Supporting Information

Approaches to Open Fullerenes: Synthesis and Kinetic Stability of Diels-Alder Adducts of Substituted Isobenzofurans and C₆₀

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Supporting Experimental Section

General Methods. All reactions were performed under an argon atmosphere unless otherwise noted. Chemicals were used as purchased without further purification. Distilled solvents were dried as follows: tetrahydrofuran and toluene from sodium/benzophenone; dichloromethane, acetonitrile, 1,2-dichloroethane, and 1,2-dichlorobenzene (ODCB) from calcium hydride. ^1H and ^{13}C NMR spectral data were recorded on 400 or 500 MHz spectrometers. UV–Vis data were recorded on diode-array single beam spectrometers. High-resolution mass spectra of non- C_{60} products were obtained after ionization using EI; high- and low-resolution mass spectra of C_{60} derivatives were obtained after ionization using FAB^+ , FAB^- , and MALDI techniques. TGA was performed using a TG/DTA instrument.

1-Acetoxymethyl-11-oxatricyclo[6.2.1.0^{2,7}]undeca-2,4,6,9-tetraene (3b). According to general procedure A, furfuryl acetate **2b** (2.28 g, 16 mmol) produced the pure bicyclic adduct **3b** (2.64 g, 75%) as a yellow oil. ^1H NMR (500 MHz, CDCl_3) δ (ppm) 2.16 (s, 3H), 4.85 (d, $J = 12.6$ Hz, 1H), 5.01 (d, $J = 12.6$ Hz, 1H), 5.73 (d, $J = 1.8$ Hz, 1H), 6.85 (d, $J = 5.5$ Hz, 1H), 6.98–7.01 (m, 2H), 7.08 (dd, $J = 5.5, 1.8$ Hz, 1H), 7.15–7.17 (m, 1H), 7.24–7.26 (m, 1H); ^{13}C NMR (125.7 MHz, CDCl_3) δ (ppm) 20.8, 61.3, 82.3, 91.0, 119.3, 120.3, 125.1, 125.3, 142.0, 144.9, 147.7, 150.0, 171.0.

1-Carbomethoxy-11-oxatricyclo[6.2.1.0^{2,7}]undeca-2,4,6,9-tetraene (3c). According to general procedure A, 2-carbomethoxyfuran **2c** (3.78 g, 30 mmol) produced pure adduct **3c** (3.94 g, 65%) as a pale orange oil. ^1H NMR (500 MHz, CDCl_3) δ (ppm) 3.98 (s, 3H), 5.81 (s, 1H), 7.00–7.02 (m, 1H), 7.06–7.08 (m, 1H), 7.26–7.28 (m, 1H), 7.35–7.37 (m,

1H); ¹³C NMR (125.7 MHz, CDCl₃) δ (ppm) 52.8, 82.5, 90.3, 119.9, 120.6, 125.2, 125.7, 142.4, 143.6, 147.2, 147.7, 168.2.

1,8-Dimethyl-11-oxatricyclo[6.2.1.0^{2,7}]undeca-2,4,6,9-tetraene (3d). According to general procedure A, 2,5-dimethylfuran (**2d**; 0.96 g, 10 mmol) produced pure **3d** (1.12 g, 65%) as a yellow oil. ¹H NMR (500 MHz, CDCl₃) δ (ppm) 1.79 (s, 6H), 6.65 (s, 2H), 6.90 (m, 4H); ¹³C NMR (125.7 MHz, CDCl₃) δ (ppm) 15.1, 88.3, 118.1, 124.5, 146.6, 152.7.

1,8-Bis-(3'-oxobutyl)-11-oxatricyclo[6.2.1.0^{2,7}]undeca-2,4,6,9-tetraene (3e). According to general procedure A, 2,5-bis(3'-oxobutyl)furan (**2e**; 3.12 g, 15 mmol) produced pure **3e** (2.69 g, 63%) as a yellow oil. ¹H NMR (500 MHz, CDCl₃) δ (ppm) 2.17 (s, 6H), 2.45–2.53 (m, 2H), 2.59–2.67 (m, 2H), 2.71–2.75 (m, 4H), 6.72 (s, 2H), 6.95–6.97 (m, 2H), 7.10–7.12 (m, 2H); ¹³C NMR (125.7 MHz, CDCl₃) δ (ppm) 22.6, 29.9, 38.6, 91.1, 118.7, 124.7, 145.8, 151.4, 207.9; IR (neat) ν (cm⁻¹) 3069, 2950, 2929, 1715, 1359, 1166, 766; CI-MS: *m/z* (rel. intensity) 284 (15, M⁺), 267 (20), 227 (54), 209 (17), 157 (51), 128 (31); HRMS (*m/z*) calcd for C₁₈H₂₀O₃, 284.1412; found, 284.1416.

2,5-Bis(*tert*-butyldimethylsilyloxymethyl)furan (2f). 2,5-Bis(hydroxymethyl)furan¹ (6.41 g, 50 mmol) was dissolved in DMF (50 mL). Imidazole (9.2 g, 135 mmol) and *tert*-butyldimethylsilyl chloride (18.8 g, 125 mmol) were added and the reaction mixture was stirred at 25 °C. After 16 h, the mixture was diluted with Et₂O (200 mL). The mixture was washed with saturated NaHCO₃ (3 × 30 mL), saturated NaCl (30 mL), and water (30 mL). The organic layer was dried (MgSO₄). After evaporation of the solvent under

¹ Timko, J. M.; Moore, S. S.; Walba, D. M.; Hilberly, P. C.; Cram, D. J. *J. Am. Chem. Soc.* **1977**, *99*, 4207–4119.

reduced pressure, a clear yellow oil was obtained as a pure product (17.8 g, 100%). ^1H NMR 400 MHz (CDCl_3) δ (ppm) 0.03 (s, 12H), 0.86 (s, 18H), 4.87 (s, 4H), 6.41 (s, 2H); ^{13}C NMR 100 MHz (CDCl_3) δ (ppm) -5.3, 18.3, 25.8, 58.1, 107.7, 153.8; IR (neat) ν (cm^{-1}) 2929, 2857, 1472, 1256, 1083, 837, 777; FAB-MS m/z (rel. intensity) 355.2 ($[\text{M}-\text{H}]^+$, 72%), 341 ($[\text{M}-\text{CH}_2]^+$, 25%), 299 ($[\text{M}-\text{C}_4\text{H}_9]^+$, 60%), 225 ($[\text{M}-\text{OSiMe}_2t\text{-Bu}]^+$, 100%); HRMS (m/z) $[\text{M}-\text{H}]^+$ calcd for $\text{C}_{18}\text{H}_{35}\text{O}_3\text{Si}_2$, 355.2125; found, 355.2125.

1,8-Bis(hydroxymethyl)-11-oxatricyclo[6.2.1.0^{2,7}]undeca-2,4,6,9-tetraene (3g). The deprotection of the bis(silyl ether) **2f** (1.30 g, 3.0 mmol) was performed by dissolving it in THF (20 mL) containing water (several drops) and then adding 1 M tetra-*n*-butylammoniumfluoride in THF (12 mL, 12.0 mmol). After stirring for 2 h at 20 °C, the solution was filtered through silica gel (eluent: THF) to give pure **3g** (590 mg, 96%) as a yellow oil. ^1H NMR (500 MHz, CDCl_3) δ (ppm) 4.26 (d, $J = 12.5$ Hz, 2H), 4.37 (d, $J = 12.5$ Hz, 2H), 4.73 (s, 2H), 6.91–6.94 (m, 4H), 7.18–7.20 (m, 2H); ^{13}C NMR (125.7 MHz, CDCl_3) δ (ppm) 60.5, 94.1, 120.3, 125.7, 145.0, 151.4; IR (neat) ν (cm^{-1}) 3418, 2925, 2871, 1732, 1713, 1645, 1454, 1305, 1248, 1044, 955. EI-MS: m/z (rel. intensity) 204 (9, M^+), 178 (18), 157 (50), 142 (28), 128 (100, $\text{C}_{10}\text{H}_8^+$); HRMS (m/z) calcd for $\text{C}_{12}\text{H}_{12}\text{O}_3$, 204.0786; found, 204.0787.

1-Hydroxymethyl-11-oxatricyclo[6.2.1.0^{2,7}]undeca-2,4,6,9-tetraene (3h). $\text{LiOH}\cdot\text{H}_2\text{O}$ (218 mg, 5.2 mmol) was added to a solution of compound **3b** (1.08 g, 5 mmol) in $\text{CH}_3\text{OH}/\text{H}_2\text{O}$ (20:1; 21 mL). The progress of the reaction was monitored by TLC until all of the starting materials had reacted. The solvents were evaporated under reduced pressure. The mixture was purified by passing the sample through a short silica gel plug ($\text{CH}_2\text{Cl}_2/\text{MeOH}$, 50:1) and drying to give **3h** (93%) as a white solid. ^1H NMR (500

MHz, CDCl₃) δ (ppm) 2.20 (br, 1H), 4.34 (d, J = 12.6 Hz, 1H), 4.38 (d, J = 12.6 Hz, 1H), 5.64 (d, J = 1.7 Hz, 1H), 6.81 (d, J = 5.5 Hz, 1H), 6.89–6.93 (m, 2H), 6.99 (dd, J = 5.5, 1.7 Hz, 1H), 7.09–7.12 (m, 1H), 7.15–7.18 (m, 1H); ¹³C NMR (125.8 MHz, CDCl₃) δ (ppm) 60.3, 82.1, 93.6, 119.5, 120.1, 125.0, 125.1, 142.3, 144.8, 147.7, 150.4; IR (neat) ν (cm⁻¹) 3448, 3075, 3003, 2921, 1452, 1067, 1036, 970, 938, 868, 762, 692; HRMS (m/z) calcd for C₁₁H₁₀O₂, 174.0681; found, 174.0682.

9,10-(11-Oxatricyclo[6.2.1.0^{2,7}]undeca-2,4,6-trieno)-1',2'-buckminsterfullerene (7a).

According to general procedure B, the reaction of compound **3a** (75 mg, 0.52 mmol), tetrazine **4a** (132 mg, 0.56 mmol), and C₆₀ (288 mg, 0.40 mmol) gave pure adduct **7a** (193 mg, 58%; 66% based on recovered C₆₀) as a brown solid after chromatography (SiO₂; toluene/cyclohexane, 1:1; R_f = 0.7). ¹H NMR (500 MHz, CS₂/CDCl₃ 9:1) δ (ppm) 6.72 (s, 2H), 7.45–7.47 (m, 2H), 7.62–7.65 (m, 2H).

9,10-(1-(2'-Acetoxymethyl)-11-oxatricyclo[6.2.1.0^{2,7}]undeca-2,4,6-trieno)-1'',2''-

buckminsterfullerene (7b). According to general procedure B, the reaction of compound **3b** (24 mg 0.11 mmol), tetrazine **4a** (28 mg, 0.12 mmol), and C₆₀ (72 mg, 0.1 mmol) gave the pure adduct **7b** (29 mg, 31%; 48% based on recovered C₆₀) as a brown solid after chromatography (SiO₂, toluene; R_f = 0.5). ¹H NMR (500 MHz, CDCl₃) δ (ppm) 2.07 (s, 3H), 5.28 (d, J = 12.5 Hz, 1H), 5.68 (d, J = 12.5 Hz, 1H), 6.73 (s, 1H), 7.46–7.48 (m, 2H), 7.57–7.58 (m, 1H), 7.63–7.64 (m, 1H); ¹³C NMR (125.7 MHz, CDCl₃) δ (ppm) 20.0, 62.5, 77.6, 78.0, 88.2, 94.8, 121.5, 121.6, 127.7, 127.9, 137.5, 137.57, 137.60, 137.8, 139.2, 139.4, 139.5, 139.9, 141.3, 141.3, 141.5, 141.68, 141.69, 141.71, 141.73, 141.74, 141.75, 141.76, 142.0, 142.27, 142.31, 142.34, 142.4, 142.71, 142.74, 143.0, 143.7, 143.8, 144.0, 144.15, 144.23, 144.4, 144.86, 144.87, 144.89,

144.91, 144.92, 144.98, 144.99, 145.02, 145.07, 145.12, 145.52, 145.53, 145.65, 145.70, 145.78, 145.79, 145.9, 146.0, 146.62, 146.64, 150.8, 151.0, 151.3, 153.8; IR (KBr) ν (cm^{-1}) 1749, 1213, 521.

9,10-(1-Carbomethoxy-11-oxatricyclo[6.2.1.0^{2,7}]undeca-2,4,6-trieno)-1',2'-

buckminsterfullerene (7c). According to general procedure B, the reaction of compound **3c** (105 mg, 0.52 mmol), tetrazine **4a** (142 mg, 0.6 mmol), and C₆₀ (288 mg, 0.4 mmol) gave the pure adduct **7c** (150 mg, 43%; 57% based on recovered C₆₀) as a brown solid after chromatography (SiO₂, toluene; R_f = 0.7). ¹H NMR (500 MHz, CS₂/CDCl₃ 1:1) δ (ppm) 4.12 (s, 3H), 6.93 (s, 1H), 7.56–7.60 (m, 2H), 7.73–7.74 (m, 1H), 7.93–7.95 (m, 1H); ¹³C NMR (125.7 MHz, CS₂/CDCl₃ 1:1) δ (ppm) 52.7, 76.9, 88.2, 95.0, 121.8, 122.1, 125.1, 128.0, 128.2, 128.6, 128.8, 137.3, 137.6, 137.75, 137.81, 138.3, 139.2, 139.56, 139.57, 140.0, 141.5, 141.70, 141.72, 141.8, 141.9, 141.89, 141.96, 141.98, 142.1, 142.38, 142.41, 142.45, 142.50, 142.8, 143.3, 143.88, 143.94, 144.2, 144.3, 144.6, 144.8, 145.0, 145.1, 145.15, 145.22, 145.24, 145.3, 145.58, 145.60, 145.67, 145.73, 145.77, 145.83, 146.0, 146.1, 146.2, 146.75, 146.81, 146.9, 150.30, 150.34, 153.3, 166.4; IR (KBr) ν (cm^{-1}) 2947, 1739, 1431, 1206, 1115, 526.

9,10-(1,8-Dimethyl-11-oxatricyclo[6.2.1.0^{2,7}]undeca-2,4,6-trieno)-1',2'-

buckminsterfullerene (7d). According to general procedure B, the reaction of compound **3d** (224 mg, 1.3 mmol), tetrazine **4a** (307 mg, 1.3 mmol), and C₆₀ (720 mg, 1.0 mmol) gave the pure adduct **7d** (740 mg, 85%; 89% based on recovered C₆₀) as a brown solid after chromatography (SiO₂) using first toluene/cyclohexane (1:1) to elute the unreacted C₆₀ and then toluene to elute the isobenzofuran adduct (R_f = 0.9; toluene). ¹H NMR (500 MHz, CS₂/CDCl₃ 1:1) δ (ppm) 2.60 (s, 6H), 7.44–7.47 (m, 2H), 7.49–7.52

(m, 2H); ^{13}C NMR (125.7 MHz, $\text{CS}_2/\text{CDCl}_3$ 1:1) δ (ppm) 17.4, 93.0, 120.6, 127.8, 137.6, 137.7, 139.7, 139.8, 141.6, 141.9, 142.0, 142.05, 142.12, 142.5, 142.6, 144.1, 144.5, 144.7, 145.06, 145.12, 145.2, 145.3, 145.7, 145.95, 146.01, 146.04, 146.1, 146.2, 146.88, 146.9, 152.3, 154.1; IR (KBr) ν (cm^{-1}) 2926, 1638, 1384, 1131, 527.

9,10-(1,8-Bis(3'-oxobutyl)-11-oxatricyclo[6.2.1.0^{2,7}]undeca-2,4,6-trieno)-1'',2''-

buckminsterfullerene (7e). According to general procedure B, the reaction of compound **3e** (148 mg, 0.52 mmol), tetrazine **4a** (132 mg, 0.56 mmol), and C_{60} (288 mg, 0.4 mmol) gave the pure adduct **7e** (69 mg, 18%; 52% based on recovered C_{60}) as a brown solid after chromatography (SiO_2) using toluene first to elute the unreacted C_{60} and then toluene/EtOAc (9:1) to elute the isobenzofuran adduct ($R_f = 0.2$; toluene/EtOAc, 9:1). This compound was also prepared using the more-reactive parent tetrazine, **4b**.^{Error!}

Bookmark not defined. Here, a mixture of compound **3e** (44 mg, 0.17 mmol), a solution of tetrazine **4b** in CH_2Cl_2 (0.37 mmol/mL, 0.8 mL), and C_{60} (103 mg, 0.14 mmol) were stirred at 20 °C for 24 h in toluene (100 mL). Chromatography (SiO_2 , toluene; $R_f = 0.2$) gave the pure adduct **7e** (53 mg, 39%; 62% based on recovered C_{60}) as a brown solid. ^1H NMR (500 MHz, $\text{CS}_2/\text{CDCl}_3$ 1:2) δ (ppm) 2.32 (s, 6H), 2.92–2.99 (m, 2H), 3.04–3.17 (m, 4H), 3.39–3.49 (m, 2H), 7.44–7.49 (m, 4H); ^{13}C NMR (125.7 MHz, $\text{CS}_2/\text{CDCl}_3$ 1:2) δ (ppm) 24.8, 29.6, 39.3, 81.1, 95.7, 121.7, 127.8, 137.4, 137.6, 139.6, 139.7, 141.5, 141.80, 141.84, 141.87, 141.91, 142.0, 142.4, 142.5, 142.9, 143.9, 144.40, 144.44, 144.7, 145.0, 145.05, 145.14, 145.3, 145.6, 145.7, 145.8, 145.86, 145.88, 146.1, 146.8, 151.5, 153.3, 205.4; IR (KBr) ν (cm^{-1}) 2929, 1717, 1650, 527.

9,10-(1,8-Bis(hydroxymethyl)-11-oxatricyclo[6.2.1.0^{2,7}]undeca-2,4,6-trieno)-1',2'-

buckminsterfullerene (7g). A mixture of diol **3g** (21 mg, 0.1 mmol), a solution of tetrazine **4b** in CH₂Cl₂ (0.37 mmol/mL, 0.55 mL), and C₆₀ (72 mg, 0.1 mmol) in *o*-dichlorobenzene (80 mL) was stirred at 20 °C for 12 h. Chromatography on SiO₂ eluting first with toluene to remove unreacted C₆₀ and then with toluene/THF (8:2) gave the pure adduct **7g** (30 mg, 32%; 50% based on recovered C₆₀) as a brown solid (R_f = 0.3; toluene/THF 8:2). ¹H NMR (500 MHz, CDCl₃/DMSO-*d*₆ 9:1) δ (ppm) 5.02 (d, *J* = 12.2 Hz, 2H), 5.19 (d, *J* = 12.2 Hz, 2H), 7.50–7.52 (m, 2H), 7.82–7.84 (m, 2H); ¹³C NMR (125.7 MHz, CDCl₃/DMSO-*d*₆ 9:1) δ (ppm) 61.6, 80.2, 96.2, 127.4, 137.2, 137.3, 139.0, 139.2, 141.3, 141.5, 141.77, 141.81, 141.84, 141.9, 142.2, 142.3, 143.8, 144.3, 144.7, 144.8, 144.86, 144.89, 144.94, 145.2, 145.8, 145.9, 146.2, 146.6, 146.7, 152.9, 153.7; FT-IR (KBr) ν (cm⁻¹) 3448, 2921, 2859, 1651, 1637, 526; FAB-MS: *m/z* (rel. intensity) 898 (14, M⁺), 720 (100, C₆₀⁺); HRMS calcd for C₇₀H₁₀O₃: 898.0630; found: 898.0676.

9,10-(1-Hydroxymethyl-11-oxatricyclo[6.2.1.0^{2,7}]undeca-2,4,6-trieno)-1',2'-

buckminsterfullerene (7h). 3,6-(Bipyridyl)-1,2,4,5-tetrazine (**4a**; 39.6 mg, 0.15 mmol), compound **3h** (19.0 mg, 0.11 mmol), and C₆₀ (751 mg, 0.10 mmol) were dissolved in distilled chlorobenzene (70 mL). The reaction was run overnight at 45 °C under Ar. The C₆₀ adducts were separated by flash chromatography, eluting with toluene. Three bands were obtained: first, a purple band for unreacted C₆₀; second, a brown band for the *cis-I* adduct **9a**; third, a slower-running band for the most-polar compound, **7h**. All fractions were rapidly concentrated to dryness. The yields of the *cis-I* **9a** adduct and compound **7h** were 57 and 12%, respectively.

Isobenzofuranol adduct 7h. ^1H NMR (500 MHz $\text{CS}_2/\text{C}_2\text{D}_2\text{Cl}_4(1:1)$) δ (ppm) 1.68 (bs, 1H), 5.15 (d, 1H, $J = 12.8$ Hz), 5.29 (d, 1H, $J = 12.8$ Hz), 6.89 (s, 1H), 7.58 (m, 2H), 7.75 (m, 1H), 7.79 (m, 1H). ^{13}C NMR (500 MHz $\text{CS}_2/\text{C}_2\text{D}_2\text{Cl}_4 (1:1)$) δ (ppm) 62.0, 77.1, 78.2, 88.0, 97.1, 121.85, 121.93, 125.0, 137.1, 137.3, 137.4, 137.50, 137.53, 137.7, 139.2, 139.3, 139.5, 139.8, 141.21, 141.23, 141.4, 141.56 (2 carbons), 141.63, 141.7 (2 carbons), 141.8 (2 carbons), 142.0, 142.1, 142.15, 142.19, 142.23, 142.3, 142.58, 142.61, 142.7, 143.6, 143.7, 144.06, 144.07, 144.2, 144.3, 144.7, 144.80 (2 carbons), 144.83 (2 carbons), 144.92, 144.94, 145.0, 145.05, 145.11, 145.45, 145.47, 145.50, 145.59, 145.61, 145.71, 145.72, 145.8, 145.85, 145.87, 146.6, 150.8, 151.1, 151.9, 153.9; FT-IR (KBr) ν (cm^{-1}) 3458, 2919, 2856, 1507, 526.

Isobenzofuran ether adduct 7h'. ^1H NMR (500 MHz $\text{CS}_2/\text{C}_2\text{D}_2\text{Cl}_4 (1:1)$) δ (ppm) 5.19 (d, 1H, $J = 11.0$ Hz), 5.30 (d, 1H, $J = 11.0$ Hz), 6.48 (s, 1H), 6.73 (s, 1H), 7.46 (m, 3H), 7.65 (d, 1H, $J = 6.89$ Hz). ^{13}C NMR (500 MHz $\text{CS}_2/\text{C}_2\text{D}_2\text{Cl}_4 (1:1)$) δ (ppm) 58.9, 65.5, 78.7, 88.9, 93.3, 99.4, 101.0, 120.8, 122.7, 127.9, 128.1, 136.0, 137.5, 137.9, 138.2, 138.5, 139.1, 140.4, 140.6, 141.2, 141.7, 141.9, 142.1, 142.15, 142.16, 142.22, 142.5, 142.6, 142.7, 142.9, 143.0, 143.46 (2 carbons), 143.50, 143.7, 143.78, 143.84, 143.85, 143.91, 143.96, 144.04, 144.1, 144.2, 144.4, 144.56, 144.62, 144.7, 144.9, 145.0, 145.25, 145.29, 145.5, 146.0, 146.2, 146.3, 146.7, 146.79, 146.83, 146.9, 147.6, 147.9, 147.95, 148.01, 148.6, 148.8, 148.9, 149.00, 150.9, 151.0. MALDI-TOF (m/z) found: 868.0537, calculated 868.0524.

Ortep drawing of compound 7a

1A.

Packing view of crystal structure

Table 1. Crystal data and structure refinement for Compound **7a**

Empirical formula	C ₇₀ H ₆ OS ₄	
Formula weight	990.99	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/n	
Unit cell dimensions	a = 9.927(3) Å	α = 90°.
	b = 31.615(10) Å	β = 108.654(6)°.
	c = 12.614(4) Å	γ = 90°.
Volume	3751(2) Å ³	
Z	4	
Density (calculated)	1.755 Mg/m ³	
Absorption coefficient	0.317 mm ⁻¹	
F(000)	1992	
Crystal size	1 x .2 x .4 mm ³	
Theta range for data collection	1.29 to 28.28°.	
Index ranges	-12 ≤ h ≤ 8, -41 ≤ k ≤ 41, -16 ≤ l ≤ 15	
Reflections collected	24415	
Independent reflections	8959 [R(int) = 0.0314]	
Completeness to theta = 28.28°	96.2 %	
Absorption correction	Empirical	
Max. and min. transmission	1.0000 and 0.8444	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	8959 / 0 / 682	
Goodness-of-fit on F ²	1.051	
Final R indices [I > 2σ(I)]	R1 = 0.0395, wR2 = 0.1008	
R indices (all data)	R1 = 0.0590, wR2 = 0.1099	
Largest diff. peak and hole	0.468 and -0.483 e.Å ⁻³	

[illegible]

¹³C NMR of compound **2f**

¹H NMR of compound **3f**

^{13}C NMR of compound **3f**

¹H NMR of compound **3g**

¹³C NMR of compound **3g**

^1H NMR of compound **7a**

¹H NMR of compound **7b**

¹³C NMR of compound **7b**

^{13}C NMR of compound **7c**

¹H NMR of compound **7d**

^{13}C NMR of compound **7d**

^1H NMR of compound **7e**

^{13}C NMR of compound **7e**

¹H NMR of compound **7f**

¹³C NMR of compound **7f**

¹H NMR of compound **7g**

^{13}C NMR of compound **7g**

¹H NMR of 7h

¹³C NMR of **7h**

^1H NMR of 7h

Rate constant of the decomposition of **7a**

Decomposition of C₆₀-IBF 7a

T (K)	k (s ⁻¹)
343	9.2850E-05
348	1.7386E-04
353	3.6889E-04
358	7.5610E-04
363	1.3406E-03
368	2.1646E-03

Rate constant of the decomposition of **7c**

C₆₀-IBFCO₂Me 7c

T (K)	k (s ⁻¹)
313	1.3584E-04
318	2.4131E-04
323	6.6316E-04
328	1.6686E-03
333	2.9800E-03