solvents, and its elemental composition corresponded to the formula  $\sim C_{78}H_{53}I$  (C/H ratio 1.5).



Likewise, photochemical reaction of acetyl iodide with an equimolar amount of bromobenzene in 50 h gave acetyl bromide, iodobenzene (9%), and biacetyl (16%). In this case, oligomeric product **2** with the composition  $C_{16}H_{13}I$  was also isolated. Oligomers **1** and **2** are products of photochemical polyphenylation of PhI formed in the first step; they were assigned the formulas  $H(C_6H_4)_{13}I$  and  $H(C_6H_4)_3I$ , respectively.

The reactions of acetyl iodide with chloro- and bromobenzene are photoinduced aromatic radical substitution processes. The source of primary radicals is MeCOI, as follows from the presence in the reaction mixture of biacetyl which is the product of combination of acetyl radicals.

Unlike bromobenzene, the reaction of acetyl iodide with 1,4-dibromobenzene afforded exclusively 1,4-diiodobenzene (yield 74%) and acetyl bromide (Scheme 2).



Ultraviolet irradiation of a mixture of acetyl iodide and fluorobenzene or hexafluorobenzene gave neither halogen exchange products nor biacetyl. These findings indicated that fluorine-substituted benzenes inhibit photochemical generation of radicals and their subsequent reactions.

No iodobenzene was formed in the photolysis of benzoyl iodide in chloro- or bromobenzene. Here, the initial halobenzene remained unchanged, whereas benzoyl iodide was converted into black oligomers **3** (in chlorobenzene) and **4** (in bromobenzene) whose compositions were estimated at  $C_{20}H_{11}O_2I$  and  $C_{14}H_7O_2I$ , respectively.

The elemental compositions of **3** and **4** and their IR spectra which contained absorption bands at 1688 and

 Table 1. Electric conductivities, parameters of ESR spectra, and relaxation parameters of polymers 3 and 4

Parameter		3	4
σ, S/cm		$2.8 \times 10^{-9}$	$1.3 \times 10^{-8}$
N, spin/g		$1.8 \times 10^{19}$	$1.9 \times 10^{19}$
$\Delta H, G$		7.4	7.3
g-Factor		2.00497	2.00542
Asymmetry factor		0.80	0.77
A/B			
$T_1$ , <sup>a</sup> ns	$T'_1$	2940.0	858.8
	$T_1''$	8913.0	3720.2
$T_2$ , <sup>a</sup> ns	$T'_2$	_	2.61
	$T_2''$	132.5	126.4

<sup>a</sup> Spin–lattice relaxation time  $T_1$  and spin–spin relaxation time  $T_2$  characterize the lifetime of an electron spin in the excited state.

1686 cm<sup>-1</sup>, respectively, indicated the presence in their molecules of a carbonyl group conjugated with a benzene ring. Presumably, oligomers **3** and **4** are products of photochemical polycondensation of benzoyl iodide via its autoacylation accompanied by photochemical iodination of the aromatic ring. Their structure may be represented by the following constitutional repeating units:  $-C_6H_4C(O)C_6H_4C(O)C_6H_3I-$  (**3**) and  $-C_6H_4C(O)C_6H_3C(O)I-$  (**4**).

The data of stationary ESR showed that oligomers 3 and 4 are fairly similar to polymers isolated previously in the photolysis of acetyl iodide in benzene or toluene [2]. They exhibited pronounced paramagnetic properties. Their ESR spectra in the g-factor region displayed sharp asymmetric signals typical of free radicals. The upper part of the ESR lines conforms to the Lorentz shape, and the lower part, to the Gauss curve, which suggests their irregular structure. The concentration of paramagnetic centers amounts to  $10^{19}$  spin/g (Table 1). However, the g-factors characterizing the contribution of spin-orbit interactions were appreciably different ( $\Delta g = 0.00045$ ). The electric conductivity of oligomers 3 and 4 is lower by an order of magnitude than that of the polymers isolated previously [2], despite a high iodine content. Presumably, unlike the polymers described in [2] which were doped with iodine, iodine atoms in 3 and 4 are covalently bound to macromolecules. The concentration of unpaired electrons and signal width (less than 10 G) in the ESR spectra of 3 and 4 (which characterize environment of paramagnetic centers) make it possible to record spin echo in the pulsed mode at room temperature and determine relaxation parameters  $T_1$  and  $T_2$ . The spin-spin relaxation time  $T_2$  was much shorter than the spin-lattice relaxation time  $T_1$ , which is typical of dilute paramagnetics [3]. The relaxation time of 3 and 4 is determined by two contributions, which indicates a complicated character of interaction between unpaired electrons and their environment and requires special study.

Polymeric products **5–10** were also obtained by UV irradiation of mixtures of acetyl iodide with bromoarenes (2- and 3-bromotoluenes and 4-bromo-1,2-dimethylbenzene). No biacetyl was detected in the reaction mixtures. According to the data of elemental analysis and IR spectroscopy, polymers **5–10** lack carbonyl group. This means that acylation of the aromatic ring does not occur, as in the photolysis of acetyl iodide in halobenzenes. Compounds **5–10** are formed as a result of photochemical polycondensation of intermediate iodo-substituted arenes generated in the first step of the photochemical process via halogen exchange in the initial bromoarenes (Scheme 3).

Scheme 3.  

$$n \operatorname{MeC}(O)I + n \operatorname{BrC}_{6}H_{5-m}\operatorname{Me}_{m}$$
  
 $\xrightarrow{hv} [n \operatorname{IC}_{6}H_{5-m}\operatorname{Me}_{m}] \xrightarrow{hv} \operatorname{H}[\operatorname{C}_{6}H_{5-m}\operatorname{Me}_{m}]_{n}I$   
 $n = 1, 2; m = 1, 2.$ 

Increase of the UV irradiation time in the reaction of acetyl iodide with bromoarenes leads to reduced concentration of iodine in the polymeric products, presumably due to profound polycondensation. In the reaction of acetyl iodide with 2- or 3-bromotoluene increase of the irradiation time from 20 to 50 h changes the polymer composition from  $C_{13}H_{13}I$  (5) to  $C_{45}H_{42}I$  (6) and from  $C_{15}H_{14}I$  (7) to  $C_{30}H_{26}I$  (8). Analogous propagation of the polyarylene chain was observed in the photolysis of acetyl iodide in 4-bromo-1,2-dimethylbenzene. In this case, the resulting polymers had the compositions  $C_{32}H_{34}I$  (9, 20 h) and  $C_{55}H_{41}I$  (10, 50 h). Polymers 5–10 were isolated as black nonfusible powders which were insoluble in water and common organic solvents. According to the differential thermal analysis data, polymers 5-10 are stable up to 200-320°C. All compounds 5-10 are characterized by gradual lowering of the weight loss curves on heating to 900°C, and in all cases a weak exothermic effect was observed at 88-90°C, presumably due to elimination of iodine. These data indicate that variation of the conditions, namely increase of the UV irradiation time from 20 to 50 h, leads to formation of more thermally stable polymers 6, 8, and 10 which

start to decompose (10% weight loss) at  $330-420^{\circ}$ C against 255-318°C for 5, 7, and 9. Samples 5-8 obtained by the photochemical reactions of acetyl iodide with *o*- and *m*-bromotoluenes showed similar thermal stabilities (up to  $200-235^{\circ}$ C). Polymers 9 and 10 synthesized from acetyl iodide and 4-bromo-1,2-dimethylbenzene were more thermally stable, and they began to decompose at a temperature higher by 118–120°C than that found for polymers 5-8.

The results of ESR study of polymers **5–10** and their electric conductivities are given in Table 2. It is seen that compounds **5–10** are semiconductors. Their ESR parameters are very similar. Polymers **5–10** exhibit pronounced paramagnetic properties and are characterized by narrow, almost symmetric ESR signals typical of free radicals. The line shape is either mixed or approaching the Lorentz shape. The concentration of paramagnetic centers was estimated at  $10^{19}$  spin/g. All polymers obtained after 50-h irradiation showed narrower ESR signals and increased concentration of the conductivity. Thus not all paramagnetic centers generated by irradiation ensure electric conductivity.

Unlike acetyl iodide, the photolysis of benzoyl iodide in 2- and 3-bromotoluene gave benzil as the only product as a result of combination of benzoyl radicals (Scheme 4).

To conclude, we have demonstrated the possibility for using acyl iodides in photochemical reactions with haloarenes to obtain iodoarenes and iodine-containing oligomers and polymers possessing paramagnetic and semiconducting properties. Our results confirm once

Table 2. Parameters of the ESR spectra and electric conductivities of polymers 5–10

Compound no.	Reactants, reaction time	g-Factor	ΔH, G	<i>N</i> , spin/g	Asymmetry factor <i>A</i> / <i>B</i>	Line shape	Electric conductivity, S/cm
5	MeCOI– <i>o</i> -bromotoluene, 20 h	2.0031	7.1	$1.4 \times 10^{19}$	0.96	Mixed	$1.5 \times 10^{-9}$
6	MeCOI– <i>m</i> -bromotoluene, 20 h	2.0028	6.3	$9.4 \times 10^{18}$	0.99	Mixed	$3.6 \times 10^{-10}$
7	MeCOI– <i>o</i> -bromotoluene, 50 h	2.0028	6.0	$3.0 \times 10^{19}$	1.0	Mixed	$7.1 \times 10^{-8}$
8	MeCOI– <i>m</i> -bromotoluene, 50 h	2.0024	5.3	2.0×10 <sup>19</sup>	1.02	Mixed, closer to Lorentz	$1.7 \times 10^{-11}$
9	MeCOI-4-bromo-1,2-di- methylbenzene, 20 h	2.0024	5.2	$2.8 \times 10^{19}$	1.02	Close to purely Lorentz	$1.7 \times 10^{-9}$
10	MeCOI-4-bromo-1,2-di- methylbenzene, 50 h	2.0026	5.0	$3.5 \times 10^{19}$	1.0	Mixed, closer to Lorentz	$7.1 \times 10^{-10}$



more a wide synthetic potential of acyl iodides as reagents and synthons in organic synthesis [4–7]. In addition, appreciable dependence of the reaction path on the arene and acyl iodide nature has been observed in their photochemical transformations (cf. [2]).

## **EXPERIMENTAL**

Photochemical reactions were carried out under irradiation with an OKN-11 mercury lamp. The IR spectra were recorded on a UR-20 spectrometer from thin films. The ESR spectra were obtained on a Radiopan SE/X-2547 spectrometer equipped with a magnetometer and a high-frequency meter (sensitivity  $5 \times 10^{10}$  spin/g, room temperature), as well as on a Bruker Elexsys E-580 radiospectrometer in continuous and pulsed modes at room temperature. The concentrations of paramagnetic centers were calculated according to the procedure described in [8] using DPPH as calibration standard and nomogram for double integration of the first derivative curve [9]. The relaxation times  $T_1$  and  $T_2$  were determined with the aid of the following pulse sequences:  $\pi$ -*T*- $\pi/2$ - $\tau$ - $\pi$  for  $T_1$  and  $\pi/2-\tau-\pi$  for  $T_2$ , where  $\pi/2 = 20$  ns,  $\tau = 200$  ns, and T = 1000 ns [10]. Thermogravimetric analysis was performed on a MOM Q-1500 derivatograph (Hungary) in air (maximal temperature 900°C, heating rate 10 deg/min; DTA sensitivity 1/10).

**Reaction of acetyl iodide with chlorobenzene.** A mixture of 8.5 g (50 mmol) of acetyl iodide and 5.6 g (50 mmol) of chlorobenzene was irradiated with UV light over a period of 29 h. Distillation of the mixture under atmospheric pressure gave 25% of acetyl chloride, bp 50–51°C,  $n_D^{20} = 1.389$  (bp 52°C,  $n_D^{20} = 1.389$  [11]); 14% of biacetyl, bp 88–89°C,  $n_D^{20} = 1.3950$  (bp 88,  $n_D^{20} = 1.3950$  [11]); and 8% of iodobenzene, bp 186–187°C,  $n_D^{20} = 1.6200$  (bp 188°C,  $n_D^{20} = 1.6200$  [11]). Found, %: I 63.17. C<sub>6</sub>H<sub>5</sub>I. Calculated, %: I 62.20.

After distillation of liquid products, the residue was washed with acetone and dried in a vacuum desiccator to obtain black nonfusible polymer 1, which was insoluble in water and common organic solvents. Found, %: C 81.4; H 5.1; I 11.3; C/H ratio 1.5.  $C_{78}H_{53}I$ . Calculated, %: C 83.8; H 4.7; I 11.4.

**Reaction of acetyl iodide with bromobenzene.** A mixture of 8.5 g (50 mmol) of acetyl iodide and 7.9 g (50 mmol) of bromobenzene was irradiated with UV light over a period of 50 h. Distillation of the mixture under atmospheric pressure gave 20% of acetyl bromide, bp 78–79°C,  $n_D^{20} = 1.4520$  (bp 75–77°C,  $n_D^{20} =$ 1.450 [11]), 9% of iodobenzene, and 16% of biacetyl. The solid residue was washed with acetone and diethyl ether to obtain polymer **2**. Found, %: C 58.8; H 3.1; I 37.8. C<sub>16</sub>H<sub>13</sub>I. Calculated, %: C 57.8; H 3.9; I 38.2.

**Photolysis of benzoyl iodide in chlorobenzene.** A mixture of 11.6 g (50 mmol) of benzoyl iodide and 5.6 g (50 mmol) of chlorobenzene was irradiated with UV light over a period of 50 h. After removal of chlorobenzene by distillation, the residue was washed with acetone and diethyl ether and dried in a vacuum desiccator. The product was powder-like nonfusible polymer **3** which was insoluble in water and common organic solvents. IR spectrum, v, cm<sup>-1</sup>: 3070, 3016 (C–H<sub>arom</sub>), 1686 (C=O), 1601, 1452, 1418 (C–C<sub>arom</sub>), 932, 707, 682 ( $\delta$ C–H<sub>arom</sub>), 545 (C–I), 430 ( $\delta$ C–C). Found, %: C 56.7; H 2.7; I 31.1. C<sub>20</sub>H<sub>11</sub>O<sub>2</sub>I. Calculated, %: C 58.5; H 2.7; I 30.9.

Photolysis of benzoyl iodide in bromobenzene. A mixture of 11.6 g (50 mmol) of benzoyl iodide and 7.9 g (50 mmol) of bromobenzene was irradiated with UV light over a period of 50 h. Distillation of the mixture under atmospheric pressure gave initial bromobenzene. The residue was treated as described above to isolate polymer **4** whose properties were similar to those of **3**. IR spectrum, v, cm<sup>-1</sup>: 3070, 3023 (C–H<sub>arom</sub>), 1688 (C=O), 1602, 1453, 1423 (C–C<sub>arom</sub>), 934, 707, 683 ( $\delta$ C–H<sub>arom</sub>), 551 (C–I), 430 ( $\delta$ C–C). Found, %: C 49.1; H 2.6; I 37.3. C<sub>14</sub>H<sub>7</sub>O<sub>2</sub>I. Calculated, %: C 50.3; H 2.1; I 38.2.

Photolysis of acetyl iodide in 2-bromotoluene. A mixture of 8.5 g (50 mmol) of acetyl iodide and 8.6 g (50 mmol) of 2-bromotoluene was irradiated with UV light over a period of 20 h. Distillation of the mixture gave acetyl bromide. The residue was washed with acetone and diethyl ether and dried in a vacuum desiccator to isolate black powder-like polymeric product 5. IR spectrum, v, cm<sup>-1</sup>: 3436 (C–H<sub>arom</sub>), 2920, 2853 (C–H<sub>aliph</sub>), 1627, 1434 (C–C<sub>arom</sub>), 1027, 868, 728 ( $\delta$ C–H), 618 (C–I), 463 ( $\delta$ C–C). Found, %: C 52.12; H 4.45; I 42.74. C<sub>13</sub>H<sub>13</sub>I. Calculated, %: C 52.70; H 4.39; I 42.94.

Irradiation of a mixture of the same amounts of acetyl iodide and 2-bromotoluene over a period of 50 h, followed by distillation of acetyl bromide, gave polymeric product **6** whose properties were similar to those of **5**. IR spectrum, v, cm<sup>-1</sup>: 3436 (C–H<sub>arom</sub>), 2918, 2850 (C–H<sub>aliph</sub>), 1628, 1440 (C–C<sub>arom</sub>), 1029, 867, 728 ( $\delta$ C–H), 587 (C–I), 463 ( $\delta$ C–C). Found, %: C 75.47; H 5.57; I 15.89. C<sub>45</sub>H<sub>42</sub>I. Calculated, %: C 76.16; H 5.92; I 17.91.

Photolysis of acetyl iodide in 3-bromotoluene. A mixture of 8.5 g (50 mmol) of acetyl iodide and 8.6 g (50 mmol) of 3-bromotoluene was irradiated with UV light over a period of 20 h. Distillation of the mixture gave acetyl bromide and unreacted acetyl iodide and 3-bromotoluene (28–30% of the initial amount). The residue was washed with acetone and diethyl ether to obtain black powder-like polymeric product 7. IR spectrum, v, cm<sup>-1</sup>: 3440 (C–H<sub>arom</sub>), 2910, 2851 (C–H<sub>aliph</sub>), 1625, 1439 (C–C<sub>arom</sub>), 1030, 863, 729 ( $\delta$ C–H), 587 (C–I), 464 ( $\delta$ C–C). Found, %: C 56.07; H 4.6; I 42.49. C<sub>15</sub>H<sub>14</sub>I. Calculated, %: C 56.07; H 4.36; I 39.56.

Likewise, after irradiation of a mixture of 17 g (100 mmol) of acetyl iodide and 17.2 g (100 mmol) of 2-bromotoluene over a period of 50 h, we isolated polymer **8** whose properties were similar to those of 7. IR spectrum, v, cm<sup>-1</sup>: 3440 (C–H<sub>arom</sub>), 2918, 2852 (C–H<sub>aliph</sub>), 1587, 1424 (C–C<sub>arom</sub>), 1038, 865, 759 ( $\delta$ C–H), 528 (C–I), 448 ( $\delta$ C–C). Found, %: C 72.93; H 4.88; I 25.30. C<sub>30</sub>H<sub>26</sub>I. Calculated, %: C 70.17; H 5.06; I 24.75.

Photolysis of acetyl iodide in 4-bromo-1,2-dimethylbenzene. A mixture of 8.5 g (50 mmol) of acetyl iodide and 9.3 g (50 mmol) of 4-bromo-1,2dimethylbenzene was irradiated with UV light over a period of 20 h. Distillation of the mixture gave acetyl bromide and unreacted acetyl iodide and 4-bromo-1,2dimethylbenzene. The solid residue was washed with acetone and diethyl ether to obtain polymeric product **9** whose properties were similar to those of **5–8**. IR spectrum, v, cm<sup>-1</sup>: 3492 (C–H<sub>arom</sub>), 2916, 2855 (C–H<sub>aliph</sub>), 1591, 1444 (C–C<sub>arom</sub>), 1019, 867, 812 ( $\delta$ C–H), 528 (C–I), 442 ( $\delta$ C–C). Found, %: C 69.72; H 5.74; I 20.64. C<sub>32</sub>H<sub>34</sub>I. Calculated, %: C 70.45; H 6.23; I 23.30.

Irradiation of a mixture of the same amounts of the reactants over a period of 50 h gave polymeric product **10** which was analogous to **9**. IR spectrum,  $\nu$ , cm<sup>-1</sup>: 3428 (C–H<sub>arom</sub>), 2915, 2852 (C–H<sub>aliph</sub>), 1576, 1442 (C–C<sub>arom</sub>), 996, 864, 808 ( $\delta$ C–H), 512 (C–I), 437

(δC–C). Found, %: C 79.89; H 5.06; I 15.35. C<sub>55</sub>H<sub>41</sub>I. Calculated, %: C 79.71; H 4.95; I 15.33.

**Photolysis of benzoyl iodide in 2-bromotoluene.** Irradiation with UV light of a mixture of 11.6 g (50 mmol) of benzoyl iodide and 8.6 g (50 mmol) of *o*-bromotoluene over a period of 24 h gave 55% of benzil, mp 99–100°C (95–96°C [2]). IR spectrum: v 1687 cm<sup>-1</sup> (C=O) (cf. 1681 cm<sup>-1</sup> [12]). Likewise, 53% of benzil was isolated after UV irradiation of a mixture of benzoyl iodide and *m*-bromotoluene over a period of 24 h.

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