J. Org. Chem. **1984**, *49*, 3051-3053

Direct Aromatic Periodination

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Received December 27, 1983

Periodic acid and iodine in concentrated sulfuric acid exhaustively iodinated unactivated aromatic substrates. Thus benzene, nitrobenzene, benzoic acid, chlorobenzene, phthalic anhydride, and toluene were all converted to their periodo derivatives. Benzonitrile was converted to pentaiodobenzamide. This direct method compared favorably with the only general periodination procedure available, a mercuration/iododemercuration sequence. Partially iodinated products were obtained under less vigorous conditions. Thus, triiodo derivatives were obtained from nitrobenzene, benzoic acid, and toluene; tetraiodo derivatives were obtained from benzene, chlorobenzene, and trifluorotoluene.

Polyiodinated and periodinated aromatics have traditionally been prepared by two methods. The diazotization/iodination of iodoanilines requires uncommon starting materials which must already be partially iodinated.[1] The Jacobsen reaction, in which the iodines of iodinated aromatics migrate to form mixtures of more and less highly iodinated products, is limited in scope.[2] Recently, Deacon and Farquharson[3] described in detail the first generally applicable periodination method.[4] It entails exhaustive mercuration followed by iododemercuration with triiodide anion. This sequence may take up to two weeks, and gives yields of about 40%, often with significant impurities as detected by mass spectroscopy.

Direct aromatic iodination procedures, employing molecular iodine, have been used only infrequently for polyiodinations. The direct reaction requires an oxidizing reagent to convert iodine to a more powerful electrophile.[5] Fuming sulfuric acid typically plays this role. For instance, hexaiodobenzene can be prepared by treatment of benzene with excess iodine and fuming sulfuric acid at 180 °C.[6] Recently, periodination of chlorobenzene with a similar mixture was reported.[7] With strongly deactivated substrates, however, incomplete iodination occurs: nitrobenzene gives only triiodonitrobenzene under such conditions.[8]

A more powerful mixture for direct iodination results when periodic acid (H_5IO_6) replaces fuming sulfuric acid as the oxidizing reagent.[9] Such a mixture is capable of hexaiodinating benzene at 100 °C.[10] I now report the use of this method to periodinate aromatics, including substrates which are strongly deactivated toward electrophilic aromatic substitution. In addition, partially iodinated aromatics can often be obtained by using less vigorous conditions.

Results and Discussion

Table I

starting material	relative equiv of "I^+"	final reactn temp, °C	product	crystallized yield, %
benzene (**1a**)	4	25	$C_6I_4H_2$ (**5a**)	71
	10	100	C_6I_6 (**2a**)	51
nitrobenzene (**1b**)	5	75	$C_6I_3H_2NO_2$ (**6b**)	32
	10	95	$C_6I_5NO_2$ (**2b**)	30
benzoic acid (**1c**)	3	50	$C_6I_3H_2COOH$ (**6c**)	31
	10	95	C_6I_5COOH (**2c**)	49
benzonitrile (**1d**)	10	90	$C_6I_5CONH_2$ (**2d**)	32
chlorobenzene (**1e**)	6	5	C_6I_4HCl (**7**)	38
	10	60	C_6I_5Cl (**2e**)	55
toluene (**1f**)	3	5	$C_6I_3H_2CH_3$ (**5f**)	29
	10	5	$C_6I_5CH_3$ (**2f**)	28
trifluorotoluene (**1g**)	6	100	$C_6I_4HCF_3$ (4)	67
phthalic anhydride	8	105	$C_6I_4(CO)_2O$ (3)	44

Iodinations were performed with a 3:1 mixture of I_2:H_5IO_6 in sulfuric acid. All of the iodine so introduced can theoretically be converted to iodonium cation (I^+), as shown in eq 1. I^+ would make an ideal electrophile for

$$^{1}/_{7}H_5IO_6 + {}^{3}/_{7}I_2 + H^+ \rightarrow I^+ + {}^{6}/_{7}H_2O \qquad (1)$$

iodination via electrophilic aromatic substitution. However, there is evidence that I^+ in H_2SO_4 is largely disproportionated to IO^+ and I_3^+ ions.[11] The identity of the actual electrophile is therefore not certain. It is helpful in calculating reagent quantities, however, to assume that eq 1 holds. This is done in Table I, where the column labeled relative equiv of "I^+" indicates the ratio of iodinating species to substrate.

Periodinations. Excess I^+ was found to improve periodination yields; a 2-fold excess was therefore routinely employed. Table I shows the results of direct periodination of simple aromatics **1**. The yields listed are those of isolated and purified products. (Crude yields were often considerably higher, indicating losses during recrystallization.)

Benzene (**1a**) was converted to hexaiodobenzene (**2a**), and deactivated substrates **1b**, **1c**, and **1e** were converted to their pentaiodo derivatives **2b**, **2c**, and **2e**. Phthalic anhydride, with two deactivating groups, was periodinated to **3**. Benzonitrile (**1d**) was also periodinated, but the

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[6] Durand, J. F.; Mancet, M. *Bull. Soc. Chim. Fr.* **1935**, *2*, 665–666.

[7] Page, S. W.; Poppiti, J. A. *Anal. Chem.* **1981**, *53*, 574–575.

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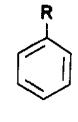
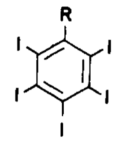
[11] Garrett, R. A.; Gillespie, R. J.; Senior, J. B. *Inorg. Chem.* **1965**, *4*, 563–566.

0022-3263/84/1949-3051$01.50/0



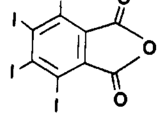
1



2

1a, R = H
1b, R = NO_2
1c, R = COOH
1d, R = CN
1e, R = Cl
1f, R = CH_3
1g, R = CF_3

2a, R = I
2b, R = NO_2
2c, R = COOH
2d, R = $CONH_2$
2e, R = Cl
2f, R = CH_3



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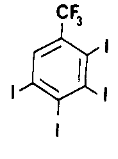
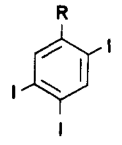


4

cyano group was partially hydrolyzed during the process to give the unsubstituted amide **2d**. The mildly activated substrate toluene (**1f**) was periodinated to **2f** at 5 °C; higher temperatures decreased the yield.

Partial Iodinations. Trifluorotoluene (**1g**) did not yield a periodinated product, even at 200 °C. Instead, the tetraiodinated **4** was obtained. Since other deactivated aromatics were easily periodinated, the failure of the reaction here was likely due to the steric hindrance afforded by the CF_3 group. The pentaiodinated derivative can, however, be prepared by the mercuration procedure.[3]

During early periodination attempts using stoichiometric amounts of I^+, it became clear that partially iodinated aromatics were significant byproducts. These could be prepared intentionally by avoiding large I^+ excesses and using lower reaction temperatures. Thus, Table I shows the production of 1,2,4,5-substituted products **5a** and **5f** from benzene and toluene, respectively. The substituents in **5** were distributed in the least hindered pattern: no substituent had two substituent neighbors. Nitrobenzene and benzoic acid gave the 1,3,4,5-substituted products **6** instead. Apparently the larger steric requirements of the nitro and carboxy groups forced the three substituting iodines in **6** to be adjacent. Attempts to prepare **6b** always resulted in troublesome amounts of diiodo or tetraiodo derivatives. The latter continued to contaminate the sample after several crystallizations. The less powerful iodine-in-fuming-sulfuric-acid mixture[8] would therefore be preferred for preparing **6b**.



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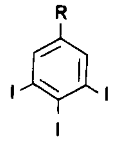
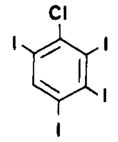


6

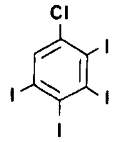
5a, R = I
5e, R = Cl
5f, R = CH_3

6b, R = NO_2
6c, R = COOH

Chlorobenzene gave a tetraiodo derivative under mild conditions. The utility of ^{13}C NMR in assigning structures to polyiodinated aromatics can be exemplified here. (^{13}C NMR spectra have apparently not been reported previously for most of these derivatives. Their ^{1}H NMR spectra are, of course, of limited value due to scarcity of protons.) The observation of more than four ^{13}C signals removed the symmetric (2,3,5,6-tetraiodo) isomer from consideration, leaving the asymmetric **7** and **8** isomers as candidates. The four CI signals were upfield due to the shielding effect of iodine.[12] In **7**, one of these signals (C_3) should be split into a small doublet during off-resonance decoupling due to three-bond splitting from the C_5 hydrogen.[13] In **8**, there should be two such doublets (C_2 and C_4, split by the C_6 hydrogen). Only one doublet was observed, confirming the structure as **7**. If **5e** was an intermediate in the production of **7**, the fourth I substituted **5e** at the position which avoided making all four iodines adjacent.



7



8

Limitations. Toluene was the only activated aromatic which was smoothly iodinated. *o*-Xylene gave a complex mixture of products; ethylbenzene largely suffered side chain oxidation. Diphenyl ether, anisole, and biphenyl gave intractable tars or high melting solids. Easily oxidized substrates did not fare well either: acetophenone and benzil gave benzoic acid products. Benzophenone underwent apparent retro-Friedel–Crafts acylation at 100 °C, giving fair yields of **2a** and **2c**. It appears, then, that the direct aromatic periodination is limited primarily to unactivated, oxidation-resistant substrates.

Summary

Recent speculation[3] that a direct procedure could not be sufficiently vigorous to periodinate aromatics without displacing other substituent groups has been shown to be unduly pessimistic. Direct periodinations of unactivated substrates proceeded with similar yields and improved reaction times and purity, compared to the mercuration/iododemercuration sequence. The direct procedure should therefore be the method of choice for preparing the periodinated products **2**.[14]

Experimental Section

Melting points were determined on a Mel-Temp apparatus and are corrected. Thin-layer chromatography (TLC) was performed on Eastman 13181 silica gel sheets; developing solvent and R_f value are given. NMR spectra were determined with a JEOL JNM-FX60Q spectrometer. ^{13}C NMR assignments were made by comparison to calculated chemical shifts,[12] by relative peak size, and by off-resonance decoupling multiplicity (including three-bond coupling).[13] One-bond C–H coupling multiplicities are given if greater than a singlet. Mass spectra were determined with a Hewlett-Packard 5985 GC/MS instrument (heated probe); parent (M^+) and base (100%) peaks are given. IR spectra (KBr pellet) were determined with a Beckman Acculab-1 spectrometer; weak peaks are not given unless noted. Microanalyses were performed by Galbraith Laboratories.

General Iodination Procedure. The following paragraph describes the iodination method. The quantities of reagents used in each case were those required to give the relative amounts of "I^+" specified in Table I,according to the stoichiometry of eq 1. As a typical example, the quantities used in the preparation of pentaiodonitrobenzene are given in parentheses.

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[12] Levy, G. C.; Lichter, R. L.; Nelson, G. L. "Carbon-13 Nuclear Magnetic Resonance Spectroscopy"; Wiley: New York, 1980; pp 111–112.

[13] Silverstein, R. M.; Bassler, G. C.; Morrill, T. C. "Spectrometric Identification of Organic Compounds"; Wiley: New York, 1981; p 272.

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Periodic acid (1.59 g, 6.97 mmol) was dissolved with stirring[15] in concentrated H_2SO_4 (25 mL). Iodine (5.20 g, 20.5 mmol) was crushed and added to the clear solution. After about 30 min of stirring, the dark mixture was placed in an ice bath. The aromatic substrate ($C_6H_5NO_2$: 0.50 mL, 4.86 mmol) was then added slowly. The reaction was allowed to stir to room temperature for 1 day and was then brought to its final reaction temperature (Table I) for 1 day. (Reactions performed at 5 °C were not allowed to warm before workup.) The mixture was then cooled and poured onto crushed ice. The resulting solid was collected by suction filtration and washed well with methanol to remove iodine. The crude product (2.66 g, 73%) was purified by crystallization.

1,2,4,5-Tetraiodobenzene (2a). The crude lavender powder (82%) was crystallized from 2-methoxyethanol, giving white needles (71%): mp 252–255 °C (lit.[2] mp 253 °C); TLC (1:1 CH_2Cl_2:cyclohexane) 0.68; ^{1}H NMR (Me_2SO-d_6) δ 8.32 (s); ^{13}C NMR (Me_2SO-d_6) δ 110.1 (CI), 147.1 (d, CH); MS, *m/e* 581.5 (M^+, 100%); IR 3080 (CH), 1425, 1400, 1265, 1110, 1080, 980, 870 cm^{-1}.

Hexaiodobenzene (5a). The crude product was washed with hot tetrahydrofuran (THF) to give an orange powder (72%). Crystallization from *N*-methylpyrrolidinone/water gave orange needles in three crops (51%): mp about 380–419 °C dec with loss of I_2 (lit.[10] mp about 370–430 °C dec); ^{1}H NMR (Me_2SO-d_6) no signal; ^{13}C NMR (Me_2SO-d_6, 75 °C) δ 120.3 (s, CI); MS, *m/e* 833.4 (M^+), 325.9 (100%); IR 1230, 1200 cm^{-1}.

3,4,5-Triiodonitrobenzene (6b). The crude yellow powder (89%) was crystallized from methanol to give yellow needles (61%) with a wide melting range. A second crystallization gave light yellow needles (32%): mp 155–161 °C (lit.[8] mp 166–167 °C); TLC (cyclohexane) 0.28; ^{1}H NMR ($CDCl_3$) δ 8.61 (s); ^{13}C NMR ($CDCl_3$) δ 106.5 (C_m), 130.5 (C_p), 132.5 (d, C_o), 147.0 (C_i); MS, *m/e* 500.8 (M^+, 100%) (a peak at *m/e* 626.6 suggested that less than 2% of $C_6I_4HNO_2$ still contaminated this sample); IR 3090 (CH), 1565, 1500 (s), 1345, 1320 (s), 1170, 1105, 885, 860, 720, 685 cm^{-1}.

Pentaiodonitrobenzene (2b). The deep yellow crude product (73%) was crystallized from 2-methoxyethanol/water to give bright yellow crystals (25%); a second crop was recrystallized to provide an additional 5%: mp about 329–335 °C dec with loss of I_2 (lit.[3] mp 330–332 °C dec); TLC (1:1 CH_2Cl_2:cyclohexane) 0.59; ^{1}H NMR (Me_2SO-d_6) no signal; ^{13}C NMR (Me_2SO-d_6) δ 100.6 (C_o), 125.6 (C_m), 125.9 (C_p), 157.9 (C_i); MS, *m/e* 752.6 (M^+), 325.9 (100%); IR 1530 (s), 1450, 1375, 1350, 1255, 1245, 1190, 1095, 855 cm^{-1}.

3,4,5-Triiodobenzoic Acid (6c). The pink crude product (62%) was crystallized from ethanol to give flowery white clumps in two crops (31%): mp 287–291 °C (lit.[16] mp 293 °C); TLC (4:1 THF:ethanol) 0.28; ^{1}H NMR (Me_2SO-d_6) δ 8.28 (s, CH), 4.0 (broad s, COOH + H_2O); ^{13}C NMR (Me_2SO-d_6) δ 109.0 (C_m), 128.4 (C_p), 132.8 (C_i), 138.2 (d, C_o), 164.5 (COOH); MS, *m/e* 499.8 (M^+, 100%); IR 2860 (broad, OH), 1690 (s), 1560, 1510, 1425, 1365, 1345, 1275 (s), 1060, 885, 750, 685 cm^{-1}.

Pentaiodobenzoic Acid (2c). The crude product was bright yellow (72%): mp 327–340 °C dec (lit.[3] mp >340 °C); TLC (4:1 ethyl acetate:ethanol) 0.15; ^{1}H NMR (Me_2SO-d_6) δ 4.7 (broad, COOH + H_2O); ^{13}C NMR (Me_2SO-d_6) δ 104.9 (C_o), 123.0 (C_p), 124.9 (C_m), 148.8 (C_i), 170.0 (COOH); MS, *m/e* 751.5 (M^+), 325.7 (100%); IR 1680 (s, CO), 1480, 1230, 1000, 815, 680 cm^{-1}. The absence of observable OH stretch has been noted before.[3]

Anal. Calcd for $C_7HO_2I_5$: C, 11.19; H, 0.13; I. 84.42. Found: C, 10.92; H, 0.09; I, 84.68.

Crystallization attempts from a variety of solvents resulted in lower melting points; for example, crystallization from acetone–hexanes gave yellow crystals (52%), mp 322–333 °C dec.

Pentaiodobenzamide (2d). The crude gold powder (69%) was crystallized from 2-methoxyethanol to give a light yellow powder (32%): mp about 371–378 °C dec with loss of I_2 (lit.[4] mp 380–385 °C) (recrystallization lowered the melting point); ^{1}H NMR (Me_2SO-d_6) δ 7.55 (s, 1 H), 7.87 (s, 1 H); ^{13}C NMR (Me_2SO-d_6) δ 105.4 (C_o), 121.6 (C_p), 124.7 (C_m), 150.5 (C_i), 172.1 ($CONH_2$); MS, *m/e* 750.6 (M^+), 127.0 (100%); IR 3430, 3280, 3180 (NH), 1665 (s, CO), 1590, 1485, 1380, 1260, 1240, 1100, 805 cm^{-1}.

Anal. Calcd for $C_7H_2NOI_5$: C, 11.20; H, 0.27; N, 1.87; I, 84.53. Found: C, 11.27; H, 0.21; N, 1.73; I. 84.58.

2,3,4,6-Tetraiodochlorobenzene (7). The crude tan powder (94%) was crystallized in three crops from methanol/water; filtration of the hot solution removed pentaiodochlorobenzene. The resulting greenish-yellow needles were recrystallized from toluene/hexanes to give light yellow needles (38%): mp 176–178 °C; TLC (cyclohexane) 0.51; ^{1}H NMR (Me_2SO-d_6) δ 8.48 (s); ^{13}C NMR (Me_2SO-d_6) δ 96.8 (CI), 105.2 (CI), 111.8 (CI), 123.6 (C_3I), 147.8 (d, CH) (the CCl signal could not be distinguished from noise); MS, *m/e* 615.6, 617.5 (M^+), 78.1 (100%); IR 3080 (w, CH), 1480, 1355, 1260, 1160, 990, 855, 695 cm^{-1}).

Anal. Calcd for C_6HClI_4: C, 11.70; H, 0.16; I, 82.39. Found: C, 11.52; H, 0.23; I, 82.60.

Pentaiodochlorobenzene (2e). The yellow-orange crude product (82%) was crystallized from THF in two crops (55%): mp 356–358 °C (lit.[7] mp 330–335 °C dec); ^{1}H NMR (Me_2SO-d_6) no signal; ^{13}C NMR (Me_2SO-d_6) δ 109.4 (C_o), 118.8 (C_p), 123.0 (C_m) (the CCl signal could not be distinguished from noise); MS, *m/e* 741.6, 743.6 (M^+), 741.6 (100%); IR 1250, 1225, 1210 cm^{-1}.

2,4,5-Triiodotoluene (5f). The tan crude powder (49%) was crystallized twice from ethanol to give tan needles (29%), mp 114.5–117 °C (lit.[17] mp 121–122 °C). The second recrystallization did not improve the melting point, but reduced contamination with tetraiodotoluene to less than 1%, as estimated from the mass spectrum: TLC (hexanes) 0.38; ^{1}H NMR ($CDCl_3$) δ 2.31 (s, 3 H), 7.70 (s, 1 H), 8.21 (s, 1 H); ^{13}C NMR ($CDCl_3$) δ 27.0 (CH_3), 101.0 (C_2), 104.9 (CI), 107.4 (CI), 139.5 (d, C_6), 143.0 (C_1), 147.4 (d, C_3); MS, *m/e* 469.8 (M^+, 100%); IR 1440, 1365, 1300, 1090, 1010 (s), 870, 835 cm^{-1}.

Pentaiodotoluene (2f). The grey-brown crude product (73%) was crystallized from *N*-methylpyrrolidinone/water to give bright yellow needles in two crops (28%): mp 310–313 °C dec (lit.[3] mp 306–309 °C dec); TLC (THF) 0.07; ^{1}H NMR (Me_2SO-d_6, 85 °C) δ 3.04 (s, CH_3); ^{13}C NMR (Me_2SO-d_6, 85 °C) δ 111.0 (C_o), 117.9 (C_p), 124.0 (C_m), 146.2 (C_i); ^{13}C NMR (C_5D_5N) δ 47.9 (CH_3); MS, *m/e* 721.5 (M^+), 340.9 (100%); IR 1365, 1300, 1265, 1225, 1125, 1000 cm^{-1}. The absence of observable CH stretch has been noted before.[3]

2,3,4,5-Tetraiodo(trifluoromethyl)benzene (4). The crude yellow powder (79%) was crystallized from Me_2SO/water to give light yellow needles (67%): mp 153–155 °C; TLC (cyclohexane) 0.57; ^{1}H NMR (Me_2SO-d_6 + $CDCl_3$ + CS_2) δ 8.02 (s); ^{13}C NMR (Me_2SO-d_6 + $CDCl_3$ + CS_2) δ 104.0 (C_2), 105.8 (C_5), 119.3 (CF_3, q, J_{CF} = 275 Hz), 126.8 (C_4), 128.4 (C_3), 133.0 (C_1, q, J_{CF} = 30.5 Hz), 134.2 (d, C_6, $J_{CF} \sim 5$ Hz); MS, *m/e* 649.6 (M^+), 269.0 (100%); IR 1315, 1250, 1130 cm^{-1}.

Anal. Calcd for $C_7HF_3I_4$: C, 12.94; H, 0.16; I, 78.13. Found: C, 13.08; H, 0.20; I, 78.39.

Tetraiodophthalic Anhydride (3). The bright greenish-yellow crude powder (64%) was crystallized from THF/hexanes, giving sparkling yellow needlets (44%): mp 327–331 °C (lit.[18] 327–328 °C); ^{1}H NMR (Me_2SO-d_6) no signal; ^{13}C NMR (Me_2SO-d_6) δ 106.6 (C_2), 126.8 (C_3), 139.7 (C_1), 168.0 (CO); MS, *m/e* 651.6 (M^+), 325.9 (100%); IR 1860, 1815, 1780 (s), 1750, 1520, 1320, 1280, 1220, 1170 (s), 1100, 905, 715 cm^{-1}.

Acknowledgment. I thank David Johnson for the mass spectral analyses. This research was supported by the donors of the Petroleum Research Fund, administered by the American Chemical Society.

Registry No. **1a**, 71-43-2; **1b**, 98-95-3; **1c**, 65-85-0; **1d**, 100-47-0; **1e**, 108-90-7; **1f**, 108-88-3; **1g**, 98-08-8; **2a**, 608-74-2; **2b**, 59875-34-2; **2c**, 64385-02-0; **2d**, 59875-33-1; **2e**, 64349-87-7; **2f**, 64349-91-3; **3**, 632-80-4; **4**, 90857-69-5; **5a**, 636-31-7; **5f**, 32704-10-2; **6b**, 53663-23-3; **6c**, 2338-20-7; **7**, 90857-70-8; phthalic anhdride, 85-44-9; *o*-xylene, 95-47-6; ethylbenzene, 100-41-4; diphenyl ether, 101-84-8; anisole, 100-66-3; biphenyl, 92-52-4; acetophenone, 98-86-2; benzil, 134-81-6; benzophenone, 119-61-9.

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(15) Although magnetic stirring is adequate for the quantities used here, Prof. Robert Hutchins has informed me that vigorous mechanical stirring is required when similar reactions are run on large scales.

(16) Goldberg, A. A.; Jefferies, H. S.; Turman, H. S.; Besly, D. M. *Q. J. Pharm. Pharmacol.* **1946**, *19*, 483–490.

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