Supplementary Material

A simple and efficient two-step synthesis of 1,2,3-triiodoarenes via consecutive C-H iodination/ ipso-iododecarboxylation strategy: A potential application towards ortho-diiodoarenes by regioselective metal-iodine exchange reaction

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1. General Information

All commercial reagents and chromatography solvents were used as obtained unless otherwise stated. Ethanol, diethyl ether, hydrochloric acid, chlorobenzene (C₆H₅Cl), chloroform, ethyl acetate, palladium (II) diacetate [pd(OAc)₂], iodobenzene diacetate PhI(OAc)₂, N-iodosuccinimide (NIS), sodium sulfate anhyd., iso-propylmagnesium chloride (iPrMgCl, 2M), iodine (I₂). Anhydrous solvents were distilled over appropriate drying agents prior to use were used as supplied. Analytical thin layer chromatography (TLC) was performed on Merck silica gel 60 F₂₅₄. Merck Silica gel 60 (0.063 – 0.2 mm) was used for column chromatography. Visualization of TLC was accomplished with UV light (254 nm). Reaction scale is between 2-5 gm. NMR spectra were recorded on a Bruker-Avance 400 MHz spectrometer. The residual solvent protons (¹H) or the solvent carbon (¹³C) were used as internal standards. ¹H-NMR data are presented as follows: chemical shift in ppm (δ) downfield from trimethylsilane (TMS) (multiplicity, integration, coupling constant). The following abbreviations are used in reporting NMR data: s, singlet; bs, broad singlet; d, doublet; t, triplet; q, quartet; dq, doublet of quartets; dd, doublet of doublets; m, multiplet. High resolution mass spectra were recorded using Chemical Ionization (CI) and electrospray ionization (ESI) techniques.

2. General Procedure for Iodination of Benzoic Acid Derivatives

(General procedure by Jin-Quan Yu, Angew. Chem. Int. Ed., 2008, 47, 5215-5219)

In a flamed dry 100 mL RBF, substrate (16.4 mmol, 1.0 equiv.), palladium acetate (0.05 equiv.), iodobenzene diacetate (1.5 equiv.) and iodine (1.5 equiv.) were dissolved in anhydrous dimethylformamide (DMF) (40 mL) under atmospheric air. The flask was then sealed with a speta and the reaction mixture was stirred at 100 °C for 24 hr. The reaction mixture was cooled to room temperature, diluted with ethyl acetate and then washed with 0.5 N HCl (20 mL × 4). The organic phase was washed with brine, dried over Na₂SO₄ and concentrated in a rotary evaporator. The

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residue was purified by column chromatography on silica gel (3:1 hexane : EtOAc) to give the iodination product.

2.1 Synthesis of 4-fluoro-2,6-diiodobenzoic acid (6)

The title compound was prepared using the general procedure for iodination of arene carboxylic acids and isolated in 85% yield as a white solid. The spectroscopic data for this compound are matched the previous report by *Angew. Chem. Int. Ed.* 2008; 47 (28): 5215-9.

2.2 Synthesis of 4-chloro-2,6-diiodobenzoic acid (7)

The title compound was prepared using the general procedure for iodination of arene carboxylic acids and isolated in 87% yield as a white solid. The spectroscopic data for this compound are matched the previous report by *Angew. Chem. Int. Ed.* 2008; 47 (28): 5215-9.

2.3 Synthesis of 2,6-diido-4-methylbenzoic acid (10)

The title compound was prepared using the general procedure for iodination of arene carboxylic acids and isolated in 85% yield as a white solid. The spectroscopic data for this compound are matched the previous report by *Angew. Chem. Int. Ed.* 2008; 47 (28): 5215-9.

2.4 Synthesis of 2-chloro-6-iodobenzoic acid (12)

The title compound was prepared using the general procedure for iodination of arene carboxylic acids and isolated in 91% yield as a yellowish solid. The spectroscopic data for this compound are matched the previous report by *Angew. Chem. Int. Ed.* 2008; 47 (28): 5215-9.

2.5 Synthesis of 2-bromo-6-iodobenzoic acid (13)

The title compound was prepared using the general procedure for iodination of arene carboxylic acids and isolated in 86% yield as a yellowish solid. The spectroscopic data for this compound are

2.6 **Synthesis of 2,6-diiodobenzoic acid (14)**

The title compound was prepared using the general procedure for iodination of arene carboxylic acids and isolated in 82% yield as a white solid. The spectroscopic data for this compound are matched the previous report by *Angew. Chem. Int. Ed.* 2008; 47 (28): 5215-9.

2.7 **Synthesis of 3-chloro-2,6-diiodobenzoic acid (15)**

The title compound was prepared using the general procedure for iodination of arene carboxylic acids and isolated in 81% yield as a white solid. δH (400 MHz, d-CDCl₃) δ: 7.74 (d, 1H, J = 8.0 Hz), 7.20 (d, 1H, J = 8.0 Hz). δC (100 MHz, d-CDCl₃) δ: 172.2, 146.8, 142.6, 139.3, 130.3, 95.7, 86.9. IR (cast film, cm⁻¹) 1790, 1652, 1548, 1152, 965. Mp: 148-150 °C. HRMS (ESI) m/z for C₇H₂Cl₂O₂ [M-H]⁻: calcd. 406.7833; found, 406.7824.

2.8 **Synthesis of 2-iodo-6-methylbenzoic acid (17)**

The title compound was prepared using the general procedure for iodination of arene carboxylic acids and isolated in 69% yield as a yellowish solid. The spectroscopic data for this compound are matched the previous report by *Angew. Chem. Int. Ed.* 2008; 47 (28): 5215-9.

2.9 **Synthesis of methyl 3,4,5-triiodobenzoate (20)**

The title compound was prepared using the general procedure for iodination of arene carboxylic acids and isolated in 77% yield as a pale-yellow solid. The spectroscopic data for this compound are matched the previous report by *Angew. Chem. Int. Ed.* 2008; 47 (28): 5215-9.

3. **General Procedure for *Ipso*-Iododecarboxylation of Iodinated Benzoic acid Derivatives**
A mixture of ortho-diiodobenzoic acid derivative (1.0 mmol, 1.0 equiv.), N-iodosuccinamide (NIS, 2.0 equiv.) and 1,2-dichloroethane (DCE, 10 mL) were added to a flamed dry 100 mL RBF and the mixture was irradiated with 100W (tungsten lamp) under reflux conditions for 24 h. The reaction mixture was then cooled to room temperature. The mixture was then washed sat. NaHSO₃, sat. NaHCO₃ and with brine. The organic layers were then dried over anhydrous Na₂SO₄, filtered and concentrated in a rotary evaporator. The residue was then purified by flash chromatography on silica gel (100% hexane) to give the desired iodinated product.

### 3.1 Synthesis of 5-fluoro-1,2,3-triiodobenzene (21)

The title compound was prepared using the general procedure for ipso-iododecarboxylation of benzoic acid derivatives and isolated in 82% yield as a white solid. The spectroscopic data for this compound are matched our previous report by *Aust. J. Chem.* 2013, 66, 1570–1575.

### 3.2 Synthesis of 5-chloro-1,2,3-triiodobenzene (22)

The title compound was prepared using the general procedure for ipso-iododecarboxylation of benzoic acid derivatives and isolated in 84% yield as a white solid. The spectroscopic data for this compound are matched our previous report by *Aust. J. Chem.* 2013, 66, 1570–1575.

### 3.3 Synthesis of 1,2,3-triiodo-5-methylbenzene (23)

The title compound was prepared using the general procedure for ipso-iododecarboxylation of benzoic acid derivatives and isolated in 61% yield as a white solid. The spectroscopic data for this compound are matched our previous report by *Aust. J. Chem.* 2013, 66, 1570–1575.

### 3.4 Synthesis of 1-chloro-2,3-diiodobenzene (24)

The title compound was prepared using the general procedure for ipso-iododecarboxylation of benzoic acid derivatives and isolated in 83% yield as a white solid. δH (400 MHz, d-CDCl₃) δ: 7.75 (d, 1H,
3.5 **Synthesis of 1-bromo-2,3-diiodobenzene (25)**

The title compound was prepared using the general procedure for ipso-iododecarboxylation of benzoic acid derivatives and isolated in 80% yield as a white solid. $\delta_H$ (400 MHz, $d$-CDCl$_3$) $\delta$: 7.81 (d, 1H, $J = 8.0$ Hz), 7.58 (d, 1H, $J = 8.0$ Hz), 6.89 (d, 1H, $J = 8.0$ Hz). $\delta_C$ (100 MHz, $d$-CDCl$_3$) $\delta$: 137.5, 131.3, 130.2, 129.1, 114.9, 108.8. IR (cast film, cm$^{-1}$) 2987, 2962, 1601, 1542, 1118, 624. **Mp**: 92–93 °C. HRMS (CI) m/z for C$_6$H$_3$BrI$_2$ [M$^+$]: calcd. 407.7507; found, 407.7497.

3.6 **Synthesis of 1,2,3-triiodobenzene (26)**

The title compound was prepared using the general procedure for ipso-iododecarboxylation of benzoic acid derivatives and isolated in 81% yield as a white solid. $\delta_H$ (400 MHz, $d$-CDCl$_3$) $\delta$: 7.85 (d, 2H, $J = 8.0$ Hz), 6.68 (t, 1H, $J = 8$ Hz). $\delta_C$ (100 MHz, $d$-CDCl$_3$) $\delta$: 138.2, 130.4, 120.7, 106.7. IR (cast film, cm$^{-1}$) 3121, 2968, 1624, 1510, 1015, 462. **Mp**: 111-112 °C. HRMS (CI) m/z for C$_6$H$_3$I$_3$ [M$^+$]: calcd. 455.7369; found, 455.7362.

3.7 **Synthesis of 1-chloro-2,3,4-triiodobenzene (27)**

The title compound was prepared using the general procedure for ipso-iododecarboxylation of benzoic acid derivatives and isolated in 65% yield as a white solid. $\delta_H$ (400 MHz, $d$-CDCl$_3$) $\delta$: 7.83 (d, 1H, $J = 8.0$ Hz), 7.13 (d, 1H, $J = 8$ Hz). $\delta_C$ (100 MHz, $d$-CDCl$_3$) $\delta$: 138.9, 137.7, 128.9, 123.1, 112.6, 103.8. IR (cast film, cm$^{-1}$) 3088, 2941, 1605, 1544, 1142, 570. **Mp**: 85-86 °C. HRMS (CI) m/z for C$_6$H$_2$ClI$_3$ [M$^+$]: calcd. 489.6979; found, 489.6970.

3.8 **Synthesis of methyl 3,4,5-triiodobenzoate (28)**

The title compound was prepared using the general procedure for ipso-iododecarboxylation of benzoic acid derivatives and isolated in 75% yield as a white solid. $\delta_H$ (400 MHz, $d$-CDCl$_3$) $\delta$: 8.40 (s, 2H).
δc (100 MHz, d-CDCl3) δ: 163.5, 139.0, 138.6, 129.3, 106.5, 52.3. IR (cast film, cm⁻¹) 3058, 2967, 1548, 1520, 1047, 654. Mp: 145-146 °C. HRMS (Cl) m/z for C₈H₅I₃O₂ [M⁺]: calcd. 513.7424; found, 513.7415.

3.9 Synthesis of 1,2-diiodo-3-methylbenzene (29)

The title compound was prepared using the general procedure for ipso-iododecarboxylation of benzoic acid derivatives and isolated in 53% yield as a white solid. The spectroscopic data for this compound are matched our previous report by J. Org. Chem., 1991, 56, 6573-6579.

4. General Procedure for Metal-Halogen Exchange Reaction of 1,2,3-triiodoarenes

To a solution of triiodoarene (0.66 mmol, 1.0 equiv.) in 15 mL of a mixture of THF at –78 °C was added dropwise isopropyl magnesium chloride (2M in THF, 0.23 mL, 0.70 mmol). The mixture was stirred at that temperature for 2 h. The electrophile was then added and the solution was slowly warmed to room temperature and stirred overnight. Saturated NH₄Cl was added and the resulting mixture was stirred for 30 min. The aqueous layer was extracted with Et₂O (2 x 50 mL). The organic layer was washed with brine, dried with anhydrous Na₂SO₄, filtered and then the solvent was evaporated under reduced pressure. The crude product was purified by flash chromatography (100% hexane) to yield the pure desired product.

4.1 Synthesis of 1,3-diiodobenzene (30)

The title compound was prepared using the general procedure for Metal-Halogen exchange reaction and isolated in 68% yield as a white solid. The spectroscopic data for this compound are matched the previous report by Adv. Syn. Cat., 2011 353(9), 1438-1442.

4.2 Synthesis of 1-fluoro-3,5-diiodobenzene (31)

The title compound was prepared using the general procedure for Metal-Halogen exchange reaction and isolated in 67% yield as a white solid. The spectroscopic data for this compound are
matched the previous report by *Revue Roumaine d Chimie*, **1989**, **34**(3), 807-810.

### 4.3 Synthesis of 1-chloro-3,5-diiodobenzene (32)

The title compound was prepared using the general procedure for Metal-Halogen exchange reaction and isolated in 69% yield as a white solid. The spectroscopic data for this compound are matched the previous report by *Aust. J. Chem.* **2013**, **66**, 1570-1575.

![Chemical Structure](image)

### 4.4 Synthesis of 2-butyl-1,3-diiodobenzene (33)

The title compound was prepared using the general procedure for Metal-Halogen exchange reaction and isolated in 68% yield as colorless oil. $\delta_H$ (300 MHz, $d$-CDCl$_3$): 7.80 (d, 2H, $J = 7.8$ Hz), 6.47 (t, 1H, $J = 7.8$ Hz), 3.04 (t, 2H, $J = 6.9$ Hz), 1.52 (m, 4H), 1.01 (t, 3H, $J = 6.9$ Hz). $\delta_C$ (75 MHz, $d$-CDCl$_3$) $\delta$: 146.5, 140.0, 129.1, 98.9, 46.6, 30.4, 22.8, 13.8. IR (cast film, cm$^{-1}$) 3050, 2971, 1612, 1594, 1217, 1078, 684. HRMS (Cl) m/z for C$_{10}$H$_{12}$I$_2$ [M$^+$]: calcd. 385.9028; found, 385.9016.

![Chemical Structure](image)

### 4.5 Synthesis of 1,3-diiodo-2-methylbenzene (34)

The title compound was prepared using the general procedure for Metal-Halogen exchange reaction and isolated in 64% yield as colorless oil. $\delta_H$ (400 MHz, $d$-CDCl$_3$): 7.82 (d, 2H, $J = 8.0$ Hz), 6.49 (t, 1H, $J = 8.0$ Hz), 2.75 (s, 3H). $\delta_C$ (100 MHz, $d$-CDCl$_3$) $\delta$: 142.5, 139.2, 128.6, 99.0, 34.6. IR (cast film, cm$^{-1}$) 3102, 2942, 2810, 1583, 1512, 1389, 1125, 1014, 894. HRMS (Cl) m/z for C$_7$H$_6$I$_2$ [M$^+$]: calcd. 343.8559; found, 343.8548.

![Chemical Structure](image)

### 4.6 Synthesis of 5-chloro-1,3-diiodo-2-methylbenzene (35)

The title compound was prepared using the general procedure for Metal-Halogen exchange reaction and isolated in 54% yield as a white solid. The spectroscopic data for this compound are matched the previous report by *Aust. J. Chem.* **2013**, **66**, 1570-
4.7 Synthesis of (2,6-diiodophenyl)(pyridin-2-yl)methanol (36)

The title compound was prepared using the general procedure for Metal-Halogen exchange reaction and isolated in 61% yield as colorless oil. δ\textsubscript{H} (400 MHz, d-CDCl\textsubscript{3}) δ: 9.79 (s, 1H), 8.02 (d, 2H, J = 8.0 Hz), 6.68 (t, 1H, J = 8.0 Hz). δ\textsubscript{C} (100 MHz, d-CDCl\textsubscript{3}) δ: 195.7, 141.6, 135.6, 134.6, 97.5. IR (cast film, cm\textsuperscript{-1}) 3114, 2974, 2711, 1713, 1234, 1019, 787. HRMS (Cl) m/z for C\textsubscript{7}H\textsubscript{4}I\textsubscript{2}O [M\textsuperscript{+}]: calcd. 357.8352; found, 357.8347.

4.8 Synthesis of (2,6-diiodophenyl)trimethylsilane (37)

The title compound was prepared using the general procedure for Metal-Halogen exchange reaction and isolated in 70% yield as colorless oil. δ\textsubscript{H} (500 MHz, d-CDCl\textsubscript{3}) δ: 7.94 (d, 2H, J = 7.5 Hz), 6.53 (t, 1H, J = 7.5 Hz), 0.64 (s, 9H). δ\textsubscript{C} (125 MHz, d-CDCl\textsubscript{3}) δ: 141.4, 131.2, 103.3, 4.9. IR (cast film, cm\textsuperscript{-1}) 2986, 2915, 1589, 1468, 1269, 1146, 849. HRMS (Cl) m/z for C\textsubscript{9}H\textsubscript{12}I\textsubscript{2}Si [M\textsuperscript{+}]: calcd. 401.8798; found, 401.8788.

4.9 Synthesis of (3-iodo-1,2-phenylene)bis(trimethylsilane) (38)

The title compound was prepared using the general procedure for Metal-Halogen exchange reaction and isolated in 34% yield as colorless oil. δ\textsubscript{H} (400 MHz, d-CDCl\textsubscript{3}) δ: 7.88 (dd, 1H, J = 8.0, J = 2.0 Hz), 7.60 (dd, 1H, J = 8.0 Hz, J = 2.0 Hz), 6.88 (t, 1H, J = 8.0 Hz), 0.55 (s, 9H), 0.36 (s, 9H). δ\textsubscript{C} (100 MHz, d-CDCl\textsubscript{3}) δ: 151.0, 150.8, 140.5, 134.1, 127.9, 107.2, 3.43, 2.54. IR (cast film, cm\textsuperscript{-1}) 2984, 2946, 1612, 1578, 1272, 1269, 1146, 844, 789. HRMS (Cl) m/z for C\textsubscript{12}H\textsubscript{21}Si\textsubscript{2} [M\textsuperscript{+}]: calcd. 348.0226; found, 348.0217.
5. NMR Spectra for New Compounds

5.1 $^1$H-NMR of 3-chloro-2,6-diiodobenzoic acid (15) in $d$-CDCl$_3$ at 25 °C.
5.2 $^{13}$C-NMR of 3-chloro-2,6-diiodobenzoic acid (15) in $d$-CDCl$_3$ at 25 °C.
5.3 $^1$H-NMR of 1-chloro-2,3-diiodobenzene (24) in $d$-CDCl$_3$ at 25 °C.
5.4 $^{13}$C-NMR of 1-chloro-2,3-diiodobenzene (24) in $d$-CDCl$_3$ at 25 °C.
5.5 $^1$H-NMR of 1-bromo-2,3-diodobenzene (25) in $d$-CDCl$_3$ at 25 °C.
13C-NMR of 1-bromo-2,3-diodobenzene (25) in d-CDCl₃ at 25 °C.
5.7 \( ^1H \)-NMR of 1,2,3-triiodobenzene (26) in \( d\text{-CDCl}_3 \) at 25 °C.
5.8

13C-NMR of 1,2,3-triiodobenzene (26) in d-CDCl3 at 25°C.
5.9 $^1$H-NMR of 1-chloro-2,3,4-triiodobenzene (27) in $d$-CDCl$_3$ at 25 °C.
5.10 $^{13}$C-NMR of 1-chloro-2,3,4-triiodobenzene (27) in $d_2$-CDCl$_3$ at 25°C.
5.11 $^1$H-NMR of methyl 3,4,5-triiodobenzoate (28) in $^d$CDCl$_3$ at 25 °C.
5.12

$^{13}$C-NMR of methyl 3,4,5-triiodobenzoate (28) in $d$-CDCl$_3$ at 25°C.
5.13 $^1$H-NMR of 2-butyl-1,3-diiodobenzene (33) in $d$-CDCl$_3$ at 25 °C.
5.14 $^{13}$C-NMR of 2-butyl-1,3-diiodobenzene (33) in $d$-CDCl$_3$ at 25 °C.
5.15  $^1$H-NMR of 1,3-diiodo-2-methylbenzene (34) in $d$-CDCl$_3$ at 25 °C.
5.16 $^{13}$C-NMR of 1,3-diiodo-2-methylbenzene (34) in d-CDCl$_3$ at 25°C.
5.17 $^1$H-NMR of 2,6-diiodobenzaldehyde (36) in $d$-CDCl$_3$ at 25 °C.
5.18 $^{13}$C-NMR of 2,6-diiodobenzaldehyde (36) in $d$-CDCl$_3$ at 25 $^\circ$C.
5.19 $^1$H-NMR of (2,6-diiodophenyl)trimethylsilane (37) in $d$-CDCl$_3$ at 25 °C.
5.20 $^{13}$C-NMR of (2,6-diiodophenyl)trimethylsilane (37) in $d$-CDCl$_3$ at 25 °C.
5.21 $^1$H-NMR of (3-iodo-1,2-phenylene)bis(trimethylsilane) (38) in $d$-CDCl$_3$ at 25 °C.
5.22 $^{13}$C-NMR of (3-iodo-1,2-phenylene)bis(trimethylsilane) (38) in $d$-CDCl$_3$ at 25 °C.