



A Simple and Efficient Iodination of Aromatic Compounds Using I₂/Choline Chloride/K₂S₂O₈

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A simple and efficient method for the iodination of aromatic compounds has been achieved in the presence of molecular iodine, choline chloride and potassium peroxodisulfate at 65 °C in acetonitrile. The rate of conversion of aromatic compounds into iodoaromatic compounds was promoted by *in situ* formed choline peroxodisulfate. This protocol provides an efficient access to iodoarenes with operational simplicity, good functional group tolerance and a moderate to good product yield.

Keywords: Iodination, Choline chloride, Potassium peroxodisulfate, Choline peroxodisulfate, Molecular iodine.

INTRODUCTION

Aryl iodides are important class of compounds for the synthesis of various pharmaceutical and bioactive compounds [1]. A large amount of research have been devoted to the synthesis of iodoaromatic compounds, including electrophilic aromatic halogenations [2], the Sandmeyer reaction [3] or *ortho*-lithiation [4]. They are the most reactive intermediates for various cross-coupling reactions such as Heck reactions, Still and Negishi cross couplings reactions and especially useful for formation of carbon-carbon and carbon-heteroatom bonds in the preparation of ethylenic and acetylenic compounds on the basis of catalytic condensations [5]. Though direct iodination of arenes is logically more attractive, however, the iodofunctionalization of aromatics and heteroarenes is frequently not straightforward due to the weak electrophilic nature of the iodonium ion and relatively weak bond strength of C–I compared to C–Br and C–Cl. Therefore the direct iodination of arenes with molecular iodine under mild conditions requires an oxidizing agent to activate less active molecular iodine to the more reactive species with an I⁺ character. Many oxidizing agents have been considered for formation of I⁺ like species either *in situ* or as stable isolable intermediates. Some examples of iodinating agents are: I₂/CuBr₂ [6], CsI₃ [7], KF/I₂ [8], I₂/HIO₃/grinding [9], I₂/Bi(NO₃)₃·5H₂O–BiCl₃ [10], I₂/TMOF [11], NIS/In(OTf)₃ [12], I₂/silver nitrate [13], NIS/CF₃CO₂H [14], *N*-iodosaccharin [15], hydroxyl radicals/nanocrystalline CeO₂/I₂ [16], *N*-chlorosuccinimide and sodium iodide [17]. Recent methods for iodination of aromatic compounds includes the reactions catalyzed by NIS/Pd(OAc)₂ [18], KIO₃/K₂S₂O₈/

Pd(OAc)₂ [19], I₂/LiOtBu/1,10-phenanthroline [6], NIS/Ball milling [20], I₂/Pd(OAc)₂/CsOAc/NaHCO₃ [21].

However, most of the described reactions in the literature require one or some of the following conditions: an excess of the reagent or strong oxidizing agent, hazardous toxic solvents, elevated temperature, the use of expensive catalysts that need to be prepared prior to use, corrosive mineral acids, expensive Lewis acids, the use of corrosive and moisture-sensitive iodine monochloride, iodine vapour irritant in solvent free conditions. Moreover, the practical application of most of these reagents suffers from disadvantages such as low regioselectivity, long reaction times, low yields and tedious work-up. Therefore, development of new methods for the preparation of iodoaromatic compounds, in order to increase the regioselectivity, yields of products and to reduce reaction times, is still in great demand.

Peroxodisulfate compounds like K₂S₂O₈, Na₂S₂O₈ and (NH₄)₂S₂O₈ are widely used for the oxidation of various organic compounds, because the peroxodisulfate ions are an excellent, versatile oxidant and possess high oxidation potential [22]. But these reagents imposes certain limitations such as high activation energy (30 kcal/mol), use of strong mineral acids and toxic transition metals catalyst for hemolysis of peroxodisulfate ion (S₂O₈²⁻) into sulfate radical (SO₄^{•-}) [23].

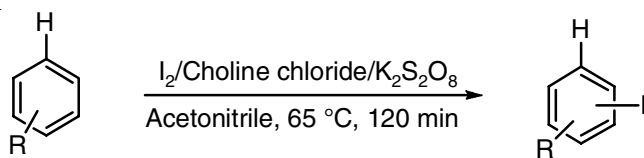
The modified peroxodisulfate compounds have becoming promising compounds for various organic transformations. For example, iodination of various aromatic compounds containing activating and deactivating functional groups by I₂/1,4-bis-(triphenylphosphonium)-2-butene peroxodisulfate [24], I₂/*n*-butyl-triphenylphosphonium peroxodisulfate [25], I₂/tetra-

butylammonium peroxodisulfate [26], I_2 /benzyltriphenylphosphonium peroxomonosulfate [27], I_2 /methyltriphenylphosphonium peroxydisulfate [28], KI/ammonium peroxodisulfate [29] are few of them to mention. In continuation to our search for simple non-hazardous methods for the organic transformations, especially for synthesis of iodinated compound [30–33]. In this article, we wish to report the use of molecular iodine/choline chloride/potassium peroxodisulfate system as an excellent iodinating agent for the iodination of both activated and deactivated aromatic compounds.

EXPERIMENTAL

Choline chloride, potassium peroxodisulfate, aromatic compounds such as phenol, aniline, anisole, toluene *etc.* and solvents like ethyl acetate, acetone, petroleum ether and dichloromethane are purchased from SD fine chemicals, India. Melting points were measured on Deep vision apparatus in open capillary tubes, values are reported uncorrected. Thin layer chromatography was carried out on pre-coated sheets of silica gel containing 60 F₂₅₄ indicator (Merck, Darmstadt, Germany). Column chromatography was performed with silica gel (60–120 mesh; SD Fine). 1H NMR spectra were recorded on 400 MHz Bruker Advance instrument in $CDCl_3$ solvent; ^{13}C NMR spectra were recorded on 100 MHz Bruker Advance instrument in $CDCl_3$ solvent, chemical shifts (δ) are expressed in ppm value relative to the internal standard TMS. FT-IR of all the samples was recorded on a Bruker alpha-P spectrophotometer. Mass spectra were recorded on a JEOL GC Mate II mass spectrometer.

General procedure for iodination of aromatic compounds: To a solution of aromatic compound (10 mmol) in acetonitrile (5 mL) in a 100 mL round-bottomed flask with a magnetic bar was added choline chloride (10 mmol), potassium peroxodisulfate (10 mmol) and iodine (15 mmol). The reaction mixture was stirred for the appropriate time at 65 °C. The progress of the reaction was monitored by TLC. The reaction mixture was poured into aqueous sodium thiosulfate solution (1 mol/L) in order to remove unreacted iodine and extracted with ethyl acetoacetate (10 mL, 3 times). Organic layer is dried over anhydrous sodium sulphate. Evaporation of the solvent under vacuum followed by column chromatography on silica gel gave the corresponding iodinated compounds (**Scheme-I**).



Spectral data

a) 4-Iodoaniline: Brown solid, m.p.: 53–55 °C (lit. 55–56.5 °C) [34]; 1H NMR (400 MHz, $CDCl_3$): δ 3.46 (br, 2H), 6.44 (d, J = 8.4 Hz, 2H), 7.38 (d, J = 8.4 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 79.41, 117.33, 137.92, 146.08; Mass m/z : calcd. for C_6H_6NI : 219.02; found: 221.08.

b) 4-Iodophenol: Brown solid, m.p.: 92–93 °C (lit. 92 °C) [33]; 1H NMR (400 MHz, $CDCl_3$): δ 5.20 (s, 1H), 6.62 (d,

J = 8.8 Hz, 2H), 7.51 (d, J = 8.4 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 82.85, 117.51, 138.19, 155.26; Mass m/z : calcd. for C_6H_5OI : 219.02; found: 219.04.

c) 4-Iodo-2-methyl phenol: m.p.: 64–66 °C; 1H NMR (400 MHz, $CDCl_3$): 2.09 (s, 3H), 5.33 (broad, 1H), 6.71 (d, J = 8.4, 1H), 6.95 (d, J = 8.4, 1H), 7.04 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): 15.78, 115.34, 121.04, 124.44, 125.50, 129.68, 153.77; Mass m/z : calcd. for C_7H_7OI : 234.03; found: 234.97.

d) 2-Chloro-4-iodoaniline: White crystalline solid, mp 65–67 (Lit. 68–70) [33]; 1H NMR (400 MHz, $CDCl_3$): δ 4.15 (br, 2H), 6.49 (d, J = 8.4 Hz, 1H), 7.28 (q, 1H), 7.51 (d, J = 1.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 117.33, 120.23, 136.33, 137.17, 142.74; Mass m/z : calcd. for C_6H_5NCII : 253.47; found: 253.08.

e) 2-Iodo-4-chloroaniline: Brown solid, m.p.: 41 °C (lit. 38–40 °C) [33]; 1H NMR (400 MHz, $CDCl_3$): δ 4.23 (brs, 2H), 6.71 (d, J = 8.6 Hz, 1H), 7.18 (dd, J = 2.4 Hz and 8.6 Hz, 1H), 7.48 (d, J = 2.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 84.7, 115.5, 125.3, 129.2, 138.5, 144.3; Mass m/z : calcd. for C_6H_5NCII : 253.47; found: 253.08.

f) 2-Iodo-4-nitroaniline: Yellow solid, m.p.: 98 °C (lit. 99–100 °C) [33]; 1H NMR (400 MHz, $CDCl_3$): δ 7.07 (d, J = 7 Hz, 1H), 8.06 (dd, J = 2 Hz and 7 Hz, 1H), 8.67 (d, J = 2 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 83.1, 115.2, 126.6, 135.9, 143.8, 163.4; Mass m/z : calcd. for $C_6H_5N_2O_2I$: 264.02; found: 265.11.

g) 4-Iodoanisole: Yellow solid, m.p.: 51 °C (mp 46–48 °C) [33]; 1H NMR (400 MHz, $CDCl_3$): δ 3.71 (s, 3H), 6.65 (d, 2H), 7.48 (d, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 55.3, 82.6, 116.5, 138.3, 159.1; Mass m/z : Calcd for C_7H_7OI : 234.03; Found 233.11.

h) 4-Iodotoluene: Brown solid, m.p.: 33–35 °C; 1H NMR (400 MHz, $CDCl_3$): δ /ppm 2.22 (s, 3H), 6.83 (d, J = 8.0 Hz, 2H), 7.48 (d, J = 8.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 21.28, 90.55, 131.37, 137.37; Mass m/z : calcd. for C_7H_7I : 218.03; found: 218.08.

i) 2-Chloro-4-iodotoluene: Off-white solid, m.p.: 83–85 °C; 1H NMR (400 MHz, $CDCl_3$): δ 2.26 (s, 3H), 6.86 (d, J = 8.0 Hz, 1H), 7.39 (q, 1H), 7.76 (d, J = 1.2 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 19.61, 90.06, 132.06, 135.00, 135.78, 136.92; Mass m/z : calcd. for C_7H_6ClI : 252.48; found: 251.05.

j) 4-Iodo-*o*-xylene: Brown solid, m.p.: 69–71 °C; 1H NMR (400 MHz, $CDCl_3$): δ 2.15 (s, 6H), 6.79 (d, J = 8.0 Hz, 1H), 7.35 (d, J = 8.0 Hz, 1H), 7.40 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 19.24, 90.55, 131.24, 134.53, 135.13, 137.41, 138.89; Mass m/z : calcd. for C_8H_9I : 232.06; found: 231.33.

k) 4-Iodo-*m*-xylene: Brown solid, m.p.: 71–72 °C; 1H NMR (400 MHz, $CDCl_3$): δ 2.23 (s, 3H), 2.35 (s, 3H), 6.63 (d, J = 8.1 Hz, 1H), 7.02 (s, 1H), 7.61 (d, J = 8.1 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 20.98, 28.04, 97.17, 128.45, 130.86, 138.11, 138.72, 141.059; Mass m/z : calcd. for C_8H_9I : 232.06; found: 232.06.

l) 3-Iodobenzoic acid: White solid, m.p.: 189 °C (lit. 187) [33]; 1H NMR (400 MHz, $CDCl_3$): δ 5.11 (s, broad), 7.23 (t, J = 8.0 Hz, 1H), 7.93 (d, J = 8.0 Hz, 1H), 8.06 (d, J = 8.0 Hz, 1H), 8.45 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 93.83, 129.34, 130.18, 131.22, 139.07, 142.58, 169.84; Mass m/z : calcd. for $C_7H_5O_2I$: 248.02; found: 248.00.

m) 1-Chloro-4-iodo-benzene: Yellowish powder, m.p.: 54 °C (lit. 56 °C) [33]; ¹H NMR (400 MHz, CDCl₃): δ 7.05 (d, *J* = 8.0 Hz, 2H), 7.57 (d, *J* = 8.0 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃): δ 91.19, 130.18, 134.24, 138.42; Mass *m/z*: calcd. for C₆H₄ClI: 238.45; found: 238.17.

n) 1-Bromo 4-iodo-benzene: White solid, m.p.: 88-89 °C (lit. 90 °C) [33]; ¹H NMR (400 MHz, CDCl₃): δ 7.20 (d, *J* = 8.0 Hz, 2H), 7.51 (d, *J* = 8.0 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃): δ 92.06, 122.23, 133.14, 138.81; Mass *m/z*: calcd. for C₆H₄BrI: 282.90; found: 282.20.

RESULTS AND DISCUSSION

In this paper, we are reporting the use of choline chloride/potassium peroxodisulfate as a highly efficient oxidant for the iodination of activated as well as deactivated aromatic compounds by molecular iodine. The iodination of aromatic compounds with this reagent was investigated in acetonitrile under reflux conditions. Iodination takes place by oxidation of molecular iodine into electrophilic iodonium cation radical (I₂^{•+}) by *in situ* formed choline peroxodisulfate. The *in situ* formation of choline peroxodisulfate was supported by the work reported by Gadilohar *et al.* [35] (Scheme-II). They found that choline peroxodisulfate is quite stable when stored below 10 °C in the absence of light for months without loss of its activity and it is soluble in polar solvents such as methanol, ethanol, acetonitrile, but insoluble in solvents such as hexane and diethyl ether.

Our initial experiment was to optimize the reaction condition, so we examined the iodination of aniline as a model substrate in the presence of molecular iodine as iodinating reagent and choline chloride/potassium peroxodisulfate as oxidizing agent in different solvent such as water, dichloromethane, ethanol, methanol, acetone, acetonitrile at reflux condition in order to study the influence of solvent (Table-1). Iodination of aniline with I₂/choline peroxodisulfate was unsuccessful in water and also in dichloromethane (entry 1, 2; Table-1). On the other hand, we performed same reaction in methanol and ethanol, unfortunately, rate of reaction in methanol and ethanol was also disappointingly sluggish even at elevated temperatures (entry 3, 5, Table-1). Then, we attempted iodination in acetone, we obtained desired product in 62 %, it was also not encouraging because of longer reaction time and poor product yield (entry

6, Table-1). Acetonitrile was found to be the good choice for iodination with optimum yield of 93 % of desired product (entry 4, Table-1).

Then we performed the reaction of aniline (10 mmol) in the presence of varying amounts of molecular iodine, choline chloride and potassium peroxodisulfate. We have realized that the molar ratio of 1.0/1.0/1.0/1.5 for aromatic substrate/iodine/choline chloride/oxidant was satisfactory, giving products in good yields at reflux temperature (entry 5, Table-2).

TABLE-2
EFFECT OF MOLE RATIO OF SUBSTRATE, CHOLINE CHLORIDE, POTASSIUM PEROXODISULFATE AND MOLECULAR IODINE

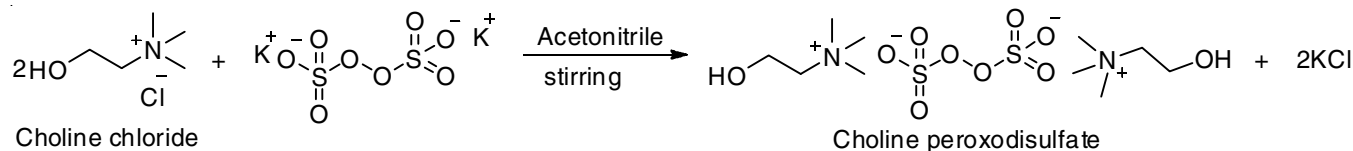
Entry	Substrate/choline chloride/oxidant/iodine	Solvent	Time	Yield (%)
1	1.0/1.0/0.5/1.0	CH ₃ CN	2.5	56
2	1.0/1.0/1.0/1.0	CH ₃ CN	2.5	73
3	1.0/1.0/1.5/1.0	CH ₃ CN	2.5	75
4	1.0/1.0/1.0/0.5	CH ₃ CN	2.5	53
5	1.0/1.0/1.0/1.5	CH ₃ CN	2.5	92
6	1.0/—/1.0/1.5	CH ₃ CN	2.5	43
7	1.0/1.0/—/1.5	CH ₃ CN	2.5	Trace

We investigated iodination reaction of all other activated and deactivated aromatic compounds in the optimized conditions using I₂/choline chloride/potassium peroxodisulfate iodinating system. The absence of any side products in TLC confirmed that the reaction was very clean. A variety of commercially available aniline, phenol, methyl and methoxy aromatic compounds were subjected to the reaction with molecular iodine and potassium peroxodisulfate in the presence of choline chloride at 65 °C. Iodination of aniline derivatives took place with high yield at 65 °C with short reaction times, between 60- 90 min (entry a; Table-3) and 90-120 min (entries d, e and f; Table-3). Surprisingly, iodination of phenol derivatives proceeded in 90-120 min at 65 °C (entry b,c; Table-3), whereas reaction of methoxy and methyl derivatives proceeded in 150-210 min with good yield (entry g,h,i,j,k; Table-3). Iodination of benzoic acid, chlorobenzene and bromobenzene required refluxing for 180-300 min with moderate yield (entry l, m, n; Table-3). Thus highest yields were observed with nucleophilic arenes, such as aniline, phenol, anisole and moderate yields were observed electron-poor arenes such as benzoic acid, chlorophenol.

Plausible mechanism: We have proposed a plausible mechanism for iodination of aromatic compounds by molecular iodine and choline chloride/potassium peroxodisulfate; by homolytic cleavage under the influence of heat, *in situ* formed choline peroxodisulfate readily forms choline sulphate radical [1], which can be easily converted into choline sulphate anion [4] by one electron transfer from molecular iodine [3], so that molecular iodine is oxidized to iodonium cation radical [4].

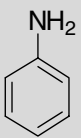
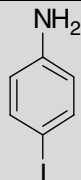
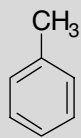
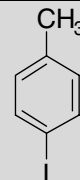
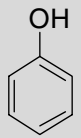
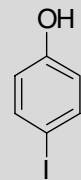
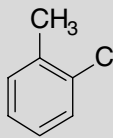
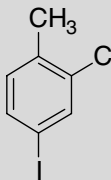
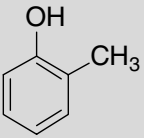
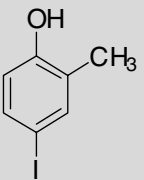
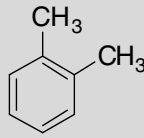
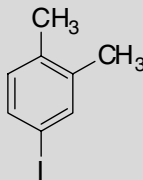
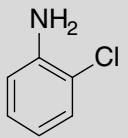
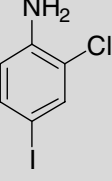
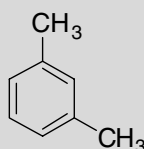
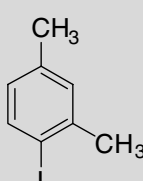
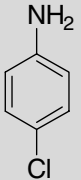
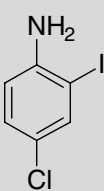
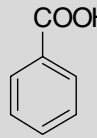
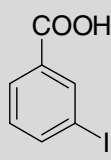
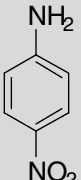
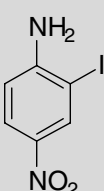
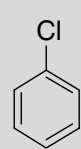
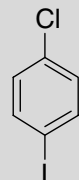
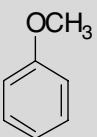
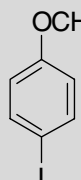
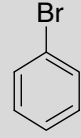
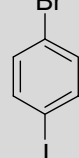
TABLE-1
OPTIMIZATION OF IODINATION REACTION

S. No.	Solvent	Time (h)	Temp. (°C)	Yield (%)
1	Water	6.0	65	—
2	Dichloromethane	6.0	65	23
3	Methanol	4.0	65	31
4	Acetonitrile	2.5	65	93
5	Ethanol	4.0	65	57
6	Acetone	3.5	65	62
7	DMSO	4.0	65	47



Scheme-II

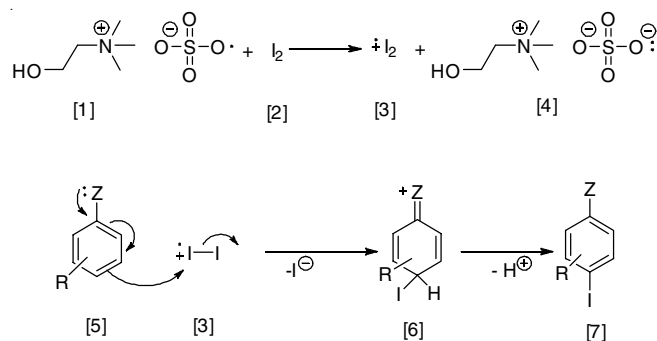
TABLE-3

Entry	Substrate	Product	Time (min)	Yield (%)	Entry	Substrate	Product	Time (min)	Yield (%)
a			90	94	h			180	71
b			90	92	i			180	67
c			120	86	j			210	69
d			120	81	k			210	65
e			120	86	l			180	61
f			120	76	m			240	57
g			150	73	n			300	51

The nucleophilic attack of iodonium cation radical on aromatic substrate produces an intermediate [6], which rapidly oxidized to iodinated aromatic compounds. The rate of the reaction may further accelerated by *in situ* formed HI acid (Scheme-III).

Conclusion

In conclusion, a simple procedure is developed for the iodination of activated as well as deactivated aromatic compounds by molecular iodine in the presence of choline chloride/potassium peroxodisulfate. I_2 /choline chloride/potassium peroxodisulfate system is a mild, efficient, inexpensive iodinating agent. The methodology involves the *in situ* generation



of choline peroxodisulfate, which in turn forms iodonium cation radical (I₂^{•+}), which acts as the electrophile for the iodination, moreover HI formed as side product will enhance the rate of conversion. A remarkable feature of this system is that harsh reaction condition such as high temperature, toxic transition metal catalyst and strong oxidizer are not required, toxic volatile solvents are not used as well as not produced during the reaction, furthermore the reagent can be easily prepared.

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