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Iodinated 1,4-naphthoquinones

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Iodination of 5-amino- and 6-amino-1,4-naphthoquinones with I_2 and HIO_3 in aqueous dioxane occurs only at the benzoid ring. Depending on the reaction conditions, either aminoiodo- or aminodiiodonaphthoquinones are produced. Diazotization of these compounds followed by reduction or replacement of the diazo group with iodine affords mono- or polyiodo derivatives of 1,4-naphthoquinone.

Key words: amino-1,4-naphthoquinones, iodination, diazotization, organoiodine compounds.

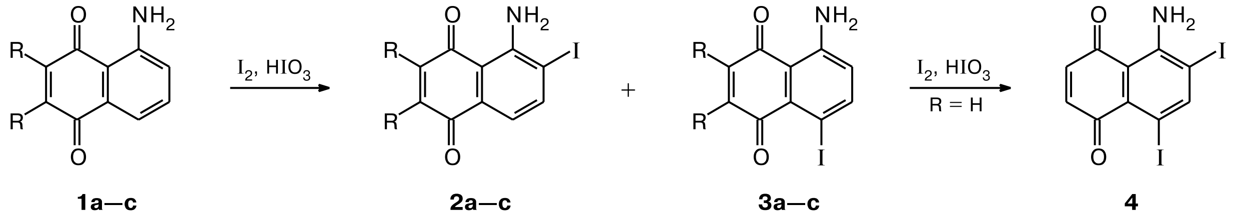
Iodo-substituted carbo- and heteroaromatic compounds serve as key intermediates in the synthesis of various acetylene, ethylene, and some functionalized aromatic derivatives.[1—3] Aryl and hetaryl iodides are often synthesized by electrophilic substitution of hydrogen.[1] However, direct iodination of polycyclic quinones (naphtho- or anthraquinone) is difficult to perform due to the deactivating effect of two electron-withdrawing carbonyl groups. In anthraquinone, this effect can be compensated by introducing the strong electron-donating amino group, which allows the amino-substituted benzoid ring to undergo iodination.[4,5] The possibility of subsequent removal and replacement of the amino group through diazonium salts provides an approach to various iodo derivatives of anthraquinone. Iodination of aminonaphthoquinones and their use as the basis for the synthesis of difficultly accessible iodo derivatives of naphthoquinone have not been described in the literature.

The direct replacement of hydrogen atoms in various aromatic substrates, including aminoanthraquinones, can be successfully performed with the use of the efficient $I_2—HIO_3—H_2SO_4$ iodinating system in AcOH.[4,6—8] Our attempt to use this procedure for iodination of more active aminonaphthoquinones failed due to rapid resinification of the reaction mixture. Earlier,[5] when studying iodination of aminoanthraquinones, we have proposed that H_2SO_4 should be excluded from the iodinating system by replacing it with an excess of HIO_3. This led to a substantial increase in the yield of the products and expansion of the scope of this reaction as applied to this series of compounds. In the present study, we demonstrated that the modified $I_2—HIO_3$ system is suitable also for iodination of aminonaphthoquinones, which allowed us to synthesize representatives of mono- and polyiodo-1,4-naphthoquinones.

Aminonaphthoquinones are iodinated at a reasonable rate in aqueous dioxane in the presence of a 5—8-fold (with respect to the stoichiometry) amount of HIO_3 at 80—87 °C. 5-Amino-1,4-naphthoquinone (**1a**) reacts with 1 equiv. of I_2 to give a mixture of isomeric 5-amino-6-iodo- (**2a**) and 5-amino-8-iodo-1,4-naphthoquinones (**3a**) (Scheme 1).

The reaction rate and the isomeric composition of the products depend on the solvent component ratio. An in-

Scheme 1



R = H (**a**), Me (**b**), Br (**c**)

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crease in the water percentage in the mixed aqueous-dioxane solvent leads to a decrease in the reaction time and a slight increase in the fraction of *para* isomer **3a** relative to *ortho* isomer **2a**. For example, an increase in dilution of dioxane with water from 7.5 : 1 to 2 : 1 leads to a decrease in the reaction time from 4 to 2.5 h, a decrease in the yield of *ortho* isomer **2a** from 54 to 23%, and an increase in the yield of *para* isomer **3a** from 22 to 29%. A decrease in the water fraction in the solvent from 1 : 7.5 to 1 : 10 leads to an increase in the reaction time to 9 h and an increase in the *ortho*-to-*para* isomer ratio from 2.4 to 2.7.

The structures of isomeric amino iodides **2a** and **3a** were proved by deamination of isomer **2a** giving rise to known 6-iodo-1,4-naphthoquinone (**5**)[9] and the transformation of isomer **3a** into 5,8-diiodo-1,4-naphthoquinone (**6**), whose structure was unambiguously established by ^{1}H NMR spectroscopy (see below).

Naphthoquinones containing the substituted quinoid ring, *viz*., 5-amino-2,3-dimethyl- (**1b**) and 5-amino-2,3-dibromo-1,4-naphthoquinone (**1c**), were also subjected to iodination under conditions favorable for the predominant formation of the *ortho* isomer (see Scheme 1). As in the case of aminoquinone **1a**, iodination of 2,3-disubstituted quinones **1b,c** occurs at the *ortho* and *para* positions with respect to the amino group. The yields of *ortho* isomers **2b,c** were 65 and 44%, respectively. The yields of *para* isomers **3b,c** were 21 and 18%, respectively. The reaction time depends substantially on the properties of the substituents in the quinoid ring of aminonaphthoquinones **1**. The presence of Me groups results in a decrease in the reaction time from 4 to 1 h. To the contrary, the presence of bromine atoms increases the reaction time to 17 h. A comparison of the ^{1}H NMR spectra of aminoiodides **2a—c** and **3a—c** revealed their characteristic features, which can be used to determine their structures (Table 1). The electron-donating amino group causes upfield shifts of the signals for the *ortho*- and *para*-protons, the shift of the signal for the *ortho*-protons being particularly large. Thus, the signal for the proton at position 6 in the spectra of 5-amino-8-iodo-1,4-naphthoquinones **3a—c** is observed at δ 6.55—6.64, whereas the signal for the proton at position 8 in the spectra of isomeric 5-amino-6-iodides **2a—c** appears at δ 7.14—7.27. The signal for the proton at position 7, which is only slightly affected by the adjacent I atom both in compounds

Table 1. Melting points, elemental analysis data, and ^{1}H NMR spectra (in $CDCl_3$) of iodinated aminonaphthoquinones **2a—c**, **3a—c**, **4**, **8**, and **10**

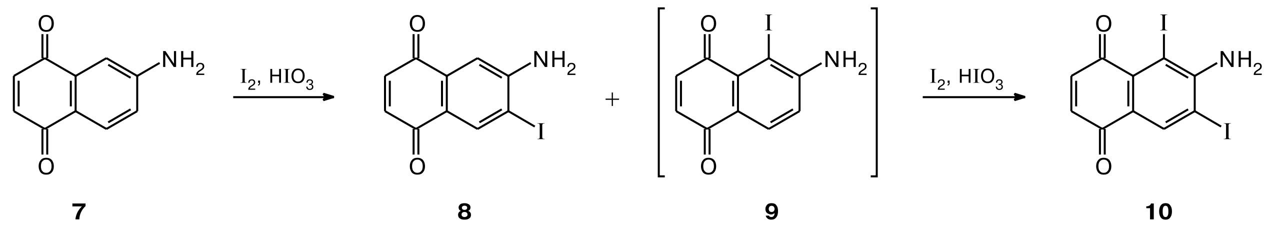
Compound	M.p./°C (solvent)	Found / Calculated (%) C	H	I	Molecular formula	^{1}H NMR, δ (*J*/Hz)
2a	153—154 (benzene)	39.89 40.16	1.97 2.02	42.26 42.43	$C_{10}H_6INO_2$	6.86, 6.87 (both d, 1 H each, H(2), H(3), *J* = 10.3); 7.15 (d, 1 H, H(8), *J* = 7.9); 7.25 (br.s, 2 H, NH_2); 8.03 (d, 1 H, H(7), *J* = 7.9)
2b	170.5—171.5 (toluene—hexane)	43.86 44.06	3.37 3.08	38.89 38.79	$C_{12}H_{10}INO_2$	2.11, 2.13 (both s, 3 H each, 2-Me, 3-Me); 7.14 (d, 1 H, H(8), *J* = 8.0); 7.20 (br.s, 2 H, NH_2); 7.94 (d, 1 H, H(7), *J* = 8.0)
2c[a]	251—252 (toluene—hexane)	26.56 26.29	0.90 0.88	27.96 27.78	$C_{10}H_4Br_2INO_2$	7.27 (d, 1 H, H(8), *J* = 7.9); 7.60 (br.s, 2 H, NH_2); 8.05 (d, 1 H, H(7), *J* = 7.9)
3a	185—186 (AcOEt)	40.15 40.16	1.97 2.02	42.44 42.43	$C_{10}H_6INO_2$	6.61 (d, 1 H, H(6), *J* = 9.0); 6.85, 6.88 (both d, 1 H each, H(2), H(3), *J* = 10.2); 6.90 (br.s, 2 H, NH_2); 7.97 (d, 1 H, H(7), *J* = 9.0)
3b	181—182 (toluene—hexane)	44.25 44.06	3.27 3.08	38.63 38.79	$C_{12}H_{10}INO_2$	2.13, 2.15 (both s, 3 H each, 2-Me, 3-Me); 6.55 (d, 1 H, H(6), *J* = 8.9); 6.82 (br.s, 2 H, NH_2); 7.91 (d, 1 H, H(7), *J* = 8.9)
3c[b]	233—234 (toluene—hexane)	26.42 26.29	1.16 0.88	28.01 27.78	$C_{10}H_4Br_2INO_2$	6.64 (d, 1 H, H(6), *J* = 8.9); 7.00 (br.s, 2 H, NH_2); 8.00 (d, 1 H, H(7), *J* = 8.9)
4	218—219 (dichloroethane)	28.42 28.26	1.33 1.19	59.89 59.72	$C_{10}H_5I_2NO_2$	6.87, 6.89 (both d, 1 H each, H(2), H(3), *J* = 10.2); 7.50 (br.s, 2 H, NH_2); 8.64 (s, 1 H, H(7))
8	>210 (decomp.) (toluene)	40.36 40.16	2.11 2.02	42.39 42.43	$C_{10}H_6INO_2$	4.87 (br.s, 2 H, NH_2); 6.84 (s, 2 H, H(2), H(3)); 7.23 (s, 1 H, H(5)); 8.37 (s, 1 H, H(8))
10	248 (decomp.) (ethanol)	28.62 28.26	1.37 1.19	59.75 59.72	$C_{10}H_5I_2NO_2$	6.36 (br.s, 2 H, NH_2); 6.85 (br.s, 2 H, H(2), H(3)); 8.23 (s, 1 H, H(8))[c]

[a] Found, Br (%): 35.21. Calculated, Br (%): 34.98.

[b] Found, Br (%): 35.27. Calculated, Br (%): 34.98.

[c] In DMSO-d_6.

Scheme 2



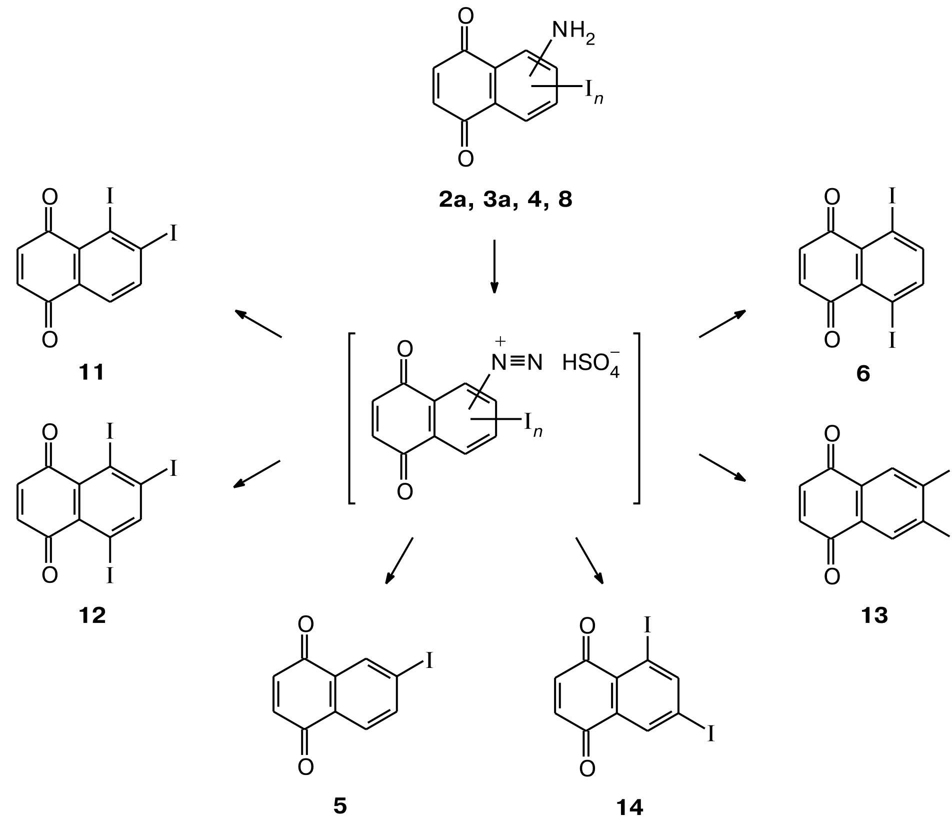
2a—c and **3a—c**, is always observed at δ 7.91—8.05. It is also noteworthy that the spin-spin coupling constant between the H(7) and H(8) protons ($J_{\mathrm{H(7),H(8)}}$ = 8.0 Hz) is slightly smaller than that between the H(7) and H(6) protons ($J_{\mathrm{H(6),H(7)}}$ = 9.0 Hz).

Iodination of 5-amino-1,4-naphthoquinone (**1a**) with 2 equiv. of I_2 in the presence of an excess of HIO_3 in aqueous dioxane affords 5-amino-6,8-diiodo-1,4-naphthoquinone (**4**) in 45% yield (see Scheme 1). The reaction performed with the use of dioxane and water in a ratio of 2.5 : 1 at 85 °C was completed in only 21 h. The ^{1}H NMR spectrum of diiodide **4** shows a downfield shift of a singlet for the H(7) proton under the influence of two vicinal I atoms (δ 8.64) (see Table 1).

6-Amino-1,4-naphthoquinone (**7**) is also iodinated under the above-described conditions used for iodination of 5-aminoquinone **1** (Scheme 2).

The reaction with 1 equiv. of I_2 and an excess of HIO_3 produces a mixture of 6-amino-7-iodo- (**8**) and 6-amino-5-iodo-1,4-naphthoquinone (**9**), with isomer **8** predominating (see Scheme 2). Due to substantially lower solubility in toluene, iodide **8** is easily separated from iodide **9** by crystallization. The yield of the former was 41%. Isomer **9** was not isolated in the individual state. The assignment of the structures of isomeric amino iodides **8** and **9** was made based on the ^{1}H NMR spectroscopic data. The spectrum of compound **8** shows singlets for the H(5) (δ 7.23) and H(8) protons (δ 8.37) (see Table 1), whereas the signals

Scheme 3



n = 1 (**2a**, **3a**, **8**), 2 (**4**) for the vicinal aromatic H(7) and H(8) protons in the spectrum of compound **9** appear as doublets at δ 7.03 and 7.73, respectively. The composition of the mixture of isomeric iodides **8** and **9** generated from 6-aminoquinone **7**, unlike that prepared by iodination of 5-aminoquinone **1**, depends only slightly on the solvent component ratio. The ratio of iodides **8** and **9** changes only from 4 to 3.5 as the dioxane-to-water ratio decreases from 6.2 to 2 (^{1}H NMR spectroscopic data). The reaction of 6-amino-1,4-naphthoquinone (**7**) with 2 equiv. of I_2 and an excess of HIO_3 produced 6-amino-5,7-diiodo-1,4-naphthoquinone (**10**) in 41% yield (see Table 1).

The development of the procedure for iodination of 5-amino- and 6-aminonaphthoquinones coupled with the possibility of performing deamination and replacing the amino group with the iodine atom or other functional substituents opens the way for the synthesis of a wide range of various naphthoquinone derivatives. We used this method to synthesize a series of mono-, di-, and triiodo-substituted naphthoquinones (Scheme 3).

Diazotization of aminoiodoquinones **2a**, **3a**, **4**, and **8** was carried out with the use of $NaNO_2$ in an aqueous solution of AcOH and H_2SO_4 at 10—15 °C. The diazo group was replaced with the iodine atom by the gradual addition of a solution of the diazonium salt to an aqueous KI solution at 40—90 °C. Deamination of aminoiodoquinones **2a** and **4** was carried out by reducing the corresponding diazonium salts with H_3PO_2 at 0—5 °C. Iodonaphthoquinones **5**, **6**, and **11—14** were prepared in 37—68% yields. Their structures were completely confirmed by analytical and spectroscopic methods (Table 2).

Experimental

The ^{1}H NMR spectra were recorded on a Bruker DPX-200 instrument (200 MHz) in $CDCl_3$ at 25 °C. The progress of the reactions was monitored and the purity of the compounds were checked by TLC on Silufol UV 254 plates. 5-Amino-1,4-naphthoquinones **1a—c** were prepared according to a known procedure.[10] 6-Amino-1,4-naphthoquinone (**7**) was synthesized according to a procedure described in the study.[9] Commercial HIO_3 was used.

5-Amino-6-iodo-1,4-naphthoquinone (2a) and 5-amino-8-iodo-1,4-naphthoquinone (3a). ***A.*** Finely ground I_2 (1.95 g, 7.7 mmol) and HIO_3 (3.00 g, 17.0 mmol) in H_2O (20 mL) were successively added to a solution of 5-amino-1,4-naphthoquinone (**1a**) (3.00 g, 17.3 mmol) in dioxane (150 mL) at 70—80 °C. The reaction mixture was refluxed for 4 h, poured into water (1 L), and extracted with $CHCl_3$ (3×100 mL). The extract was washed with a 1% Na_2SO_3 solution (2×100 mL) and water. The solvent was distilled off *in vacuo* and the residue was dissolved in toluene and chromatographed on SiO_2 (200—315 μm, 40×330 mm) using the 0→4% gradient of acetone in toluene as the eluent. Amino iodide **2a** (R_f 0.15) and isomer **3a** (R_f 0.07) were prepared in yields of 2.80 g (54%) and 1.18 g (23%), respectively (see Table 1). Iodination of the same amount of aminoquinone **1a** in a mixture of dioxane (150 mL) and water (15 mL) was completed in only 9 h. The yields of *ortho* isomer **2a** and *para* isomer **3a** were 2.40 g (46%) and 0.90 g (17%), respectively.

B. Iodination of aminonaphthoquinone **1a** (1.00 g, 5.8 mmol) was performed as described above in the presence of I_2 (0.70 g, 2.8 mmol) and HIO_3 (1.70 g, 9.6 mmol) in a mixture of dioxane (50 mL) and H_2O (25 mL) with refluxing for 2.5 h. The yields of *ortho* isomer **2a** and *para* isomer **3a** were 0.40 g (23%) and 0.50 g (29%), respectively.

5-Amino-6,8-diiodo-1,4-naphthoquinone (4). A solution of amine **1a** (0.90 g, 5.2 mmol), I_2 (1.50 g, 5.9 mmol), and HIO_3 (1.80 g, 10.2 mmol) in dioxane (50 mL) and water (20 mL) was refluxed for 21 h. After chromatography on Al_2O_3 with the use of $CHCl_3$ as the eluent and recrystallization from 1,2-dichloroethane, diiodide **4** was isolated in a yield of 1.00 g (45%) (see Table 1).

5-Amino-6-iodo-2,3-dimethyl-1,4-naphthoquinone (2b) and 5-amino-8-iodo-2,3-dimethyl-1,4-naphthoquinone (3b). Iodination of 5-amino-2,3-dimethyl-1,4-naphthoquinone (**1b**) (3.50 g, 17.4 mmol) was performed analogously to aminonaphthoquinone

Table 2. Melting points, elemental analysis data, and ^{1}H NMR spectra of di- and triiodo-substituted 1,4-naphthoquinones **6** and **11—14**

Compound	M.p./°C (solvent)	Found / Calculated (%) C	H	I	Molecular formula	^{1}H NMR, δ (*J*/Hz)
6	166—167 (benzene)	29.15 29.30	0.94 0.98	61.86 61.91	$C_{10}H_4I_2O_2$	6.98 (s, 2 H, H(2), H(3)); 7.94 (s, 2 H, H(6), H(7))
11	198—199 (benzene)	29.32 29.30	1.06 0.98	62.09 61.91	$C_{10}H_4I_2O_2$	6.93, 7.04 (both d, 1 H each, H(2), H(3), *J* = 10.2); 7.85 (d, 1 H, H(7), *J* = 9.0); 8.37 (d, 1 H, H(8), *J* = 9.0)
12	217—218 (benzene)	22.30 22.41	0.59 0.56	71.36 71.05	$C_{10}H_3I_3O_2$	6.97, 6.98 (both d, 1 H each, H(2), H(3), *J* = 10.2); 8.99 (s, 1 H, H(7))
13	232—233 (decomp.) (benzene—hexane)	29.43 29.30	1.11 0.98	61.76 61.91	$C_{10}H_4I_2O_2$	6.97 (s, 2 H, H(2), H(3)); 8.49 (s, 2 H, H(5), H(8))
14	178—179 (benzene—hexane)	29.48 29.30	1.03 0.98	61.72 61.91	$C_{10}H_4I_2O_2$	6.96, 6.98 (both d, 1 H each, H(2), H(3), *J* = 10.2); 8.46, 8.77 (both s, 1 H each, H(6), H(8))

1a with the use of I_2 (2.00 g, 7.9 mmol) and HIO_3 (3.05 g, 17.3 mmol) in a mixture of dioxane (150 mL) and H_2O (20 mL) with refluxing for 1 h. Chromatography on Al_2O_3 (40×110 mm) (toluene as the eluent) afforded *ortho* isomer **2b** in a yield of 3.70 g (65%) and *para* isomer **3b** in a yield of 1.20 g (21%) (see Table 1).

5-Amino-2,3-dibromo-6-iodo-1,4-naphthoquinone (2c) and 5-amino-2,3-dibromo-8-iodo-1,4-naphthoquinone (3c). A mixture of 5-amino-2,3-dibromo-1,4-naphthoquinone (**1c**) (1.00 g, 3.0 mmol), I_2 (0.35 g, 1.4 mmol), and HIO_3 (0.52 g, 2.9 mmol) in dioxane (26 mL) and H_2O (3 mL) was refluxed with stirring for 17 h. After standard work-up and separation of isomers, *ortho* isomer **2c** and *para* isomer **3c** were obtained in yields of 0.60 g (44%) and 0.25 g (18%), respectively (see Table 1).

6-Amino-7-iodo-1,4-naphthoquinone (8). 6-Amino-1,4-naphthoquinone (**7**) (1.00 g, 5.8 mmol), I_2 (0.65 g, 2.6 mmol), and HIO_3 (1.00 g, 5.7 mmol) in a mixture of dioxane (50 mL) and water (25 mL) were refluxed for 1 h. After standard isolation, the product was dissolved in a toluene—$CHCl_3$ mixture and filtered through an Al_2O_3 layer. Then the solvent was removed *in vacuo*, and the residue (1.00 g) consisting of 6-amino-7-iodo- (**8**) and 6-amino-5-iodo-1,4-naphthoquinone (**9**) in a ratio of 4 : 1 (^1H NMR spectroscopic data) was recrystallized from toluene. Iodide **8** was isolated in a yield of 0.70 g (41%) (see Table 1).

6-Amino-5,7-diiodo-1,4-naphthoquinone (10). A solution of 6-amino-1,4-naphthoquinone (**7**) (0.90 g, 5.2 mmol), I_2 (1.50 g, 5.9 mmol), and HIO_3 (1.80 g, 10.2 mmol) in dioxane (50 mL) and water (25 mL) was refluxed for 1 h. Diiodide **10** was obtained in a yield of 0.91 g (41%) (see Table 1).

5,7-Diiodo-1,4-naphthoquinone (14). A solution of $NaNO_2$ (0.32 g, 4.5 mmol) in water (4 mL) was added to a solution of 5-amino-6,8-diiodo-1,4-naphthoquinone (**4**) (0.88 g, 2.1 mmol) in a mixture of AcOH (15 mL), concentrated H_2SO_4 (27 mL), and water (10 mL) at 10 °C. The reaction mixture was stirred at this temperature for 30 min. The resulting solution of the diazonium salt was carefully poured into a mixture of 48% H_3PO_2 (11 mL) and ice (200 g), and the mixture was allowed to stand for ~14 h. The precipitate that formed was filtered off, washed with water, and dried. Chromatography on Al_2O_3 (benzene and $CHCl_3$ as the eluents) afforded diiodide **14** in a yield of 0.44 g (52%) (see Table 2).

6-Iodo-1,4-naphthoquinone (5). Deamination of 5-amino-6-iodo-1,4-naphthoquinone (**2a**) (1.0 g, 3.3 mmol) was performed under the same conditions as amine **4**. Compound **5** was obtained in a yield of 0.35 g (37%), m.p. 123—124 °C (*cf.* lit. data[9]).

5,6-Diiodo-1,4-naphthoquinone (11). A solution of $NaNO_2$ (0.60 g, 8.5 mmol) in water (1 mL) and 60% H_2SO_4 (3.5 mL) were added to a solution of 5-amino-6-iodo-1,4-naphthoquinone (**2a**) (1.50 g, 5.0 mmol) in AcOH (36 mL) at 15 °C. The reaction mixture was cooled to 10 °C. Then 10% H_2SO_4 (35 mL) was added and the mixture was stirred for 30 min. The resulting solution of the diazonium salt was added with vigorous stirring to a solution of KI (2.80 g, 16.9 mmol) in water (180 mL) at 40 °C. The precipitate that formed was filtered off, washed with water, dried, and chromatographed on SiO_2 using benzene as the eluent. The yield of diiodide **11** was 1.40 g (68%) (see Table 2).

5,8-Diiodo-1,4-naphthoquinone (6). Diazotization of 5-amino-8-iodo-1,4-naphthoquinone (**3a**) (0.80 g, 2.7 mmol) was performed as described above in the synthesis of diiodide **11**. The resulting solution of the diazonium salt was added to a stirred solution of KI (1.50 g, 9.0 mmol) in water (60 mL) preheated to 90 °C. The resulting suspension was heated to boiling and then cooled. The precipitate that formed was filtered off, washed with water, dried, and chromatographed on Al_2O_3 using 1,2-dichloroethane as the eluent. The yield of diiodide **6** was 0.50 g (46%) (see Table 2).

6,7-Diiodo-1,4-naphthoquinone (13). A solution of $NaNO_2$ (0.33 g, 4.6 mmol) in water (3 mL) and 50% H_2SO_4 (8 mL) were gradually added to a solution of aminonaphthoquinone **8** (0.90 g, 3.0 mmol) in AcOH (16 mL) at 10 °C. The reaction mixture was stirred at this temperature for 1 h. The resulting solution of the diazonium salt was poured into a stirred mixture of KI (2.90 g, 17.5 mmol) in water (50 mL) and toluene (50 mL), which was preheated to 60 °C. After cooling, the toluene layer was separated, and the aqueous layer was extracted with toluene. The combined extracts were washed with an aqueous Na_2CO_3 solution and water and filtered through an Al_2O_3 layer. Diiodide **13** was obtained in a yield of 0.80 g (65%) (see Table 2).

5,6,8-Triiodo-1,4-naphthoquinone (12). Initially, 5-amino-6,8-diiodo-1,4-naphthoquinone (**4**) (0.50 g, 1.2 mmol) was subjected to diazotization, and then the diazo group was replaced with iodine, as described above in the synthesis of diiodide **11**. After chromatography on SiO_2 with the use of 1,2-dichloroethane as the eluent, triiodide **12** was isolated in a yield of 0.26 g (41%) (see Table 2).

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