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Synthetic Communications: An International Journal for Rapid Communication of Synthetic Organic Chemistry

Publication details, including instructions for authors and subscription information: <u>http://www.tandfonline.com/loi/lsyc20</u>

Iodination of Aromatic Compounds with Iodine and n - Butyl- triphenylphosphonium Peroxodisulfate

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To cite this article: H. Tajik, A. A. Esmaeili, I. Mohammadpoor-Baltork, A. Ershadi & H. Tajmehri (2003) Iodination of Aromatic Compounds with Iodine and n-Butyl- triphenylphosphonium Peroxodisulfate, Synthetic Communications: An International Journal for Rapid Communication of Synthetic Organic Chemistry, 33:8, 1319-1323, DOI: <u>10.1081/SCC-120018691</u>

To link to this article: <u>http://dx.doi.org/10.1081/SCC-120018691</u>

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SYNTHETIC COMMUNICATIONS[®] Vol. 33, No. 8, pp. 1319–1323, 2003

Iodination of Aromatic Compounds with Iodine and *n*-Butyltriphenylphosphonium Peroxodisulfate

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ABSTRACT

Iodine and *n*-butyltriphenylphosphonium peroxodisulfate can be successfully used for the iodination of some aromatic compounds in acetonitrile as solvent. The yields obtained are good to excellent.

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Iodoarenes are useful compounds in organic synthesis. Because of the low reactivity of iodine, the general procedure for halogenation of aromatic compounds cannot be successfully used for preparation of aryl iodides. In order to obtain aryl iodides some procedures have been employed among them we can refer to the following; addition of an oxidizing agent^[1-7] or base,^[8,9] iodine monochloride^[10,11] (alone or in the presence of a Lewis acid), *N*-iodosuccinimide^[12] and metal iodideoxidizing agent systems.^[13–15] Nevertheless, most of them employ acidic or severe conditions. In most cases the yields of reactions are not high. Furthermore, there are not simple and reliable methods for their preparation. Recently, some new systems have been reported especially for direct iodination of aromatic compounds.^[16,17]

In this research we have chosen *n*-butyltriphenylphosphonium peroxodisulfate,^[18] [$(n-BuPPh_3)_2S_2O_8$], as oxidizing agent and molecular iodine to iodinate aryl ethers, phenols, and aromatic carboxylic acids as shown in Table 1.

According to the optained results, this method describes a mild and convenient way of iodinating aromatic rings. Aromatic ethers can be quantitatively iodinated by this system. The compounds which contain electron donating groups give para substituted products (Table 1). In some cases where the para position is blocked, no reaction was observed even after a long reaction time (Table 1, Entries 6,7). Aromatic compounds which are deactivated were also directly iodinated in meta position in relatively good yields (Table 1, Entries 8,11), which to the best of our knowledge was not reported in the literatures. Phenols and aromatic amines, however, gave mostly the oxidation products that was not intended in this research.

In all of the cases, parallel reactions were performed to check the oxidation products. According to the results obtained no oxidation product was observed for all of the substrate given in Table 1.

EXPERIMENTAL

All products were identified by comparison of their spectral and physical data with those of the known samples.^[19] The purity of the products was checked by TLC on silica gel polygram SIL G UV 254 plates. *n*-Butyltriphenylphosphonium peroxodisulfate was prepared according to Ref.^[18] Other chemicals were purchased from the Merck chemical company. Products were separated and identified by different chromatography techniques, and also identified by comparison

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Table 1. Iodination of some aromatic compounds with iodine and $(n-Bu PPh_3)_3S_2O_8$ in acetonitrile under reflux (substrate/iodine/oxidant; 1:1:1).

Entry	Substrate	Product	Reaction time (h)	Yield (%)
1	<i>С</i> ОМе	I-OMe	0.5	100
2	OMe OMe		0.4	100
3			0.5	100
4	-ОМе		0.5	100
5	ОН	он	2	100
6		_	20	_
7	Br-C-OMe	—	20	_
8	Сресоон	Соон	3	80
9	соон	соон	15	70
10	соон	соон	15	80
11			2	70

of their m.p., b.p., IR, and NMR spectra and refractive index with those reported for the authentic samples.

General procedure: To a solution of aromatic compound (1 mmol), iodine (1 mmol) and *n*-butyltriphenylphosphonium peroxodisulfate were added and refluxed for 0.25–20 h. Progress of the reactions was monitored by TLC or GC (capillary column). The reaction mixture was cooled to room temperature and the excess of iodine was removed by dropwise addition of sodium thiosulfate solution (1 M). The colorless solution was transferred to a separatory funnel and the organic layer was separated

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and dried with magnesium sulfate or calcium chloride. Evaporation of the solvent followed by column chromatography on silica gel gave the corresponding compounds from good to excellent yields (Table 1).

ACKNOWLEDGMENT

We are thankful of Guilan University Research Council for partial support of this work.

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Received in the USA February 4, 2002



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