Supplementary Material (ESI) for Chemical Communications

Supporting Information

for

A Novel One-Pot Three-Component Synthesis of 3-Halo Furans and Sequential Suzuki Coupling

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SUPPORTING INFORMATION

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I. Experimental section

General Considerations. All reactions involving water-sensitive compounds were carried out in oven-dried Schlenk glassware under a nitrogen atmosphere. The solvents were dried according to standard procedures[1] and were distilled prior to use. Column chromatography: silica gel 60 M (mesh 230-400) Macherey-Nagel or aluminum oxide 90 active neutral (mesh 70-230) Merck. Thin layer chromatography (TLC): silica gel layered aluminium foil (60 F_{254} Merck, Darmstadt). Melting points (uncorrected): Reichert-Jung Thermovar and Büchi Melting Point B-540. Acid chlorides **1** and boronic acids **4** were purchased from ACROS or Merck and used without further purification. THP-protected propargyl alcohols **2** were prepared from propargyl alcohols by literature protocols.[2] ^{1}H and ^{13}C NMR spectra: Bruker ARX250, Bruker DRX 300 with Aceton-d_6, $CDCl_3$ or DMSO-d_6 as solvents. The assignments of quaternary C, CH, CH_2 and CH_3 were made on the basis of DEPT spectra. IR: Bruker Vector 22 FT-IR. UV/Vis: Hewlett Packard HP8452 A. MS: Jeol JMS-700 und Finnigan TSQ 700. Elemental analyses were carried out in the microanalitycal laboratory of the Department Chemie der Universität Heidelberg.

General procedure for the synthesis of chloro furans 3

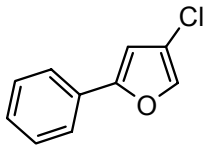
In a screw cap pressure vessel 14 mg (0.02 mmol) of $Pd(PPh_3)_2Cl_2$ and 7 mg (0.04 mmol) of CuI were dissolved in 5 mL of degassed THF. Then 1 mmol of acid chloride **1**, 1 mmol of THP protected propargyl alcohol **2**, as well as 0.14 mL (1.00 mmol) of triethylamine were successively added to the solution. The reaction mixture was stirred for 1-2 h at room temperature until the conversion was complete (monitored by TLC). Then 117 mg (2.00 mmol) of sodium chloride, 209 mg (1.10 mmol) of *p*-toluenesulfonic acid monohydrate and 3 mL of methanol were added and the reaction mixture was heated at 70°C for 2 h. After complete conversion of the alkynone to the furan (TLC), the reaction mixture was diluted with a saturated solution of $NaHCO_3$ (20 mL) and extracted with dichloromethane (3 × 20 mL). The combined organic layers were dried with sodium sulfate, evaporated and applied to column chromatography on neutral aluminium oxide (hexane/ethyl acetate 9:1) to give the analytically pure chloro furans **3a-f** as oils or as solids (crystallization from pentane or methanol) (for experimental details see Table 1).

Supplementary Material (ESI) for Chemical Communications

Table 1. Experimental details for one-pot synthesis of chloro-furans **3a-f**.

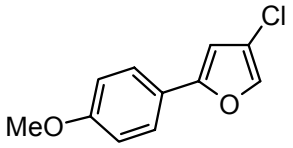
Acid chloride 1	Propargylic alcohol 2	3-Halo furan **3**
141mg (1.00 mmol) of **1a**	141 mg (1.00 mmol) of **2a**	113 mg (63 %) of **3a**
171 mg (1.00 mmol) of **1b**	141 mg (1.00 mmol) of **2a**	138 mg (71 %) of **3b**
141 mg (1.00 mmol) of **1a**	168 mg (1.00 mmol) of **2b**	145 mg (70 %) of **3c**
147 mg (1.00 mmol) of **1c**	168 mg (1.00 mmol) of **2b**	125 mg (59 %) of **3d**
167 mg (1.00 mmol) of **1d**	168 mg (1.00 mmol) of **2b**	170 mg (73 %) of **3e**
145 mg (1.00 mmol) of **1e**	141 mg (1.00 mmol) of **2a**	117 mg (64 %) of **3f**

4-Chloro-2-phenyl-furan (3a)



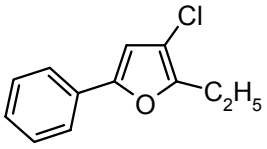
According to standard procedure the reaction gave rise to 113 mg (63 %) of **3a** as a colorless solid, R_f = 0.75 (hexane/ethyl acetate 9:1). ^{1}H NMR (CD_2Cl_2, 300 MHz): δ = 6.68 (d, J = 0.7 Hz, 1 H), 7.24–7.34 (m, 1 H), 7.37–7.44 (m, 2 H), 7.49 (d, J = 0.7 Hz, 1 H), 7.61–7.65 (m, 2 H).^{13}C NMR (CD_2Cl_2, 75 MHz): δ = 106.8 (CH), 117.8 (C_{quat}), 124.2 (CH), 128.6 (CH), 129.2 (CH), 130.3 (C_{quat}), 138.5 (CH), 154.7 (C_{quat}). EI MS (*m/z* (%)): 180 (^{37}Cl-M^+, 33), 178 (^{35}Cl-M^+, 100), 149 (^{35}Cl-M^+ – CHO, 29), 115 (M^+ – CHO – Cl, 57). Anal. calcd. for $C_{10}H_7ClO$ (178.6): C 67.24, H 3.95, Cl 19.85. Found: C 67.52, H 4.44.

4-Chloro-2-(4-methoxy-phenyl)-furan (3b)



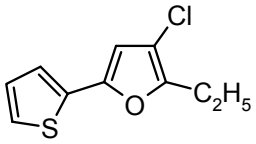
According to standard procedure the reaction gave rise to 148 mg (71 %) of **3b** as a colorless solid, R_f = 0.60 (hexane/ethyl acetate 9:1). ^{1}H NMR (CD_2Cl_2, 300 MHz): δ = 3.81 (s, 3 H), 6.52 (d, J = 1.1 Hz, 1 H), 6.92 (d, J = 8.8 Hz, 2 H), 7.43 (d, J = 1.1 Hz, 1 H), 7.55 (d, J = 8.8 Hz, 2 H). ^{13}C NMR (CD_2Cl_2, 75 MHz): δ = 55.7 (CH_3), 105.2 (CH), 114.6 (CH), 117.7 (C_{quat}), 123.2 (C_{quat}), 125.7 (CH), 137.8 (CH), 154.8 (C_{quat}), 160.2 (C_{quat}). EI MS (*m/z* (%)): 210 (^{37}Cl-M^+, 33), 208 (^{35}Cl-M^+, 100), 195 (^{37}Cl-M^+ – CH_3, 17), 193 (^{35}Cl-M^+ – CH_3, 51), 179 (^{35}Cl-M^+ – CHO, 11), 145 (M^+ – CHO – Cl, 49).

3-Chloro-2-ethyl-5-phenyl-furan (3c)



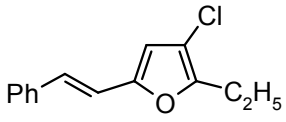
According to standard procedure the reaction gave rise to 145 mg (70 %) of **3c** as a colorless oil, R_f = 0.87 (hexane/ethyl acetate 9:1). ^{1}H NMR (CD_2Cl_2, 300 MHz): δ = 1.29 (t, J = 7.6 Hz, 3 H), 2.74 (q, J = 7.6 Hz, 2 H), 6.60 (s, 1 H), 7.24–7.32 (m, 2 H), 7.35–7.42 (m, 2 H), 7.58–7.64 (m, 2 H). ^{13}C NMR (CD_2Cl_2, 75 MHz): δ = 12.4 (CH_3), 19.5 (CH_2), 107.0 (CH), 112.2 (C_{quat}), 123.8 (CH), 128.0 (CH), 129.1 (CH), 130.7 (C_{quat}), 151.7 (C_{quat}), 152.5 (C_{quat}). EI MS (*m/z* (%)): 208 (^{37}Cl-M$^+$, 16), 206 (^{35}Cl-M$^+$, 44), 193 (^{37}Cl-M$^+$ – CH_3, 33), 191 (^{35}Cl-M$^+$ – CH_3, 100). HRMS calcd. for $C_{12}H_{11}{}^{37}ClO$: 208.0469. Found: 208.0462; HRMS calcd. for $C_{12}H_{11}{}^{35}ClO$: 208.0498. Found: 206.0479.

3-Chloro-2-ethyl-5-thiophen-2-yl-furan (3d)



According to standard procedure the reaction gave rise to 125 mg (59 %) of **3d** as a yellow oil, R_f = 0.72 (hexane/ethyl acetate 9:1). ^{1}H NMR (CD_2Cl_2, 300 MHz): δ = 1.25 (t, J = 7.6 Hz, 3 H), 2.69 (q, J = 7.6 Hz, 2 H), 6.44 (s, 1 H), 7.03 (dd, J = 3.7, 5.1 Hz, 1 H), 7.22 (dd, J = 1.1, 3.7 Hz, 1 H), 7.25 (dd, J = 1.1, 5.1 Hz, 1 H). ^{13}C NMR (CD_2Cl_2, 75 MHz): δ = 12.3 (CH_3), 19.4 (CH_2), 106.7 (CH), 112.0 (C_{quat}), 123.1 (CH), 124.8 (CH), 128.1 (CH), 133.4 (C_{quat}), 147.4 (C_{quat}), 152.1 (C_{quat}). EI MS (*m/z* (%)): 214 (^{37}Cl-M$^+$, 23), 212 (^{35}Cl-M$^+$, 69), 199 (^{37}Cl-M$^+$ – CH_3, 33), 197 (^{35}Cl-M$^+$ – CH_3, 100). HRMS calcd. for $C_{10}H_9{}^{37}ClOS$: 214.0033. Found: 214.0005; HRMS calcd. for $C_{10}H_9{}^{35}ClOS$: 212.0063. Found: 212.0036.

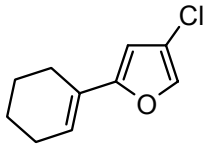
3-Chloro-2-ethyl-5-styryl-furan (3e)



According to standard procedure the reaction gave rise to 170 mg (73 %) of **3e** as a yellow oil, R_f = 0.70 (hexane/ethyl acetate 9:1). ^{1}H NMR (CD_2Cl_2, 300 MHz): δ = 1.29 (t, J = 7.6 Hz, 3 H), 2.69 (q, J = 7.6 Hz, 2 H), 6.29 (s, 1 H), 6.79 (d, J = 16.5 Hz, 1 H), 6.99 (d, J = 16.5 Hz, 1 H), 7.21–7.27 (m, 1 H), 7.30–7.38 (m, 2 H), 7.42–7.48 (m, 2 H). ^{13}C NMR (CD_2Cl_2, 75 MHz): δ = 12.3 (CH_3), 19.5 (CH_2), 110.2 (CH), 112.1 (C_{quat}), 116.2 (CH), 126.7 (CH), 127.4 (CH), 128.1

(CH), 129.1 (CH), 137.1 (C_{quat}), 151.0 (C_{quat}), 152.6 (C_{quat}). EI MS (*m/z* (%)): 234 (^{37}Cl-M^+, 28), 232 (^{35}Cl-M^+, 83), 219 (^{37}Cl-M^+ – CH_3, 35), 217 (^{35}Cl-M^+ – CH_3, 100). HRMS calcd. for $C_{14}H_{13}{}^{37}ClO$: 234.0625. Found: 234.0617; HRMS calcd. for $C_{14}H_{13}{}^{35}ClO$: 232.0655. Found: 232.0664.

4-Chloro-2-cyclohex-1-enyl-furan (3f)



According to standard procedure the reaction gave rise to 117 mg (64 %) of **3h** as a yellow oil, R_f = 0.77 (hexane/ethyl acetate 9:1). ^{13}C NMR (acetone-d_6, 75 MHz): δ = 22.7 (CH_2), 22.9 (CH_2), 25.2 (CH_2), 25.7 (CH_2), 105.8 (CH), 117.3 (C_{quat}), 124.6 (CH), 127.6 (C_{quat}), 138.3 (CH), 156.7 (C_{quat}). EI MS (*m/z* (%)): 184 (^{37}Cl-M^+, 32), 182 (^{35}Cl-M^+, 100), 169 (^{37}Cl-M^+ – CH_3, 10), 167 (^{35}Cl-M^+ – CH_3, 23). HRMS calcd. for $C_{10}H_{11}{}^{37}ClO$: 184.0469. Found: 184.0463; HRMS calcd. for $C_{10}H_{11}{}^{35}ClO$: 182.0498. Found: 182.0501.

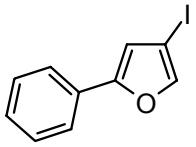
General procedure for the synthesis of Iodo furans 3g-j

In a screw cap pressure vessel 14 mg (0.02 mmol) of $Pd(PPh_3)_2Cl_2$ and 7 mg (0.04 mmol) of CuI were dissolved in 5 mL of degassed THF. Then 1 mmol of acid chloride **1**, 1 mmol of THP protected propargyl alcohol **2**, as well as 0.14 mL (1.00 mmol) of triethylamine were successively added to the solution (for experimental details see Table 10). The reaction mixture was stirred for 1-2 h at room temp until the conversion was complete (monitored by TLC). Afterwards 750 mg (5.00 mmol) of sodium iodide, 209 mg (1.10 mmol) of *p*-toluenesulfonic acid monohydrate and 3 mL of methanol were added and the reaction mixture was stirred at room temp for 2 h. After complete conversion of the alkynone to the furan (TLC), the reaction mixture was diluted with a saturated solution of $NaHCO_3$ (20 mL) and extracted with dichloromethane (3 × 20 mL). The combined organic layers were dried with sodium sulfate, evaporated and applied to column chromatography on neutral aluminium oxide (hexane/ethyl acetate 9:1) to give the analytically pure iodo furans **3g-j** as oils or as solids (crystallization from pentane or methanol) (for experimental details see Table 2).

Table 2. Experimental details for one-pot synthesis of iodo-furans **3g-j**.

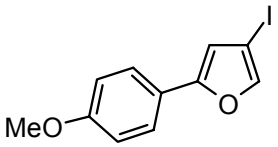
Acid chloride **1**	Propargylic alcohol **2**	3-Halo furan **3**
141mg (1.00 mmol) of **1a**	141 mg (1.00 mmol) of **2a**	170 mg (63%) of **3g**
171 mg (1.00 mmol) of **1b**	141 mg (1.00 mmol) of **2a**	190 mg (63%) of **3h**
186 mg (1.00 mmol) of **1f**	141 mg (1.00 mmol) of **2a**	128 mg (40%) of **3i**
141 mg (1.00 mmol) of **1a**	168 mg (1.00 mmol) of **2b**	215 mg (72%) of **3j**

4-Iodo-2-phenyl-furan (3g)



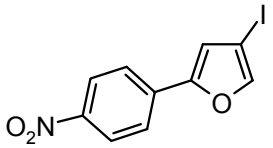
According to standard procedure the reaction gave rise to 170 mg (63 %) of **3g** as a colorless solid, R_f = 0.75 (hexane/ethyl acetate 9:1), Mp. 64 °C (64-65 °C[3]). ^{1}H NMR (acetone-d_6, 300 MHz): δ = 7.02 (d, ^4J = 0.7 Hz, 1 H), 7.33 (tt, ^3J = 2.2, 7.4 Hz, 1 H), 7.40–7.48 (m, 2 H), 7.70–7.75 (m, 2 H), 7.75 (d, ^4J = 0.7 Hz, 1 H). ^{13}C NMR (acetone -d_6, 75 MHz): δ = 66.5 (C_{quat}), 112.9 (CH), 124.5 (CH), 128.9 (CH), 129.6 (CH), 130.4 (C_{quat}), 146.4 (CH), 156.2 (C_{quat}).

4-Iodo-2-(4-methoxy-phenyl)-furan (3h)



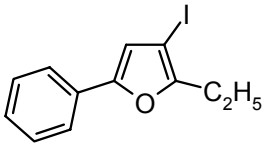
According to standard procedure the reaction gave rise to 190 mg (63 %) of **3h** as a colorless solid, R_f = 0.60 (hexane/ethyl acetate 9:1), Mp. 84-85 °C. ^{1}H NMR (acetone-d_6, 300 MHz): δ = 3.83 (s, 3 H), 6.84 (s, 1 H), 6.99 (dt, J = 2.0, 8.8 Hz, 2 H), 7.65 (dt, J = 2.2, 8.8 Hz, 2 H), 7.67 (br s, 1 H). ^{13}C NMR (acetone-d_6, 75 MHz): δ = 55.7 (CH_3), 66.5 (C_{quat}), 111.3 (CH), 115.2 (CH), 123.4 (C_{quat}), 126.2 (CH), 145.7 (CH), 156.6 (C_{quat}), 160.7 (C_{quat}). EI MS (*m/z* (%)): 300 (M^+, 100), 285 $(M^+ - CH_3)^+$, 12), 173 (M^+ – I, 20). IR (KBr): $\tilde{\nu}$ 3107 cm^{-1}, 2958, 1612, 1513, 1485, 1291, 1255, 1181, 1103, 1036, 910, 833, 795, 588. UV/Vis (CH_2Cl_2): λ_{max} (ε) 288 nm (27100), 306 (15700). Anal. calcd. for $C_{11}H_9O_2I$ (300.1): C 44.03, H 3.02. Found: C 44.42, H 3.24.

4-Iodo-2-(4-nitro-phenyl)-furan (3i)



According to standard procedure the reaction gave rise to 126 mg (40 %) of **3i** as yellow crystals, R_f = 0.78 (hexane/ethyl acetate 4:1), Mp. 160 °C. ^{1}H NMR (acetone-d_6, 300 MHz): δ = 7.35 (br s, 1 H), 7.90 (d, ^4J = 0.7 Hz, 1 H), 7.97 (dt, J = 2.2, 9.2 Hz, 2 H), 8.31 (dt, J = 2.2, 9.2 Hz, 2 H). ^{13}C NMR (acetone-d_6, 75 MHz): δ = 67.1 (C_{quat}), 116.8 (CH), 125.0 (CH), 125.2 (CH), 135.9 (C_{quat}), 147.8 (C_{quat}), 148.4 (CH), 154.0 (C_{quat}). EI MS (*m/z* (%)): 315 (M^+, 100). IR (KBr): $\tilde{\nu}$ 3135 cm^{-1}, 1600, 1569, 1514, 1336, 1279, 1143, 1111, 1098, 1023, 913, 854, 827, 816, 773, 752, 692, 587, 515. UV/Vis (CH_2Cl_2): λ_{max} (ε) 244 nm (9200), 350 (18000). Anal. calcd. for $C_{10}H_6NO_3I$ (315.1): C 38.12, H 1.92, N 4.45. Found: C 38.22, H 2.09, N 4.38.

2-Ethyl-3-iodo-5-phenyl-furan (3j)



According to standard procedure the reaction gave rise to 215 mg (72 %) of **3j** as a light yellow liquid, R_f = 0.87 (hexane/ethyl acetate 9:1). ^{1}H NMR (acetone-d_6, 300 MHz): δ = 1.26 (t, ^3J = 7.7 Hz, 3 H), 2.76 (q, ^4J = 7.7 Hz, 2H), 6.89 (s, 1H), 7.29 (tt, J = 3.0, 7.4 Hz, 1 H), 7.38–7.45 (m, 2 H), 7.66–7.71 (m, 2 H). ^{13}C NMR (acetone-d_6, 75 MHz): δ = 12.8 (CH_3), 21.6 (CH_2), 63.9 (C_{quat}), 113.6 (CH), 124.2 (CH), 128.5 (CH), 129.7 (CH), 131.0 (C_{quat}), 154.4 (C_{quat}), 158.1 (C_{quat}). EI MS (*m/z* (%)): 298 (M^+, 94), 283 ($M^+ - CH_3$, 100), 105 ($C_6H_5CO^+$, 22), 77 ($C_6H_5^+$, 13). IR (KBr): $\tilde{\nu}$ 2973 cm^{-1}, 1550, 1487, 1444, 1281, 1142, 1065, 1008, 754, 686. UV/Vis (CH_2Cl_2): λ_{max} (ε) 294 nm (15300), 308 (10400). HRMS calcd. for $C_{12}H_{11}IO$: 297.9855. Found: 297.9861.

General procedure for the synthesis of 2,5-disubstituted 3-Aryl-furans 5 via Suzuki-Coupling

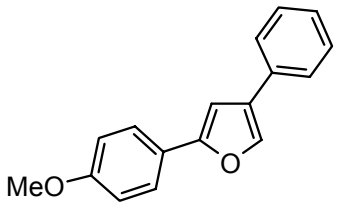
In a screw cap pressure vessel 35 mg (0.05 mmol) of $Pd(PPh_3)_2Cl_2$ and 7 mg (0.04 mmol) of CuI were dissolved in 5 mL of degassed THF. Then 1 mmol of acid chloride **1**, 1 mmol of THP protected propargyl alcohol **2**, as well as 0.14 mL (1.00 mmol) of triethylamine were added successively to the solution. The reaction mixture was stirred for 1-2 h at room temp until the conversion was complete (monitored by TLC). Afterwards 750 mg (5.00 mmol) of sodium iodide, 209 mg (1.10 mmol) of *p*-toluenesulfonic acid monohydrate and 3 mL of methanol were

added and the reaction mixture was stirred at room temperature for 2 h. After complete conversion of alkynone to the furan (TLC), 4 mL of 2 *M* solution of sodium carbonate (8 mmol) and 1.05 mmol of boronic acid **4** were added to the reaction mixture and heated at 90°C for 16-46h. After complete consumption of iodo furan (TLC), the reaction mixture was diluted with water (20 mL) and extracted with dichloromethane (3 × 20 mL). The combined organic layers were dried with sodium sulfate and the solvents were evaporated in vacuo. The residue was chromatographed on silica gel (hexane/ethyl acetate 9:1–20:1) to give the analytically pure 2,5-disubstituted 3-aryl-furans **5** as oils or as solids (crystallization from pentane) (for experimental details, see Table 3).

Table 3. Experimental details for the Sonogashira-cyclocondensation-Suzuki sequence.

Acid chloride **1**	THP Propargyl ether **2**	Boronic acid **4**	Furan **5**
171 mg (1.00 mmol) of **1b**	141 mg (1.00 mmol) of **2a**	128 mg (1.05 mmol) of **4a**	115 mg (50%) of **5a**
147 mg (1.00 mmol) of **1c**	247 mg (1.00 mmol) of **2c**	128 mg (1.05 mmol) of **4a**	174 mg (52%) of **5b**
167 mg (1.00 mmol) of **1d**	168 mg (1.00 mmol) of **2b**	137 mg (1.05 mmol) of **4b**	117 mg (42%) of **5c**
107 mg (1.00 mmol) of **1h**	141 mg (1.00 mmol) of **2a**	188 mg (1.05 mmol) of **4c**	122 mg (52%) of **5d**

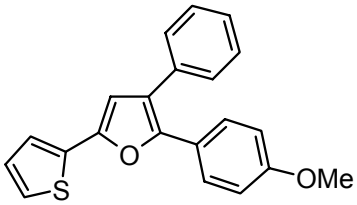
2-(4-Methoxy-phenyl)-4-phenyl-furan (5a)



According to standard procedure the reaction gave rise to 115 mg (50 %) of **5a** as a colorless solid. R_f = 0.42 (hexane/ethyl acetate 9:1), Mp 129 °C. ^{1}H NMR (acetone-d_6, 300 MHz): δ = 3.84 (s, 3 H), 7.02 (d, ^3J = 8.8 Hz, 2 H), 7.16 (d, J = 0.7 Hz, 1 H), 7.28 (tt, J = 1.3, 7.4 Hz, 1 H), 7.36-7.44 (m, 2 H), 7.63-7.68 (m, 2 H), 7.72 (d, ^3J = 8.8 Hz, 2 H), 8.02 (d, J = 0.7 Hz, 1 H). ^{13}C NMR (acetone-d_6, 75 MHz): δ = 55.6 (CH_3), 103.3 (CH), 115.1 (CH), 124.5 (C_{quat}), 126.1 (CH), 126.5 (CH), 127.8 (CH), 129.3 (C_{quat}), 129.6 (CH), 133.4 (C_{quat}), 138.6 (CH), 155.8 (C_{quat}), 160.4 (C_{quat}). EI MS (*m/z* (%)):250 (M^+, 100), 235 ($M^+ - CH_3$, 14), 221 ($M^+ - CHO$, 15), 207 (16), 178 (21). IR (KBr): $\tilde{\nu}$ 1611 cm^{-1}, 1500, 1251, 1179, 1037, 1023, 913, 838, 804, 749, 692. UV/Vis

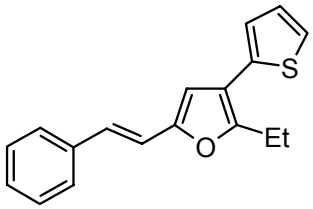
(CH_2Cl_2): λ_{max} (ε): 248 nm (15300), 280 (23600), 290 (20800). Anal calcd. for $C_{17}H_{14}O_2$ (250.30): C 81.58, H 5.64. Found: C 81.20, H 5.63.

2-(4-Methoxy-phenyl)-3-phenyl-5-thiophen-2-yl-furan (5b)



According to standard procedure the reaction gave rise to 174 mg (52 %) of **5b** as a colorless oil, R_f = 0.62 (hexane/ethyl acetate 9:1). ^{1}H NMR ($CDCl_3$, 300 MHz): δ = 3.82 (s, 3 H), 6.68 (s, 1 H), 6.87 (d, ^3J = 8.8 Hz, 2 H), 7.08 (dd, J = 3.7, 5.1 Hz, 1 H), 7.23–7.27 (m, 1 H), 7.31–7.42 (m, 4 H), 7.44–7.49 (m, 2 H), 7.54 (d, ^3J = 9.2 Hz, 2 H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ = 55.1 (CH_3), 109.0 (CH), 113.7 (CH), 122.3 (CH), 122.8 (C_{quat}), 123.5 (C_{quat}), 123.9 (CH), 127.0 (CH), 127.51 (CH), 127.54 (CH), 128.43 (CH), 128.47 (CH), 133.5 (C_{quat}), 134.0 (C_{quat}), 147.5 (C_{quat}), 147.57 (C_{quat}), 159.0 (C_{quat}).

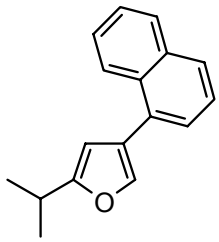
2-Ethyl-5-styryl-3-thiophen-2-yl)-furan (5c)



According to standard procedure the reaction gave rise to 117 mg (42%) of **5c** as a yellow oil, R_f = 0.62 (hexane/ethyl acetate 9:1). ^{1}H NMR ($CDCl_3$, 300 MHz): δ = 1.38 (t, ^3J = 7.5 Hz, 3 H), 2.94 (q, ^3J = 7.4 Hz, 2 H), 6.50 (s, 1 H), 6.87 (d, ^3J = 16.5 Hz, 1 H), 7.02-7.10 (m, 3 H), 7.23-7.30 (m, 2 H), 7.37 (t, J = 7.5 Hz, 2 H), 7.50 (d, J = 7.0 Hz, 2 H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ = 12.5 (CH_3), 20.7 (CH_2), 109.6 (CH), 116.0 (CH), 116.1 (C_{quat}), 123.5 (CH), 123.6 (CH), 126.1 (CH), 126.5 (CH), 127.2 (CH), 127.3 (CH), 128.5 (CH), 135.5 (C_{quat}), 136.9 (C_{quat}), 150.9 (C_{quat}), 152.8 (C_{quat}). EI MS (*m/z* (%)): 282 (^{34}S-M^+, 6), 280 (^{32}S-M^+, 100), 267 (^{34}S-M^+ – CH_3, 10), 265 (^{32}S-M^+ – CH_3, 69), 223 (M^+ – $C_3H_5CO^+$, 11).

Supplementary Material (ESI) for Chemical Communications

2-Isopropyl-4-naphthalen-1-yl-furan (5d)



According to standard procedure the reaction gave rise to 122 mg (52%) of **5d** as a colorless oil, R_f = 0.62 (hexane/ethyl acetate 20:1). ^{1}H NMR ($CDCl_3$, 300 MHz): δ = 1.36 (d, ^3J = 7.0 Hz, 6 H), 3.06 (sept, ^3J = 7.0 Hz, 1 H), 6.32 (t, 1 H), 7.45–7.57 (m, 5 H), 7.79–7.85 (m, 1 H), 7.86–7.93 (m, 1 H), 8.19–8.26 (m, 1 H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ = 20.9 (CH_3), 27.8 (CH), 105.5 (CH), 124.9 (C_{quat}), 125.2 (CH), 125.56 (CH), 125.58 (CH), 125.8 (CH), 126.4 (CH), 127.3 (CH), 128.2 (CH), 131.2 (C_{quat}), 131.6 (C_{quat}), 133.7 (C_{quat}), 138.2 (CH), 161.8 (C_{quat}).

II. X-Ray structure data of 3i

Table 4: Crystal data and structure refinement for **3i**.

Identification code	aka14 (**3i**)
Empirical formula	$C_{10}H_6INO_3$
Formula weight	315.06
Temperature	200(2) K
Wavelength	0.71073 Å
Crystal system	Triclinic
Space group	$P\bar{1}$
Z	8
Unit cell dimensions	a = 8.2679(1) Å α = 84.927(1) deg. b = 11.0675(1) Å β = 83.749(1) deg. c = 22.2477(2) Å γ = 88.385(1) deg.
Volume	2015.39(4) $Å^3$
Density (calculated)	2.08 g/cm^3
Absorption coefficient	3.16 mm^{-1}
Crystal shape	polyhedron
Crystal size	0.50 x 0.34 x 0.30 mm^3
Theta range for data collection	0.9 to 27.5 deg.
Index ranges	$-10\le h\le 10$, $-14\le k\le 14$, $-28\le l\le 28$
Reflections collected	20833
Independent reflections	9160 (R(int) = 0.0201)
Observed reflections	8200 ($I > 2\sigma(I)$)
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.45 and 0.30
Refinement method	Full-matrix least-squares on F^2
Data/restraints/parameters	9160 / 0 / 542
Goodness-of-fit on F^2	1.10
Final R indices ($I>2\sigma(I)$)	R1 = 0.021, wR2 = 0.049
Largest diff. peak and hole	0.69 and -0.35 $eÅ^{-3}$

Table 5: Atomic coordinates and equivalent isotropic displacement parameters ($Å^2$) for **3i**. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

Atom	x	y	z	U_{eq}
I11	0.7953(1)	0.3887(1)	0.1785(1)	0.0297(1)
C11	0.9512(3)	0.2439(2)	0.1594(1)	0.0258(4)
N11	1.5350(2)	-0.3171(2)	0.2257(1)	0.0296(4)
C21	1.0352(3)	0.1676(2)	0.2015(1)	0.0259(4)
C31	1.1141(3)	0.0811(2)	0.1695(1)	0.0243(4)
O41	1.0843(2)	0.0994(1)	0.1095(1)	0.0304(3)
C51	0.9850(3)	0.1998(2)	0.1051(1)	0.0307(5)
C61	1.2178(2)	-0.0226(2)	0.1852(1)	0.0242(4)
C71	1.2881(3)	-0.0326(2)	0.2401(1)	0.0269(5)
C81	1.3913(3)	-0.1288(2)	0.2538(1)	0.0272(5)
C91	1.4247(3)	-0.2153(2)	0.2124(1)	0.0240(4)
C101	1.3550(3)	-0.2091(2)	0.1583(1)	0.0286(5)
O111	1.6026(2)	-0.3209(2)	0.2722(1)	0.0400(4)
C111	1.2508(3)	-0.1128(2)	0.1455(1)	0.0275(5)
O121	1.5545(2)	-0.3953(2)	0.1896(1)	0.0464(5)
I22	0.6609(1)	0.1085(1)	0.3166(1)	0.0303(1)
N12	1.3586(2)	-0.6215(2)	0.2774(1)	0.0314(4)
C12	0.8055(3)	-0.0424(2)	0.3373(1)	0.0271(4)
C22	0.8733(3)	-0.1274(2)	0.2965(1)	0.0268(4)

C32	0.9627(3)	-0.2085(2)	0.3294(1)	0.0250(4)
O42	0.9532(2)	-0.1777(1)	0.3884(1)	0.0329(4)
C52	0.8564(3)	-0.0766(2)	0.3914(1)	0.0344(5)
C62	1.0642(3)	-0.3133(2)	0.3150(1)	0.0239(4)
C72	1.0540(3)	-0.3694(2)	0.2617(1)	0.0280(5)
C82	1.1510(3)	-0.4693(2)	0.2489(1)	0.0287(5)
C92	1.2580(3)	-0.5138(2)	0.2898(1)	0.0251(4)
C102	1.2721(3)	-0.4598(2)	0.3429(1)	0.0285(5)
O112	1.4535(3)	-0.6572(2)	0.3142(1)	0.0504(5)
C112	1.1760(3)	-0.3594(2)	0.3548(1)	0.0275(5)
O122	1.3436(2)	-0.6717(2)	0.2314(1)	0.0411(4)
I33	0.3350(1)	0.9223(1)	0.4484(1)	0.0310(1)
N13	1.0629(2)	0.2131(2)	0.3981(1)	0.0318(4)
C13	0.4866(3)	0.7752(2)	0.4673(1)	0.0265(4)
C23	0.5641(3)	0.6965(2)	0.4251(1)	0.0272(5)
C33	0.6481(3)	0.6118(2)	0.4572(1)	0.0250(4)
O43	0.6273(2)	0.6333(1)	0.5177(1)	0.0346(4)
C53	0.5280(3)	0.7340(2)	0.5221(1)	0.0345(5)
C63	0.7517(3)	0.5087(2)	0.4416(1)	0.0239(4)
C73	0.7778(3)	0.4773(2)	0.3816(1)	0.0278(5)
C83	0.8794(3)	0.3804(2)	0.3672(1)	0.0284(5)
C93	0.9534(3)	0.3148(2)	0.4129(1)	0.0244(4)
C103	0.9282(3)	0.3422(2)	0.4730(1)	0.0297(5)
C113	0.8281(3)	0.4400(2)	0.4865(1)	0.0290(5)
O113	1.1050(2)	0.1990(2)	0.3447(1)	0.0470(5)
O123	1.1071(3)	0.1454(2)	0.4402(1)	0.0538(5)
I44	0.1396(1)	0.5906(1)	0.0540(1)	0.0304(1)
C14	0.2857(3)	0.4407(2)	0.0355(1)	0.0266(4)
N14	0.8377(2)	-0.1376(2)	0.1010(1)	0.0307(4)
C24	0.3600(3)	0.3595(2)	0.0779(1)	0.0279(5)
C34	0.4431(3)	0.2751(2)	0.0451(1)	0.0259(4)
O44	0.4234(2)	0.3002(2)	-0.0154(1)	0.0365(4)
C54	0.3270(3)	0.4023(2)	-0.0195(1)	0.0354(5)
C64	0.5439(3)	0.1702(2)	0.0604(1)	0.0254(4)
C74	0.5599(3)	0.1295(2)	0.1206(1)	0.0251(4)
C84	0.6559(3)	0.0282(2)	0.1343(1)	0.0262(4)
C94	0.7360(3)	-0.0311(2)	0.0874(1)	0.0251(4)
C104	0.7245(3)	0.0075(2)	0.0270(1)	0.0343(5)
O114	0.8299(2)	-0.1837(2)	0.1533(1)	0.0461(5)
C114	0.6275(3)	0.1073(2)	0.0141(1)	0.0337(5)
O124	0.9287(2)	-0.1767(2)	0.0591(1)	0.0471(5)

Table 6: Hydrogen coordinates and isotropic displacement parameters ($Å^2$) for **3i**.

Atom	x	y	z	U_{eq}
H21	1.0357	0.1759	0.2436	0.031
H51	0.9453	0.2336	0.0686	0.037
H71	1.2645	0.0274	0.2680	0.032
H81	1.4387	-0.1358	0.2911	0.033
H101	1.3783	-0.2697	0.1307	0.034
H111	1.2008	-0.1080	0.1089	0.033
H22	0.8589	-0.1274	0.2547	0.032
H52	0.8288	-0.0358	0.4270	0.041
H72	0.9798	-0.3385	0.2342	0.034
H82	1.1445	-0.5071	0.2125	0.034
H102	1.3463	-0.4914	0.3703	0.034
H112	1.1855	-0.3206	0.3907	0.033
H23	0.5578	0.7024	0.3825	0.033

H53	0.4933	0.7695	0.5588	0.041
H73	0.7256	0.5228	0.3507	0.033
H83	0.8982	0.3591	0.3265	0.034
H103	0.9785	0.2951	0.5038	0.036
H113	0.8106	0.4611	0.5272	0.035
H24	0.3531	0.3634	0.1206	0.033
H54	0.2939	0.4405	-0.0562	0.042
H74	0.5047	0.1715	0.1524	0.030
H84	0.6664	0.0001	0.1753	0.031
H104	0.7820	-0.0338	-0.0046	0.041
H114	0.6168	0.1342	-0.0270	0.040

Table 7: Anisotropic displacement parameters ($Å^2$) for **3i**. The anisotropic displacement factor exponent takes the form: $-2\ pi^2\ (h^2\ a^{*2}\ U_{11} + ... + 2\ h\ k\ a^*\ b^*\ U_{12})$

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
I11	0.0291(1)	0.0239(1)	0.0356(1)	-0.0012(1)	-0.0041(1)	0.0075(1)
C11	0.0240(10)	0.0241(11)	0.0289(11)	-0.0006(9)	-0.0032(8)	0.0032(8)
N11	0.0281(10)	0.0263(10)	0.0332(10)	0.0013(8)	-0.0033(8)	0.0048(8)
C21	0.0269(11)	0.0267(11)	0.0242(11)	-0.0028(8)	-0.0043(8)	0.0061(8)
C31	0.0252(10)	0.0276(11)	0.0201(10)	-0.0009(8)	-0.0043(8)	0.0028(8)
O41	0.0349(9)	0.0346(9)	0.0219(7)	-0.0037(6)	-0.0053(6)	0.0117(7)
C51	0.0314(12)	0.0321(12)	0.0281(11)	0.0020(9)	-0.0073(9)	0.0094(9)
C61	0.0202(10)	0.0280(11)	0.0234(10)	-0.0006(8)	-0.0001(8)	0.0034(8)
C71	0.0316(11)	0.0268(11)	0.0225(11)	-0.0036(8)	-0.0042(9)	0.0041(9)
C81	0.0280(11)	0.0302(12)	0.0240(11)	-0.0032(9)	-0.0056(9)	0.0033(9)
C91	0.0242(10)	0.0204(10)	0.0269(11)	0.0007(8)	-0.0029(8)	0.0027(8)
C101	0.0310(12)	0.0279(12)	0.0277(11)	-0.0078(9)	-0.0047(9)	0.0054(9)
O111	0.0434(10)	0.0420(10)	0.0350(9)	0.0024(8)	-0.0142(8)	0.0128(8)
C111	0.0276(11)	0.0327(12)	0.0236(11)	-0.0066(9)	-0.0073(8)	0.0058(9)
O121	0.0545(12)	0.0354(10)	0.0523(11)	-0.0158(8)	-0.0151(9)	0.0225(9)
I22	0.0308(1)	0.0229(1)	0.0364(1)	-0.0027(1)	-0.0015(1)	0.0064(1)
N12	0.0337(11)	0.0280(10)	0.0317(10)	-0.0030(8)	-0.0022(8)	0.0072(8)
C12	0.0274(11)	0.0216(11)	0.0316(12)	-0.0018(9)	-0.0019(9)	0.0027(8)
C22	0.0312(11)	0.0258(11)	0.0231(11)	-0.0014(8)	-0.0033(9)	0.0045(9)
C32	0.0264(11)	0.0252(11)	0.0234(10)	-0.0031(8)	-0.0020(8)	0.0006(8)
O42	0.0424(9)	0.0323(9)	0.0252(8)	-0.0068(7)	-0.0081(7)	0.0118(7)
C52	0.0421(14)	0.0295(12)	0.0322(12)	-0.0098(10)	-0.0045(10)	0.0106(10)
C62	0.0254(10)	0.0214(10)	0.0240(10)	0.0014(8)	-0.0011(8)	0.0018(8)
C72	0.0290(11)	0.0312(12)	0.0242(11)	-0.0022(9)	-0.0066(9)	0.0042(9)
C82	0.0317(12)	0.0305(12)	0.0248(11)	-0.0063(9)	-0.0044(9)	0.0040(9)
C92	0.0269(11)	0.0230(11)	0.0248(11)	-0.0024(8)	-0.0003(8)	0.0029(8)
C102	0.0334(12)	0.0281(12)	0.0244(11)	-0.0010(9)	-0.0083(9)	0.0054(9)
O112	0.0574(12)	0.0453(11)	0.0521(12)	-0.0145(9)	-0.0222(10)	0.0306(9)
C112	0.0350(12)	0.0257(11)	0.0224(10)	-0.0037(8)	-0.0059(9)	0.0059(9)
O122	0.0512(11)	0.0384(10)	0.0344(9)	-0.0126(8)	-0.0029(8)	0.0115(8)
I33	0.0330(1)	0.0230(1)	0.0371(1)	-0.0042(1)	-0.0054(1)	0.0087(1)
N13	0.0326(10)	0.0278(10)	0.0352(11)	-0.0057(8)	-0.0045(8)	0.0069(8)
C13	0.0269(11)	0.0199(10)	0.0326(12)	-0.0038(9)	-0.0030(9)	0.0048(8)
C23	0.0321(12)	0.0228(11)	0.0271(11)	-0.0034(9)	-0.0050(9)	0.0047(9)
C33	0.0279(11)	0.0230(11)	0.0243(10)	-0.0032(8)	-0.0026(8)	0.0015(8)
O43	0.0467(10)	0.0331(9)	0.0238(8)	-0.0040(7)	-0.0055(7)	0.0153(7)
C53	0.0431(14)	0.0297(12)	0.0304(12)	-0.0073(10)	-0.0033(10)	0.0147(10)
C63	0.0265(11)	0.0194(10)	0.0252(11)	-0.0003(8)	-0.0018(8)	0.0004(8)
C73	0.0321(12)	0.0258(11)	0.0257(11)	-0.0003(9)	-0.0068(9)	0.0038(9)
C83	0.0336(12)	0.0282(11)	0.0234(11)	-0.0035(9)	-0.0023(9)	0.0033(9)
C93	0.0268(11)	0.0183(10)	0.0279(11)	-0.0025(8)	-0.0024(8)	0.0041(8)
C103	0.0364(12)	0.0262(11)	0.0264(11)	0.0001(9)	-0.0066(9)	0.0079(9)
C113	0.0393(13)	0.0270(11)	0.0207(10)	-0.0034(9)	-0.0033(9)	0.0072(9)

O113	0.0550(12)	0.0481(11)	0.0377(10)	-0.0151(8)	0.0005(8)	0.0199(9)
O123	0.0708(14)	0.0436(11)	0.0461(11)	-0.0033(9)	-0.0116(10)	0.0352(10)
I44	0.0299(1)	0.0239(1)	0.0384(1)	-0.0053(1)	-0.0087(1)	0.0097(1)
C14	0.0229(10)	0.0224(10)	0.0341(12)	-0.0013(9)	-0.0043(9)	0.0061(8)
N14	0.0333(10)	0.0262(10)	0.0332(11)	-0.0035(8)	-0.0084(8)	0.0103(8)
C24	0.0303(11)	0.0254(11)	0.0273(11)	-0.0014(9)	-0.0026(9)	0.0065(9)
C34	0.0258(11)	0.0262(11)	0.0247(11)	0.0004(8)	-0.0014(8)	0.0047(8)
O44	0.0451(10)	0.0387(10)	0.0245(8)	-0.0014(7)	-0.0051(7)	0.0224(8)
C54	0.0386(13)	0.0357(13)	0.0307(12)	0.0024(10)	-0.0074(10)	0.0171(10)
C64	0.0233(10)	0.0246(11)	0.0272(11)	0.0002(8)	-0.0009(8)	0.0049(8)
C74	0.0254(10)	0.0248(11)	0.0244(11)	-0.0040(8)	0.0012(8)	0.0045(8)
C84	0.0289(11)	0.0247(11)	0.0237(10)	0.0021(8)	-0.0014(8)	0.0018(8)
C94	0.0265(11)	0.0195(10)	0.0288(11)	-0.0002(8)	-0.0040(8)	0.0058(8)
C104	0.0403(13)	0.0352(13)	0.0264(12)	-0.0043(10)	-0.0017(10)	0.0159(10)
O114	0.0594(12)	0.0406(10)	0.0356(10)	0.0080(8)	-0.0072(8)	0.0221(9)
C114	0.0410(13)	0.0353(13)	0.0234(11)	-0.0007(9)	-0.0026(10)	0.0171(10)
O124	0.0579(12)	0.0422(11)	0.0403(10)	-0.0094(8)	-0.0058(9)	0.0321(9)

Table 8: Bond lengths (Å) and angles (deg) for **3i**.

I11-C11	2.072(2)	O43-C53	1.370(3)
C11-C51	1.344(3)	C63-C113	1.396(3)
C11-C21	1.428(3)	C63-C73	1.403(3)
N11-O111	1.225(2)	C73-C83	1.382(3)
N11-O121	1.227(2)	C83-C93	1.384(3)
N11-C91	1.460(3)	C93-C103	1.390(3)
C21-C31	1.354(3)	C103-C113	1.378(3)
C31-O41	1.379(2)	I44-C14	2.069(2)
C31-C61	1.454(3)	C14-C54	1.336(3)
O41-C51	1.366(3)	C14-C24	1.421(3)
C61-C111	1.393(3)	N14-O114	1.223(2)
C61-C71	1.403(3)	N14-O124	1.234(2)
C71-C81	1.380(3)	N14-C94	1.459(3)
C81-C91	1.390(3)	C24-C34	1.360(3)
C91-C101	1.384(3)	C34-O44	1.378(3)
C101-C111	1.381(3)	C34-C64	1.448(3)
I22-C12	2.074(2)	O44-C54	1.366(3)
N12-O122	1.226(2)	C64-C74	1.393(3)
N12-O112	1.227(3)	C64-C114	1.403(3)
N12-C92	1.463(3)	C74-C84	1.386(3)
C12-C52	1.337(3)	C84-C94	1.380(3)
C12-C22	1.426(3)	C94-C104	1.385(3)
C22-C32	1.361(3)	C104-C114	1.374(3)
C32-O42	1.378(2)	C51-C11-C21	106.79(19)
C32-C62	1.451(3)	C51-C11-I11	125.91(16)
O42-C52	1.359(3)	C21-C11-I11	127.20(16)
C62-C72	1.397(3)	O111-N11-O121	123.28(19)
C62-C112	1.404(3)	O111-N11-C91	118.78(18)
C72-C82	1.380(3)	O121-N11-C91	117.94(18)
C82-C92	1.388(3)	C31-C21-C11	106.46(19)
C92-C102	1.388(3)	C21-C31-O41	109.74(18)
C102-C112	1.376(3)	C21-C31-C61	133.9(2)
I33-C13	2.069(2)	O41-C31-C61	116.35(18)
N13-O113	1.224(2)	C51-O41-C31	106.52(16)
N13-O123	1.228(3)	C11-C51-O41	110.49(19)
N13-C93	1.462(3)	C111-C61-C71	118.97(19)
C13-C53	1.341(3)	C111-C61-C31	120.36(19)
C13-C23	1.426(3)	C71-C61-C31	120.66(19)
C23-C33	1.352(3)	C81-C71-C61	120.5(2)
C33-O43	1.379(3)	C71-C81-C91	118.9(2)
C33-C63	1.451(3)	C101-C91-C81	122.0(2)

C101-C91-N11	118.22(19)	C94-C84-C74	118.9(2)
C81-C91-N11	119.78(19)	C84-C94-C104	122.26(19)
C111-C101-C91	118.38(19)	C84-C94-N14	119.58(19)
C101-C111-C61	121.3(2)	C104-C94-N14	118.16(19)
O122-N12-O112	123.42(19)	C114-C104-C94	118.2(2)
O122-N12-C92	118.79(18)	C104-C114-C64	121.4(2)
O112-N12-C92	117.78(18)		
C52-C12-C22	106.87(19)		
C52-C12-I22	126.31(16)		
C22-C12-I22	126.80(16)		
C32-C22-C12	106.29(19)		
C22-C32-O42	109.36(18)		
C22-C32-C62	134.1(2)		
O42-C32-C62	116.55(18)		
C52-O42-C32	106.72(17)		
C12-C52-O42	110.77(19)		
C72-C62-C112	118.93(19)		
C72-C62-C32	121.24(19)		
C112-C62-C32	119.83(19)		
C82-C72-C62	120.5(2)		
C72-C82-C92	119.0(2)		
C82-C92-C102	122.0(2)		
C82-C92-N12	119.62(19)		
C102-C92-N12	118.40(19)		
C112-C102-C92	118.4(2)		
C102-C112-C62	121.18(19)		
O113-N13-O123	123.3(2)		
O113-N13-C93	118.76(19)		
O123-N13-C93	117.94(19)		
C53-C13-C23	106.76(19)		
C53-C13-I33	126.30(16)		
C23-C13-I33	126.92(16)		
C33-C23-C13	106.83(19)		
C23-C33-O43	109.46(19)		
C23-C33-C63	134.2(2)		
O43-C33-C63	116.31(18)		
C53-O43-C33	106.62(17)		
C13-C53-O43	110.33(19)		
C113-C63-C73	119.1(2)		
C113-C63-C33	120.05(19)		
C73-C63-C33	120.82(19)		
C83-C73-C63	120.2(2)		
C73-C83-C93	119.0(2)		
C83-C93-C103	122.3(2)		
C83-C93-N13	119.49(19)		
C103-C93-N13	118.18(19)		
C113-C103-C93	118.0(2)		
C103-C113-C63	121.4(2)		
C54-C14-C24	107.22(19)		
C54-C14-I44	125.65(17)		
C24-C14-I44	127.13(16)		
O114-N14-O124	123.07(19)		
O114-N14-C94	118.83(18)		
O124-N14-C94	118.10(19)		
C34-C24-C14	106.3(2)		
C24-C34-O44	109.50(18)		
C24-C34-C64	134.1(2)		
O44-C34-C64	116.37(18)		
C54-O44-C34	106.53(17)		
C14-C54-O44	110.46(19)		
C74-C64-C114	118.73(19)		
C74-C64-C34	121.34(19)		
C114-C64-C34	119.9(2)		
C84-C74-C64	120.48(19)		

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