Supporting Information for

Difluorobenzocyclooctyne: synthesis, reactivity, and stabilization by β-cyclodextrin

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General Experimental Procedure

All chemical reagents were purchased from Sigma-Aldrich, Acros, and TCI and used without purification unless noted otherwise. Anhydrous DMF and MeOH were purchased from Aldrich or Acros in sealed bottles; all other solvents were purified as described by Pangborn *et al.*[1] In all cases, solvent was removed by reduced pressure with a Buchi Rotovapor R-114 equipped with a Welch self-cleaning dry vacuum. Products were further dried by reduced pressure with an Edwards RV5 high vacuum. Lyophilization was performed on a LABCONCO FreeZone® instrument equipped with an Edwards RV2 pump. Thin layer chromatography was performed with EMD 60 Å silica gel plates. Flash chromatography was performed using Silicycle® 60 Å 230-400 mesh silica. All ^{1}H, ^{13}C, and ^{19}F NMR spectra are reported in ppm and referenced to solvent peaks (^{1}H and ^{13}C). Spectra were obtained on Bruker AVQ-400, AVB-400, DRX-500, AV-500, or AV-600 instruments. CPMAS ^{13}C-NMR was performed on an AV-500 spectrometer (see below for further detail). Electron impact (EI) and electrospray ionization (ESI) mass spectra were obtained from the UC Berkeley Mass Spectrometry Facility. X-ray crystallography structures were obtained from the UC Berkeley X-ray Crystallography Facility (see page S56-S92 for details).

CPMAS ^{13}C-NMR procedure

Solid-state NMR measurements of β-cyclodextrin and DIFBO-β-cyclodextrin complex were taken at rt on a 11.75 T Bruker Avance NMR spectrometer equipped with a double resonance magic angle spinning (MAS) probe. Samples were spun at 10 kHz using zirconia rotors of 4 mm outer diameter and 80 μL sample volume. The 1-D ^{13}C cross-polarization (CP) experiments were conducted using a ^{1}H 90° pulse of 4.2 μs, a 59 kHz ^{1}H-decoupling field, 2 ms contact time, 2 s recycle delay and 1800-4000 scans. The ^{13}C chemical shifts were referenced against the adamantane ^{13}C resonances at 29.45 and 38.48 ppm.

Oligomerization products of DIFBO

Trimer 9. Elutes from reverse phase (C-18) HPLC at 29.7 min using CH_3CN/H_2O solvent system with a gradient of 50% to 100% CH_3CN over 25 min followed by 10 min of 100% CH_3CN. $R_f = 0.45$ in 4:1 hexane/EtOAc. ^{1}H-NMR (500 MHz, $CDCl_3$): δ 7.51 (bs, 1H), 7.33 (td, J = 7.5, 0.9 Hz, 1H), 7.23 (J = 7.5, 1.0 Hz, 1H), 7.18 (d, J = 7.5, 1H), 7.00-7.09 (m, 4H), 6.83-6.89 (m, 3H), 6.68 (d, J = 7.3 Hz, 1H), 2.79-2.84 (m, 1H), 2.53-2.59 (m, 3H), 2.40 (apparent t, J = 6.4 Hz, 2H), 2.31 (td, J = 12.9, 5.6 Hz, 1H), 1.88-2.08 (m, 4H), 1.76-1.80 (m, 1H), 1.64-1.73 (m, 2H), 1.55-1.62 (m, 1H), 1.24-1.47 (m, 3H). ^{13}C-NMR (150 MHz, $CDCl_3$): δ 141.6 (dd, J = 5, 4 Hz), 141.02, 140.99, 139.9 (dd, J = 30, 22 Hz), 139.6 (t, J =4 Hz), 138.9, 138.7 (apparent t, J = 3 Hz), 138.6 (d, J = 3 Hz), 138.3, 137.4, 136.8 (t, J = 23 Hz), 135.5 (dd, J = 33, 26 Hz), 133.4 (q, J = 5 Hz), 132.1, 131.2, 128.5, 128.08, 128.07, 128.01, 127.2, 127.1, 125.1 (dd, J = 247, 244 Hz), 125.0, 124.7, 124.1, 123.1 (dd, J = 247, 244 Hz), 122.8 (dd, J = 254, 240 Hz), 38.7 (td, J =

[1] Pangborn, A. B.; Giardello, M. A.;Grubbs, R. H.; Rosen, R. K.; Timmers, F. J. *Organometallics* **1996**, *15*, 1518.

28, 7 Hz), 37.5 (t, J = 30 Hz), 35.8 (t, J = 28 Hz), 30.64, 30.61, 30.1, 24.0 (apparent q, J = 4 Hz), 23.6 (dd, J = 7, 3 Hz), 21.2 (d, J = 10 Hz). ^{19}F-NMR (564 MHz, $CDCl_3$): δ -59.1 (d, J =255 Hz, 1F), -66.7 (d, J = 274 Hz, 1F), -70.0 (dd, J = 276, 71 Hz, 1F), -71.4 (dtt, J = 257, 70, 13 Hz, 1F), -75.8 (ddd, J = 275, 33, 10 Hz, 1F), -76.9 (dquin, J = 277, 30 Hz, 1F). HRMS (EI): Calcd. for $C_{36}H_{30}F_6^+$ $[M]^+$ 576.2252, found 576.2265.

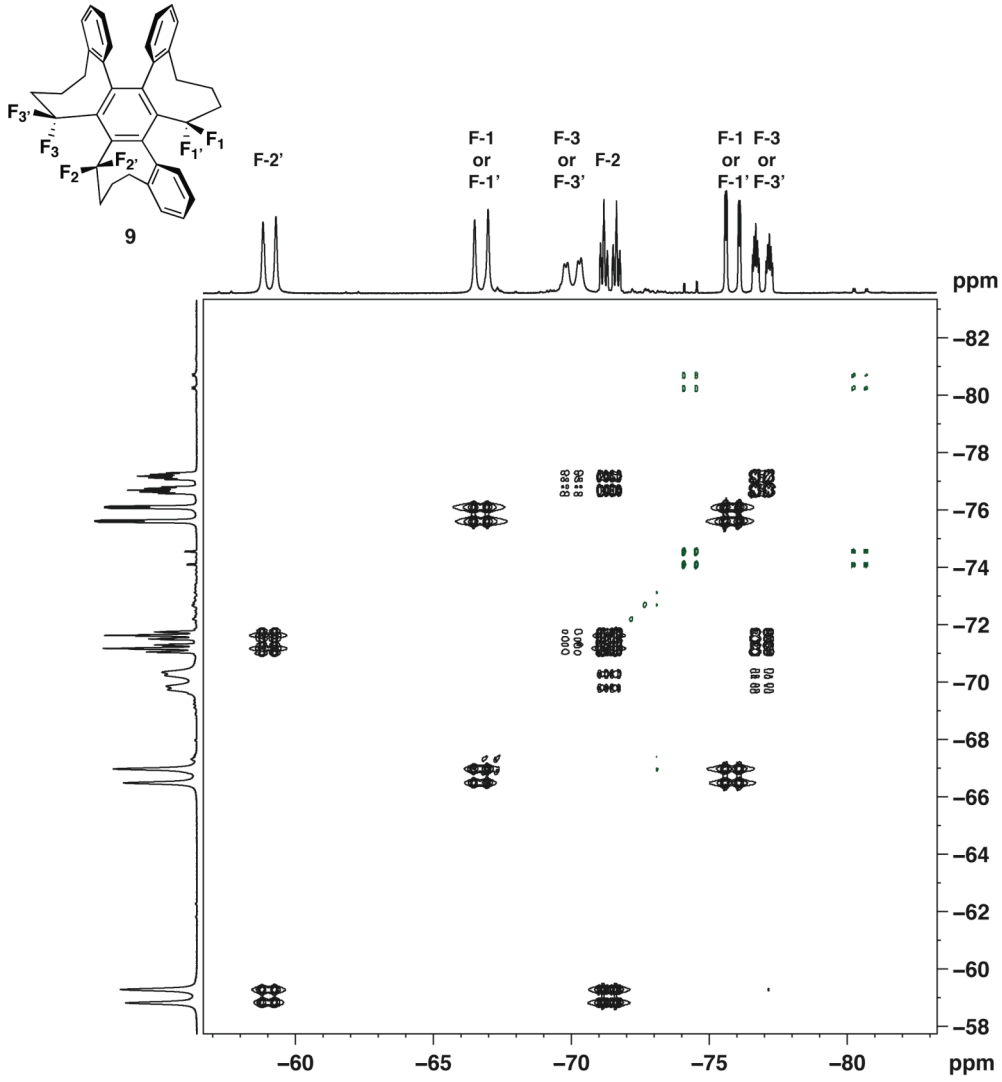


Figure S1. ^{19}F-^{19}F COSY spectrum of trimer **9** in $CDCl_3$. COSY spectra demonstrate spatial interactions of ^{19}F because fluorine atoms exhibit significant through space coupling.[2,3] The ^{19}F-

[2] Ng, S.; Sederholm, C.H. *J. Chem. Phys.* **1964**, *40*, 2090.

[3] ^{19}F-^{19}F COSY spectra are known to exhibit ^{4}J coupling as opposed to the ^{3}J coupling traditionally observed in ^{1}H-^{1}H COSY experiment due to the large through space coupling for fluorine atoms. See Buchanan, G.W.; Munteanu, E.; Dawson, B.A.; Hodgson, D. *Magn. Res. Chem.* **2005**, *43*, 528.

^{19}F NOESY spectrum of trimer **9** contains the same cross peaks observed in the COSY with decreased signal-to-noise.[4] The green peaks represent an impurity.

Trimer 10. Elutes from reverse phase (C-18) HPLC at 31.7 min using CH_3CN/H_2O solvent system with a gradient of 50% to 100% CH_3CN over 25 min followed by 10 min of 100% CH_3CN. R_f = 0.55 in 4:1 hexane/EtOAc. ^{1}H-NMR (500 MHz, $CDCl_3$): δ 7.51-7.45 (m, 1H), 7.31 (td, *J* = 7.5, 1.2 Hz, 1H), 7.21 (td, *J* = 7.7, 1.1 Hz, 1H), 7.14 (d, *J* = 7.5 Hz, 1H), 6.96-7.05 (m, 4H), 6.72 (td, *J* = 7.4, 1.4 Hz, 1H), 6.66 (td, *J* = 7.7, 0.9 Hz, 1H), 6.47 (d, *J* = 7.6 Hz, 1H), 6.33 (d, *J* =7.5 Hz, 1H), 2.67-2.82 (m, 4H), 2.52-2.65 (m, 2H), 2.45-2.51 (m, 1H), 2.33-2.39 (m, 1H), 2.05-2.20 (m, 3H), 1.87-1.98 (m, 2H), 1.76-1.84 (m, 1H), 1.59-1.71 (m, 3H), 1.48-1.58 (m, 1H). ^{13}C-NMR (125 MHz, $CDCl_3$): δ 141.2-141.3 (m), 140.6 (d, *J* = 7 Hz), 140.0 (d, *J* = 4 Hz), 139.7-139.8 (m), 139.5 (d, *J* = 5 Hz), 139.2-139.3 (m), 139.1 (d, *J* = 1 Hz), 138.2 (apparent q, *J* = 22 Hz), 137.1 (apparent t, *J* = 24 Hz), 133.7-133.8 (m), 130.5 (d, *J* = 3 Hz), 129.5, 128.2, 128.03, 127.99, 127.94, 127.2, 127.0, 125.7 (t, *J* = 245 Hz), 125.4, 124.6, 124.3 (apparent d, *J* =245 Hz), 123.8, 123.3 (t, *J* = 246 Hz), 39.0 (td, *J* = 39, 27 Hz), 38.1 (td, *J* = 27, 11 Hz), 36.0 (t, *J* = 27 Hz), 31.1, 30.7, 30.2, 24.8-24.9 (m), 24.3 (apparent quin, *J* = 3 Hz), 23.6 (apparent q, *J* = 4 Hz). ^{19}F-NMR (564 Hz, $CDCl_3$): δ -62.2 (dt, *J* = 255, 16 Hz, 1F), -67.7 (dd, *J* = 270, 17 Hz, 1F), -69.1 (dd, *J* = 255, 25 Hz, 1F), -71.3 (dd, *J* = 253, 81 Hz, 1F), -72.6- -73.6 (m, 2F). HRMS (EI): Calcd. for $C_{36}H_{30}F_6^+$ $[M]^+$ 576.2252, found 576.2252.

[4] Battiste, J.L.; Jing, N.; Newmark, R.A. *J. Fluorine Chem.* **2004**, *125*, 1331.

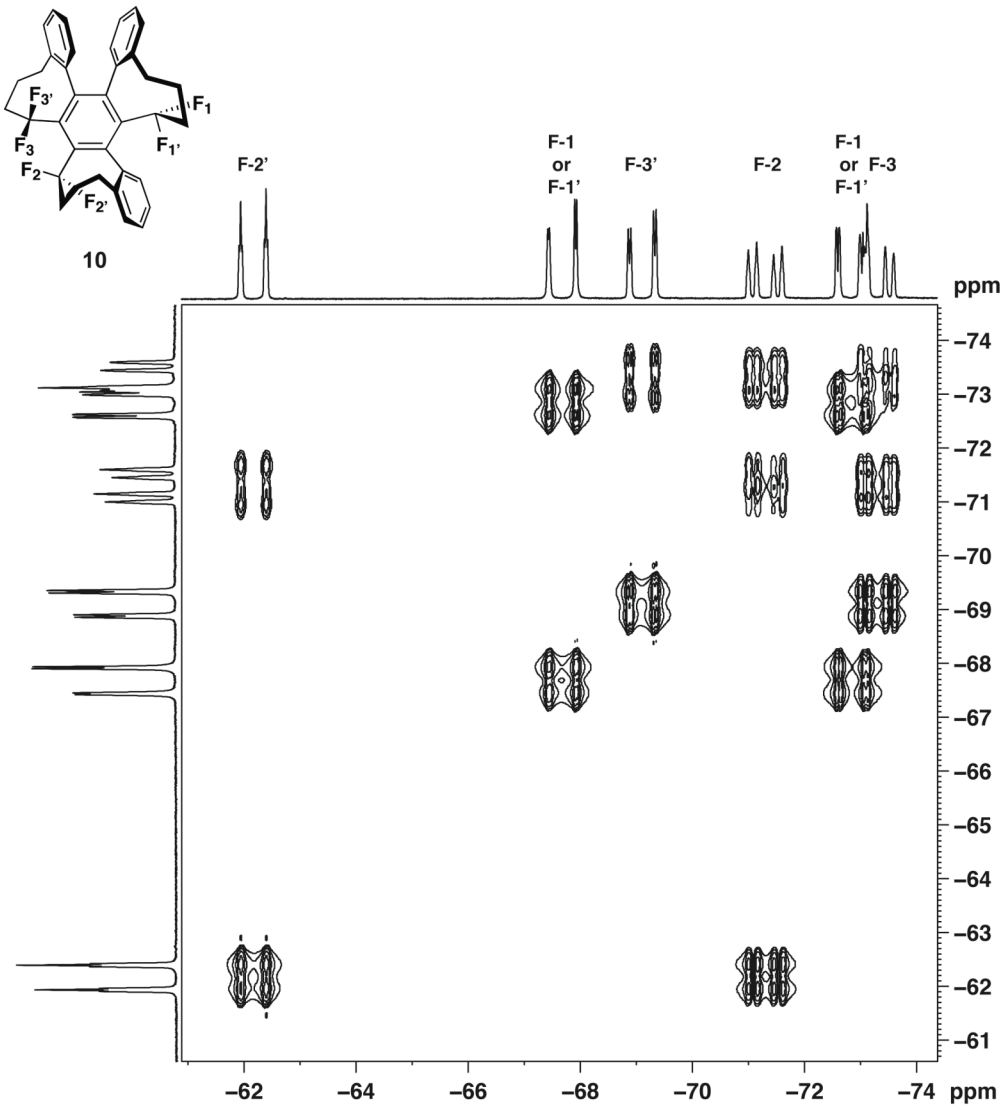


Figure S2. ^{19}F-^{19}F COSY spectrum of trimer **10** in $CDCl_3$. COSY spectra demonstrate spatial interactions of ^{19}F because fluorine atoms exhibit significant through space coupling.[5,6] The ^{19}F-^{19}F NOESY spectrum of trimer **10** contains the same cross peaks observed in the COSY with decreased signal-to-noise.[7]

[5] Ng, S.; Sederholm, C.H. *J. Chem. Phys.* **1964**, *40*, 2090.

[6] ^{19}F-^{19}F COSY spectra are known to exhibit ^{4}J coupling as opposed to the ^{3}J coupling traditionally observed in ^{1}H-^{1}H COSY experiment due to the large through space coupling for fluorine atoms. See Buchanan, G.W.; Munteanu, E.; Dawson, B.A.; Hodgson, D. *Magn. Res. Chem.* **2005**, *43*, 528.

[7] Battiste, J.L.; Jing, N.; Newmark, R.A. *J. Fluorine Chem.* **2004**, *125*, 1331.

Dimer 13. Elutes from reverse phase (C-18) HPLC at 15.3 min using CH_3CN/H_2O solvent system with a gradient of 50% to 100% CH_3CN over 25 min followed by 10 min of 100% CH_3CN. R_f = 0.15 in 4:1 hexane/EtOAc. In solution this compound sits as a mixture of ketal and keto forms in a 3:1 ratio. Minor compound designated as H' (only major product tabulated for ^{13}C and ^{19}F NMR). ^1H-NMR (500 MHz, $CDCl_3$): δ 8.09 (d, *J* = 7.9 Hz, 1H'), 7.53 (d, *J* = 7.9 Hz, 1H), 7.34 (td, *J* = 7.5, 1.2 Hz, 1H), 7.18-7.28 (m, 4H, 3H'), 7.09-7.11 (m, 2H'),7.04 (bs, 1H'), 6.94 (d, *J* = 7.4, 1H'), 6.85 (td, *J* = 7.8, 1.0 Hz, 1H), 6.15 (d, *J* = 7.8 Hz, 1H), 4.21 (bs, 1H'), 4.15 (bs, 1H), 4.04 (bs, 1H'), 3.36 (bs, 1H), 2.94-3.01 (m, 2H), 2.86 (apparent dd, *J* = 14.5, 3.5 Hz, 1H), 2.78 (apparent dd, *J* = 13.3, 6.0 Hz, 1H), 2.64-2.67 (m, 1H'), 2.51 (dt, *J* = 14, 3.5 Hz, 1H'), 2.21-2.39 (m, 2H, 5H'), 1.93-2.19 (m, 4H, 1H'), 1.70-1.83 (m, 1H, 3H'), 1.55-1.61 (m, 1H, 1H'). ^{13}C-NMR (150 MHz, $CDCl_3$): δ 146.7 (t, *J* = 8.4 Hz), 141.8, 140.1 (d, *J* = 3 Hz), 135.6, 133.8 (t, *J* = 30 Hz), 131.5, 130.3, 129.9, 128.9, 128.5, 126.9, 126.7, 125.8, 121.6 (dd, *J* = 256, 250 Hz), 120.0 (dd, *J* = 240, 232 Hz), 110.3, 107.0 (dd, *J* = 33, 25 Hz), 36.6 (apparent td, *J* = 25, 6Hz), 34.4, 32.2 (t, *J* = 26 Hz), 30.4, 24.9 (d, *J* = 9 Hz), 23.1 (d, *J* = 11 Hz). ^{19}F-NMR (564 MHz, $CDCl_3$): δ -74.8 (d, *J* = 270 Hz, 1F), -93.6 (d, *J* = 287 Hz, 1F), -99.1 (d, *J* = 259 Hz, 1F), -110.2 (d, *J* = 260 Hz, 1F). HRMS (ESI): Calcd. for $C_{24}H_{22}F_4O_3Na^+$ $[M+Na]^+$ 457.1403, found 457.1413.

Dimer 14. Elutes from reverse phase (C-18) HPLC at 21.3 min using CH_3CN/H_2O solvent system with a gradient of 50% to 100% CH_3CN over 25 min followed by 10 min of 100% CH_3CN. R_f = 0.4 in 4:1 hexane/EtOAc. ^1H-NMR (500 MHz, $CDCl_3$): δ 7.90 (dd, *J* = 8.0, 1.4 Hz, 1H), 7.52 (td, *J* = 7.5, 1.5 Hz, 1H), 7.30 (td, *J* = 6.7, 1.2 Hz, 1H), 7.25 (d, *J* = 9.2 Hz, 1H), 3.42 (bs, 2H), 2.33 (bs, 2H), 2.01 (bs, 2H). ^{13}C-NMR (125 MHz, $CDCl_3$): δ 193.3 (d, *J* = 3 Hz), 145.1-145.6 (m), 143.0, 135.6, 134.8, 131.8, 129.9, 127.1, 120.4 (ddd, *J* = 253, 246, 8 Hz), 35.8-36.0 (m), 31.3, 23.3 (t, *J* = 2 Hz). ^{19}F-NMR (564 MHz, $CDCl_3$): δ -86.5 (bs, 2F). HRMS (ESI): Calcd. for $C_{24}H_{20}O_2F_4Na^+$ $[M+Na]^+$ 439.1292, found 439.1270.

Determination of the regiochemistry of triazole isomers 11 and 12

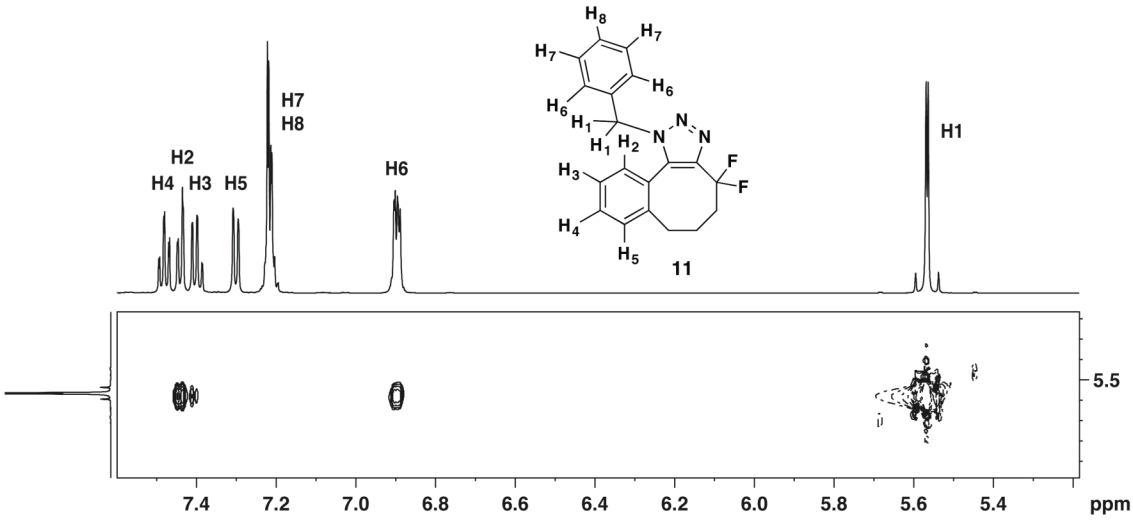


Figure S3. Portion of a ^{1}H-^{1}H NOESY spectrum in CD_3CN of triazole **11** depicting the interactions between benzylic protons H1 and other aryl protons within compound **11**.

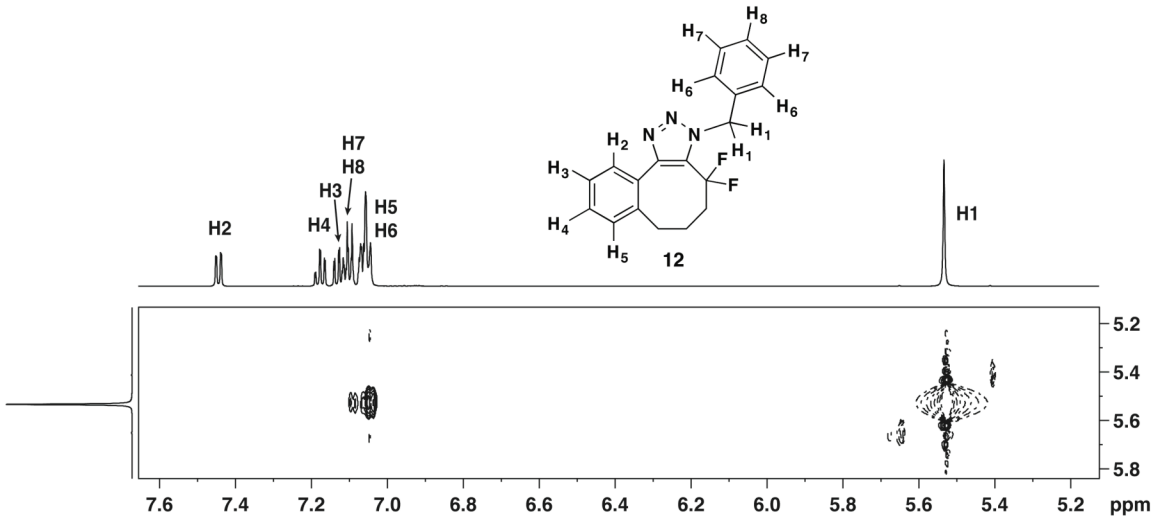


Figure S4. Portion of a ^{1}H-^{1}H NOESY spectrum in CD_3CN of triazole **12** depicting the interactions between benzylic protons H1 and other aryl protons within compound **12**.

NMR of DIFBO-β-cyclodextrin complex

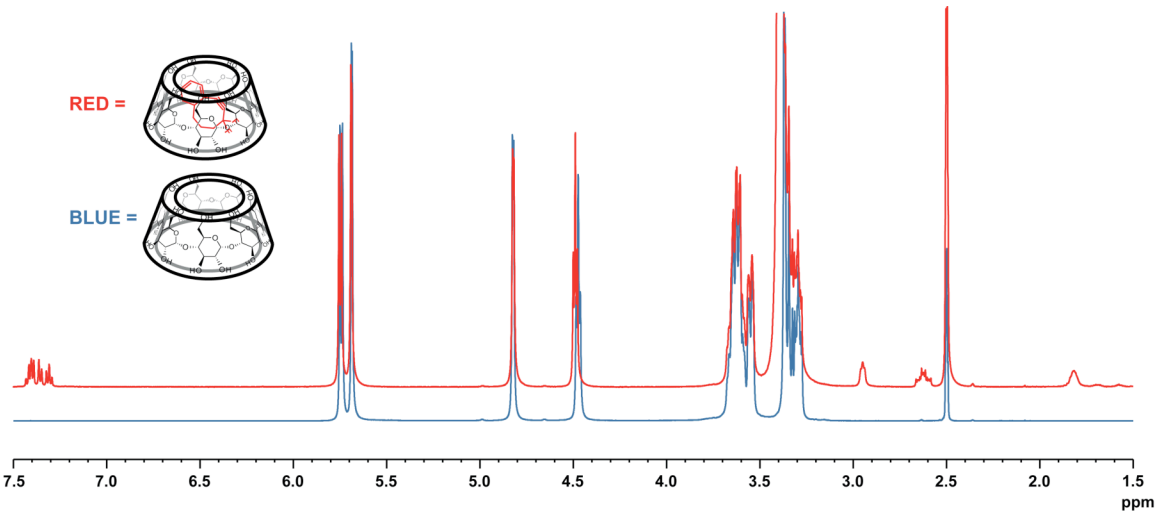


Figure S5. ^{1}H-NMR spectrum in d_6-DMSO of the DIFBO-β-cyclodextrin complex (red) and of β-cyclodextrin alone (blue). The lack of change in chemical shift of the β-cyclodextrin ^{1}H resonances indicates that the complex is not intact in DMSO.

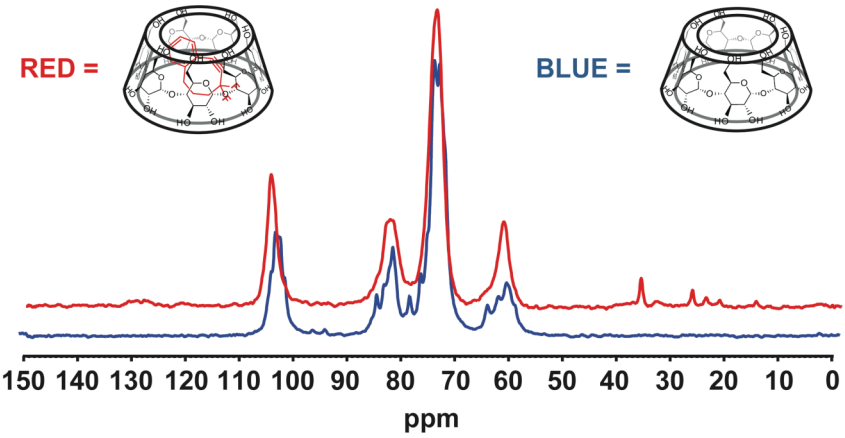


Figure S6. CPMAS ^{13}C-NMR of the β-cyclodextrin-DIFBO complex (red) compared to β-cyclodextrin alone (blue).

HPLC traces of synthetic standards

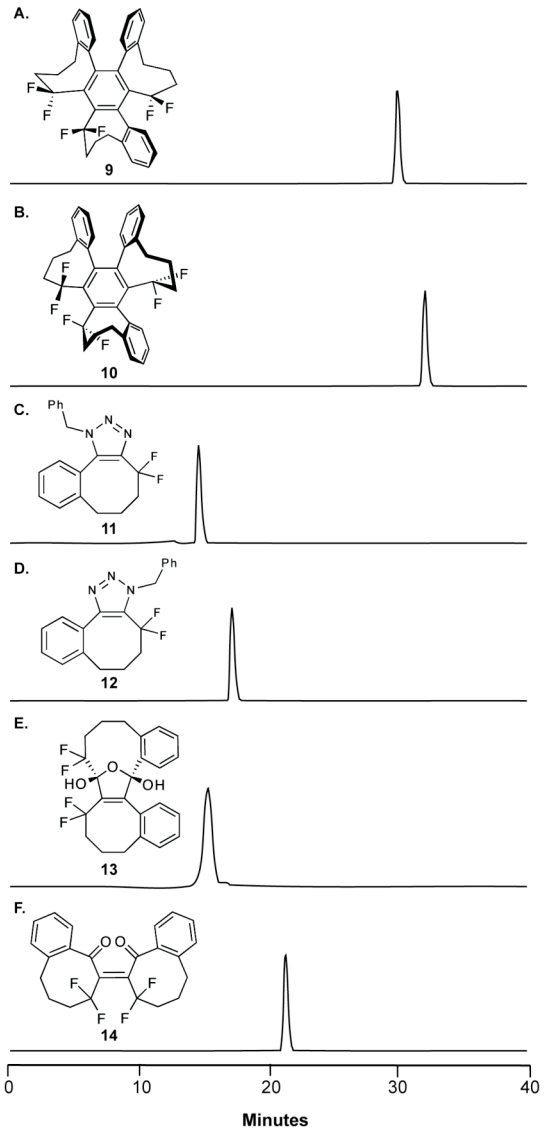


Figure S7. HPLC traces generated by monitoring the absorbance at 254 nm over time of compounds **9-14** (A-F, respectively). All traces were generated on a C-18 column using a H_2O/CH_3CN solvent system with a gradient of 50-100% CH_3CN over 25 min followed by 100% CH_3CN for 10 min before re-equilibration to 50% CH_3CN.

Degradation assay for DIFBO-β-cyclodextrin complex

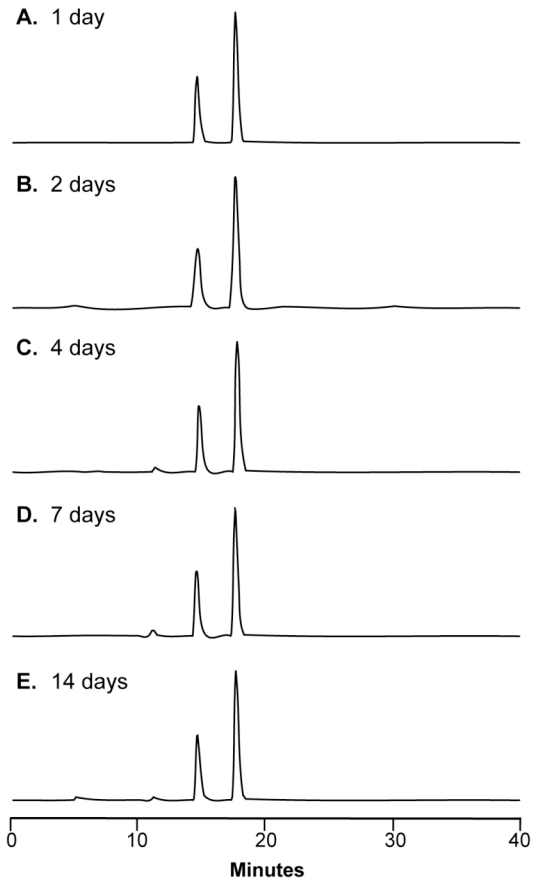


Figure S8. HPLC traces of 150 nmol of DIFBO-β-cyclodextrin complex dissolved in 1:1 CH_3CN/H_2O and reacted with benzyl azide A) 1 day, B) 2 days, C) 4 days, D) 7 days, or E) 14 days after encapsulation of DIFBO in cyclodextrin. Shown are the traces generated by monitoring the absorbance at 254 nm over time. The compounds were eluted from a C-18 column using a H_2O/CH_3CN solvent system with a gradient of 50-100% CH_3CN over 25 min followed by 100% CH_3CN for 10 min before re-equilibration to 50% CH_3CN.

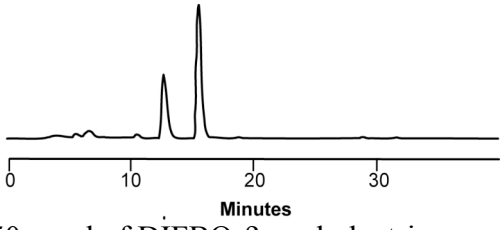
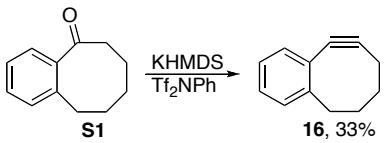


Figure S9. HPLC trace of 150 nmol of DIFBO-β-cyclodextrin complex dissolved in 1:1 CH_3CN/H_2O and reacted with benzyl azide two months after encapsulation of DIFBO in cyclodextrin. Shown is the trace generated by monitoring the absorbance at 254 nm over time. The compounds were eluted from a C-18 column using a H_2O/CH_3CN solvent system with a gradient of 50-100% CH_3CN over 25 min followed by 100% CH_3CN for 10 min before re-equilibration to 50% CH_3CN. The slight difference in retention time of the two triazole peaks as compared to Figure S8 is attributed to a different HPLC column.

Synthesis of monobenzocyclooctyne 16 (MOBO)



Scheme S1. Synthesis of monobenzocyclooctyne (MOBO).

Monobenzocyclooctyne (16). Known 7,8,9,10-tetrahydrobenzo[8]anulen-5(*6H*)-one (**S1**)[8] (100 mg, 0.57 mmol, 1 equiv) was dissolved in THF (3 mL) and cooled to -78 °C. To this solution, potassium bis(trimethylsilyl)amide (0.5 M solution in toluene, 2.28 mL, 1.14 mmol, 2 equiv) was added and the mixture was stirred at -78 °C. After 1 hr, *N*-phenyl-bis(trifluoromethanesulfonimide) (234 mg, 0.66 mmol, 1.1 equiv in 5 mL THF) was added. The reaction mixture was allowed to warm to 0 °C. After 1 hr at 0 °C, the reaction was quenched with MeOH and evaporated to dryness to result in crude monobenzocyclooctyne. Silica gel chromatography eluting with hexane yielded monobenzocyclooctyne **16** as a volatile, foul smelling oil. (30 mg, 0.19 mmol, 33%). R_f = 0.85 in 4:1 hexane/EtOAc. ^{1}H-NMR (600 MHz, $CDCl_3$): δ 7.14-7.20 (m, 4H), 2.80 (bs, 2H), 2.58 (t, J = 6.8 Hz, 2H), 2.13 (bs, 2H), 1.76 (bs, 2H). ^{13}C-NMR (150 MHz, $CDCl_3$): δ 151.6, 128.9, 127.1, 126.1, 125.8, 124.3, 112.8, 93.8, 39.2, 33.8, 25.7, 20.5. HRMS (EI): Calcd. for $C_{12}H_{12}^+$ $[M]^+$ 156.0939, found 156.0940.

[8] Huisgen, R.; Rapp, W. *Chem. Ber.* **1952**, *85*, 826.

Determination of second-order rate constants

DIFBO (4). DIFBO-β-cyclodextrin complex (~200 mg) was dissolved in CD_3CN (~4 mL) and the β-cyclodextrin was removed by filtration. The white solid was washed with CD_3CN (~1.5 mL). The resulting eluent was transferred to NMR tubes (600 μL each). A 59 mM solution of BnN_3 in CD_3CN was prepared. To the first NMR tube containing DIFBO, an approximate amount of BnN_3 solution was added. Using integration, the amount of BnN_3 necessary for an exact 1:1 mixture of BnN_3:DIFBO to be obtained was calculated. This amount (90-100 μL) was added to the remaining DIFBO solutions and each reaction was monitored by ^{1}H-NMR over 15 min. The concentration of each sample was determined relative to the amount of BnN_3 added. A plot of 1/[DIFBO] (M^{-1}) vs. time (s) resulted in a linear regression (Figure S10) whose slope was the second-order rate constant. This procedure was repeated four times with a concentration of 7-8 mM, to yield a second-order rate constant of 0.22 $\pm$ 0.01 $M^{-1}s^{-1}$.

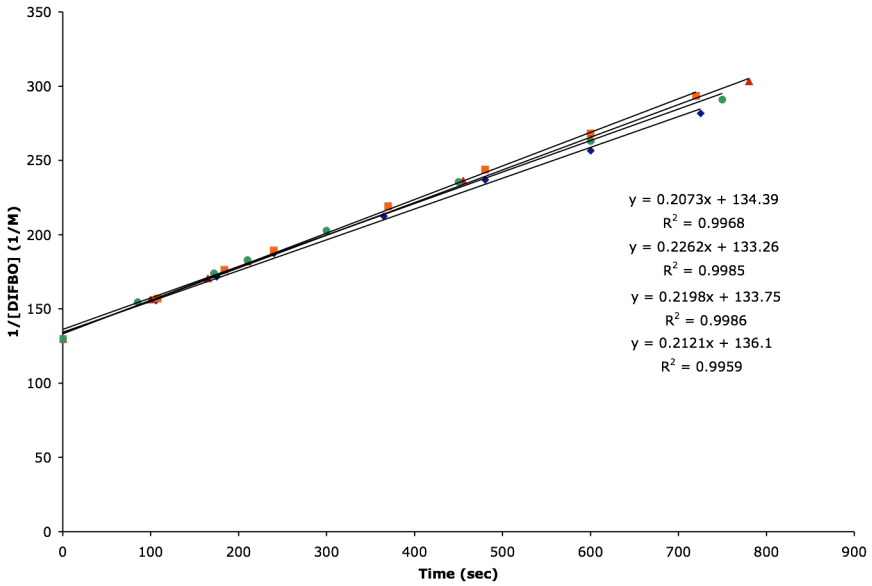


Figure S10. Plot of 1/[DIFBO] vs. time for the reaction of DIFBO and BnN_3 in CD_3CN as monitored by ^{1}H-NMR.

MOBO (16). A solution of monobenzocyclooctyne (24 mM) in CD_3CN with a small amount of hexamethyldisilane was prepared. A solution of BnN_3 (146 mM) was also prepared using the same solution of CD_3CN containing a small amount of hexamethyldisilane. ^{1}H-NMR spectra of each of these solutions were obtained, and using the ratio of BnN_3 or MOBO to hexamethyldisilane the appropriate amount of each solution necessary for a 1:1 mixture of BnN_3:MOBO was calculated. The correct amount of BnN_3 was added to MOBO and the reaction was monitored by ^{1}H-NMR over 45 min. The concentration of each sample was determined based on the concentration of the initial monobenzocyclooctyne solution. A plot of 1/[MOBO] (M^{-1}) vs. time (s) resulted in a linear regression whose slope was the second-order rate constant (Figure S11). This procedure was repeated three times with a concentration of 19 mM to yield a second-order rate constant of 0.0095 $\pm$ 0.0003 $M^{-1}s^{-1}$.

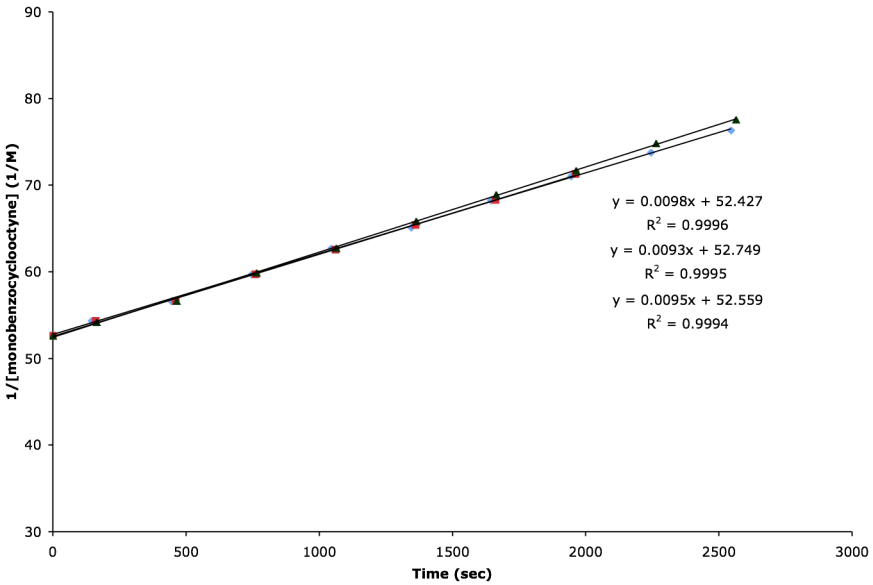
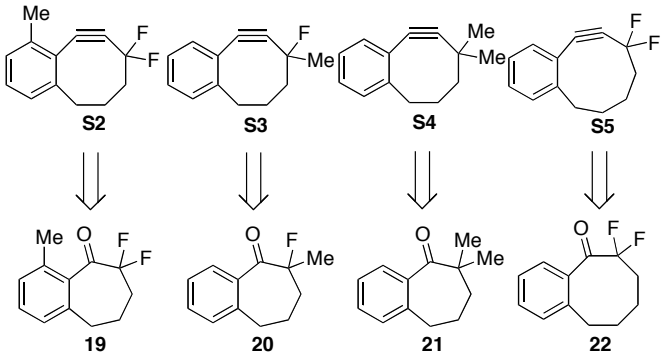


Figure S11. Plot of 1/[MOBO] vs. time for the reaction of **16** and BnN_3 in CD_3CN as monitored by ^{1}H-NMR.

Analogues of DIFBO

In order for DIFBO to be useful for reactions with azides in a biological setting, it has to have a substantially longer half-life. Physical organic chemists have spent considerable effort synthesizing highly strained molecules that are able to persist due to kinetic stability.[9] Considering the main degradation product of DIFBO was a trimer, we envisioned that steric protection of the alkyne through an appropriately placed methyl group on the aryl ring (compound **S2**) or the addition of methyl groups at the propargylic position (compounds **S3** and **S4**) would result in a more kinetically stable cyclooctyne. Additionally, we looked to stabilize DIFBO by raising the LUMO levels (compounds **S3** and **S4**) or by decreasing the ring strain (cyclononyne **S5**). We hoped that these cycloalkynes (**S2**-**S5**) would remain sufficiently reactive with azides, even though they are less activated for 1,3-dipolar cycloaddition. We envisioned cycloalkynes **S2**-**S5** could be synthesized from ketones **19**-**22** through the same homologation strategy used for DIFBO.

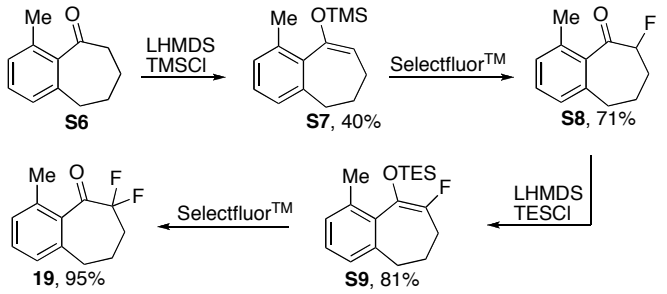
Benzosuberones **19**-**21** and ketone **22** were synthesized by standard procedures (see Schemes S2-S5). Unfortunately, when these ketones were subjected to trimethyl aluminum and trimethylsilyl diazomethane, no ring expansion was observed. Other attempts at homologation using an array of Lewis acids and diazo species failed to produce any desired product.[10] Thus, while the homologation approach to DIFBO was highly efficient, it does not appear to be a generalizable method for cycloalkyne synthesis.



Scheme S2. Analogues of DIFBO and their potential ketone precursors.

[9] a) Liebman, J.F.; Greenberg, A. *Chem. Rev.* **1976**, *76*, 311. b) Maier, G. *Angew. Chem. Int. Ed.* **1988**, *27*, 309. c) Tsuji, T.; Ohkita, M.; Kawai, H. *Bull. Chem. Soc. Jpn.* **2002**, *75*, 415. d) Tokitoh, N. *Bull. Chem. Soc. Jpn.* **2004**, *77*, 429.

[10] A selection of other conditions involved varying the Lewis acid (TMSOTf, $BF_3{\cdot}OEt_2$, $Sc(OTf)_3$, MAD, $AlCl_3$), diazo species (CH_2N_2, $TMSCLiN_2$, $N_2CCOOEt$), and temperature (including microwave radiation). Cyclononyne **S5** could unreliably be formed via treatment of **22** with $TMSCLiN_2$. (Similar conditions to those reported in Miwa, K.; Aoyama, T.; Shioiri, T. *Synlett*, **1994**, *2*, 107.) The small amount of **S5** isolated appeared to react very slowly with azides ($k \sim 10^{-5}$ $M^{-1}s^{-1}$).



Scheme S3. Synthesis of 2,2-difluoro-9-methyl-1-benzosuberone **19**.

9-Methyl-1-benzosuberone (S6). 9-Methyl-1-benzosuberone (**S6**) was prepared as decribed by R. McCague.[11] Briefly, TMS-enol ether of benzosuberone (2.55 g, 11 mmol, 1 equiv) was dissolved in hexanes (32 mL, dried over molecular sieves overnight). To this solution, tetramethylethylenediamine (4.0 mL, 27 mmol, 2.5 equiv, freshly distilled) was added followed by nBuLi (1.42 M solution in hexanes, 16.0 mL, 23.0 mmol, 2.1 equiv). The resulting orange mixture was heated to reflux. After 2 hr, MeI (0.87 mL, 14 mmol, 1.3 equiv) in hexanes (5.1 mL) was added and the reaction was immediately quenched with H_2O (25 mL). Upon cooling to rt, the product was extracted into hexane (3 x 50 mL). The organics were combined, dried with $MgSO_4$, decanted and evaporated to dryness. The crude product was purified by silica gel chromatography in a hexane/ether solvent system (40:1 to 30:1). This procedure yielded desired product **S6** (980 mg, 5.6 mmol, 51%) plus some recovered benzosuberone and 2,9-dimethyl benzosuberone. R_f = 0.5 in 5:1 hexanes/EtOAc. ^{1}H-NMR (500 MHz, $CDCl_3$): δ 7.14 (d, J = 7.6 Hz, 1H), 7.02 (d, J = 7.6 Hz, 1H), 6.90 (d, J = 7.5 Hz, 1H), 2.69 (t, J = 6.6 Hz, 2H), 2.53-2.55 (m, 2H), 2.27 (s, 3H), 1.67-1.76 (m, 4H). ^{13}C-NMR (125 MHz, $CDCl_3$): δ 210.3, 139.5, 138.0, 135.3, 129.9, 129.0, 126.3, 42.0, 32.3, 25.5, 22.3, 19.7. HRMS (ESI): Calcd. for $C_{12}H_{15}O^+$ $[M+H]^+$ 175.1117, found 175.1115.

Compound S7. 9-Methyl-1-benzosuberone (**S6**, 440 mg, 2.5 mmol, 1 equiv) was dissolved in THF (44 mL) and cooled to -78 °C. Lithium bis(trimethylsilyl)amide (1 M solution in THF, 4.7 mL, 4.7 mmol, 1.9 equiv) was added and the resulting mixture was stirred at -78 °C. After 1.5 hr, TMSCl (1.07 mL, 8.4 mmol, 3.4 equiv) was added and the reaction was warmed to 0 °C. Upon reaching 0 °C, the reaction was quenched with MeOH and evaporated to dryness. The crude product was purified by silica gel chromatography eluting with hexane to yield **S7** (255 mg, 1.0 mmol, 40%). R_f = 0.9 in 4:1 hexane/EtOAc. ^{1}H-NMR (600 MHz, $CDCl_3$): δ 7.13 (t, J = 7.5 Hz, 1H), 7.08 (d, J = 7.3 Hz, 1H), 7.03 (d, J = 7.5 Hz, 1H), 5.45 (t, J = 7.7 Hz, 1H), 2.62 (t, J = 7.0 Hz, 2H), 2.39 (s, 3H), 1.98 (apparent quin, J = 7.0 Hz, 2H), 1.73 (bs, 2H), 0.17 (s, 9H). ^{13}C-NMR (150 MHz, $CDCl_3$): 150.3, 140.6, 137.2, 136.7, 128.8, 127.7, 126.2, 108.4, 34.0, 32.7, 22.1, 21.0, 0.3. HRMS (EI): Calcd. for $C_{15}H_{22}OSi^+$ $[M]^+$ 246.1440, found 246.1440.

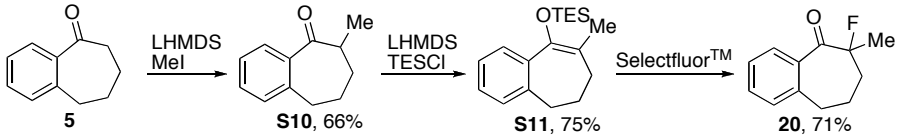
2-Fluoro-9-methyl-1-benzosuberone (S8). Selectfluor™ (475 mg, 1.3 mmol, 1.3 equiv) was dissolved in DMF (25 mL) and cooled to 0 °C. Compound **S7** (255 mg, 1.0 mmol, 1 equiv) was dissolved in DMF (12 mL) and this solution was added to the Selectfluor™ solution over 30 min at 0 °C. After addition of **S7** was complete, the reaction mixture was warmed to rt and stirred for an additional 1.5 hr. The reaction was then quenched with H_2O (25 mL) and extracted with Et_2O

[11] McCague, R. *Tetrahedron: Asymmetry* **1990**, *1*, 97.

(2 x 50 mL). The Et_2O extracts were combined, dried with $MgSO_4$, decanted and evaporated to dryness. The crude product was purified by silica gel chromatography eluting with 40:1 hexanes/ether. This procedure resulted in 142 mg of pure **S8** (0.74 mmol, 71%). R_f = 0.7 in 3:1 hexanes/EtOAc. ^{1}H-NMR (600 MHz, $CDCl_3$): δ 7.17 (t, *J* = 7.7 Hz, 1H), 7.07 (d, *J* = 7.6 Hz, 1H), 6.96 (d, *J* = 7.5 Hz, 1H), 5.07 (ddd, J_F = 50.5 Hz, portion at 5.11 apparent t, *J* = 5.8 Hz, portion at 5.03 dd, *J* = 7.6, 4.1 Hz, 1H), 2.74-2.76 (m, 2H), 2.26 (s, 3H), 2.13-2.17 (m, 2H), 1.99-2.05 (m, 1H), 1.78-1.84 (m, 1H). ^{13}C-NMR (150 MHz, $CDCl_3$): δ 206.3 (d, *J* = 24 Hz), 138.1, 137.9, 135.9, 130.2, 129.0, 127.0, 94.9 (d, *J* = 183 Hz), 35.2, 33.7 (d, *J* = 22 Hz), 23.2 (d, *J* = 6 Hz), 19.6. ^{19}F-NMR (564 MHz, $CDCl_3$): δ -101.2 (t, *J* =11 Hz, 1F). HRMS (EI): Calcd. for $C_{12}H_{13}OF^+$ $[M]^+$ 192.0950, found 192.0954.

Compound S9. 2-Fluoro-9-methyl-1-benzosuberone (**S8**, 114 mg, 0.59 mmol, 1 equiv) was dissolved in THF (10 mL) and cooled to -78 °C. Lithium bis(trimethylsilyl)amide (1M solution in THF, 1.1 mL, 1.1 mmol, 1.9 equiv) was added. The reaction mixture was stirred for 30 min at -78 °C at which point TESCl (0.33 mL, 2.0 mmol, 3.4 equiv) was added. The reaction was warmed to rt. After 10 min at rt, the reaction was quenched with MeOH, evaporated to dryness, and chromatographed on silica gel eluting with hexane/EtOAc (20:1). This procedure resulted in pure **S9** (147 mg, 0.48 mmol, 81%). R_f = 0.95 in 3:1 hexane/EtOAc. ^{1}H-NMR (500 MHz, $CDCl_3$): δ 7.10 (apparent t, *J* = 7.4 Hz, 1H), 7.05 (d, *J* = 7.2 Hz, 1H), 6.98 (d, *J* = 7.4 Hz, 1H), 2.65 (bs, 2H), 2.39 (s, 3H), 2.07-2.15 (bm, 4H), 0.89 (t, *J* =7.9 Hz, 9H), 0.60 (q, *J* = 7.9 Hz, 6H). ^{13}C-NMR (125 MHz, $CDCl_3$): δ 148.8 (d, *J* = 255 Hz), 139.9, 138.2 (d, *J* = 4 Hz), 135.5 (d, *J* = 4 Hz), 129.8 (d, *J* = 14 Hz), 129.1, 127.8, 126.4, 33.4, 32.5, 25.3 (d, *J* = 25 Hz), 21.0, 5.3, 5.2. ^{19}F-NMR (564 MHz, $CDCl_3$): δ -126.1 (t, *J* = 23 Hz, 1F). HRMS (EI): Calcd. for $C_{18}H_{27}FOSi^+$ $[M]^+$ 306.1815, found.

2,2-Difluoro-9-methyl-1-benzosuberone (19). Selectfluor™ (121 mg, 0.34 mmol, 1.6 equiv) was dissolved in DMF (6 mL). Compound **S9** (64 mg, 0.21 mmol, 1 equiv) was dissolved in DMF (3 mL) and this solution was added to the Selectfluor™ solution over 30 min. After addition of **S9** was complete, the reaction was stirred for an additional 30 min, at which point H_2O (5 mL) was added and the product was extracted with Et_2O (2 x 15 mL). The Et_2O extracts were combined, dried with $MgSO_4$, decanted and evaporated to dryness. The crude product was purified by silica gel chromatography (hexanes/EtOAc). This procedure resulted in 42 mg of pure **19** (0.20 mmol, 95%). R_f = 0.7 in 3:1 hexane/EtOAc. ^{1}H-NMR (500 MHz, $CDCl_3$): δ 7.25 (t, *J* = 7.6 Hz, 1H), 7.13 (d, *J* = 7.7 Hz, 1H), 7.00 (d, *J* = 7.7 Hz, 1H), 2.79-2.81 (m, 2H), 2.30 (s, 3H), 2.26-2.33 (m, 2H), 1.94-1.98 (m, 2H). ^{13}C-NMR (125 MHz, $CDCl_3$): δ 198.5 (t, *J* = 30 Hz), 138.2, 137.0, 136.2, 131.0 (apparent d, *J* = 12 Hz), 129.3 (apparent d, *J* = 19 Hz), 127.1 (apparent d, *J* = 18 Hz), 118.1 (t, *J* = 250 Hz), 35.8 (t, *J* = 24 Hz), 34.3, 22.5 (bs), 19.7. ^{19}F-NMR (564 MHz, $CDCl_3$): δ -106.0 (apparent t, *J* = 11 Hz, 2F). HRMS (EI): Calcd. for $C_{12}H_{12}OF_2$ 210.0856, found 210.0860.



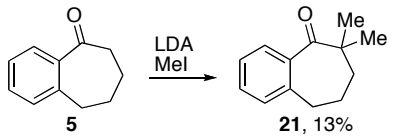
Scheme S4. Synthesis of 2-fluoro-2-methyl-1-benzosuberone **20**.

2-Methyl-1-benzosuberone (S10). Lithium bis(trimethylsilyl)amide (1M solution in THF, 0.80 mL, 0.80 mmol, 1.2 equiv) and THF were cooled to -78 °C. To this solution, benzosuberone (**5**, 100 μL, 0.68 mmol, 1 equiv) was added dropwise. This solution was stirred for 1.5 hr at which point MeI (210 μL, 3.4 mmol, 5 equiv) was added. The reaction was warmed to rt over 4 hr and then quenched with MeOH. The mixture was evaporated to dryness and the crude oil was purified by silica gel chromatography (35:1 hexane/EtOAc). This procedure yielded **S10** as a clear oil (79 mg, 0.45 mmol, 66%). R_f = 0.6 in 9:1 hexane/EtOAc. ^{1}H-NMR (600 MHz, $CDCl_3$): δ 7.44 (d, *J* = 7.7 Hz, 1H), 7.35 (t, *J* = 7.1 Hz, 1H), 7.25 (t, *J* = 7.6 Hz, 1H), 7.19 (d, *J* = 7.3 Hz, 1H), 2.88-3.01 (m, 3H), 2.03-2.06 (m, 1H), 1.84-1.91 (m, 1H), 1.64-1.68 (m, 2H), 1.21 (d, *J* = 6.5 Hz, 3H). ^{13}C-NMR (150 MHz, $CDCl_3$): δ 207.9, 142.0, 139.8, 131.4, 129.9, 128.5, 126.5, 44.3, 33.8, 32.1, 25.7, 16.6. HRMS (EI): Calcd. for $C_{12}H_{14}O^+$ $[M]^+$ 174.1045, found 174.1042.

Compound S11. 2-Methyl-1-benzosuberone (**S10**, 318 mg, 1.8 mmol, 1 equiv) was dissolved in THF (31 mL) and cooled to -78 °C. Lithium bis(trimethylsilyl)amide (1M solution in THF, 2.0 mL, 2.0 mmol, 1.1 equiv) was added to this solution. After 1 hr, TESCl (490 μL, 2.9 mmol, 1.6 equiv) was added and the reaction mixture was warmed to rt overnight. The following morning the reaction was quenched with water (1 mL) and evaporated to dryness. The crude product was extracted with Et_2O (3 x 10 mL, 15 mL H_2O). The organic extracts were combined, dried with $MgSO_4$, decanted, and evaporated to dryness. The product was purified by silica gel chromatography with hexane/EtOAc (100% hexane to 100:1 hexane/EtOAc) to yield 394 mg of silyl enol ether **S11**. (1.4 mmol, 75%). R_f = 0.9 in 9:1 hexane/ethyl acetate. ^{1}H-NMR (500 MHz, $CDCl_3$): δ 7.46 (d, *J* = 8.0 Hz, 1H), 7.23-7.28 (m, 1H), 7.17-7.21 (m, 2H), 2.62 (t, *J* = 7.1 Hz, 2H), 2.13 (apparent quin, *J* = 7.2 Hz, 2H), 1.96 (s, 3H), 1.84 (t, *J* = 7.1 Hz, 2H), 0.94 (t, *J* = 7.9 Hz, 9H), 0.57 (q, *J* =8.0 Hz, 6H). ^{13}C-NMR (150 MHz, $CDCl_3$): δ 143.2, 140.2, 140.1, 128.6, 127.1, 126.9, 125.8, 117.3, 33.8, 32.7, 30.0, 17.8, 6.9, 5.4. HRMS (EI): Calcd. for $C_{18}H_{28}OSi^+$ $[M]^+$ 288.1909, found 288.1913.

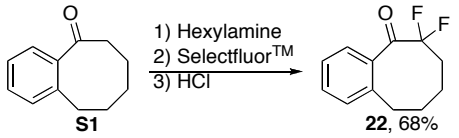
2-Fluoro-2-methyl-1-benzosuberone (20). Selectfluor™ (533 mg, 1.5 mmol, 1.5 equiv) was dissolved in DMF (20 mL) and cooled to 0 °C. Compound **S11** (294 mg, 1.0 mmol, 1 equiv) was dissolved in DMF (15 mL) and this solution was added to the Selectfluor™ solution over 30 min. After addition of **S11**, the reaction mixture was warmed to rt and stirred for an additional 30 min. The reaction was then quenched with H_2O (20 mL) and extracted with Et_2O (2 x 40 mL). The Et_2O extracts were combined, dried with $MgSO_4$, decanted and evaporated to dryness. The crude product was purified by silica gel chromatography eluting with 15:1 hexanes/EtOAc. This procedure resulted in 139 mg of pure **20** (0.72 mmol, 71%). R_f = 0.4 in 9:1 hexanes/EtOAc. ^{1}H-NMR (600 MHz, $CDCl_3$): δ 7.52 (dd, *J* = 7.6, 1.1 Hz, 1H), 7.40 (td, *J* = 7.5, 1.3 Hz, 1H), 7.30 (t, *J* = 7.4 Hz, 1H), 7.21 (d, *J* = 7.6 Hz, 1H), 3.14 (dd, *J* = 15.9, 10.7 Hz, 1H), 2.91 (dd, *J* = 16.1, 8.3 Hz, 1H), 2.20-2.28 (m, 1H), 2.10-2.16 (m, 1H), 2.04 (dtd, *J* = 32.2, 14.5, 5.1 Hz, 1H), 1.78-1.85 (m, 1H), 1.59 (d, *J* = 21.6 Hz, 3H). ^{13}C-NMR (150 MHz, $CDCl_3$): δ 204.6 (d, *J* = 29 Hz), 141.1, 137.7, 131.5, 129.7, 128.9, 126.5, 100.8 (d, *J* = 178 Hz), 38.3 (*J* = 23 Hz), 34.5, 24.1 (*J* =

2 Hz), 23.5 (d, J = 24 Hz). ^{19}F-NMR (564 MHz, $CDCl_3$): δ -144.9- -144.6 (m, 1F). HRMS (EI): Calcd. for $C_{12}H_{13}OF^+$ $[M]^+$ 192.0950, found 192.0952.



Scheme S5. Synthesis of 2,2-dimethyl-1-benzosuberone (**21**).

2,2-Dimethyl-1-benzosuberone (21). A 0.5 M solution of LDA was prepared (2.5 ml 1.4 M nBuLi, 4.0 mL THF, 0.70 mL diisopropylamine combined at -78 °C and let stir for 30 min) and benzosuberone (**5**, 0.50 mL, 3.4 mmol, 1 equiv) was added. After this mixture was stirred for 1 hr at -78 °C, MeI (0.60 mL, 9.6 mmol, 2.8 equiv) was added. The resulting yellow solution was warmed to rt overnight. The following day the reaction mixture was quenched with aqueous sat. NH_4Cl (7 mL) and the product was extracted into CH_2Cl_2 (3 x 10 mL). The organics were combined, dried with $MgSO_4$, decanted and evaporated to dryness. The crude mixture was purified by silica gel chromatography, eluting with 20:1 hexane/EtOAC to yield **21** (80 mg, 0.43 mmol, 13%) as well as a mixture of mono- and dimethylated benzosuberones. R_f = 0.6 in 9:1 hexane/EtOAc. ^{1}H-NMR (400 MHz, $CDCl_3$): δ 7.33-7.36 (m, 1H), 7.25-7.28 (m, 2H), 7.11 (d, J = 7.4, 1H), 2.77 (t, J = 6.7 Hz, 2H), 1.91 (quin, J = 6.6 Hz, 2H), 1.66-1.69 (m, 2H), 1.18 (s, 6H). ^{13}C-NMR (150 MHz, $CDCl_3$): δ 215.1, 141.5, 137.4, 130.8, 128.7, 127.2, 126.7, 46.1, 37.8, 33.2, 25.9, 23.4. HRMS (EI): Calcd. for $C_{13}H_{16}O^+$ $[M]^+$ 188.1201, found 18.1201.



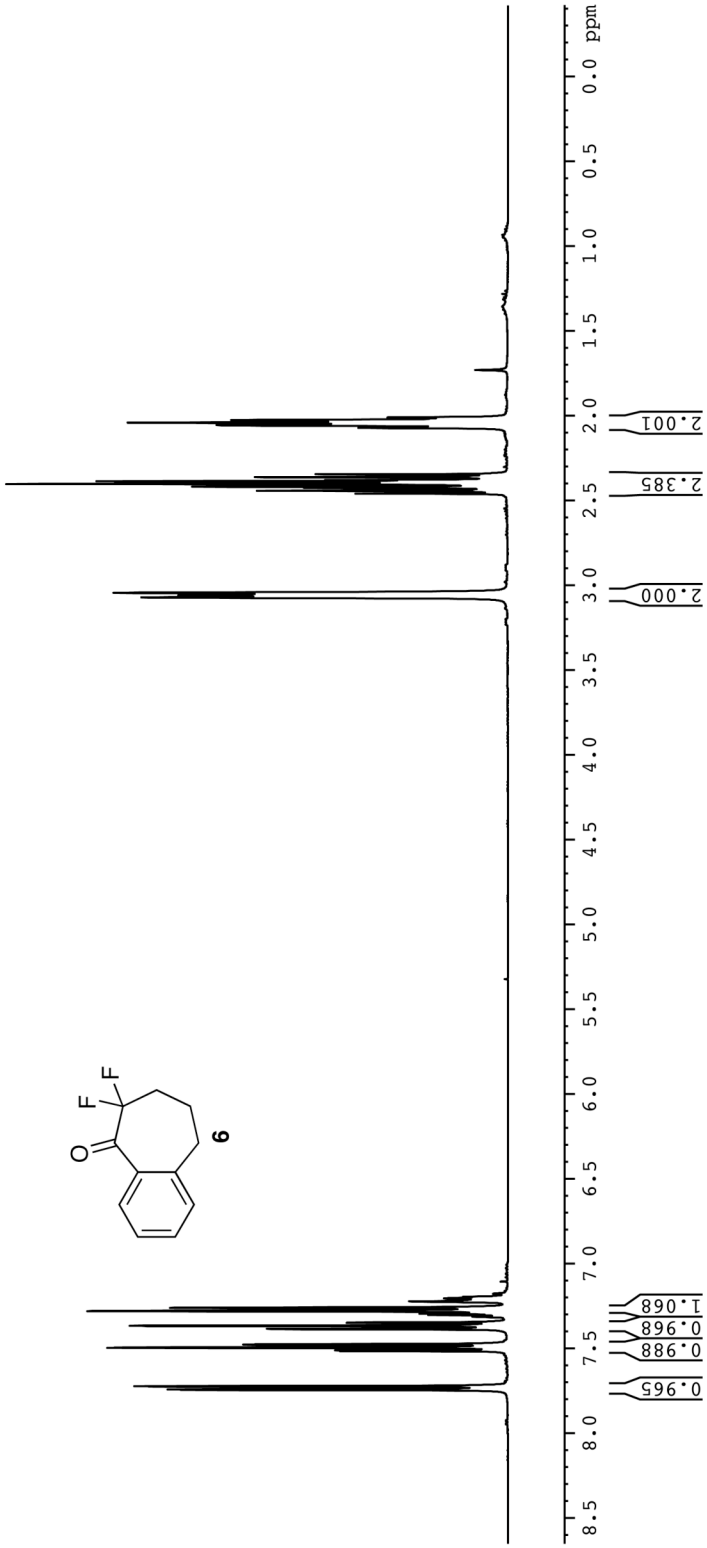
Scheme S6. Synthesis of ketone **22**.

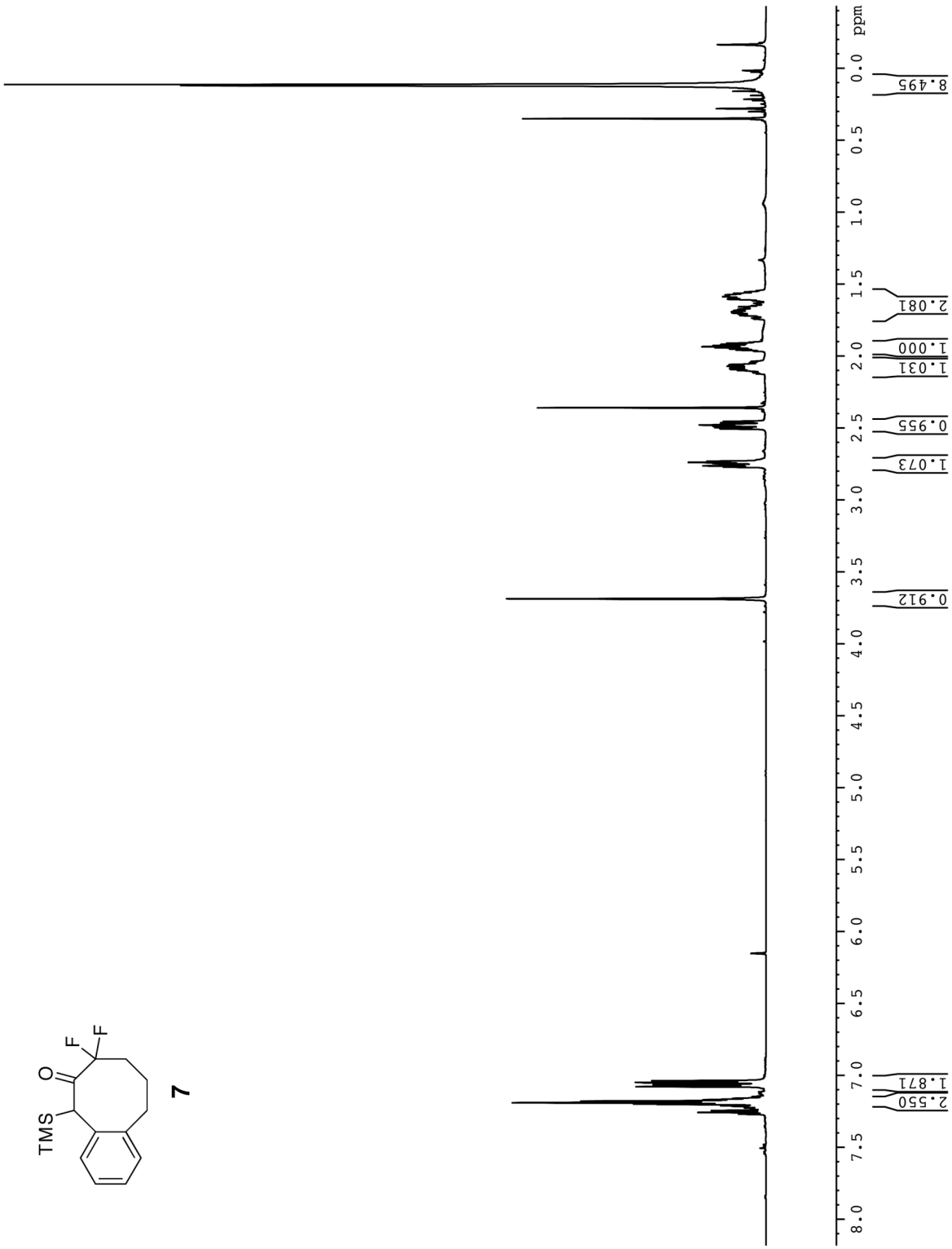
Compound 22. Ketone **S1**[12] (88 mg, 0.51 mmol, 1 equiv) was dissolved in cyclohexane (12 mL) and hexylamine (90 μL, 0.68 mmol, 1.3 equiv) and trifluoroacetic acid (1drop) were added. The reaction was heated to reflux overnight. The following morning the reaction mixture was washed with sat. $NaHCO_3$ (1 x 15 mL) and brine (1 x 15 mL). The organic solution was dried over $MgSO_4$ and evaporated to dryness. The resulting crude imine (155 mg) was dissolved in CH_3CN (5 mL). To this solution Selectfluor™ (354 g, 1.0 mmol, 2.0 equiv) and Na_2SO_4 (50.3 mg, 0.35 mmol) were added. The reaction mixture was heated to reflux overnight. The following morning 3M HCl was added to hydrolyze the imine. After 10 min at reflux, the solution was cooled to rt and evaporated to dryness. The residue was dissolved in Et_2O (10 mL), washed with sat. $NaHCO_3$ (1 x 5 mL) and brine (1 x 5 mL). The organic solution was dried over $MgSO_4$ and evaporated to dryness. The crude mixture was purified by flash chromatography with a hexane/EtOAc solvent system (96:4 hexane/EtOAc). This procedure yielded pure **22** (72

[12] McCague, R. *Tetrahedron: Asymmetry* **1990**, *1*, 97.

mg, 0.34 mmol, 68%). R_f = 0.8 in 3:1 hexane/EtOAc). ^{1}H-NMR (600 MHz, $CDCl_3$): δ 7.41 (td, *J* = 7.6, 1.4 Hz, 1H), 7.30 (td, *J* = 7.5, 1.0 Hz, 1H), 7.23 (dd, *J* = 7.6, 1.0 Hz, 1H), 7.21 (dd, *J* = 7.7, 0.2 Hz, 1H), 2.73-2.75 (m, 2H), 2.14-2.21 (m, 2H), 1.78 (apparent quin, *J* =6.2 Hz, 2H), 1.61 apparent quin, *J* = 5.3 Hz, 2H). ^{13}C-NMR (150 MHz, $CDCl_3$): δ 198.3 (t, *J* = 29 Hz), 137.8, 137.0, 130.9, 129.7, 126.5, 126.3, 119.1 (t, *J* = 251 Hz), 33.1 (t, *J* = 24 Hz), 31.4, 28.9, 19.0 (t, *J* = 6 Hz). ^{19}F-NMR (564 MHz, $CDCl_3$): δ -105.1 (t, *J* = 16 Hz, 2F). HRMS (EI): Calcd. for $C_{12}H_{12}OF_2^+$ $[M]^+$ 210.0856, found 210.0859.

^{1}H-NMR of compounds 4-6, 8-14, 16

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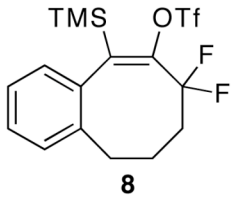
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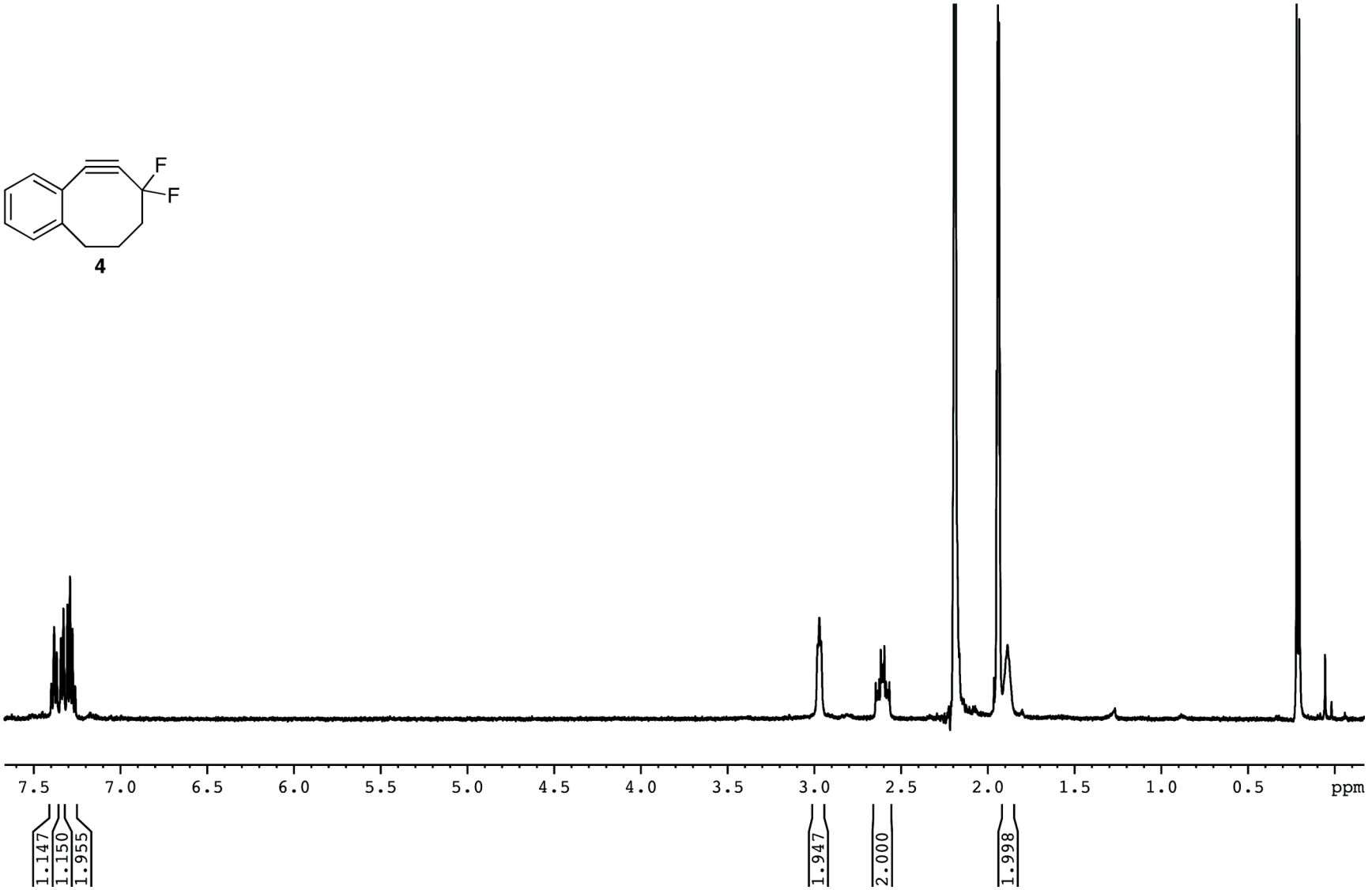
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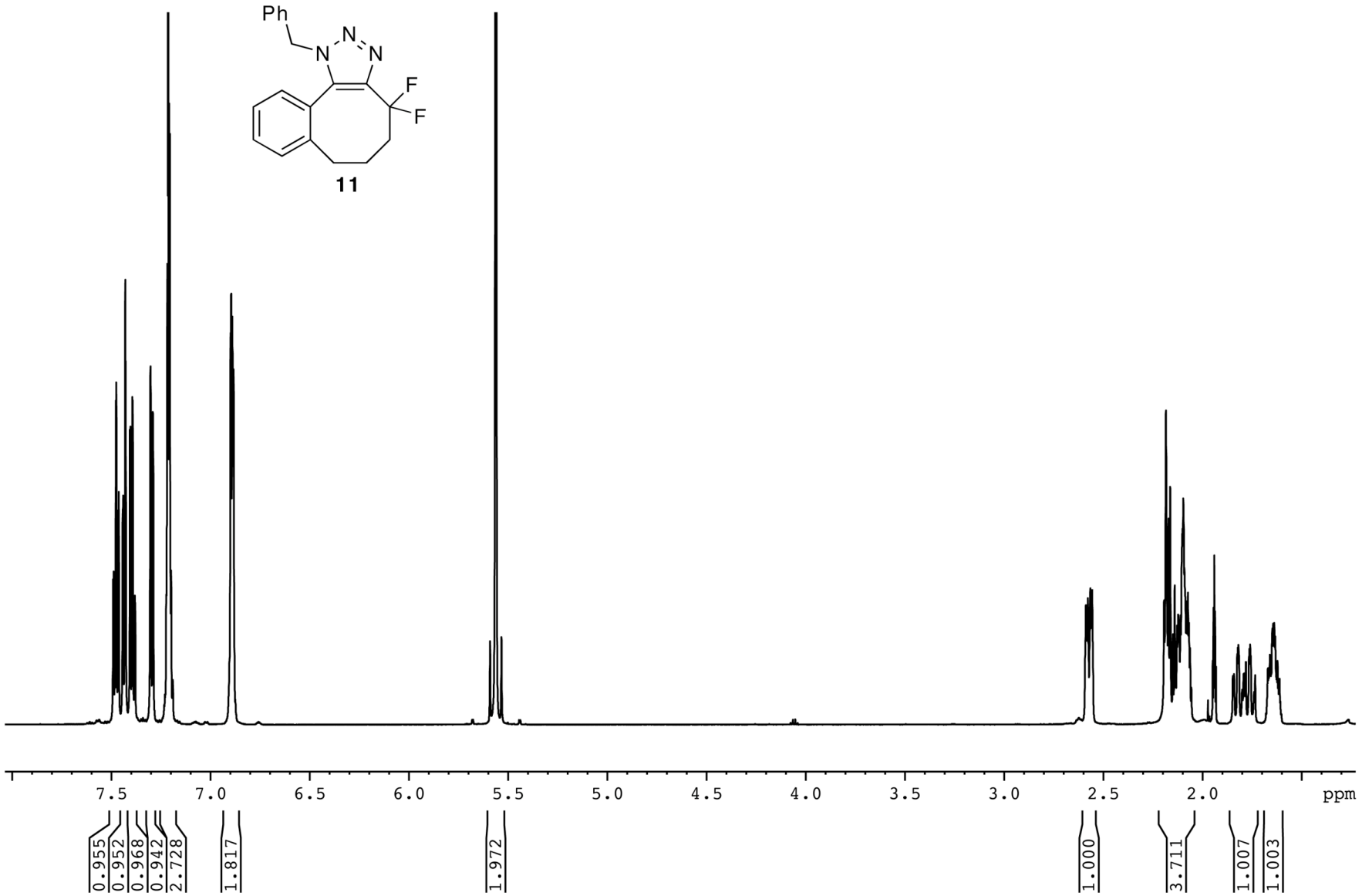
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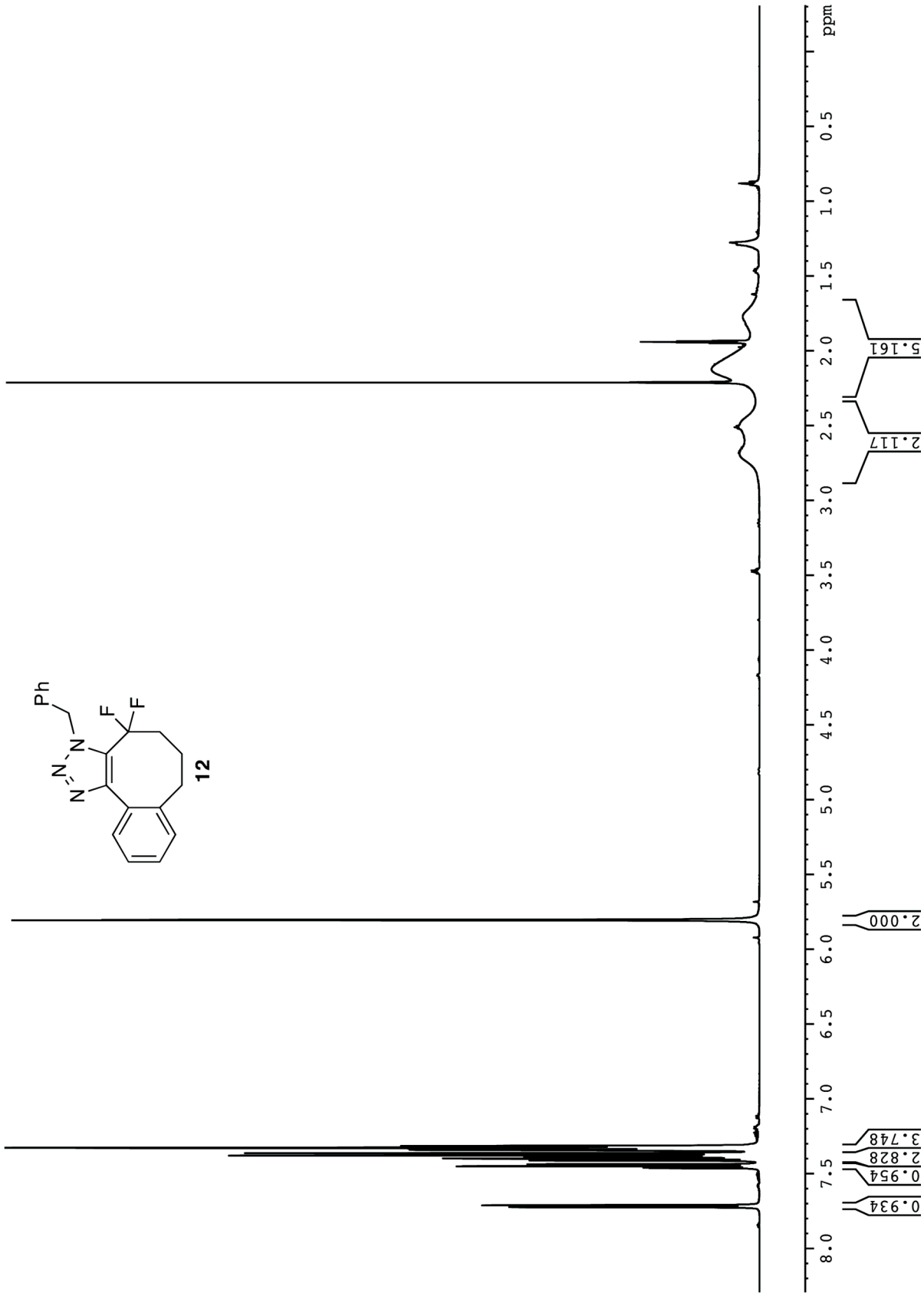
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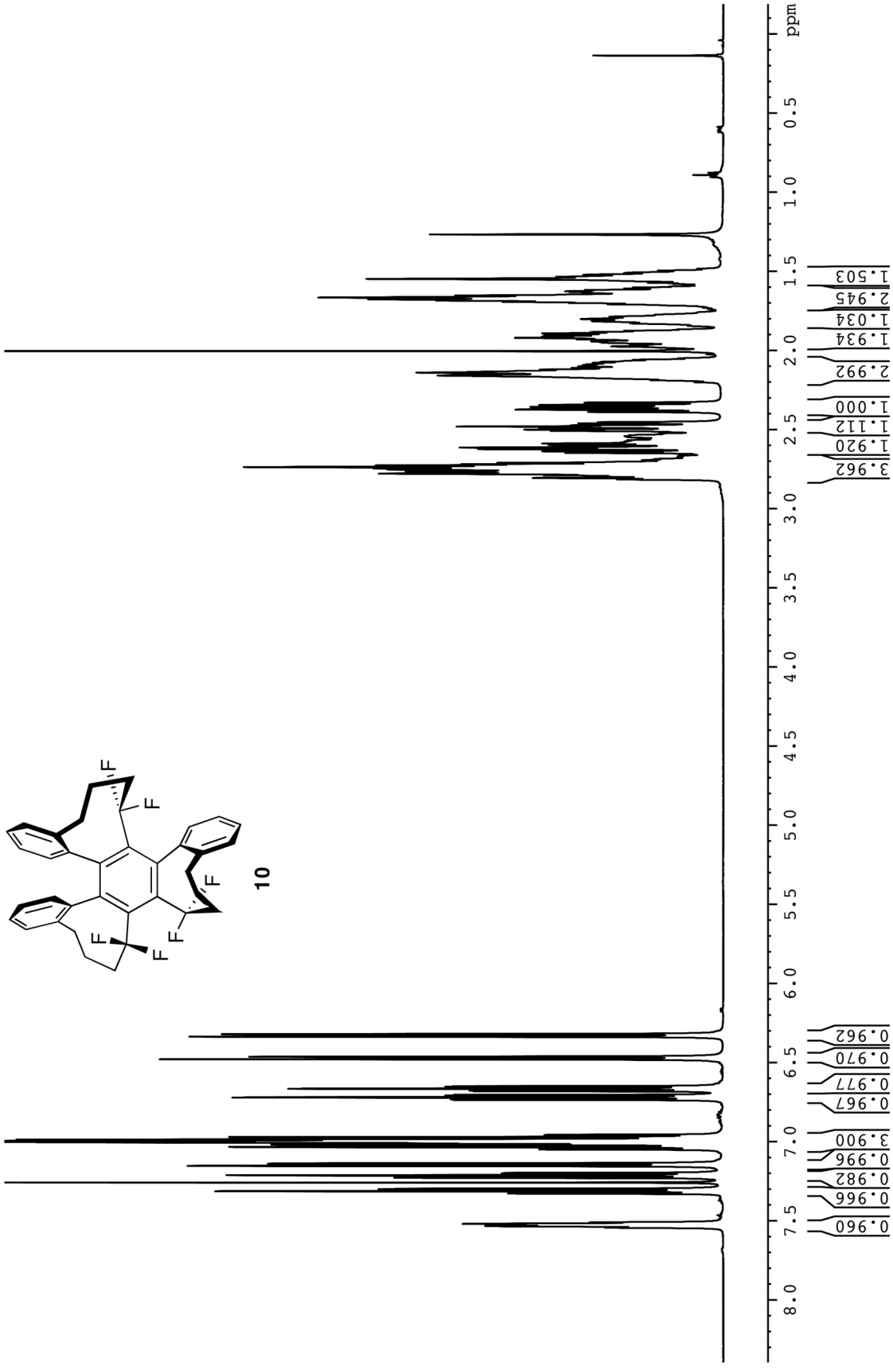
3.022
1.949
1.049
4.572
1.225
2.004
1.329
3.190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F
F
F
F
10
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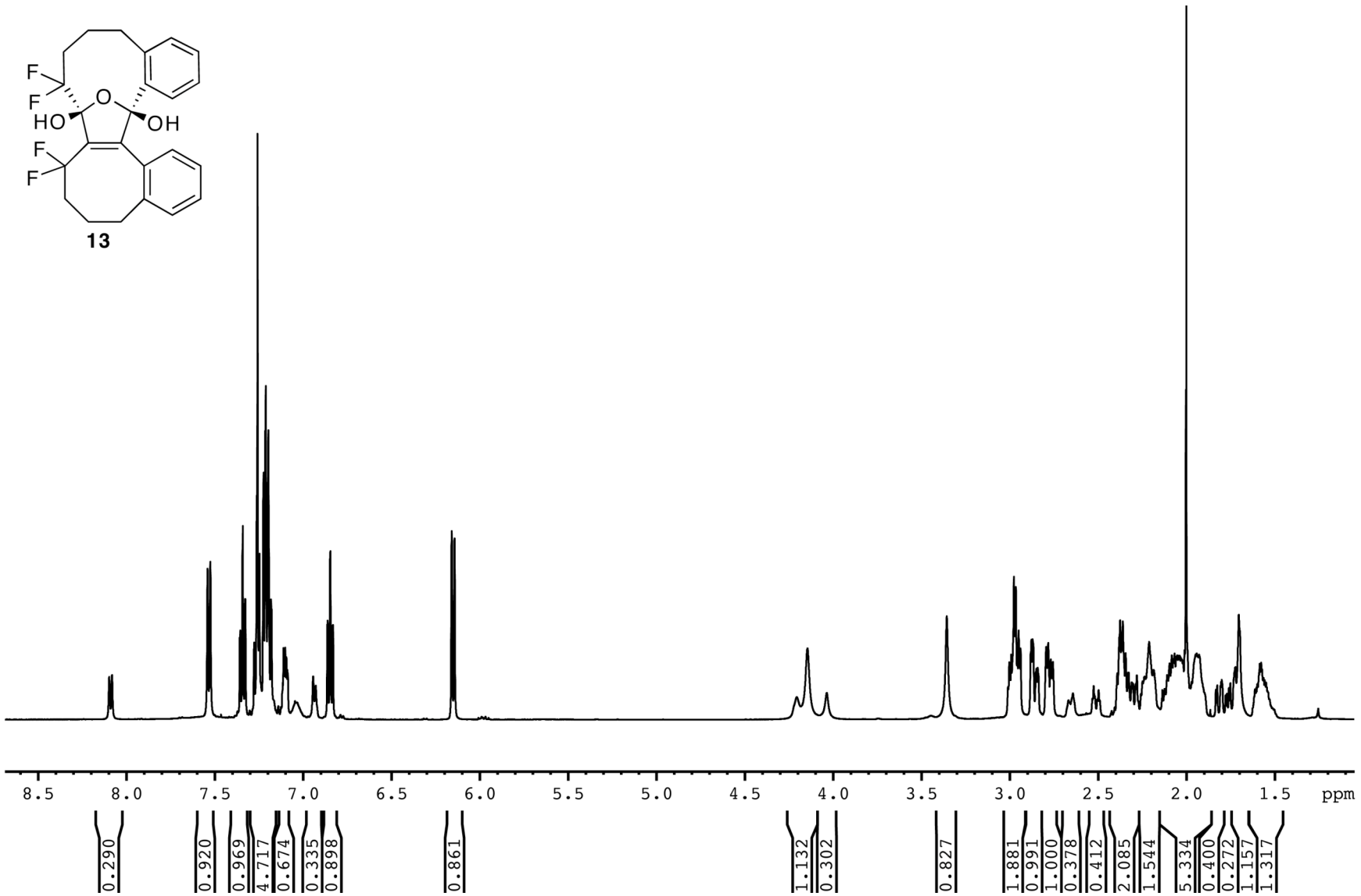
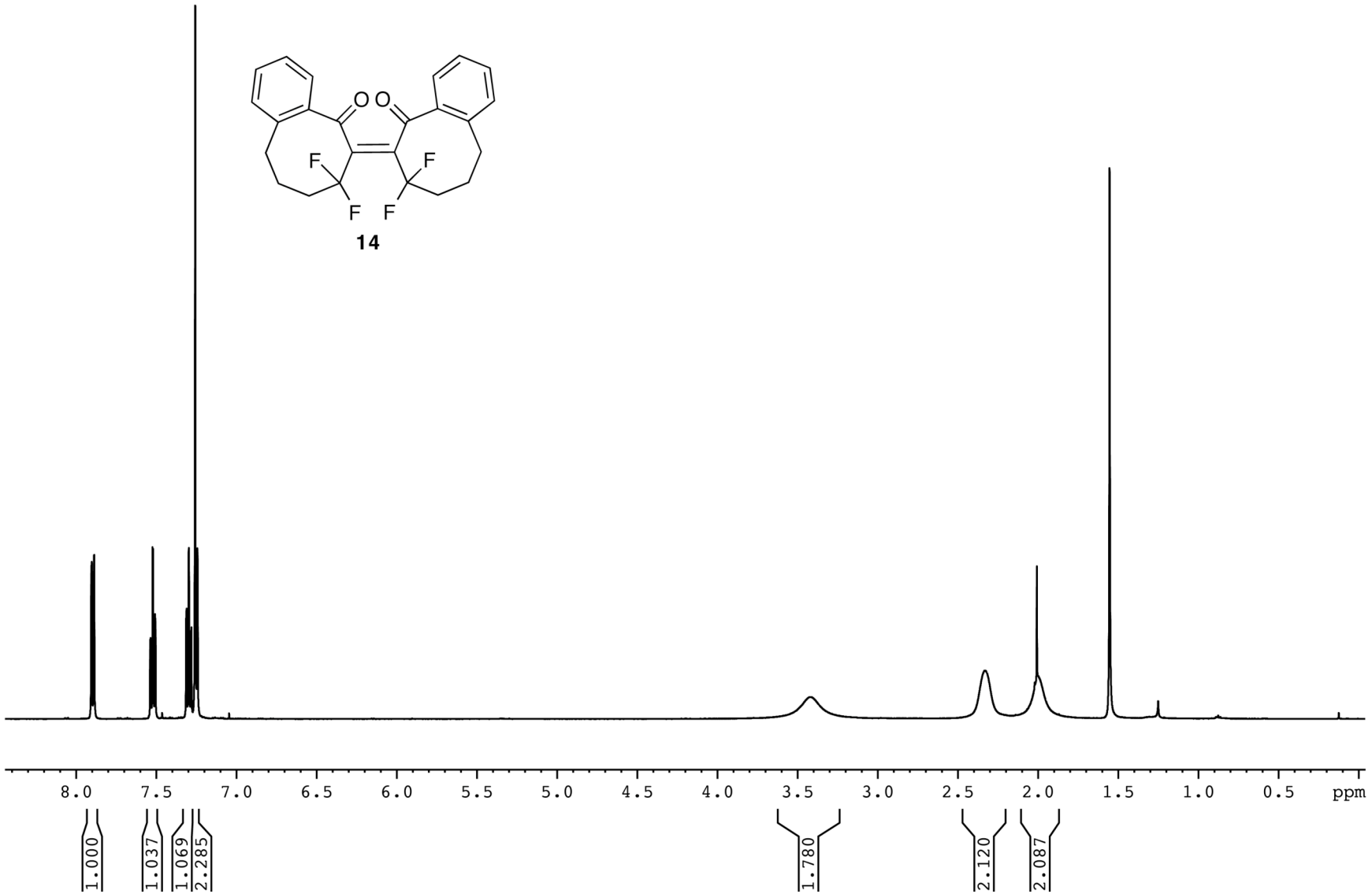
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
0.960
0.966
0.982
0.996
3.900
0.967
0.977
0.970
0.962
3.962
1.920
1.112
1.000
2.992
1.934
1.034
2.945
1.503


F
F
HO
O
OH
F
F
13
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
ppm
0.290
0.920
0.969
4.717
0.674
0.335
0.898
0.861
1.132
0.302
0.827
1.881
0.991
1.000
0.378
0.412
2.085
1.544
5.334
0.400
0.272
1.157
1.317


O O
F
F
F F
14
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
1.000
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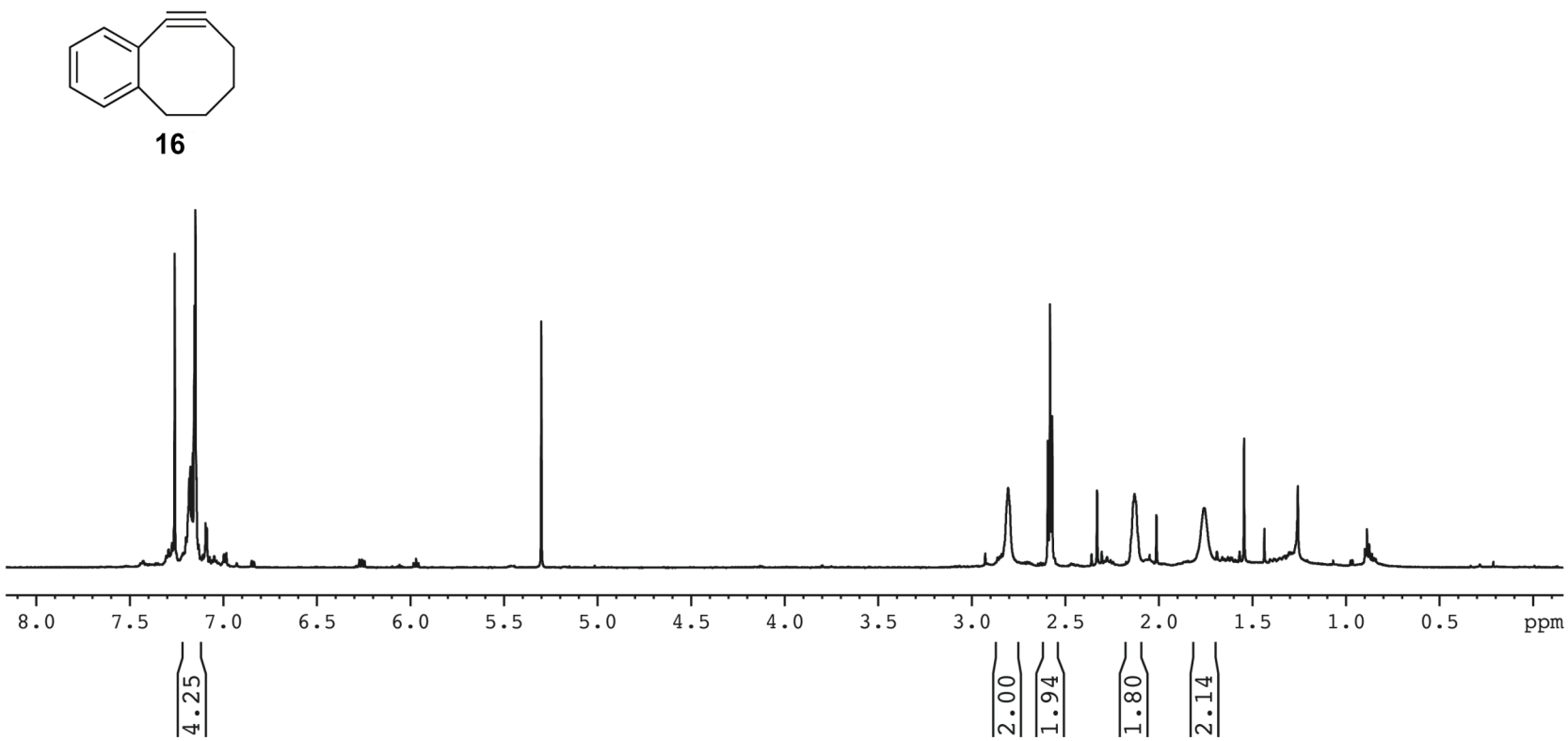
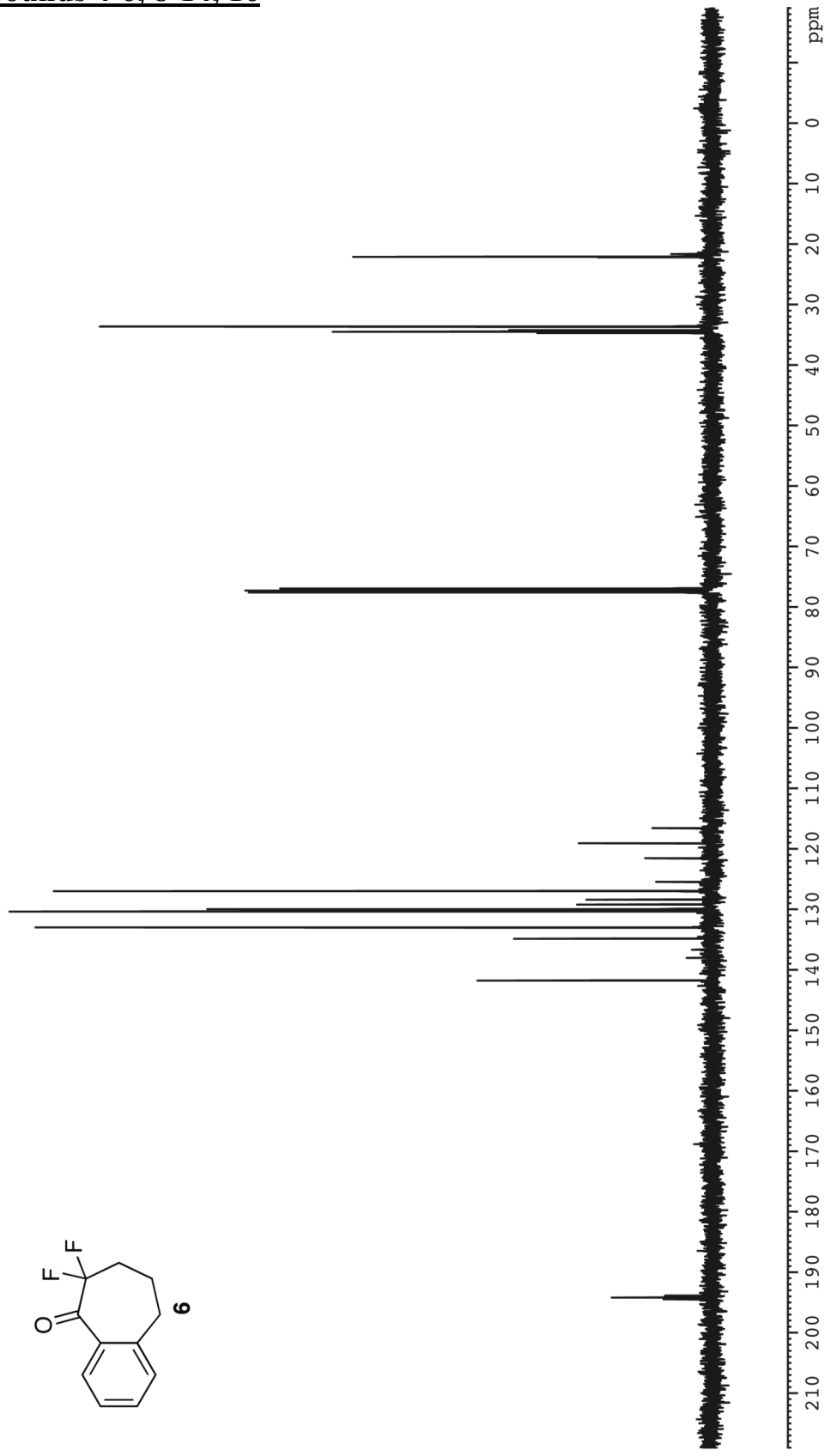
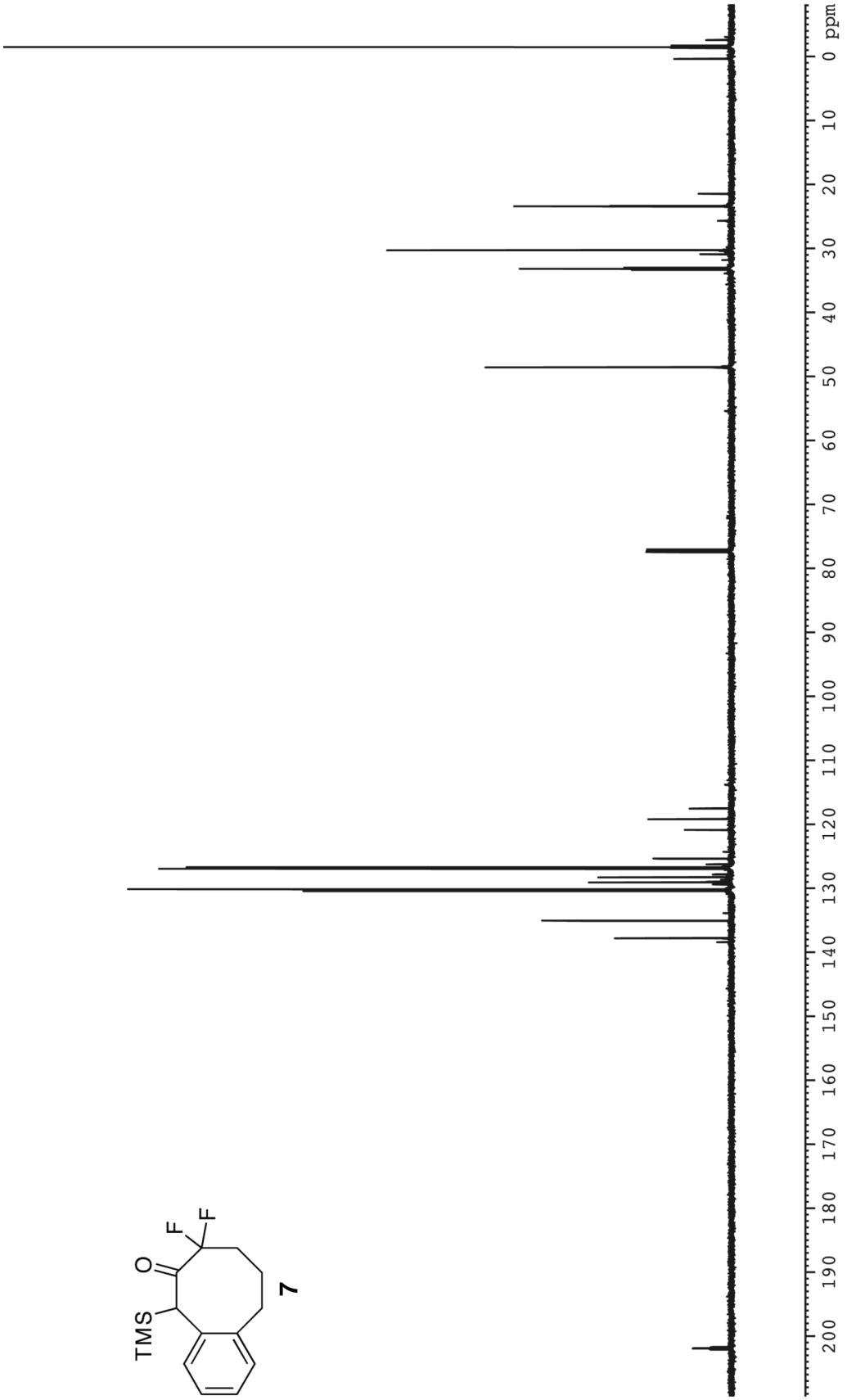
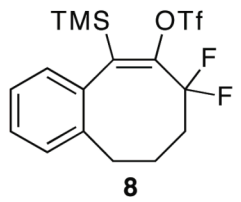
1.069
2.285
1.780
2.120
2.087


16
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
4.25
2.00
1.94
1.80
2.14

^{13}C-NMR of compounds 4-6, 8-14, 16

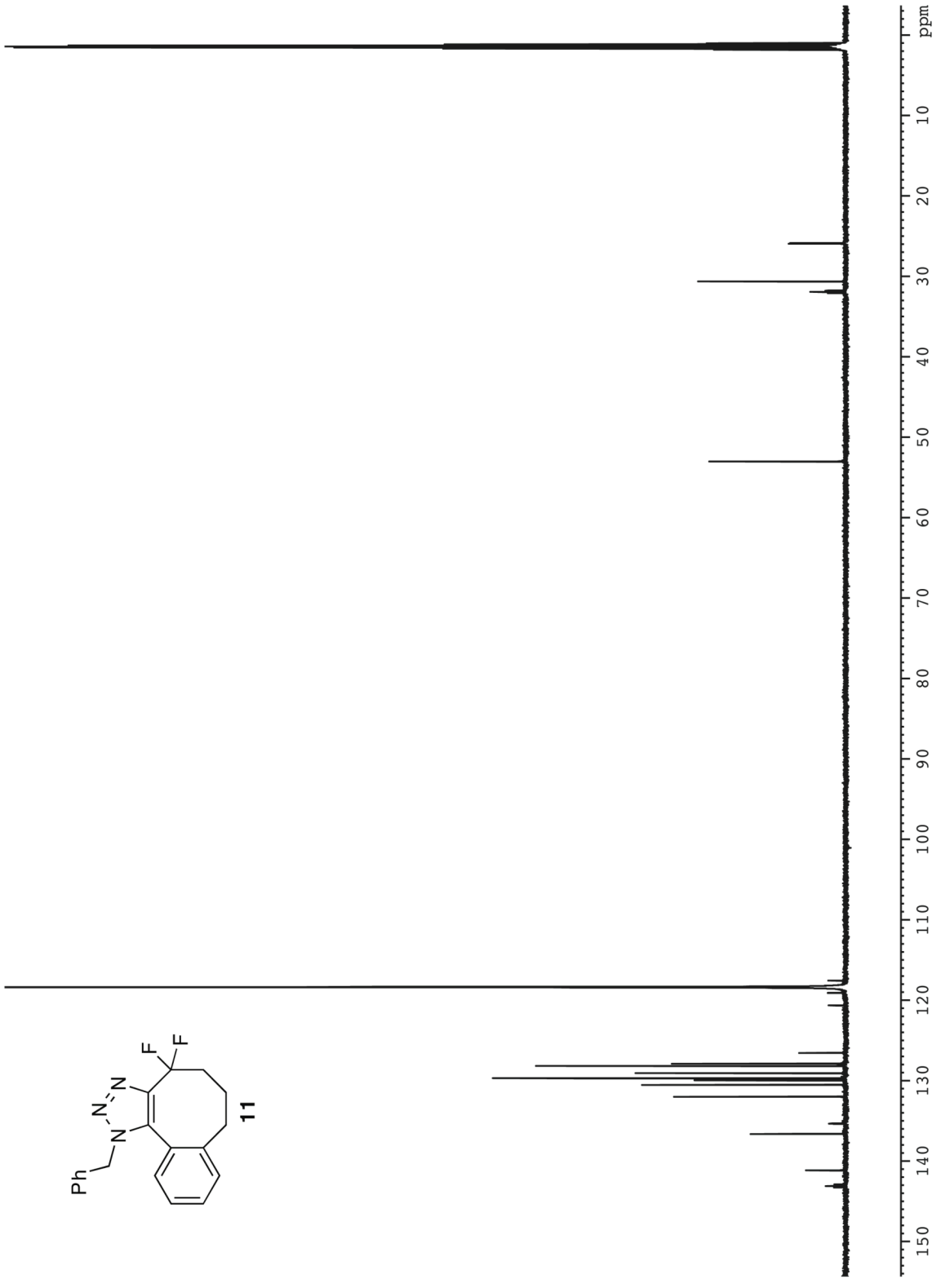


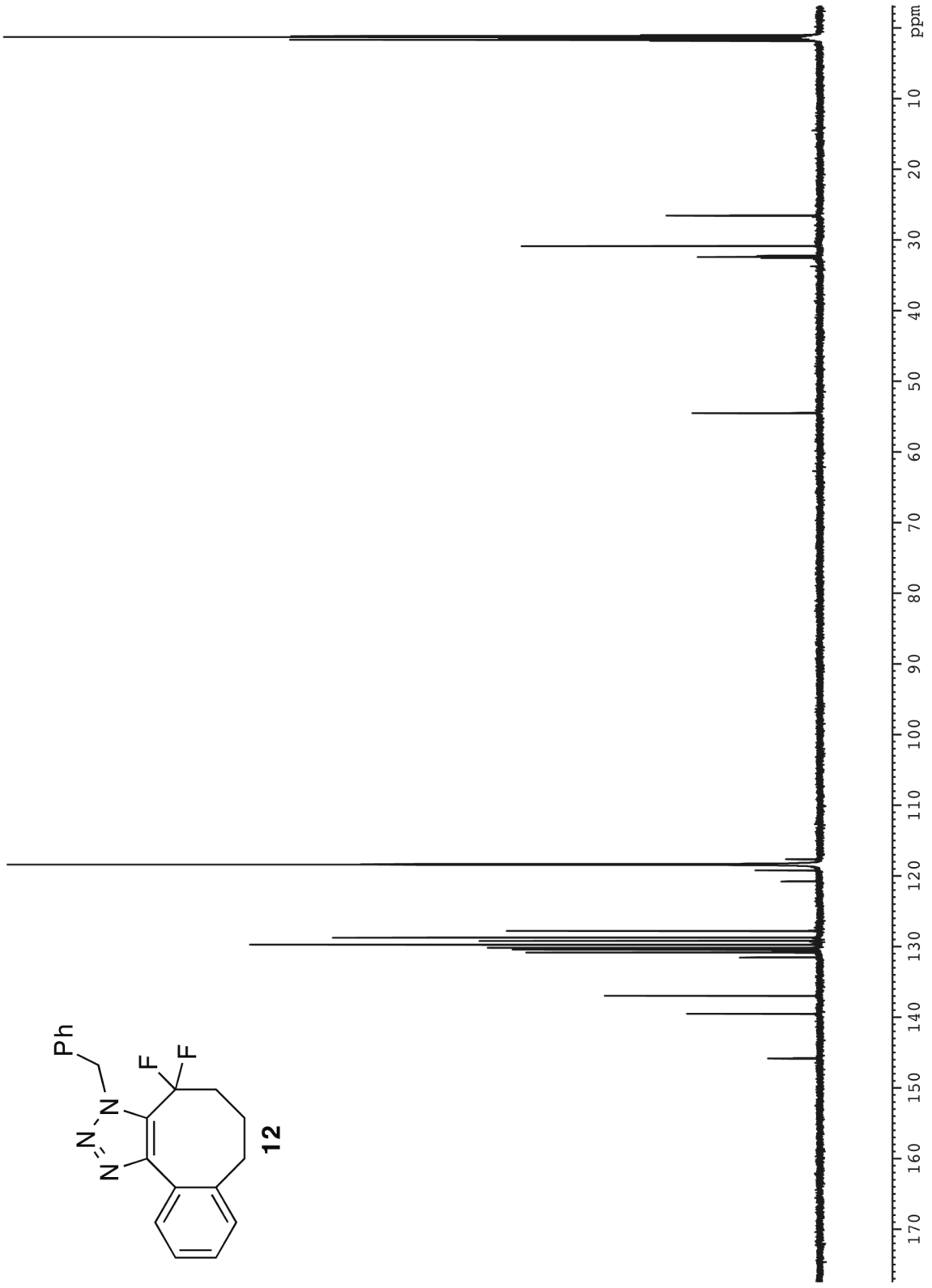

TMS
O
F
F
7
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

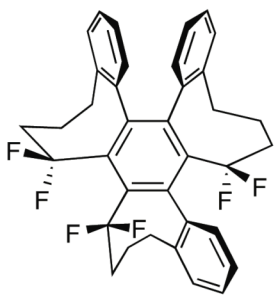

TMS
OTf
F
F

8

180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

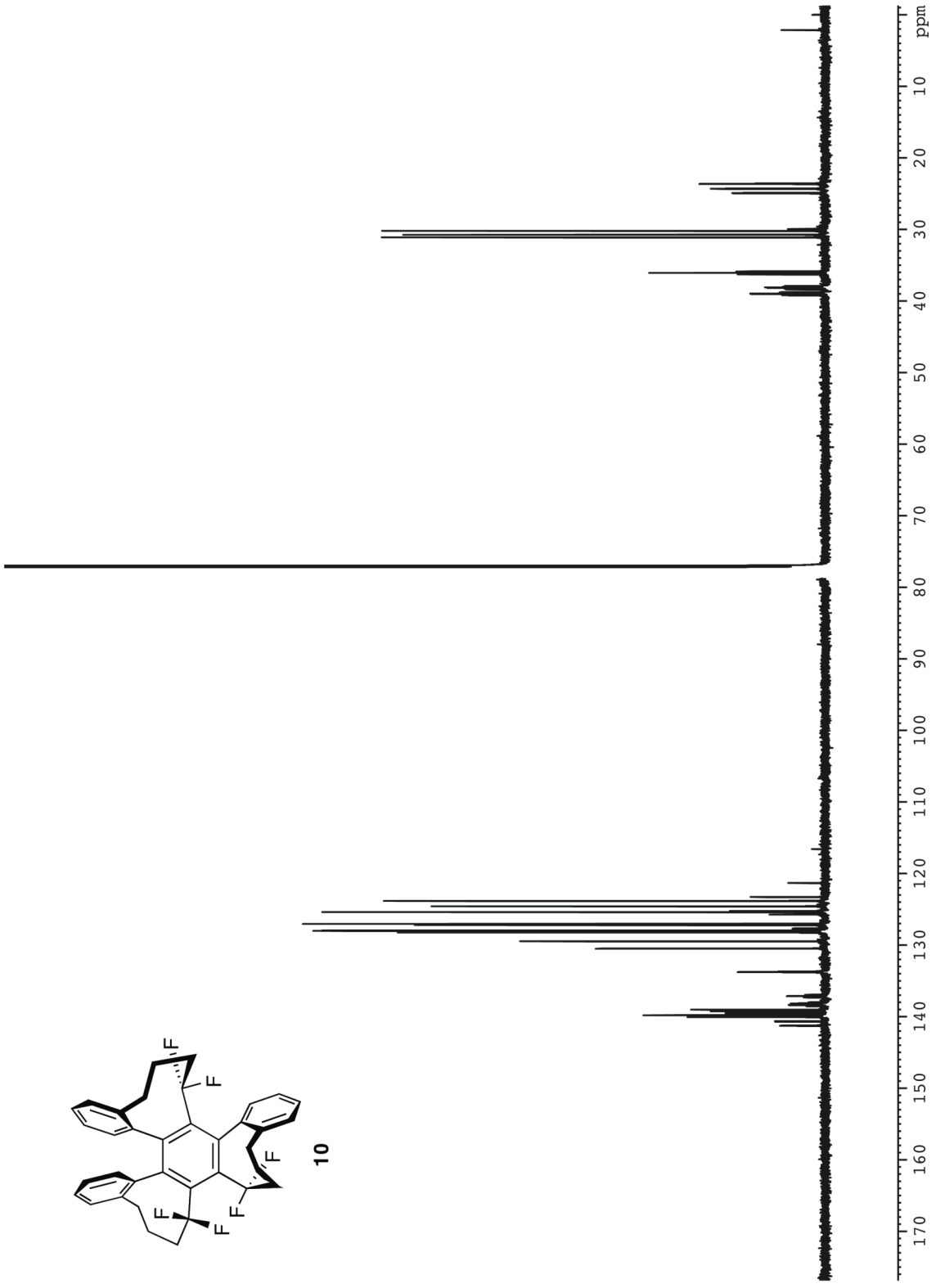

F
F
N
N
N
Ph
11
ppm
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150

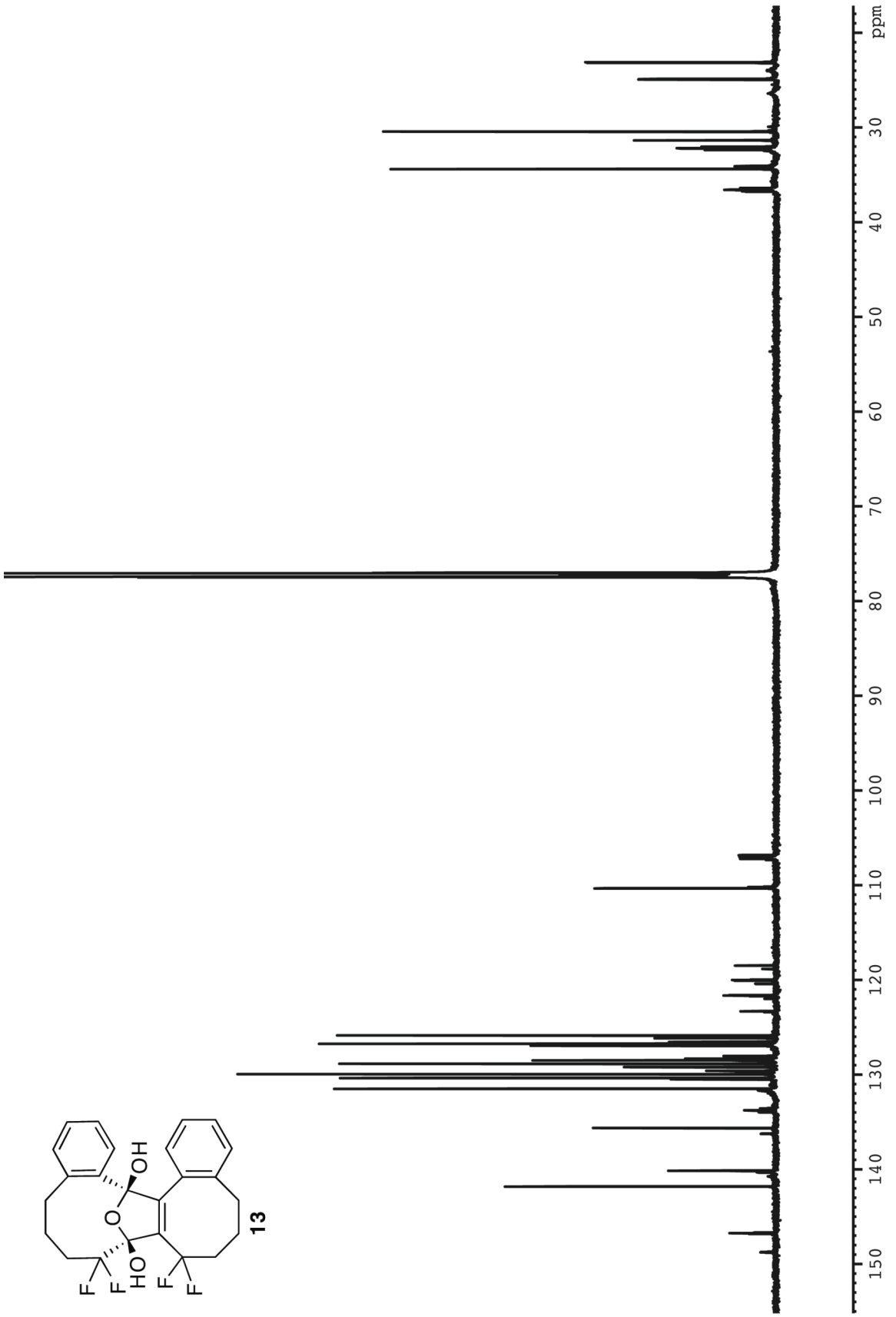

Ph
F
F
N
N
N
12
ppm
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170

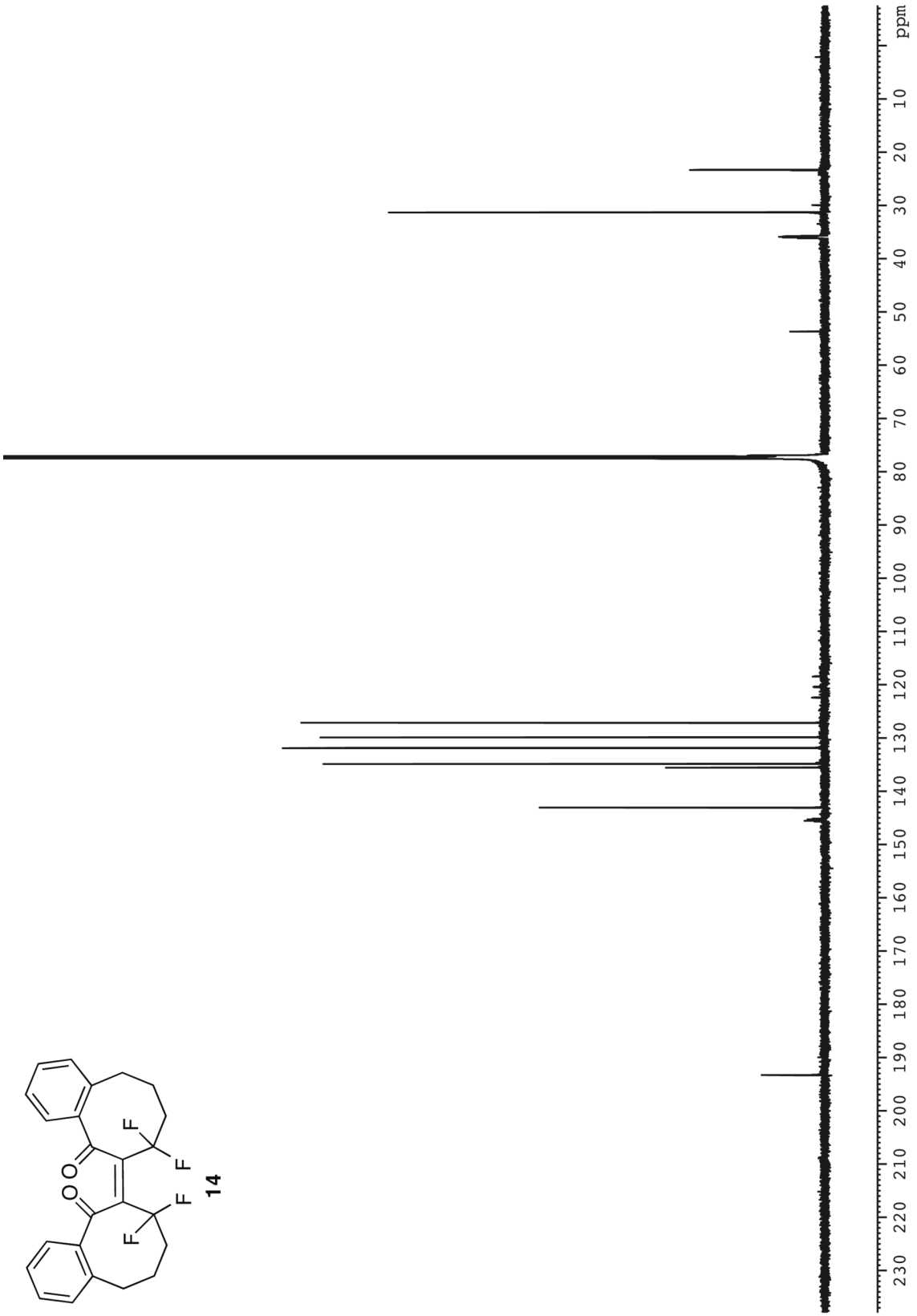

F
F
F
F
F
F

9

190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


F
F
F
F
F
F
10
ppm
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170


OH
O
HO
F
F
F
F
13
ppm
30
40
50
60
70
80
90
100
110
120
130
140
150


O
O
F
F
F
F
14
ppm
10
20
30
40
50
60
70
80
90
100
110
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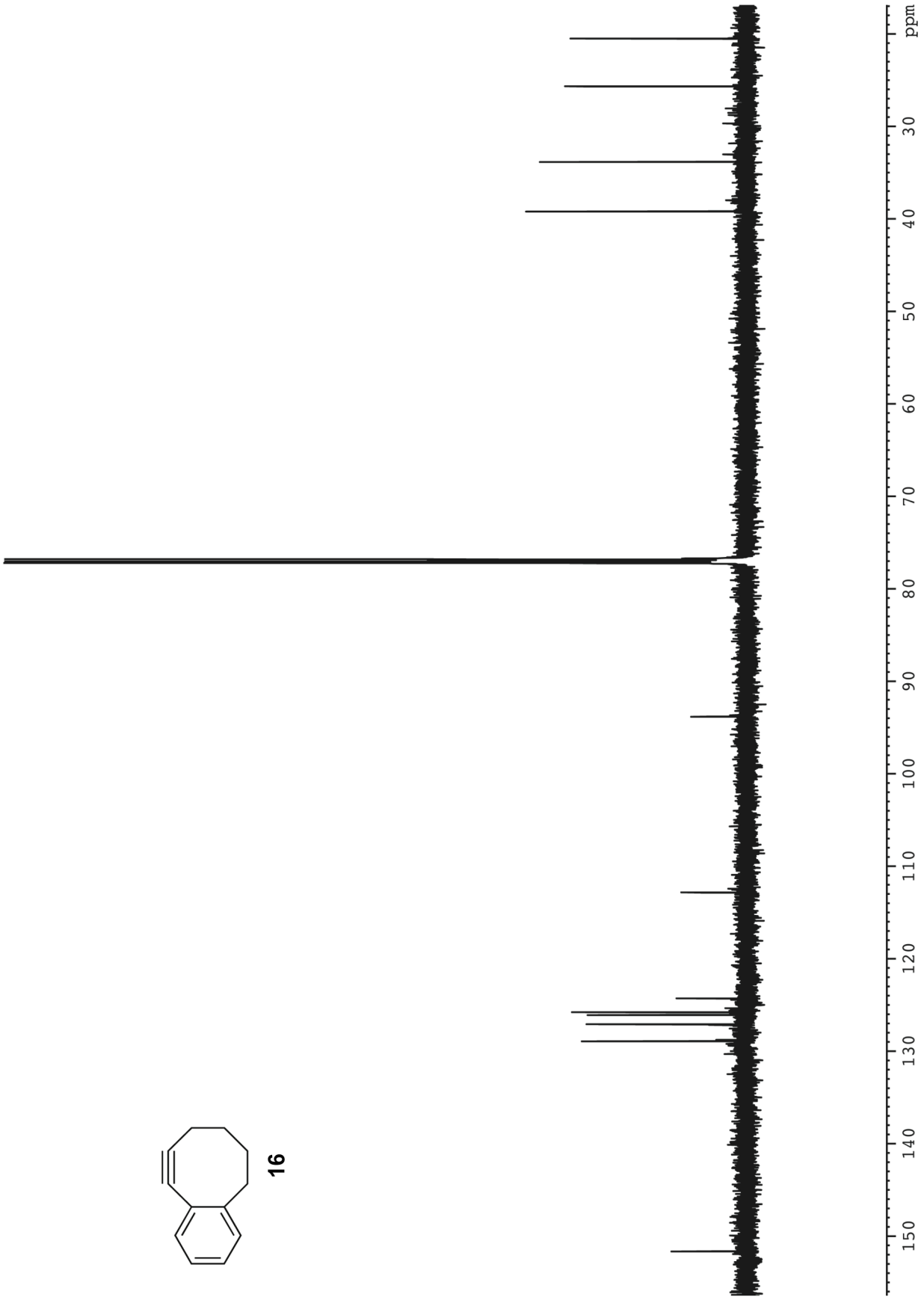
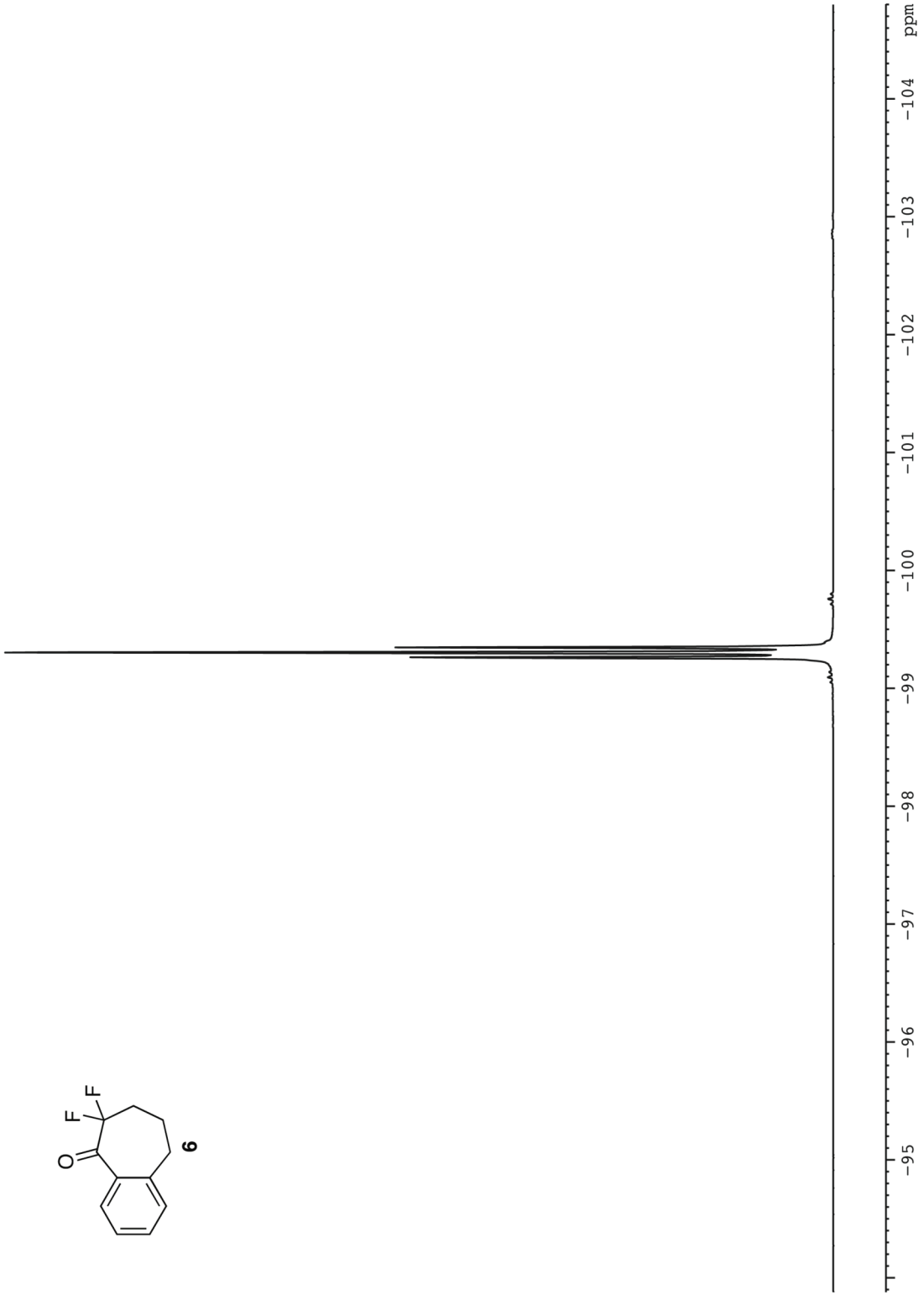
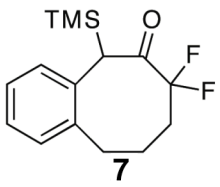
140
150
160
170
180
190
200
210
220
230



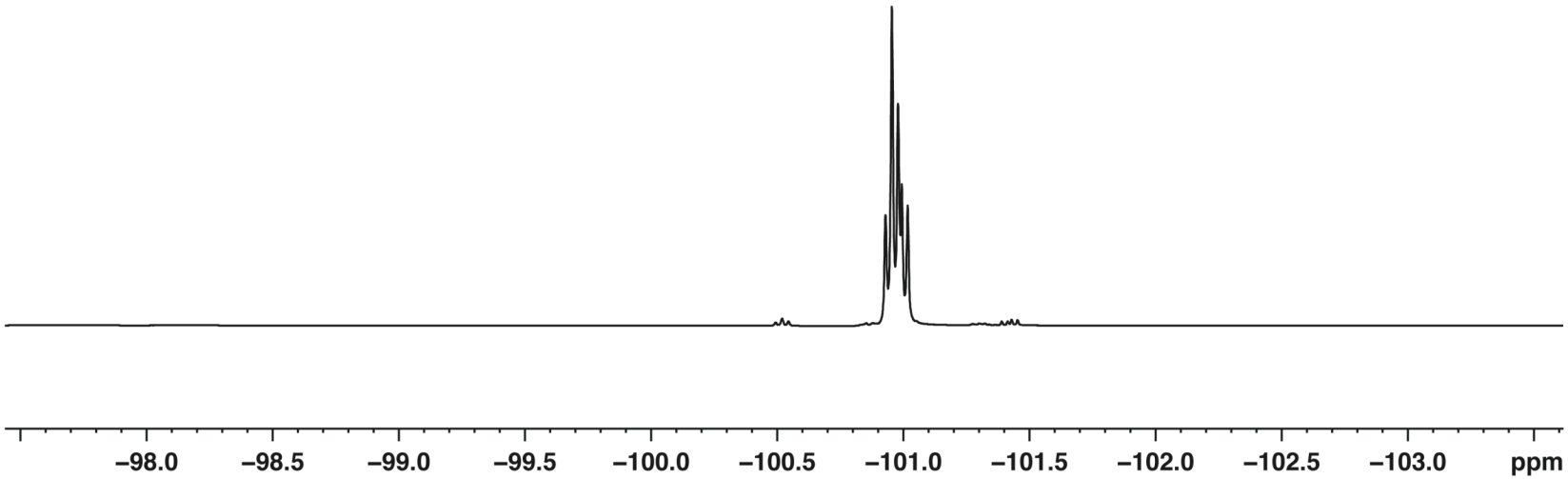
16

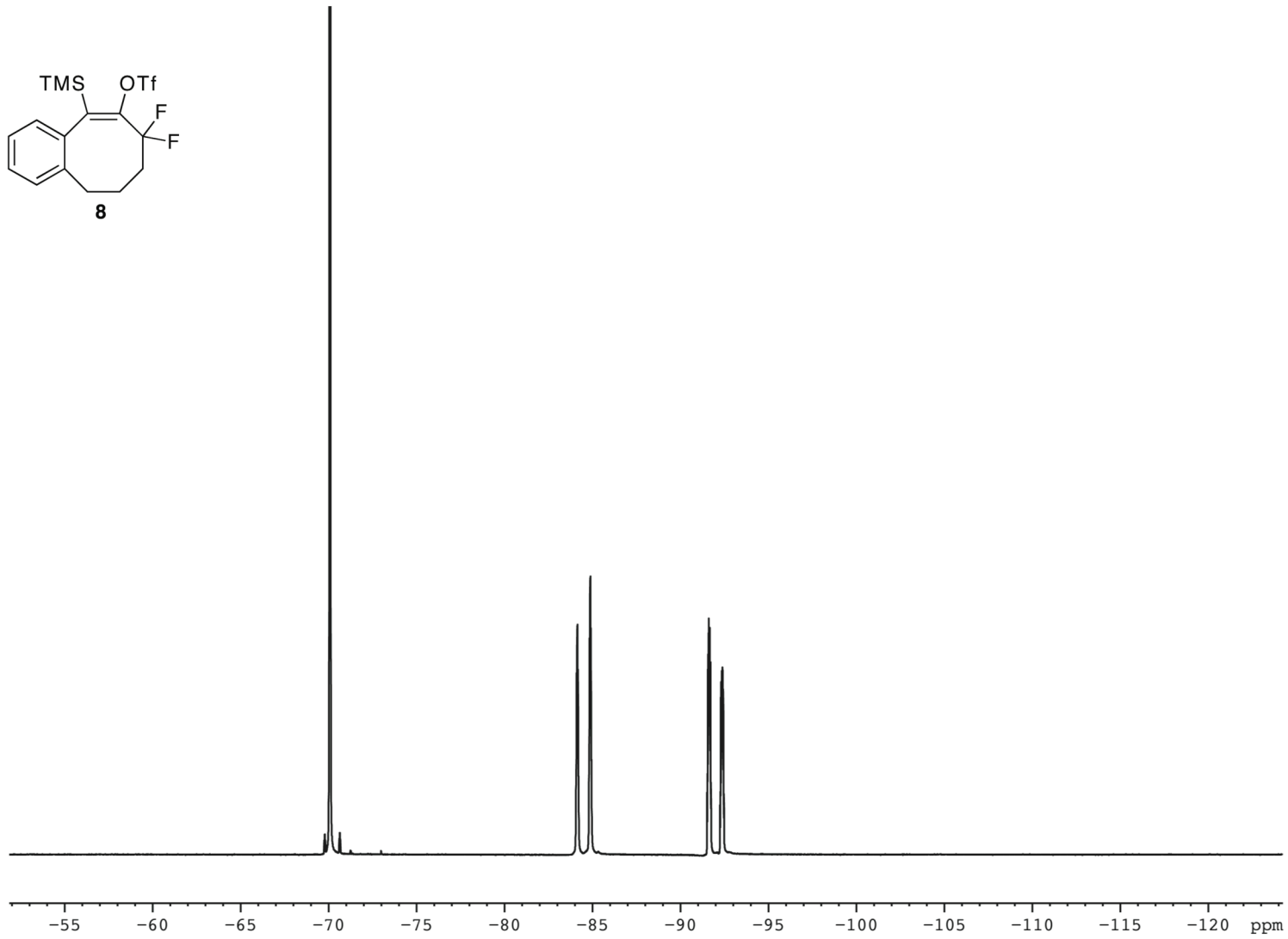
^{19}F-NMR of compounds 4-6, 8-14

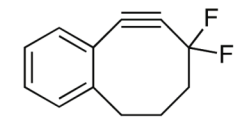




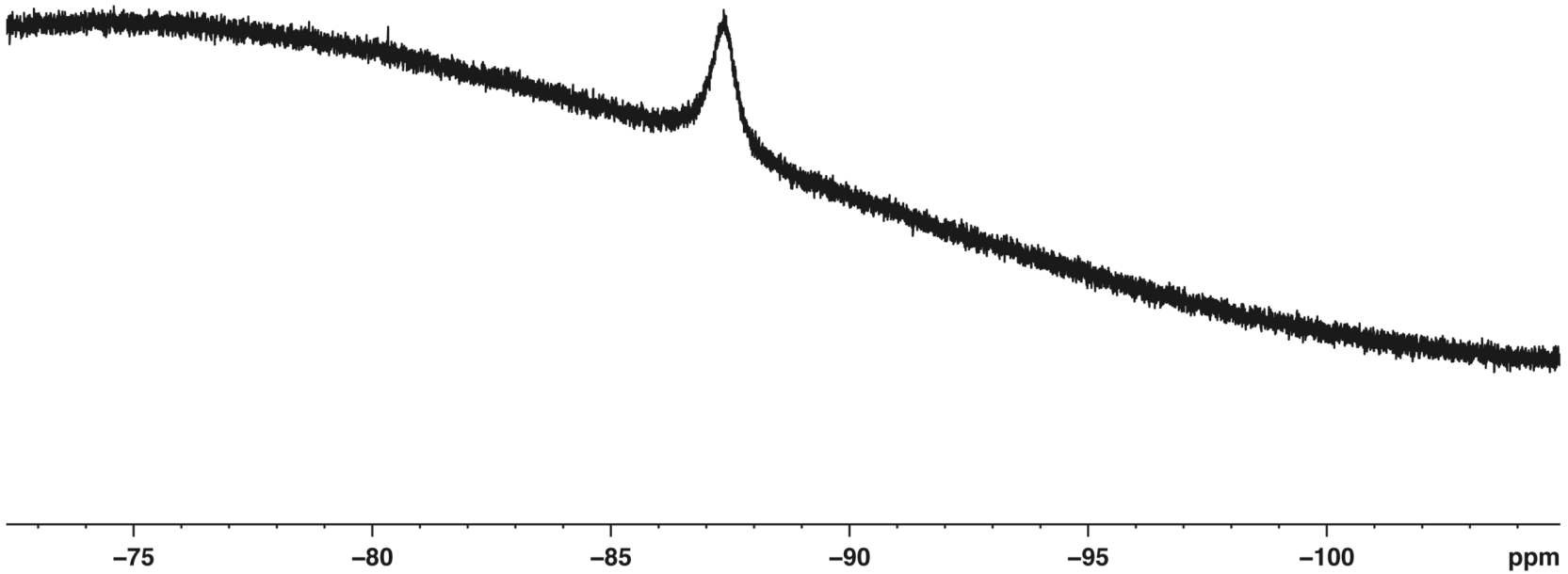
7

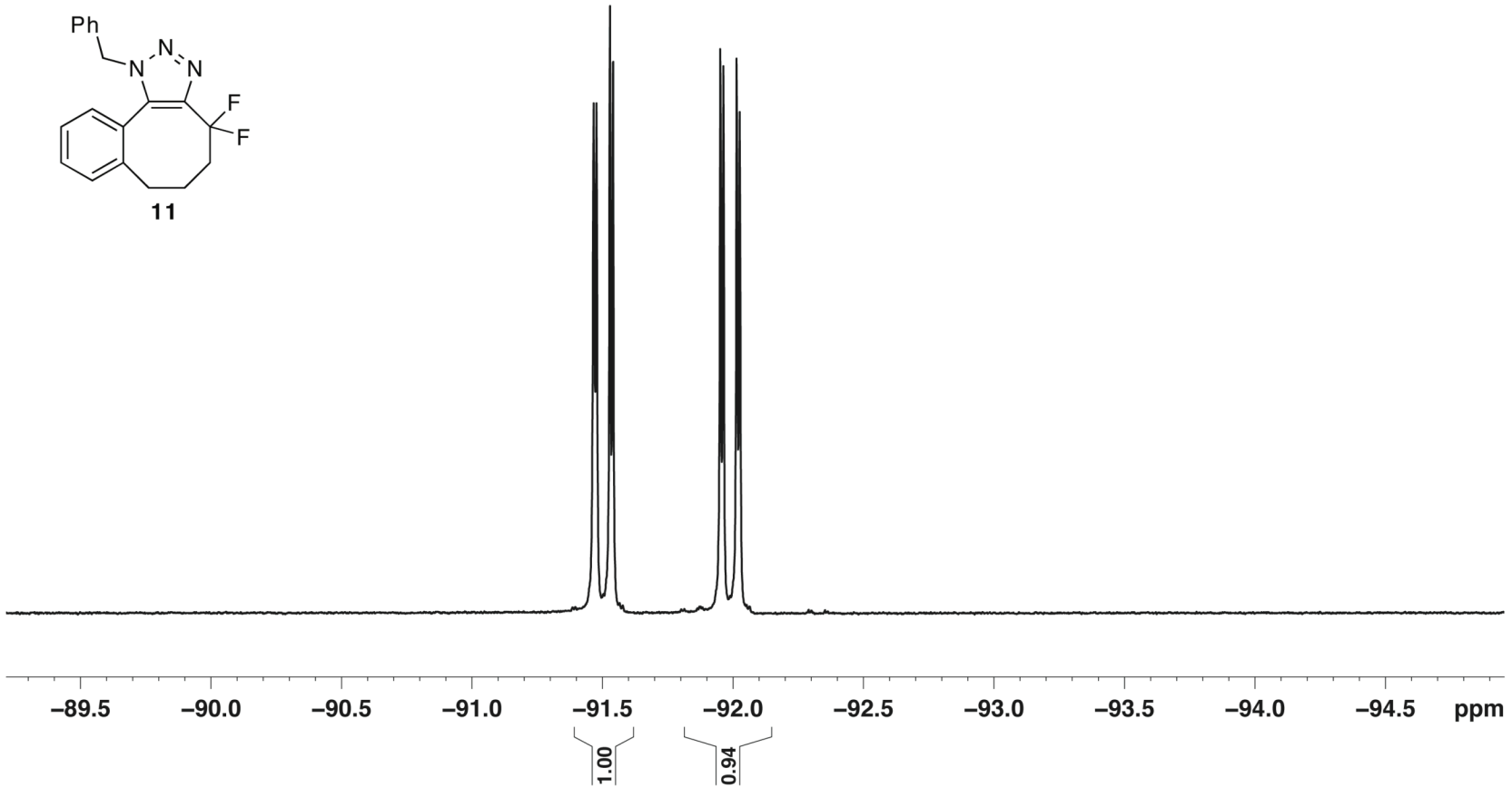


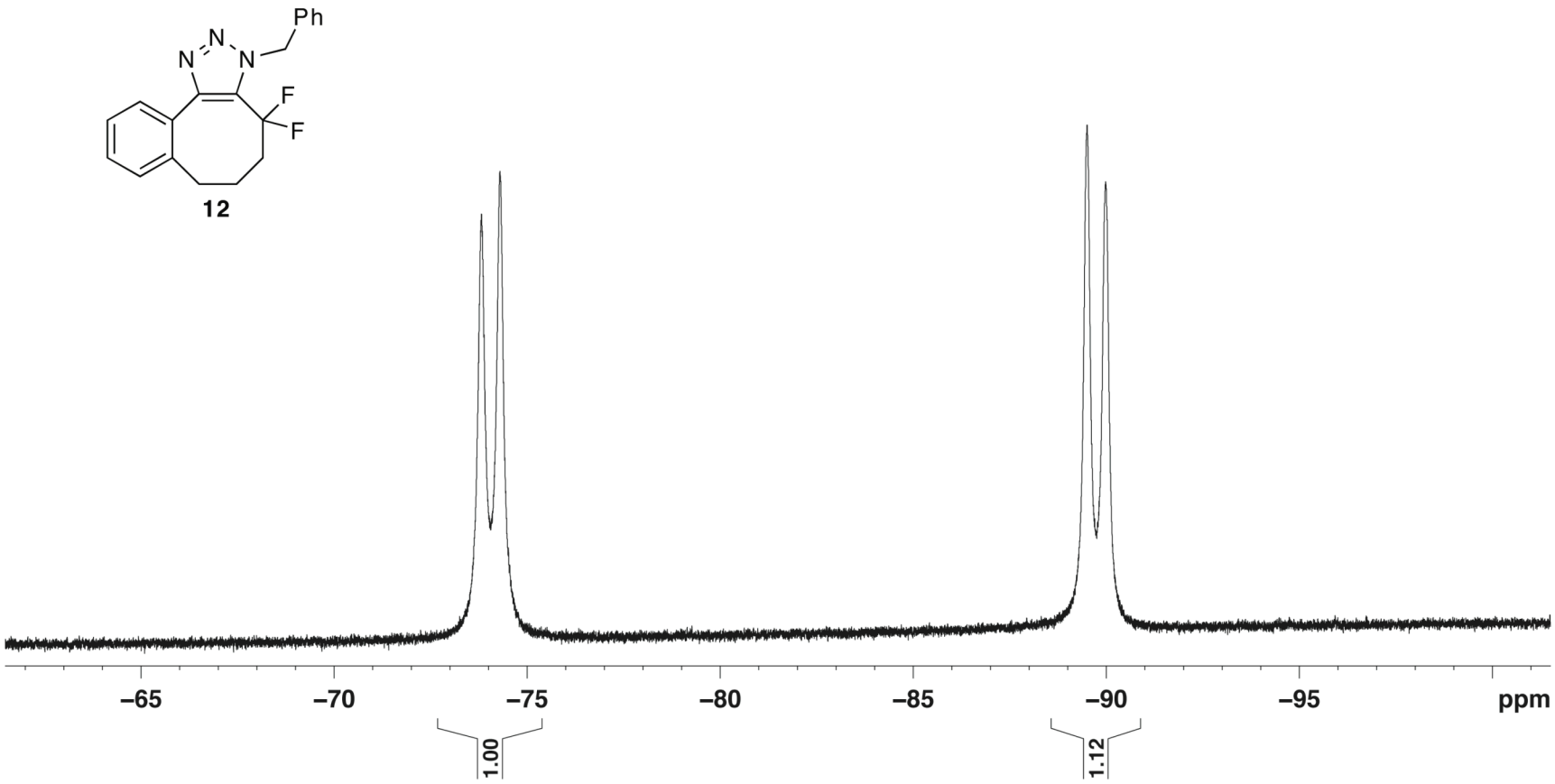

TMS
OTf
F
F
8
-55
-60
-65
-70
-75
-80
-85
-90
-95
-100
-105
-110
-115
-120
ppm



4



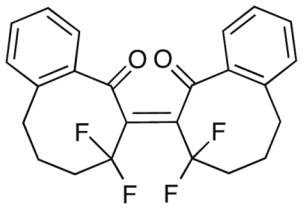

Ph
N
N
N
F
F
11
−89.5
−90.0
−90.5
−91.0
−91.5
−92.0
−92.5
−93.0
−93.5
−94.0
−94.5
ppm
1.00
0.94


Ph
N
N
N
F
F
12
–65
–70
–75
–80
–85
–90
–95
ppm
1.00
1.12

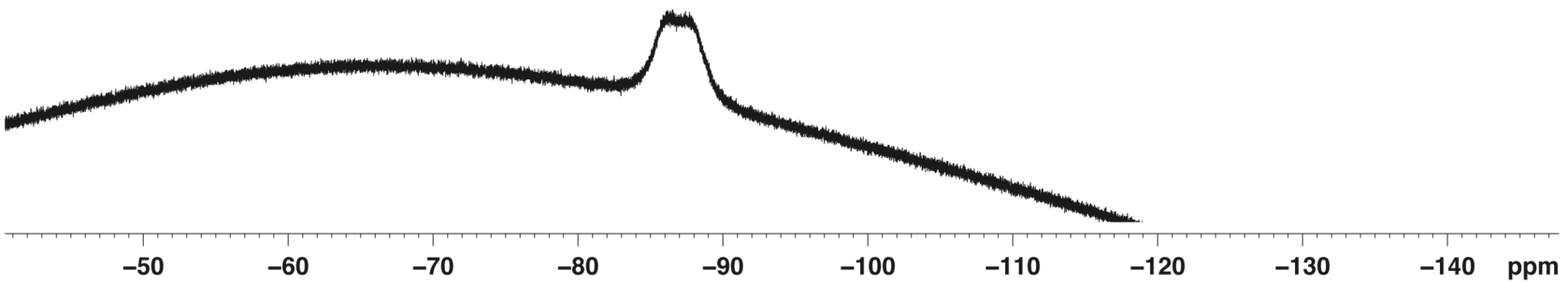
F
F
F
F
F
F
9
ppm
−55
−60
−65
−70
−75
−80
−85
1.00
1.04
1.02
1.09
1.07
1.05

F
F
F
F
F
10
ppm
−50
−55
−60
−65
−70
−75
−80
1.00
1.03
1.06
1.08
2.25

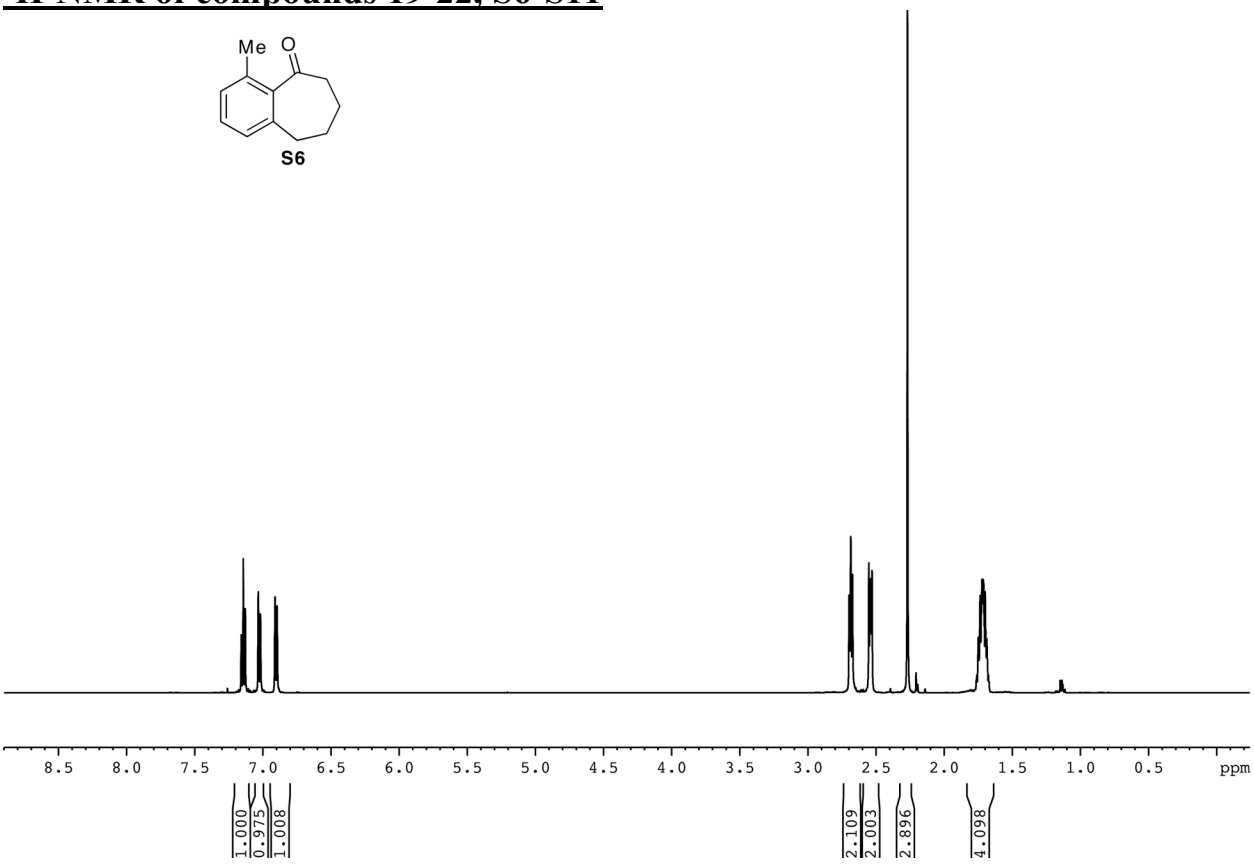
ppm
−65
−70
−75
−80
−85
−90
−95
−100
−105
−110
−115
−120
0.53
0.25
0.46
1.00
1.06
0.48
1.26
1.13
OH
HO
O
F
F
F
F
13

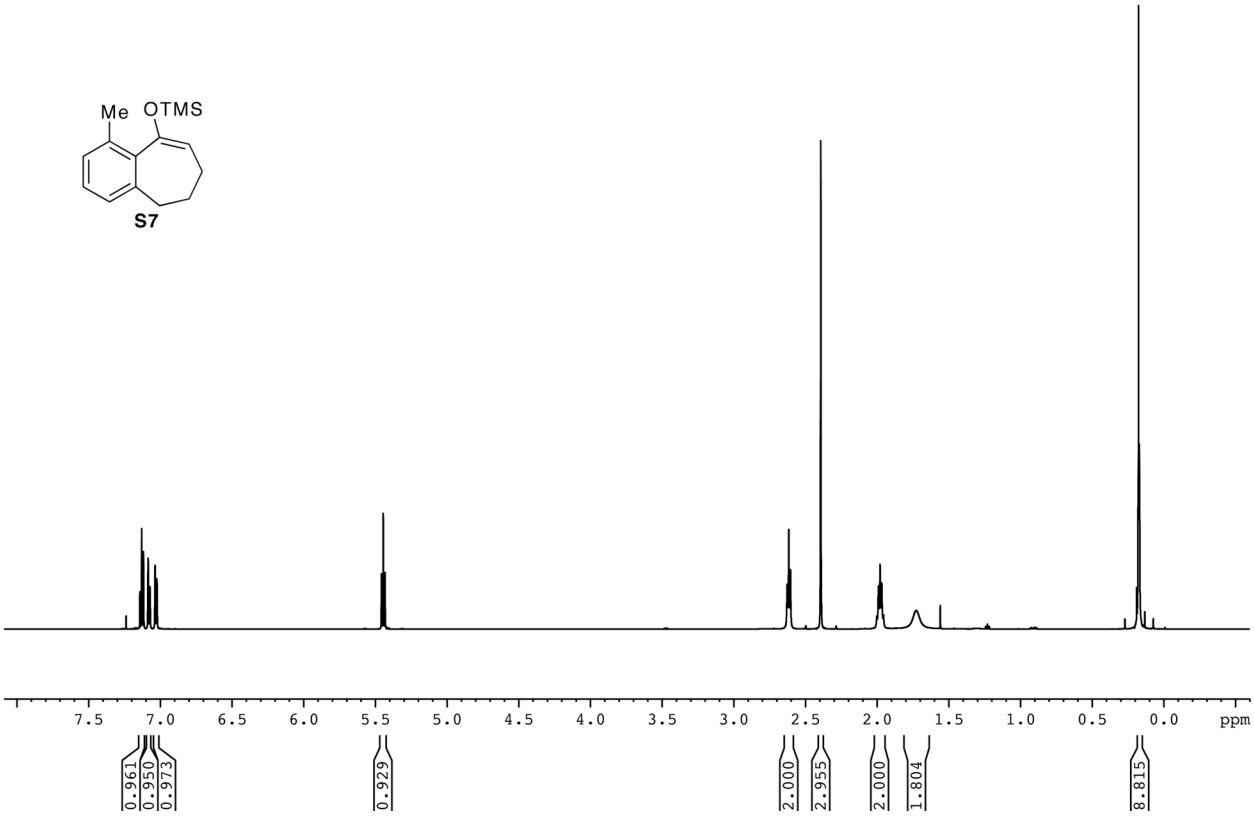


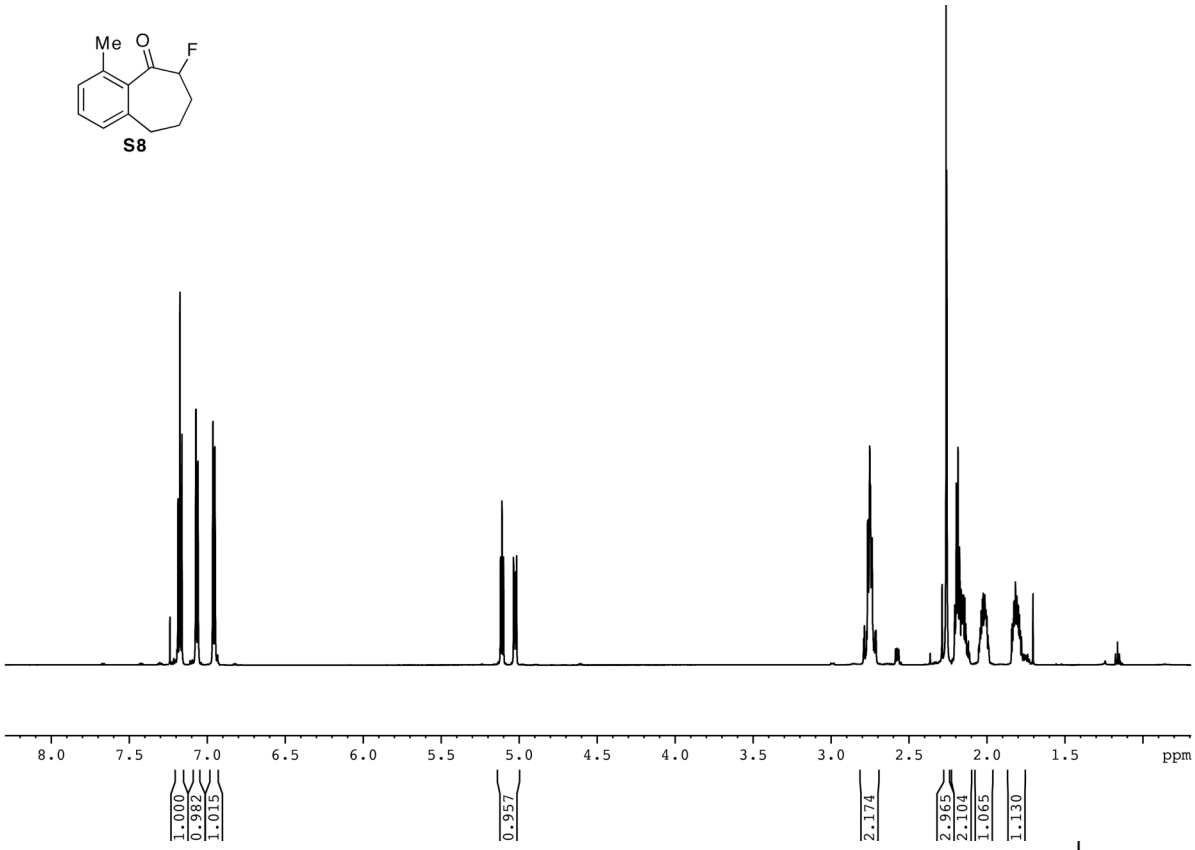
14

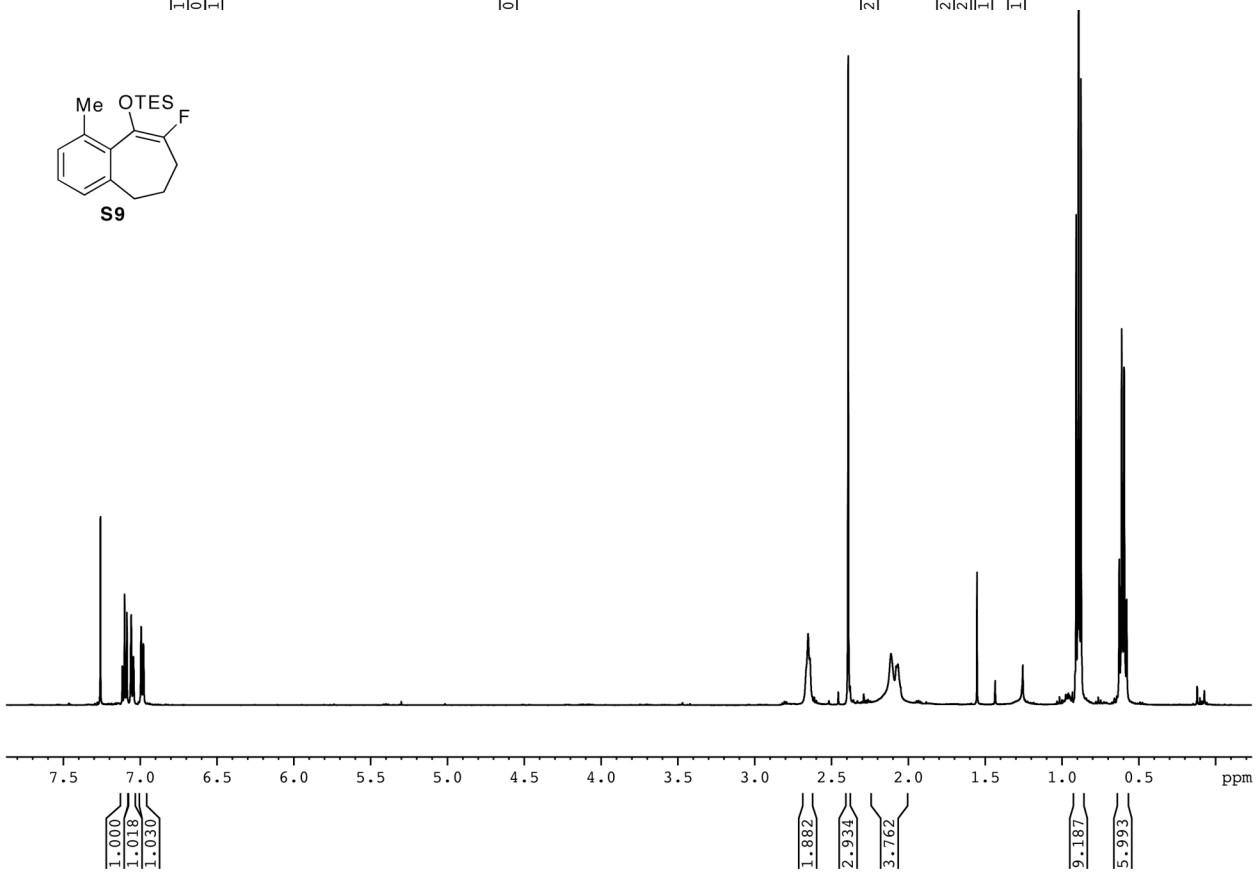


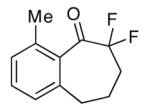
^{1}H-NMR of compounds 19-22, S6-S11





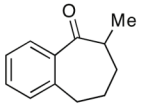

Me
O
F
S8
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
ppm
1.000
0.982
1.015
0.957
2.174
2.965
2.104
1.065
1.130


Me
OTES
F
S9
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
1.000
1.018
1.030
1.882
2.934
3.762
9.187
5.993


Me
O
F
F

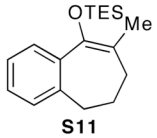
19

7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5 ppm
1.12
0.97
0.98
2.00
4.93
1.99


O
Me

S10

8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0 ppm
0.90
1.00
2.10
3.04
1.00
1.26
2.20
2.91


OTES
Me

S11

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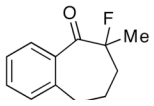
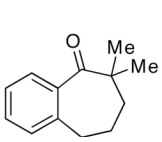
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
0.962
1.006
2.018
2.000
2.059
3.040
2.071
9.112
6.017


O
F
Me

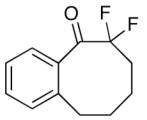
20

8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
1.000
1.028
1.065
1.007
1.028
1.029
1.053
2.088
1.044
3.088


O
Me
Me

21

7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
1.000
2.072
0.987
2.028
2.191
2.324
6.101


O
F
F

22

10
9
8
7
6
5
4
3
2
1
ppm
0.982
1.003
1.954
1.978
2.000
2.031
2.037

X-ray crystallography data

Major Trimer (9)

A colorless prism 0.12 x 0.08 x 0.04 mm in size was mounted on a Cryoloop with Paratone oil. Data were collected in a nitrogen gas stream at 100(2) K using phi and omega scans. Crystal-to-detector distance was 60 mm and exposure time was 5 seconds per frame using a scan width of 1.0°. Data collection was 99.9% complete to 67.00° in θ. A total of 12405 reflections were collected covering the indices, -10<=*h*<=10, -15<=*k*<=14, -14<=*l*<=14. 4349 reflections were found to be symmetry independent, with an R_{int} of 0.0149. Indexing and unit cell refinement indicated a primitive, monoclinic lattice. The space group was found to be P2(1) (No. 4). The data were integrated using the Bruker SAINT software program and scaled using the SADABS software program. Solution by direct methods (SIR-2008) produced a complete heavy-atom phasing model consistent with the proposed structure. All non-hydrogen atoms were refined anisotropically by full-matrix least-squares (SHELXL-97). All hydrogen atoms were placed using a riding model. Their positions were constrained relative to their parent atom using the appropriate HFIX command in SHELXL-97.

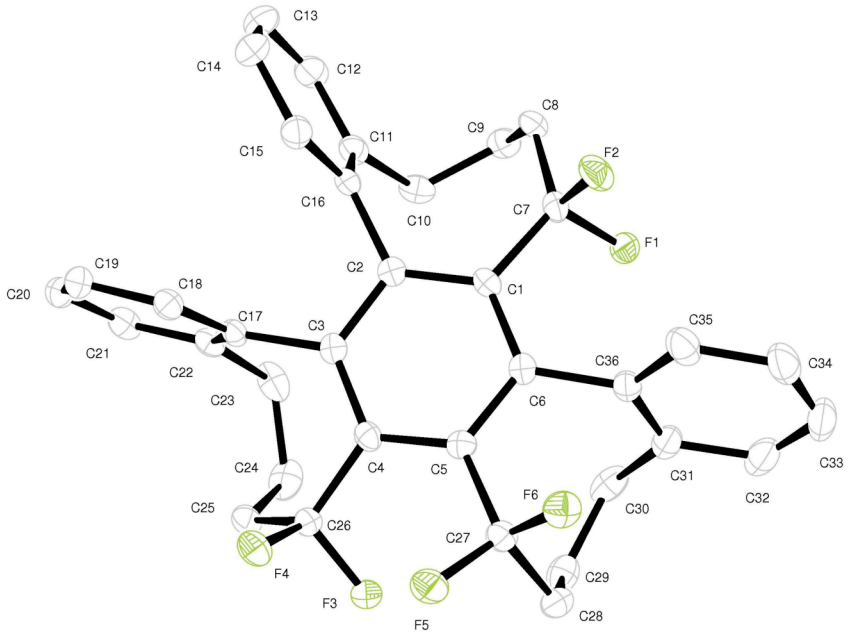


Figure S12. X-ray structure of trimer **9** with labeled carbon and fluorine atoms. ORTEPs are shown at 50% probability.

Table S1. Crystal data and structure refinement for major trimer (**9**).

Empirical formula	C36 H30 F6	
Formula weight	576.60	
Temperature	100(2) K	
Wavelength	1.54178 Å	
Crystal system	Monoclinic	
Space group	P2(1)	
Unit cell dimensions	a = 9.0552(4) Å	α= 90°.
	b = 12.8869(6) Å	β= 103.344(2)°.
	c = 12.0073(5) Å	γ = 90°.
Volume	1363.34(10) $Å^3$	
Z	2	
Density (calculated)	1.405 Mg/m^3	
Absorption coefficient	0.916 mm^{-1}	
F(000)	600	
Crystal size	0.12 x 0.08 x 0.04 mm^3	
Crystal color/habit	colorless prism	
Theta range for data collection	3.78 to 68.17°.	
Index ranges	-10<=h<=10, -15<=k<=14, -14<=l<=14	
Reflections collected	12405	
Independent reflections	4349 [R(int) = 0.0149]	
Completeness to theta = 67.00°	99.9 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.9643 and 0.8980	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4349 / 1 / 379	
Goodness-of-fit on F^2	1.038	
Final R indices [I>2sigma(I)]	R1 = 0.0274, wR2 = 0.0735	
R indices (all data)	R1 = 0.0282, wR2 = 0.0744	
Absolute structure parameter	0.07(7)	
Largest diff. peak and hole	0.247 and -0.132 e.$Å^{-3}$	

Table S2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for major trimer (**9**). U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	6239(2)	3861(1)	280(1)	17(1)
C(2)	6327(2)	3431(1)	-776(1)	17(1)
C(3)	7552(2)	2772(1)	-842(1)	17(1)
C(4)	8631(2)	2496(1)	147(1)	18(1)
C(5)	8354(2)	2758(1)	1225(1)	18(1)
C(6)	7221(2)	3485(1)	1297(1)	18(1)
C(7)	5100(2)	4718(1)	394(1)	20(1)
C(8)	4213(2)	5371(1)	-584(1)	22(1)
C(9)	5138(2)	6080(1)	-1190(2)	25(1)
C(10)	6134(2)	5496(2)	-1861(2)	24(1)
C(11)	5223(2)	4600(2)	-2461(1)	22(1)
C(12)	4186(2)	4759(2)	-3503(1)	28(1)
C(13)	3141(2)	4002(2)	-3972(2)	34(1)
C(14)	3114(2)	3070(2)	-3402(2)	31(1)
C(15)	4148(2)	2899(2)	-2361(1)	24(1)
C(16)	5196(2)	3657(1)	-1886(1)	19(1)
C(17)	7656(2)	2411(1)	-2019(1)	17(1)
C(18)	6879(2)	1536(1)	-2504(1)	20(1)
C(19)	6867(2)	1255(2)	-3626(1)	23(1)
C(20)	7668(2)	1832(2)	-4247(1)	26(1)
C(21)	8496(2)	2682(2)	-3755(1)	24(1)
C(22)	8491(2)	2994(1)	-2643(1)	21(1)
C(23)	9501(2)	3856(2)	-2059(2)	29(1)
C(24)	10902(2)	3430(2)	-1178(2)	33(1)
C(25)	10871(2)	2280(2)	-920(2)	28(1)
C(26)	10126(2)	1971(1)	44(1)	23(1)
C(27)	9212(2)	2249(1)	2350(1)	21(1)
C(28)	10528(2)	2846(2)	3119(1)	25(1)
C(29)	10954(2)	3902(2)	2695(2)	28(1)
C(30)	9712(2)	4757(2)	2495(2)	29(1)
C(31)	8382(2)	4534(1)	3025(1)	25(1)
C(32)	8341(3)	4959(2)	4090(2)	37(1)
C(33)	7159(3)	4740(2)	4604(2)	42(1)
C(34)	5993(2)	4086(2)	4059(2)	38(1)
C(35)	6004(2)	3678(2)	2984(2)	29(1)
C(36)	7181(2)	3909(1)	2468(1)	22(1)
F(1)	5874(1)	5429(1)	1182(1)	25(1)
F(2)	4033(1)	4275(1)	902(1)	28(1)
F(3)	11210(1)	2193(1)	1030(1)	27(1)
F(4)	9961(1)	913(1)	21(1)	30(1)
F(5)	9674(1)	1275(1)	2129(1)	28(1)
F(6)	8148(1)	2025(1)	2983(1)	25(1)

Table S3. Bond lengths [Å] and angles [°] for major trimer (**9**).

C(1)-C(2)	1.402(2)	C(19)-H(19)	0.9500
C(1)-C(6)	1.419(2)	C(20)-C(21)	1.381(3)
C(1)-C(7)	1.539(2)	C(20)-H(20)	0.9500
C(2)-C(3)	1.414(2)	C(21)-C(22)	1.395(2)
C(2)-C(16)	1.510(2)	C(21)-H(21)	0.9500
C(3)-C(4)	1.398(2)	C(22)-C(23)	1.506(2)
C(3)-C(17)	1.512(2)	C(23)-C(24)	1.553(3)
C(4)-C(5)	1.414(2)	C(23)-H(23A)	0.9900
C(4)-C(26)	1.544(2)	C(23)-H(23B)	0.9900
C(5)-C(6)	1.408(2)	C(24)-C(25)	1.515(3)
C(5)-C(27)	1.540(2)	C(24)-H(24A)	0.9900
C(6)-C(36)	1.516(2)	C(24)-H(24B)	0.9900
C(7)-F(2)	1.3794(19)	C(25)-C(26)	1.520(2)
C(7)-F(1)	1.3858(19)	C(25)-H(25A)	0.9900
C(7)-C(8)	1.516(2)	C(25)-H(25B)	0.9900
C(8)-C(9)	1.532(2)	C(26)-F(4)	1.371(2)
C(8)-H(8A)	0.9900	C(26)-F(3)	1.3815(19)
C(8)-H(8B)	0.9900	C(27)-F(5)	1.368(2)
C(9)-C(10)	1.537(2)	C(27)-F(6)	1.389(2)
C(9)-H(9A)	0.9900	C(27)-C(28)	1.535(2)
C(9)-H(9B)	0.9900	C(28)-C(29)	1.533(3)
C(10)-C(11)	1.503(2)	C(28)-H(28A)	0.9900
C(10)-H(10A)	0.9900	C(28)-H(28B)	0.9900
C(10)-H(10B)	0.9900	C(29)-C(30)	1.554(3)
C(11)-C(12)	1.395(2)	C(29)-H(29A)	0.9900
C(11)-C(16)	1.401(2)	C(29)-H(29B)	0.9900
C(12)-C(13)	1.386(3)	C(30)-C(31)	1.514(3)
C(12)-H(12)	0.9500	C(30)-H(30A)	0.9900
C(13)-C(14)	1.385(3)	C(30)-H(30B)	0.9900
C(13)-H(13)	0.9500	C(31)-C(36)	1.394(3)
C(14)-C(15)	1.396(2)	C(31)-C(32)	1.399(3)
C(14)-H(14)	0.9500	C(32)-C(33)	1.383(3)
C(15)-C(16)	1.388(2)	C(32)-H(32)	0.9500
C(15)-H(15)	0.9500	C(33)-C(34)	1.391(4)
C(17)-C(18)	1.384(2)	C(33)-H(33)	0.9500
C(17)-C(22)	1.400(2)	C(34)-C(35)	1.396(3)
C(18)-C(19)	1.393(2)	C(34)-H(34)	0.9500
C(18)-H(18)	0.9500	C(35)-C(36)	1.383(2)
C(19)-C(20)	1.373(3)	C(35)-H(35)	0.9500
C(2)-C(1)-C(6)	119.23(14)	C(5)-C(6)-C(36)	118.09(14)
C(2)-C(1)-C(7)	122.86(14)	C(1)-C(6)-C(36)	122.05(14)
C(6)-C(1)-C(7)	117.90(13)	F(2)-C(7)-F(1)	105.50(12)
C(1)-C(2)-C(3)	119.76(14)	F(2)-C(7)-C(8)	105.79(13)
C(1)-C(2)-C(16)	123.63(14)	F(1)-C(7)-C(8)	104.84(14)
C(3)-C(2)-C(16)	116.58(13)	F(2)-C(7)-C(1)	107.14(14)
C(4)-C(3)-C(2)	120.59(14)	F(1)-C(7)-C(1)	106.94(12)
C(4)-C(3)-C(17)	122.25(14)	C(8)-C(7)-C(1)	125.17(14)
C(2)-C(3)-C(17)	117.14(14)	C(7)-C(8)-C(9)	116.69(13)
C(3)-C(4)-C(5)	118.68(14)	C(7)-C(8)-H(8A)	108.1
C(3)-C(4)-C(26)	119.79(14)	C(9)-C(8)-H(8A)	108.1
C(5)-C(4)-C(26)	121.45(14)	C(7)-C(8)-H(8B)	108.1
C(6)-C(5)-C(4)	120.04(15)	C(9)-C(8)-H(8B)	108.1
C(6)-C(5)-C(27)	117.15(14)	H(8A)-C(8)-H(8B)	107.3
C(4)-C(5)-C(27)	122.77(14)	C(8)-C(9)-C(10)	114.10(14)
C(5)-C(6)-C(1)	119.66(14)	C(8)-C(9)-H(9A)	108.7

C(10)-C(9)-H(9A)	108.7
C(8)-C(9)-H(9B)	108.7
C(10)-C(9)-H(9B)	108.7
H(9A)-C(9)-H(9B)	107.6
C(11)-C(10)-C(9)	107.64(13)
C(11)-C(10)-H(10A)	110.2
C(9)-C(10)-H(10A)	110.2
C(11)-C(10)-H(10B)	110.2
C(9)-C(10)-H(10B)	110.2
H(10A)-C(10)-H(10B)	108.5
C(12)-C(11)-C(16)	118.98(17)
C(12)-C(11)-C(10)	119.89(17)
C(16)-C(11)-C(10)	120.06(14)
C(13)-C(12)-C(11)	121.08(18)
C(13)-C(12)-H(12)	119.5
C(11)-C(12)-H(12)	119.5
C(14)-C(13)-C(12)	119.87(16)
C(14)-C(13)-H(13)	120.1
C(12)-C(13)-H(13)	120.1
C(13)-C(14)-C(15)	119.63(18)
C(13)-C(14)-H(14)	120.2
C(15)-C(14)-H(14)	120.2
C(16)-C(15)-C(14)	120.69(18)
C(16)-C(15)-H(15)	119.7
C(14)-C(15)-H(15)	119.7
C(15)-C(16)-C(11)	119.74(15)
C(15)-C(16)-C(2)	119.14(15)
C(11)-C(16)-C(2)	121.08(15)
C(18)-C(17)-C(22)	119.83(14)
C(18)-C(17)-C(3)	120.58(14)
C(22)-C(17)-C(3)	119.52(15)
C(17)-C(18)-C(19)	120.53(15)
C(17)-C(18)-H(18)	119.7
C(19)-C(18)-H(18)	119.7
C(20)-C(19)-C(18)	119.84(16)
C(20)-C(19)-H(19)	120.1
C(18)-C(19)-H(19)	120.1
C(19)-C(20)-C(21)	120.00(15)
C(19)-C(20)-H(20)	120.0
C(21)-C(20)-H(20)	120.0
C(20)-C(21)-C(22)	121.10(16)
C(20)-C(21)-H(21)	119.4
C(22)-C(21)-H(21)	119.4
C(21)-C(22)-C(17)	118.62(16)
C(21)-C(22)-C(23)	121.51(15)
C(17)-C(22)-C(23)	119.45(15)
C(22)-C(23)-C(24)	111.72(16)
C(22)-C(23)-H(23A)	109.3
C(24)-C(23)-H(23A)	109.3
C(22)-C(23)-H(23B)	109.3
C(24)-C(23)-H(23B)	109.3
H(23A)-C(23)-H(23B)	107.9
C(25)-C(24)-C(23)	115.71(15)
C(25)-C(24)-H(24A)	108.4
C(23)-C(24)-H(24A)	108.4
C(25)-C(24)-H(24B)	108.4
C(23)-C(24)-H(24B)	108.4
H(24A)-C(24)-H(24B)	107.4
C(24)-C(25)-C(26)	116.37(15)
C(24)-C(25)-H(25A)	108.2
C(26)-C(25)-H(25A)	108.2
C(24)-C(25)-H(25B)	108.2
C(26)-C(25)-H(25B)	108.2
H(25A)-C(25)-H(25B)	107.3
F(4)-C(26)-F(3)	105.95(13)
F(4)-C(26)-C(25)	108.11(14)
F(3)-C(26)-C(25)	104.26(14)
F(4)-C(26)-C(4)	110.10(14)
F(3)-C(26)-C(4)	107.63(13)
C(25)-C(26)-C(4)	119.83(15)
F(5)-C(27)-F(6)	101.29(13)
F(5)-C(27)-C(28)	110.29(13)
F(6)-C(27)-C(28)	108.35(13)
F(5)-C(27)-C(5)	109.57(13)
F(6)-C(27)-C(5)	107.21(12)
C(28)-C(27)-C(5)	118.65(14)
C(29)-C(28)-C(27)	117.64(14)
C(29)-C(28)-H(28A)	107.9
C(27)-C(28)-H(28A)	107.9
C(29)-C(28)-H(28B)	107.9
C(27)-C(28)-H(28B)	107.9
H(28A)-C(28)-H(28B)	107.2
C(28)-C(29)-C(30)	116.87(15)
C(28)-C(29)-H(29A)	108.1
C(30)-C(29)-H(29A)	108.1
C(28)-C(29)-H(29B)	108.1
C(30)-C(29)-H(29B)	108.1
H(29A)-C(29)-H(29B)	107.3
C(31)-C(30)-C(29)	114.75(15)
C(31)-C(30)-H(30A)	108.6
C(29)-C(30)-H(30A)	108.6
C(31)-C(30)-H(30B)	108.6
C(29)-C(30)-H(30B)	108.6
H(30A)-C(30)-H(30B)	107.6
C(36)-C(31)-C(32)	118.87(18)
C(36)-C(31)-C(30)	121.07(15)
C(32)-C(31)-C(30)	120.06(18)
C(33)-C(32)-C(31)	120.9(2)
C(33)-C(32)-H(32)	119.5
C(31)-C(32)-H(32)	119.5
C(32)-C(33)-C(34)	119.73(18)
C(32)-C(33)-H(33)	120.1
C(34)-C(33)-H(33)	120.1
C(33)-C(34)-C(35)	119.78(19)
C(33)-C(34)-H(34)	120.1
C(35)-C(34)-H(34)	120.1
C(36)-C(35)-C(34)	120.3(2)
C(36)-C(35)-H(35)	119.9
C(34)-C(35)-H(35)	119.9
C(35)-C(36)-C(31)	120.35(16)
C(35)-C(36)-C(6)	121.61(17)
C(31)-C(36)-C(6)	118.03(15)

Symmetry transformations used to generate equivalent atoms:

Table S4. Anisotropic displacement parameters ($Å^2 x\ 10^3$)for major trimer (**9**). The anisotropic displacement factor exponent takes the form: $-2p^2[\ h^2a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	17(1)	14(1)	22(1)	0(1)	7(1)	0(1)
C(2)	18(1)	14(1)	19(1)	1(1)	5(1)	-3(1)
C(3)	19(1)	14(1)	19(1)	0(1)	7(1)	-3(1)
C(4)	20(1)	14(1)	22(1)	-2(1)	6(1)	0(1)
C(5)	17(1)	16(1)	20(1)	0(1)	3(1)	-2(1)
C(6)	20(1)	15(1)	19(1)	0(1)	7(1)	-2(1)
C(7)	21(1)	19(1)	21(1)	-4(1)	7(1)	1(1)
C(8)	19(1)	21(1)	25(1)	-3(1)	4(1)	6(1)
C(9)	27(1)	17(1)	29(1)	2(1)	1(1)	2(1)
C(10)	21(1)	21(1)	31(1)	8(1)	6(1)	1(1)
C(11)	21(1)	24(1)	23(1)	1(1)	9(1)	3(1)
C(12)	30(1)	34(1)	20(1)	7(1)	7(1)	10(1)
C(13)	30(1)	49(1)	20(1)	-1(1)	-2(1)	7(1)
C(14)	25(1)	37(1)	29(1)	-10(1)	-1(1)	-2(1)
C(15)	22(1)	24(1)	25(1)	-3(1)	6(1)	-1(1)
C(16)	16(1)	22(1)	19(1)	-2(1)	7(1)	3(1)
C(17)	19(1)	15(1)	18(1)	1(1)	5(1)	4(1)
C(18)	19(1)	19(1)	22(1)	1(1)	7(1)	0(1)
C(19)	23(1)	23(1)	23(1)	-6(1)	2(1)	1(1)
C(20)	27(1)	33(1)	16(1)	0(1)	5(1)	9(1)
C(21)	27(1)	26(1)	23(1)	8(1)	13(1)	6(1)
C(22)	19(1)	17(1)	27(1)	3(1)	8(1)	2(1)
C(23)	29(1)	20(1)	45(1)	-5(1)	20(1)	-7(1)
C(24)	27(1)	41(1)	34(1)	-10(1)	11(1)	-16(1)
C(25)	16(1)	38(1)	29(1)	-9(1)	5(1)	4(1)
C(26)	23(1)	21(1)	22(1)	-5(1)	0(1)	5(1)
C(27)	22(1)	19(1)	22(1)	0(1)	4(1)	2(1)
C(28)	26(1)	26(1)	21(1)	-1(1)	0(1)	3(1)
C(29)	29(1)	31(1)	24(1)	-4(1)	6(1)	-8(1)
C(30)	37(1)	22(1)	23(1)	3(1)	-3(1)	-10(1)
C(31)	38(1)	17(1)	19(1)	0(1)	1(1)	6(1)
C(32)	54(1)	27(1)	23(1)	-4(1)	-4(1)	12(1)
C(33)	65(1)	43(1)	19(1)	-2(1)	8(1)	26(1)
C(34)	48(1)	45(1)	25(1)	9(1)	18(1)	23(1)
C(35)	35(1)	28(1)	28(1)	6(1)	13(1)	10(1)
C(36)	30(1)	18(1)	17(1)	4(1)	6(1)	9(1)
F(1)	30(1)	20(1)	23(1)	-5(1)	0(1)	8(1)
F(2)	24(1)	34(1)	30(1)	2(1)	14(1)	5(1)
F(3)	20(1)	36(1)	24(1)	-7(1)	0(1)	6(1)
F(4)	35(1)	20(1)	34(1)	-3(1)	4(1)	8(1)
F(5)	34(1)	19(1)	30(1)	3(1)	3(1)	7(1)
F(6)	28(1)	25(1)	24(1)	7(1)	7(1)	-1(1)

Table S5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for major trimer (**9**).

	x	y	z	U(eq)
H(8A)	3493	5808	-285	26
H(8B)	3605	4898	-1162	26
H(9A)	5797	6527	-611	30
H(9B)	4431	6538	-1725	30
H(10A)	6430	5965	-2426	29
H(10B)	7069	5240	-1332	29
H(12)	4197	5397	-3897	33
H(13)	2445	4121	-4684	41
H(14)	2394	2550	-3717	38
H(15)	4136	2258	-1973	28
H(18)	6348	1124	-2068	24
H(19)	6307	665	-3961	28
H(20)	7653	1646	-5015	31
H(21)	9079	3061	-4180	29
H(23A)	8913	4316	-1662	35
H(23B)	9849	4273	-2641	35
H(24A)	11821	3576	-1466	40
H(24B)	11001	3817	-452	40
H(25A)	11929	2022	-730	33
H(25B)	10333	1920	-1626	33
H(28A)	10274	2954	3870	30
H(28B)	11440	2397	3252	30
H(29A)	11266	3790	1966	34
H(29B)	11851	4168	3257	34
H(30A)	10184	5419	2810	35
H(30B)	9324	4851	1660	35
H(32)	9137	5405	4464	44
H(33)	7144	5034	5326	51
H(34)	5193	3917	4418	45
H(35)	5199			

Minor Trimer (10)

A colorless rod 0.20 x 0.20 x 0.20 mm in size was mounted on a Cryoloop with Paratone oil. Data were collected in a nitrogen gas stream at 100(2) K using phi and omega scans. Crystal-to-detector distance was 60 mm and exposure time was 5 seconds per frame using a scan width of 1.0°. Data collection was 97.5% complete to 67.00° in θ. A total of 46641 reflections were collected covering the indices, -13<=*h*<=13, -13<=*k*<=13, -26<=*l*<=29. 9620 reflections were found to be symmetry independent, with an R_{int} of 0.0356. Indexing and unit cell refinement indicated a primitive, triclinic lattice. The space group was found to be P-1 (No. 2). The data were integrated using the Bruker SAINT software program and scaled using the SADABS software program. Solution by direct methods (SIR-2008) produced a complete heavy-atom phasing model consistent with the proposed structure. All non-hydrogen atoms were refined anisotropically by full-matrix least-squares (SHELXL-97). All hydrogen atoms were placed using a riding model. Their positions were constrained relative to their parent atom using the appropriate HFIX command in SHELXL-97.

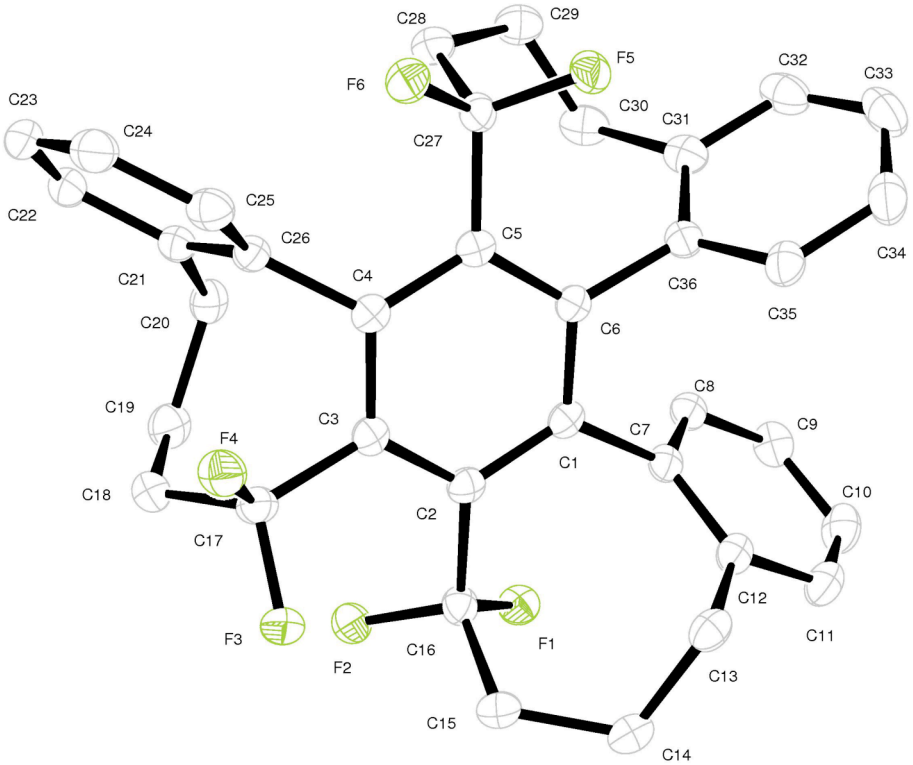


Figure S13. X-ray structure of trimer **10** with carbon and fluorine atoms labeled. Shown are ORTEPs with 50% probability.

Table S6. Crystal data and structure refinement for minor trimer (**10**).

Empirical formula	C36 H30 F6	
Formula weight	576.60	
Temperature	100(2) K	
Wavelength	1.54178 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 10.9610(4) Å	α= 90.207(2)°.
	b = 11.2443(4) Å	β= 100.731(2)°.
	c = 24.9515(9) Å	γ = 115.162(2)°.
Volume	2722.88(17) Å^3	
Z	4	
Density (calculated)	1.407 Mg/m^3	
Absorption coefficient	0.917 mm^{-1}	
F(000)	1200	
Crystal size	0.20 x 0.20 x 0.20 mm^3	
Crystal color/habit	colorless rod	
Theta range for data collection	3.62 to 68.10°.	
Index ranges	-13<=h<=13, -13<=k<=13, -26<=l<=29	
Reflections collected	46641	
Independent reflections	9620 [R(int) = 0.0356]	
Completeness to theta = 67.00°	97.5 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.8378 and 0.8378	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	9620 / 0 / 803	
Goodness-of-fit on F^2	1.028	
Final R indices [I>2sigma(I)]	R1 = 0.0428, wR2 = 0.1070	
R indices (all data)	R1 = 0.0464, wR2 = 0.1100	
Largest diff. peak and hole	0.931 and -0.549 e.Å^{-3}	

Table S7. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for minor trimer (**10**). U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	8284(2)	8473(2)	5894(1)	18(1)
C(2)	7833(2)	9349(2)	6101(1)	19(1)
C(3)	8217(2)	9753(2)	6668(1)	19(1)
C(4)	9188(2)	9429(2)	7013(1)	18(1)
C(5)	9618(2)	8537(2)	6803(1)	18(1)
C(6)	9131(2)	8033(2)	6250(1)	17(1)
C(7)	7932(2)	7981(2)	5299(1)	20(1)
C(8)	9002(2)	8321(2)	5016(1)	22(1)
C(9)	8753(2)	7881(2)	4471(1)	27(1)
C(10)	7413(2)	7065(2)	4201(1)	31(1)
C(11)	6351(2)	6724(2)	4478(1)	28(1)
C(12)	6580(2)	7176(2)	5026(1)	21(1)
C(13)	5349(2)	6860(2)	5285(1)	24(1)
C(14)	4770(2)	7891(2)	5181(1)	26(1)
C(15)	5416(2)	9095(2)	5603(1)	24(1)
C(16)	6977(2)	9875(2)	5697(1)	21(1)
C(17)	7661(2)	10576(2)	6954(1)	23(1)
C(18)	8565(2)	12064(2)	7083(1)	28(1)
C(19)	9897(2)	12705(2)	6862(1)	29(1)
C(20)	10983(2)	12157(2)	7041(1)	24(1)
C(21)	10823(2)	11525(2)	7569(1)	22(1)
C(22)	11522(2)	12284(2)	8069(1)	27(1)
C(23)	11273(2)	11764(2)	8558(1)	31(1)
C(24)	10296(2)	10473(2)	8555(1)	30(1)
C(25)	9608(2)	9704(2)	8065(1)	25(1)
C(26)	9882(2)	10209(2)	7568(1)	20(1)
C(27)	10696(2)	8196(2)	7168(1)	21(1)
C(28)	12178(2)	9163(2)	7180(1)	26(1)
C(29)	12824(2)	8943(2)	6718(1)	29(1)
C(30)	12006(2)	8772(2)	6129(1)	28(1)
C(31)	10877(2)	7386(2)	5969(1)	24(1)
C(32)	11200(2)	6396(2)	5794(1)	32(1)
C(33)	10225(2)	5102(2)	5677(1)	37(1)
C(34)	8880(2)	4768(2)	5723(1)	34(1)
C(35)	8532(2)	5738(2)	5890(1)	25(1)
C(36)	9526(2)	7038(2)	6023(1)	20(1)
C(37)	5823(2)	5746(2)	8269(1)	21(1)
C(38)	5041(2)	6466(2)	8270(1)	22(1)
C(39)	5225(2)	7243(2)	8752(1)	22(1)
C(40)	6179(2)	7289(2)	9223(1)	26(1)
C(41)	7101(2)	6727(2)	9196(1)	32(1)
C(42)	6907(2)	5940(2)	8719(1)	25(1)
C(43)	5592(2)	4735(2)	7817(1)	21(1)
C(44)	5130(2)	3421(2)	7931(1)	23(1)
C(45)	5030(2)	2458(2)	7554(1)	27(1)
C(46)	5408(2)	2807(2)	7058(1)	31(1)
C(47)	5856(2)	4107(2)	6939(1)	29(1)
C(48)	5947(2)	5085(2)	7311(1)	24(1)
C(49)	6275(2)	6457(2)	7151(1)	28(1)
C(50)	4928(2)	6549(2)	6899(1)	29(1)
C(51)	3765(2)	5835(2)	7200(1)	26(1)

C(52)	3947(2)	6419(2)	7772(1)	24(1)
C(53)	4547(2)	8160(2)	8763(1)	23(1)
C(54)	3157(2)	7718(2)	8771(1)	28(1)
C(55)	2588(2)	8608(2)	8774(1)	37(1)
C(56)	3397(3)	9939(2)	8761(1)	43(1)
C(57)	4781(2)	10381(2)	8760(1)	38(1)
C(58)	5383(2)	9512(2)	8768(1)	27(1)
C(59)	6913(2)	10026(2)	8813(1)	34(1)
C(60)	7710(2)	10222(2)	9419(1)	38(1)
C(61)	6890(2)	9486(2)	9836(1)	30(1)
C(62)	6155(2)	7981(2)	9751(1)	28(1)
C(63)	8261(3)	6883(4)	9700(1)	29(1)
C(64)	7955(3)	5888(4)	10123(1)	33(1)
C(65)	8052(3)	4605(3)	9999(1)	35(1)
C(66)	7151(2)	3885(2)	9447(1)	42(1)
C(63A)	8649(10)	7382(9)	9514(4)	21(2)
C(64A)	8832(9)	6469(7)	9941(3)	26(2)
C(65A)	7665(10)	5170(10)	9967(4)	24(2)
C(66A)	7151(2)	3885(2)	9447(1)	42(1)
C(67)	7917(2)	4327(2)	8991(1)	26(1)
C(68)	8804(2)	3779(2)	8902(1)	34(1)
C(69)	9592(2)	4188(2)	8508(1)	35(1)
C(70)	9517(2)	5163(2)	8192(1)	32(1)
C(71)	8644(2)	5706(2)	8268(1)	26(1)
C(72)	7842(2)	5304(2)	8662(1)	22(1)
F(1)	7371(1)	9986(1)	5201(1)	24(1)
F(2)	7352(1)	11173(1)	5870(1)	26(1)
F(3)	6372(1)	10362(1)	6668(1)	26(1)
F(4)	7359(1)	10025(1)	7431(1)	27(1)
F(5)	10500(1)	6935(1)	7031(1)	24(1)
F(6)	10488(1)	8141(1)	7693(1)	26(1)
F(7)	4124(1)	7701(1)	7708(1)	33(1)
F(8)	2687(1)	5785(1)	7919(1)	31(1)
F(9)	6589(1)	7452(1)	10202(1)	45(1)
F(10)	4793(1)	7569(1)	9777(1)	33(1)
F(11)	8744(2)	8146(2)	9949(1)	34(1)
F(12)	9387(1)	6958(2)	9503(1)	31(1)
F(11A)	9045(6)	8634(6)	9749(3)	26(1)
F(12A)	9617(4)	7650(5)	9195(2)	28(2)

Table S8. Bond lengths [Å] and angles [°] for minor trimer (**10**).

C(1)-C(6)	1.407(2)	C(27)-F(6)	1.3686(19)
C(1)-C(2)	1.410(2)	C(27)-F(5)	1.3717(19)
C(1)-C(7)	1.502(2)	C(27)-C(28)	1.517(2)
C(2)-C(3)	1.413(2)	C(28)-C(29)	1.530(3)
C(2)-C(16)	1.539(2)	C(28)-H(28A)	0.9900
C(3)-C(4)	1.413(2)	C(28)-H(28B)	0.9900
C(3)-C(17)	1.541(2)	C(29)-C(30)	1.538(3)
C(4)-C(5)	1.412(2)	C(29)-H(29A)	0.9900
C(4)-C(26)	1.516(2)	C(29)-H(29B)	0.9900
C(5)-C(6)	1.406(2)	C(30)-C(31)	1.513(3)
C(5)-C(27)	1.536(2)	C(30)-H(30A)	0.9900
C(6)-C(36)	1.502(2)	C(30)-H(30B)	0.9900
C(7)-C(8)	1.396(2)	C(31)-C(32)	1.394(3)
C(7)-C(12)	1.400(2)	C(31)-C(36)	1.395(2)
C(8)-C(9)	1.384(2)	C(32)-C(33)	1.380(3)
C(8)-H(8)	0.9500	C(32)-H(32)	0.9500
C(9)-C(10)	1.391(3)	C(33)-C(34)	1.385(3)
C(9)-H(9)	0.9500	C(33)-H(33)	0.9500
C(10)-C(11)	1.378(3)	C(34)-C(35)	1.386(3)
C(10)-H(10)	0.9500	C(34)-H(34)	0.9500
C(11)-C(12)	1.397(2)	C(35)-C(36)	1.393(2)
C(11)-H(11)	0.9500	C(35)-H(35)	0.9500
C(12)-C(13)	1.510(2)	C(37)-C(38)	1.408(2)
C(13)-C(14)	1.542(2)	C(37)-C(42)	1.415(2)
C(13)-H(13A)	0.9900	C(37)-C(43)	1.506(2)
C(13)-H(13B)	0.9900	C(38)-C(39)	1.416(2)
C(14)-C(15)	1.529(2)	C(38)-C(52)	1.541(2)
C(14)-H(14A)	0.9900	C(39)-C(40)	1.405(2)
C(14)-H(14B)	0.9900	C(39)-C(53)	1.508(2)
C(15)-C(16)	1.524(2)	C(40)-C(41)	1.412(2)
C(15)-H(15A)	0.9900	C(40)-C(62)	1.537(2)
C(15)-H(15B)	0.9900	C(41)-C(42)	1.407(3)
C(16)-F(1)	1.375(2)	C(41)-C(63)	1.563(3)
C(16)-F(2)	1.3795(19)	C(41)-C(63A)	1.575(9)
C(17)-F(4)	1.374(2)	C(42)-C(72)	1.504(2)
C(17)-F(3)	1.3780(19)	C(43)-C(44)	1.394(2)
C(17)-C(18)	1.534(2)	C(43)-C(48)	1.399(3)
C(18)-C(19)	1.537(3)	C(44)-C(45)	1.388(2)
C(18)-H(18A)	0.9900	C(44)-H(44)	0.9500
C(18)-H(18B)	0.9900	C(45)-C(46)	1.382(3)
C(19)-C(20)	1.557(2)	C(45)-H(45)	0.9500
C(19)-H(19A)	0.9900	C(46)-C(47)	1.383(3)
C(19)-H(19B)	0.9900	C(46)-H(46)	0.9500
C(20)-C(21)	1.502(3)	C(47)-C(48)	1.394(3)
C(20)-H(20A)	0.9900	C(47)-H(47)	0.9500
C(20)-H(20B)	0.9900	C(48)-C(49)	1.502(3)
C(21)-C(22)	1.398(2)	C(49)-C(50)	1.539(3)
C(21)-C(26)	1.398(2)	C(49)-H(49A)	0.9900
C(22)-C(23)	1.377(3)	C(49)-H(49B)	0.9900
C(22)-H(22)	0.9500	C(50)-C(51)	1.526(3)
C(23)-C(24)	1.389(3)	C(50)-H(50A)	0.9900
C(23)-H(23)	0.9500	C(50)-H(50B)	0.9900
C(24)-C(25)	1.380(3)	C(51)-C(52)	1.510(2)
C(24)-H(24)	0.9500	C(51)-H(51A)	0.9900
C(25)-C(26)	1.396(2)	C(51)-H(51B)	0.9900
C(25)-H(25)	0.9500	C(52)-F(8)	1.380(2)

C(52)-F(7)	1.385(2)	C(64)-H(64A)	0.9900
C(53)-C(54)	1.391(2)	C(64)-H(64B)	0.9900
C(53)-C(58)	1.401(3)	C(65)-C(66)	1.527(3)
C(54)-C(55)	1.388(3)	C(65)-H(65A)	0.9900
C(54)-H(54)	0.9500	C(65)-H(65B)	0.9900
C(55)-C(56)	1.382(3)	C(66)-C(67)	1.500(3)
C(55)-H(55)	0.9500	C(66)-H(66A)	0.9900
C(56)-C(57)	1.382(3)	C(66)-H(66B)	0.9900
C(56)-H(56)	0.9500	C(63A)-F(12A)	1.375(12)
C(57)-C(58)	1.391(3)	C(63A)-F(11A)	1.376(10)
C(57)-H(57)	0.9500	C(63A)-C(64A)	1.529(12)
C(58)-C(59)	1.504(3)	C(64A)-C(65A)	1.489(12)
C(59)-C(60)	1.562(3)	C(64A)-H(64C)	0.9900
C(59)-H(59A)	0.9900	C(64A)-H(64D)	0.9900
C(59)-H(59B)	0.9900	C(65A)-H(65C)	0.9900
C(60)-C(61)	1.516(3)	C(65A)-H(65D)	0.9900
C(60)-H(60A)	0.9900	C(67)-C(72)	1.395(3)
C(60)-H(60B)	0.9900	C(67)-C(68)	1.401(3)
C(61)-C(62)	1.527(3)	C(68)-C(69)	1.381(3)
C(61)-H(61A)	0.9900	C(68)-H(68)	0.9500
C(61)-H(61B)	0.9900	C(69)-C(70)	1.374(3)
C(62)-F(10)	1.376(2)	C(69)-H(69)	0.9500
C(62)-F(9)	1.377(2)	C(70)-C(71)	1.374(3)
C(63)-F(12)	1.380(3)	C(70)-H(70)	0.9500
C(63)-F(11)	1.385(4)	C(71)-C(72)	1.389(3)
C(63)-C(64)	1.516(5)	C(71)-H(71)	0.9500
C(64)-C(65)	1.526(4)		

C(6)-C(1)-C(2)	120.01(15)	C(10)-C(11)-C(12)	121.84(17)
C(6)-C(1)-C(7)	116.55(14)	C(10)-C(11)-H(11)	119.1
C(2)-C(1)-C(7)	123.43(14)	C(12)-C(11)-H(11)	119.1
C(1)-C(2)-C(3)	119.21(15)	C(11)-C(12)-C(7)	118.45(17)
C(1)-C(2)-C(16)	118.83(14)	C(11)-C(12)-C(13)	118.52(16)
C(3)-C(2)-C(16)	121.93(14)	C(7)-C(12)-C(13)	122.86(15)
C(2)-C(3)-C(4)	120.44(15)	C(12)-C(13)-C(14)	111.54(14)
C(2)-C(3)-C(17)	124.27(14)	C(12)-C(13)-H(13A)	109.3
C(4)-C(3)-C(17)	115.27(14)	C(14)-C(13)-H(13A)	109.3
C(5)-C(4)-C(3)	119.28(15)	C(12)-C(13)-H(13B)	109.3
C(5)-C(4)-C(26)	122.12(14)	C(14)-C(13)-H(13B)	109.3
C(3)-C(4)-C(26)	117.99(14)	H(13A)-C(13)-H(13B)	108.0
C(6)-C(5)-C(4)	119.84(15)	C(15)-C(14)-C(13)	115.61(14)
C(6)-C(5)-C(27)	119.99(14)	C(15)-C(14)-H(14A)	108.4
C(4)-C(5)-C(27)	120.02(14)	C(13)-C(14)-H(14A)	108.4
C(5)-C(6)-C(1)	120.47(15)	C(15)-C(14)-H(14B)	108.4
C(5)-C(6)-C(36)	120.82(14)	C(13)-C(14)-H(14B)	108.4
C(1)-C(6)-C(36)	118.70(14)	H(14A)-C(14)-H(14B)	107.4
C(8)-C(7)-C(12)	119.38(15)	C(16)-C(15)-C(14)	116.15(14)
C(8)-C(7)-C(1)	118.37(15)	C(16)-C(15)-H(15A)	108.2
C(12)-C(7)-C(1)	122.24(15)	C(14)-C(15)-H(15A)	108.2
C(9)-C(8)-C(7)	121.36(16)	C(16)-C(15)-H(15B)	108.2
C(9)-C(8)-H(8)	119.3	C(14)-C(15)-H(15B)	108.2
C(7)-C(8)-H(8)	119.3	H(15A)-C(15)-H(15B)	107.4
C(8)-C(9)-C(10)	119.33(17)	F(1)-C(16)-F(2)	102.03(13)
C(8)-C(9)-H(9)	120.3	F(1)-C(16)-C(15)	109.05(13)
C(10)-C(9)-H(9)	120.3	F(2)-C(16)-C(15)	109.80(13)
C(11)-C(10)-C(9)	119.63(17)	F(1)-C(16)-C(2)	109.96(13)
C(11)-C(10)-H(10)	120.2	F(2)-C(16)-C(2)	108.68(13)
C(9)-C(10)-H(10)	120.2	C(15)-C(16)-C(2)	116.36(14)

F(4)-C(17)-F(3)	101.32(13)	C(30)-C(29)-H(29B)	108.0
F(4)-C(17)-C(18)	109.14(14)	H(29A)-C(29)-H(29B)	107.3
F(3)-C(17)-C(18)	109.48(14)	C(31)-C(30)-C(29)	112.36(15)
F(4)-C(17)-C(3)	107.22(13)	C(31)-C(30)-H(30A)	109.1
F(3)-C(17)-C(3)	110.10(13)	C(29)-C(30)-H(30A)	109.1
C(18)-C(17)-C(3)	118.21(14)	C(31)-C(30)-H(30B)	109.1
C(17)-C(18)-C(19)	119.17(15)	C(29)-C(30)-H(30B)	109.1
C(17)-C(18)-H(18A)	107.5	H(30A)-C(30)-H(30B)	107.9
C(19)-C(18)-H(18A)	107.5	C(32)-C(31)-C(36)	118.18(17)
C(17)-C(18)-H(18B)	107.5	C(32)-C(31)-C(30)	119.51(17)
C(19)-C(18)-H(18B)	107.5	C(36)-C(31)-C(30)	122.19(15)
H(18A)-C(18)-H(18B)	107.0	C(33)-C(32)-C(31)	121.73(19)
C(18)-C(19)-C(20)	116.37(15)	C(33)-C(32)-H(32)	119.1
C(18)-C(19)-H(19A)	108.2	C(31)-C(32)-H(32)	119.1
C(20)-C(19)-H(19A)	108.2	C(32)-C(33)-C(34)	119.75(17)
C(18)-C(19)-H(19B)	108.2	C(32)-C(33)-H(33)	120.1
C(20)-C(19)-H(19B)	108.2	C(34)-C(33)-H(33)	120.1
H(19A)-C(19)-H(19B)	107.3	C(33)-C(34)-C(35)	119.50(18)
C(21)-C(20)-C(19)	111.55(15)	C(33)-C(34)-H(34)	120.2
C(21)-C(20)-H(20A)	109.3	C(35)-C(34)-H(34)	120.2
C(19)-C(20)-H(20A)	109.3	C(34)-C(35)-C(36)	120.67(18)
C(21)-C(20)-H(20B)	109.3	C(34)-C(35)-H(35)	119.7
C(19)-C(20)-H(20B)	109.3	C(36)-C(35)-H(35)	119.7
H(20A)-C(20)-H(20B)	108.0	C(35)-C(36)-C(31)	120.12(16)
C(22)-C(21)-C(26)	119.17(17)	C(35)-C(36)-C(6)	118.86(15)
C(22)-C(21)-C(20)	120.01(16)	C(31)-C(36)-C(6)	120.95(15)
C(26)-C(21)-C(20)	120.56(15)	C(38)-C(37)-C(42)	119.88(15)
C(23)-C(22)-C(21)	121.03(18)	C(38)-C(37)-C(43)	124.82(15)
C(23)-C(22)-H(22)	119.5	C(42)-C(37)-C(43)	115.30(15)
C(21)-C(22)-H(22)	119.5	C(37)-C(38)-C(39)	119.31(15)
C(22)-C(23)-C(24)	119.64(17)	C(37)-C(38)-C(52)	122.97(15)
C(22)-C(23)-H(23)	120.2	C(39)-C(38)-C(52)	117.70(15)
C(24)-C(23)-H(23)	120.2	C(40)-C(39)-C(38)	120.00(15)
C(25)-C(24)-C(23)	120.10(18)	C(40)-C(39)-C(53)	117.64(15)
C(25)-C(24)-H(24)	119.9	C(38)-C(39)-C(53)	121.97(15)
C(23)-C(24)-H(24)	119.9	C(39)-C(40)-C(41)	120.19(16)
C(24)-C(25)-C(26)	120.70(18)	C(39)-C(40)-C(62)	116.36(15)
C(24)-C(25)-H(25)	119.6	C(41)-C(40)-C(62)	123.44(16)
C(26)-C(25)-H(25)	119.6	C(42)-C(41)-C(40)	119.12(16)
C(25)-C(26)-C(21)	119.26(16)	C(42)-C(41)-C(63)	119.70(17)
C(25)-C(26)-C(4)	124.15(15)	C(40)-C(41)-C(63)	121.02(18)
C(21)-C(26)-C(4)	116.57(15)	C(42)-C(41)-C(63A)	112.2(4)
F(6)-C(27)-F(5)	102.60(12)	C(40)-C(41)-C(63A)	122.9(3)
F(6)-C(27)-C(28)	108.22(14)	C(63)-C(41)-C(63A)	28.2(3)
F(5)-C(27)-C(28)	110.18(14)	C(41)-C(42)-C(37)	120.26(16)
F(6)-C(27)-C(5)	109.22(13)	C(41)-C(42)-C(72)	122.07(16)
F(5)-C(27)-C(5)	111.48(13)	C(37)-C(42)-C(72)	117.66(15)
C(28)-C(27)-C(5)	114.42(14)	C(44)-C(43)-C(48)	119.35(16)
C(27)-C(28)-C(29)	116.55(15)	C(44)-C(43)-C(37)	117.95(15)
C(27)-C(28)-H(28A)	108.2	C(48)-C(43)-C(37)	122.41(15)
C(29)-C(28)-H(28A)	108.2	C(45)-C(44)-C(43)	121.10(17)
C(27)-C(28)-H(28B)	108.2	C(45)-C(44)-H(44)	119.4
C(29)-C(28)-H(28B)	108.2	C(43)-C(44)-H(44)	119.4
H(28A)-C(28)-H(28B)	107.3	C(46)-C(45)-C(44)	119.57(17)
C(28)-C(29)-C(30)	117.10(15)	C(46)-C(45)-H(45)	120.2
C(28)-C(29)-H(29A)	108.0	C(44)-C(45)-H(45)	120.2
C(30)-C(29)-H(29A)	108.0	C(45)-C(46)-C(47)	119.70(17)
C(28)-C(29)-H(29B)	108.0	C(45)-C(46)-H(46)	120.1

C(47)-C(46)-H(46)	120.1	C(61)-C(60)-H(60B)	108.0
C(46)-C(47)-C(48)	121.53(18)	C(59)-C(60)-H(60B)	108.0
C(46)-C(47)-H(47)	119.2	H(60A)-C(60)-H(60B)	107.2
C(48)-C(47)-H(47)	119.2	C(60)-C(61)-C(62)	118.41(16)
C(47)-C(48)-C(43)	118.72(17)	C(60)-C(61)-H(61A)	107.7
C(47)-C(48)-C(49)	120.85(17)	C(62)-C(61)-H(61A)	107.7
C(43)-C(48)-C(49)	120.12(16)	C(60)-C(61)-H(61B)	107.7
C(48)-C(49)-C(50)	109.43(14)	C(62)-C(61)-H(61B)	107.7
C(48)-C(49)-H(49A)	109.8	H(61A)-C(61)-H(61B)	107.1
C(50)-C(49)-H(49A)	109.8	F(10)-C(62)-F(9)	100.24(15)
C(48)-C(49)-H(49B)	109.8	F(10)-C(62)-C(61)	109.20(14)
C(50)-C(49)-H(49B)	109.8	F(9)-C(62)-C(61)	109.96(15)
H(49A)-C(49)-H(49B)	108.2	F(10)-C(62)-C(40)	107.09(14)
C(51)-C(50)-C(49)	113.42(15)	F(9)-C(62)-C(40)	110.19(15)
C(51)-C(50)-H(50A)	108.9	C(61)-C(62)-C(40)	118.55(16)
C(49)-C(50)-H(50A)	108.9	F(12)-C(63)-F(11)	101.8(3)
C(51)-C(50)-H(50B)	108.9	F(12)-C(63)-C(64)	109.6(2)
C(49)-C(50)-H(50B)	108.9	F(11)-C(63)-C(64)	110.1(3)
H(50A)-C(50)-H(50B)	107.7	F(12)-C(63)-C(41)	107.8(2)
C(52)-C(51)-C(50)	116.51(15)	F(11)-C(63)-C(41)	106.6(2)
C(52)-C(51)-H(51A)	108.2	C(64)-C(63)-C(41)	119.5(3)
C(50)-C(51)-H(51A)	108.2	C(63)-C(64)-C(65)	117.2(2)
C(52)-C(51)-H(51B)	108.2	C(63)-C(64)-H(64A)	108.0
C(50)-C(51)-H(51B)	108.2	C(65)-C(64)-H(64A)	108.0
H(51A)-C(51)-H(51B)	107.3	C(63)-C(64)-H(64B)	108.0
F(8)-C(52)-F(7)	105.36(13)	C(65)-C(64)-H(64B)	108.0
F(8)-C(52)-C(51)	106.19(14)	H(64A)-C(64)-H(64B)	107.2
F(7)-C(52)-C(51)	104.62(14)	C(64)-C(65)-C(66)	112.2(2)
F(8)-C(52)-C(38)	106.49(14)	C(64)-C(65)-H(65A)	109.2
F(7)-C(52)-C(38)	107.57(13)	C(66)-C(65)-H(65A)	109.2
C(51)-C(52)-C(38)	125.14(15)	C(64)-C(65)-H(65B)	109.2
C(54)-C(53)-C(58)	119.99(17)	C(66)-C(65)-H(65B)	109.2
C(54)-C(53)-C(39)	122.94(16)	H(65A)-C(65)-H(65B)	107.9
C(58)-C(53)-C(39)	117.07(15)	C(67)-C(66)-C(65)	111.41(19)
C(55)-C(54)-C(53)	120.29(19)	C(67)-C(66)-H(66A)	109.3
C(55)-C(54)-H(54)	119.9	C(65)-C(66)-H(66A)	109.3
C(53)-C(54)-H(54)	119.9	C(67)-C(66)-H(66B)	109.3
C(56)-C(55)-C(54)	120.02(19)	C(65)-C(66)-H(66B)	109.3
C(56)-C(55)-H(55)	120.0	H(66A)-C(66)-H(66B)	108.0
C(54)-C(55)-H(55)	120.0	F(12A)-C(63A)-F(11A)	99.7(7)
C(55)-C(56)-C(57)	119.71(18)	F(12A)-C(63A)-C(64A)	109.2(7)
C(55)-C(56)-H(56)	120.1	F(11A)-C(63A)-C(64A)	111.6(7)
C(57)-C(56)-H(56)	120.1	F(12A)-C(63A)-C(41)	115.7(7)
C(56)-C(57)-C(58)	121.4(2)	F(11A)-C(63A)-C(41)	112.6(6)
C(56)-C(57)-H(57)	119.3	C(64A)-C(63A)-C(41)	108.0(7)
C(58)-C(57)-H(57)	119.3	C(65A)-C(64A)-C(63A)	119.9(7)
C(57)-C(58)-C(53)	118.52(18)	C(65A)-C(64A)-H(64C)	107.3
C(57)-C(58)-C(59)	120.38(19)	C(63A)-C(64A)-H(64C)	107.3
C(53)-C(58)-C(59)	121.01(17)	C(65A)-C(64A)-H(64D)	107.3
C(58)-C(59)-C(60)	112.45(16)	C(63A)-C(64A)-H(64D)	107.3
C(58)-C(59)-H(59A)	109.1	H(64C)-C(64A)-H(64D)	106.9
C(60)-C(59)-H(59A)	109.1	C(64A)-C(65A)-H(65C)	107.4
C(58)-C(59)-H(59B)	109.1	C(64A)-C(65A)-H(65D)	107.4
C(60)-C(59)-H(59B)	109.1	H(65C)-C(65A)-H(65D)	106.9
H(59A)-C(59)-H(59B)	107.8	C(72)-C(67)-C(68)	117.60(17)
C(61)-C(60)-C(59)	117.24(17)	C(72)-C(67)-C(66)	123.43(18)
C(61)-C(60)-H(60A)	108.0	C(68)-C(67)-C(66)	118.90(18)
C(59)-C(60)-H(60A)	108.0	C(69)-C(68)-C(67)	121.92(18)

C(69)-C(68)-H(68)	119.0
C(67)-C(68)-H(68)	119.0
C(70)-C(69)-C(68)	119.63(18)
C(70)-C(69)-H(69)	120.2
C(68)-C(69)-H(69)	120.2
C(71)-C(70)-C(69)	119.49(19)
C(71)-C(70)-H(70)	120.3
C(69)-C(70)-H(70)	120.3
C(70)-C(71)-C(72)	121.62(18)
C(70)-C(71)-H(71)	119.2
C(72)-C(71)-H(71)	119.2
C(71)-C(72)-C(67)	119.73(16)
C(71)-C(72)-C(42)	118.53(16)
C(67)-C(72)-C(42)	121.73(16)

Symmetry transformations used to generate equivalent atoms:

Table S9. Anisotropic displacement parameters ($Å^2 x\ 10^3$)for minor trimer (**10**). The anisotropic displacement factor exponent takes the form: $-2p^2[\ h^2a^{*2}U^{11} + ... + 2\ h\ k\ a^*b^*U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	16(1)	15(1)	19(1)	3(1)	4(1)	2(1)
C(2)	16(1)	17(1)	21(1)	3(1)	3(1)	4(1)
C(3)	16(1)	15(1)	22(1)	2(1)	5(1)	4(1)
C(4)	16(1)	16(1)	19(1)	4(1)	6(1)	4(1)
C(5)	15(1)	17(1)	19(1)	5(1)	5(1)	4(1)
C(6)	16(1)	15(1)	19(1)	3(1)	5(1)	4(1)
C(7)	25(1)	15(1)	18(1)	4(1)	3(1)	9(1)
C(8)	25(1)	19(1)	22(1)	5(1)	4(1)	10(1)
C(9)	34(1)	26(1)	22(1)	6(1)	11(1)	13(1)
C(10)	43(1)	30(1)	18(1)	0(1)	5(1)	14(1)
C(11)	31(1)	23(1)	21(1)	1(1)	-1(1)	7(1)
C(12)	26(1)	15(1)	20(1)	4(1)	2(1)	7(1)
C(13)	22(1)	21(1)	22(1)	3(1)	1(1)	4(1)
C(14)	20(1)	28(1)	24(1)	2(1)	1(1)	7(1)
C(15)	22(1)	28(1)	23(1)	4(1)	1(1)	13(1)
C(16)	24(1)	20(1)	20(1)	2(1)	3(1)	10(1)
C(17)	20(1)	28(1)	22(1)	1(1)	3(1)	12(1)
C(18)	27(1)	26(1)	31(1)	-5(1)	0(1)	14(1)
C(19)	30(1)	19(1)	33(1)	2(1)	2(1)	8(1)
C(20)	22(1)	18(1)	27(1)	0(1)	6(1)	3(1)
C(21)	18(1)	23(1)	26(1)	-2(1)	3(1)	11(1)
C(22)	22(1)	28(1)	31(1)	-8(1)	-1(1)	13(1)
C(23)	31(1)	43(1)	23(1)	-11(1)	-5(1)	24(1)
C(24)	34(1)	48(1)	19(1)	3(1)	6(1)	28(1)
C(25)	23(1)	31(1)	24(1)	5(1)	7(1)	14(1)
C(26)	18(1)	24(1)	19(1)	0(1)	3(1)	11(1)
C(27)	26(1)	21(1)	17(1)	2(1)	4(1)	12(1)
C(28)	20(1)	27(1)	29(1)	0(1)	0(1)	11(1)
C(29)	20(1)	33(1)	33(1)	4(1)	6(1)	11(1)
C(30)	23(1)	35(1)	29(1)	10(1)	12(1)	14(1)
C(31)	29(1)	32(1)	17(1)	7(1)	8(1)	17(1)
C(32)	42(1)	46(1)	24(1)	7(1)	11(1)	31(1)
C(33)	62(1)	41(1)	25(1)	2(1)	8(1)	38(1)
C(34)	54(1)	23(1)	23(1)	0(1)	0(1)	18(1)
C(35)	32(1)	22(1)	19(1)	2(1)	3(1)	11(1)
C(36)	26(1)	22(1)	14(1)	4(1)	4(1)	12(1)
C(37)	18(1)	18(1)	24(1)	0(1)	5(1)	5(1)
C(38)	18(1)	22(1)	22(1)	1(1)	3(1)	6(1)
C(39)	18(1)	23(1)	23(1)	1(1)	4(1)	7(1)
C(40)	24(1)	28(1)	26(1)	-4(1)	-1(1)	13(1)
C(41)	25(1)	36(1)	34(1)	-12(1)	-6(1)	17(1)
C(42)	20(1)	22(1)	30(1)	-3(1)	2(1)	8(1)
C(43)	15(1)	20(1)	23(1)	-1(1)	1(1)	6(1)
C(44)	20(1)	21(1)	24(1)	2(1)	0(1)	7(1)
C(45)	24(1)	20(1)	34(1)	-3(1)	-2(1)	9(1)
C(46)	29(1)	33(1)	31(1)	-10(1)	-1(1)	15(1)
C(47)	24(1)	38(1)	22(1)	-2(1)	4(1)	11(1)
C(48)	16(1)	25(1)	24(1)	1(1)	2(1)	5(1)
C(49)	25(1)	25(1)	27(1)	4(1)	8(1)	5(1)
C(50)	31(1)	28(1)	25(1)	8(1)	7(1)	9(1)
C(51)	26(1)	25(1)	25(1)	3(1)	2(1)	10(1)
C(52)	22(1)	24(1)	26(1)	2(1)	5(1)	9(1)

C(53)	25(1)	29(1)	16(1)	0(1)	2(1)	14(1)
C(54)	24(1)	42(1)	18(1)	0(1)	2(1)	16(1)
C(55)	36(1)	67(2)	22(1)	-4(1)	0(1)	36(1)
C(56)	62(2)	57(1)	29(1)	-5(1)	-4(1)	48(1)
C(57)	58(1)	33(1)	25(1)	-1(1)	-2(1)	26(1)
C(58)	36(1)	29(1)	18(1)	0(1)	3(1)	15(1)
C(59)	35(1)	29(1)	28(1)	-1(1)	10(1)	4(1)
C(60)	28(1)	40(1)	33(1)	-6(1)	4(1)	5(1)
C(61)	33(1)	34(1)	23(1)	-6(1)	1(1)	17(1)
C(62)	31(1)	36(1)	21(1)	-1(1)	-1(1)	20(1)
C(63)	23(2)	34(2)	32(2)	-11(1)	-3(1)	17(2)
C(64)	38(2)	47(2)	20(1)	-2(1)	-4(1)	28(2)
C(65)	43(2)	43(2)	26(1)	2(1)	-1(1)	28(2)
C(66)	36(1)	52(1)	24(1)	4(1)	2(1)	7(1)
C(63A)	20(5)	14(4)	27(5)	-2(4)	-1(4)	6(4)
C(64A)	36(5)	18(4)	25(4)	3(3)	1(4)	14(4)
C(65A)	40(5)	16(4)	18(4)	3(4)	8(4)	14(4)
C(66A)	36(1)	52(1)	24(1)	4(1)	2(1)	7(1)
C(67)	21(1)	27(1)	24(1)	1(1)	-2(1)	6(1)
C(68)	31(1)	23(1)	40(1)	2(1)	-8(1)	12(1)
C(69)	27(1)	34(1)	45(1)	-10(1)	-1(1)	19(1)
C(70)	23(1)	40(1)	32(1)	-4(1)	5(1)	13(1)
C(71)	19(1)	24(1)	29(1)	2(1)	2(1)	6(1)
C(72)	18(1)	19(1)	24(1)	-4(1)	-1(1)	6(1)
F(1)	27(1)	24(1)	21(1)	7(1)	5(1)	11(1)
F(2)	28(1)	19(1)	30(1)	2(1)	2(1)	12(1)
F(3)	20(1)	32(1)	28(1)	-2(1)	1(1)	14(1)
F(4)	28(1)	36(1)	23(1)	3(1)	9(1)	16(1)
F(5)	32(1)	21(1)	22(1)	3(1)	4(1)	14(1)
F(6)	34(1)	29(1)	16(1)	4(1)	4(1)	16(1)
F(7)	45(1)	29(1)	27(1)	3(1)	2(1)	21(1)
F(8)	18(1)	42(1)	29(1)	4(1)	3(1)	10(1)
F(9)	64(1)	46(1)	26(1)	-3(1)	-13(1)	35(1)
F(10)	32(1)	44(1)	22(1)	4(1)	8(1)	14(1)
F(11)	27(1)	38(1)	34(1)	-17(1)	-8(1)	17(1)
F(12)	21(1)	37(1)	34(1)	-11(1)	-4(1)	15(1)
F(11A)	30(3)	15(3)	29(3)	-2(2)	-6(2)	10(2)
F(12A)	15(2)	30(3)	33(3)	-1(2)	6(2)	5(2)

Table S10. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for minor trimer (**10**).

	x	y	z	U(eq)
H(8)	9919	8867	5202	26
H(9)	9491	8132	4282	32
H(10)	7229	6745	3828	37
H(11)	5439	6165	4291	33
H(13A)	5622	6837	5684	29
H(13B)	4621	5976	5132	29
H(14A)	4897	8201	4816	31
H(14B)	3767	7450	5169	31
H(15A)	4994	9700	5485	29
H(15B)	5171	8797	5957	29
H(18A)	8821	12252	7486	33
H(18B)	7987	12520	6944	33
H(19A)	10338	13662	6979	35
H(19B)	9644	12603	6457	35
H(20A)	10878	11499	6751	29
H(20B)	11920	12887	7087	29
H(22)	12180	13172	8072	33
H(23)	11766	12286	8895	37
H(24)	10101	10118	8890	36
H(25)	8940	8822	8066	30
H(28A)	12749	9136	7533	31
H(28B)	12227	10062	7173	31
H(29A)	12985	8148	6780	34
H(29B)	13736	9702	6748	34
H(30A)	11592	9405	6093	33
H(30B)	12645	8981	5873	33
H(32)	12115	6619	5755	39
H(33)	10475	4443	5565	44
H(34)	8201	3880	5640	41
H(35)	7606	5515	5915	30
H(44)	4880	3181	8273	27
H(45)	4704	1564	7635	33
H(46)	5359	2156	6800	38
H(47)	6109	4338	6597	35
H(49A)	6880	6672	6882	33
H(49B)	6767	7101	7478	33
H(50A)	5111	7491	6901	35
H(50B)	4626	6169	6512	35
H(51A)	2903	5804	6977	32
H(51B)	3642	4913	7222	32
H(54)	2596	6802	8775	33
H(55)	1642	8303	8786	45
H(56)	3003	10547	8752	52
H(57)	5334	11298	8755	45
H(59A)	7259	10881	8651	40
H(59B)	7098	9399	8600	40
H(60A)	8466	9957	9424	45
H(60B)	8141	11176	9541	45
H(61A)	6187	9811	9852	36
H(61B)	7523	9734	10199	36
H(64A)	7012	5660	10178	39
H(64B)	8599	6325	10474	39

H(65A)	7766	4020	10292	42
H(65B)	9023	4803	9998	42
H(66A)	6841	2923	9463	51
H(66B)	6323	4056	9372	51
H(64C)	9615	6296	9886	31
H(64D)	9114	6967	10306	31
H(65C)	6852	5333	9981	28
H(65D)	7901	4838	10320	28
H(66C)	7312	3147	9607	51
H(66D)	6151	3554	9296	51
H(68)	8865	3106	9119	40
H(69)	10183	3798	8455	41
H(70)	10064	5459	7923	39
H(71)	8588	6372	8046	31

Dimer 13

A colorless prism 0.12 x 0.10 x 0.06 mm in size was mounted on a Cryoloop with Paratone oil. Data were collected in a nitrogen gas stream at 100(2) K using phi and omega scans. Crystal-to-detector distance was 60 mm and exposure time was 5 seconds per frame using a scan width of 1.0°. Data collection was 97.9% complete to 67.00° in θ. A total of 38046 reflections were collected covering the indices, -13<=*h*<=13, -13<=*k*<=17, -18<=*l*<=18. 7750 reflections were found to be symmetry independent, with an R_{int} of 0.0245. Indexing and unit cell refinement indicated a primitive, triclinic lattice. The space group was found to be P-1 (No. 2). The data were integrated using the Bruker SAINT software program and scaled using the SADABS software program. Solution by direct methods (SIR-2008) produced a complete heavy-atom phasing model consistent with the proposed structure. All non-hydrogen atoms were refined anisotropically by full-matrix least-squares (SHELXL-97). All hydrogen atoms, except for the water hydrogen atoms H8x and H8y, were placed using a riding model. Their positions were constrained relative to their parent atom using the appropriate HFIX command in SHELXL-97. The hydrogen atoms H8x and H8y were located from the Fourier difference map and their distances were restrained to the parent oxygen atom O8.

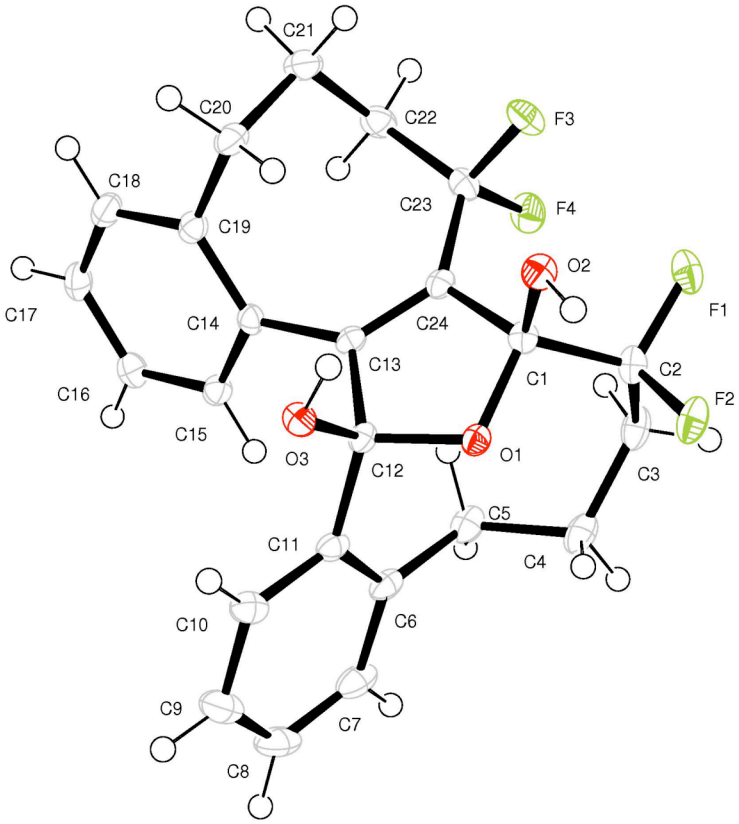


Figure S14. X-ray structure of dimer **13** with carbon, fluorine, and oxygen atoms labeled. ORTEPs are shown at 50% probability.

Table S11. Crystal data and structure refinement for dimer **13**.

Empirical formula	C51 H52 F8 O8	
Formula weight	944.93	
Temperature	100(2) K	
Wavelength	1.54178 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 10.9513(5) Å	α= 63.010(2)°.
	b = 14.9709(7) Å	β= 75.207(2)°.
	c = 15.4572(7) Å	γ = 80.666(2)°.
Volume	2180.20(17) $Å^3$	
Z	2	
Density (calculated)	1.439 Mg/m^3	
Absorption coefficient	1.012 mm^{-1}	
F(000)	988	
Crystal size	0.12 x 0.10 x 0.06 mm^3	
Crystal color/habit	colorless prism	
Theta range for data collection	3.28 to 68.36°.	
Index ranges	-13<=h<=13, -13<=k<=17, -18<=l<=18	
Reflections collected	38046	
Independent reflections	7750 [R(int) = 0.0245]	
Completeness to theta = 67.00°	97.9 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.9418 and 0.8882	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	7750 / 2 / 616	
Goodness-of-fit on F^2	1.025	
Final R indices [I>2sigma(I)]	R1 = 0.0336, wR2 = 0.0839	
R indices (all data)	R1 = 0.0358, wR2 = 0.0860	
Largest diff. peak and hole	0.347 and -0.428 $e.Å^{-3}$	

Table S12. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for dimer **13**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	3429(1)	8602(1)	-1061(1)	15(1)
C(2)	3980(1)	9348(1)	-837(1)	19(1)
C(3)	3214(1)	10276(1)	-813(1)	23(1)
C(4)	2680(1)	10975(1)	-1737(1)	24(1)
C(5)	1339(1)	10712(1)	-1679(1)	21(1)
C(6)	1118(1)	10683(1)	-2592(1)	19(1)
C(7)	539(1)	11530(1)	-3256(1)	25(1)
C(8)	250(1)	11550(1)	-4085(1)	29(1)
C(9)	521(1)	10708(1)	-4264(1)	28(1)
C(10)	1101(1)	9854(1)	-3618(1)	21(1)
C(11)	1409(1)	9834(1)	-2789(1)	16(1)
C(12)	2053(1)	8889(1)	-2105(1)	14(1)
C(13)	1359(1)	8350(1)	-1019(1)	14(1)
C(14)	40(1)	8052(1)	-772(1)	15(1)
C(15)	-934(1)	8796(1)	-1026(1)	17(1)
C(16)	-2184(1)	8542(1)	-758(1)	19(1)
C(17)	-2478(1)	7542(1)	-231(1)	21(1)
C(18)	-1518(1)	6801(1)	7(1)	21(1)
C(19)	-254(1)	7039(1)	-262(1)	17(1)
C(20)	754(1)	6223(1)	90(1)	20(1)
C(21)	860(1)	6037(1)	1135(1)	24(1)
C(22)	828(1)	6990(1)	1270(1)	21(1)
C(23)	1937(1)	7639(1)	688(1)	18(1)
C(24)	2159(1)	8174(1)	-429(1)	14(1)
C(25)	3978(1)	5930(1)	7351(1)	15(1)
C(26)	4773(1)	4934(1)	7801(1)	17(1)
C(27)	5046(1)	4190(1)	7358(1)	20(1)
C(28)	3897(1)	3804(1)	7258(1)	19(1)
C(29)	3503(1)	4448(1)	6247(1)	18(1)
C(30)	2105(1)	4733(1)	6291(1)	16(1)
C(31)	1372(1)	4145(1)	6154(1)	18(1)
C(32)	96(1)	4379(1)	6143(1)	19(1)
C(33)	-477(1)	5225(1)	6260(1)	19(1)
C(34)	234(1)	5821(1)	6398(1)	17(1)
C(35)	1513(1)	5584(1)	6426(1)	15(1)
C(36)	2235(1)	6247(1)	6613(1)	15(1)
C(37)	3373(1)	6772(1)	5813(1)	14(1)
C(38)	3215(1)	7417(1)	4777(1)	15(1)
C(39)	2910(1)	6981(1)	4231(1)	16(1)
C(40)	2760(1)	7563(1)	3262(1)	19(1)
C(41)	2884(1)	8590(1)	2835(1)	22(1)
C(42)	3171(1)	9030(1)	3374(1)	20(1)
C(43)	3352(1)	8455(1)	4342(1)	17(1)
C(44)	3813(1)	8938(1)	4859(1)	19(1)
C(45)	5272(1)	8890(1)	4642(1)	22(1)
C(46)	5902(1)	7866(1)	4735(1)	20(1)
C(47)	5655(1)	7016(1)	5762(1)	18(1)
C(48)	4355(1)	6612(1)	6240(1)	14(1)
C(49)	7370(1)	7795(1)	7563(1)	20(1)
C(50)	7342(2)	6677(1)	8115(1)	27(1)
C(51)	8624(2)	8219(1)	6987(1)	38(1)
O(1)	3190(1)	9169(1)	-2029(1)	15(1)

O(2)	4307(1)	7801(1)	-1021(1)	18(1)
O(3)	2324(1)	8197(1)	-2525(1)	16(1)
O(4)	2767(1)	5619(1)	7463(1)	16(1)
O(5)	3922(1)	6492(1)	7879(1)	17(1)
O(6)	1363(1)	6977(1)	6788(1)	18(1)
O(7)	6428(1)	8348(1)	7584(1)	23(1)
O(8)	3992(1)	5755(1)	9811(1)	30(1)
F(1)	4376(1)	8819(1)	43(1)	25(1)
F(2)	5072(1)	9647(1)	-1547(1)	24(1)
F(3)	3019(1)	7073(1)	955(1)	23(1)
F(4)	1824(1)	8370(1)	1029(1)	23(1)
F(5)	5908(1)	5167(1)	7851(1)	20(1)
F(6)	4153(1)	4458(1)	8778(1)	20(1)
F(7)	6477(1)	6208(1)	5733(1)	25(1)
F(8)	6049(1)	7275(1)	6387(1)	27(1)

Table S13. Bond lengths [Å] and angles [°] for dimer **13**.

C(1)-O(2)	1.3985(15)	C(17)-H(17)	0.9500
C(1)-O(1)	1.4156(16)	C(18)-C(19)	1.3954(19)
C(1)-C(24)	1.5252(17)	C(18)-H(18)	0.9500
C(1)-C(2)	1.5522(18)	C(19)-C(20)	1.5085(19)
C(2)-F(1)	1.3706(16)	C(20)-C(21)	1.541(2)
C(2)-F(2)	1.3752(16)	C(20)-H(20A)	0.9900
C(2)-C(3)	1.5126(19)	C(20)-H(20B)	0.9900
C(3)-C(4)	1.532(2)	C(21)-C(22)	1.527(2)
C(3)-H(3A)	0.9900	C(21)-H(21A)	0.9900
C(3)-H(3B)	0.9900	C(21)-H(21B)	0.9900
C(4)-C(5)	1.5516(19)	C(22)-C(23)	1.5033(19)
C(4)-H(4A)	0.9900	C(22)-H(22A)	0.9900
C(4)-H(4B)	0.9900	C(22)-H(22B)	0.9900
C(5)-C(6)	1.512(2)	C(23)-F(3)	1.3780(15)
C(5)-H(5A)	0.9900	C(23)-F(4)	1.3924(16)
C(5)-H(5B)	0.9900	C(23)-C(24)	1.5081(18)
C(6)-C(7)	1.398(2)	C(25)-O(5)	1.3991(15)
C(6)-C(11)	1.408(2)	C(25)-O(4)	1.4197(15)
C(7)-C(8)	1.382(2)	C(25)-C(48)	1.5317(17)
C(7)-H(7)	0.9500	C(25)-C(26)	1.5524(18)
C(8)-C(9)	1.380(2)	C(26)-F(5)	1.3743(15)
C(8)-H(8)	0.9500	C(26)-F(6)	1.3806(15)
C(9)-C(10)	1.392(2)	C(26)-C(27)	1.5097(18)
C(9)-H(9)	0.9500	C(27)-C(28)	1.5353(19)
C(10)-C(11)	1.392(2)	C(27)-H(27A)	0.9900
C(10)-H(10)	0.9500	C(27)-H(27B)	0.9900
C(11)-C(12)	1.5218(17)	C(28)-C(29)	1.5493(18)
C(12)-O(3)	1.4131(15)	C(28)-H(28A)	0.9900
C(12)-O(1)	1.4243(15)	C(28)-H(28B)	0.9900
C(12)-C(13)	1.5372(17)	C(29)-C(30)	1.5137(18)
C(13)-C(24)	1.3353(18)	C(29)-H(29A)	0.9900
C(13)-C(14)	1.4854(17)	C(29)-H(29B)	0.9900
C(14)-C(15)	1.4011(18)	C(30)-C(31)	1.3958(19)
C(14)-C(19)	1.4025(19)	C(30)-C(35)	1.4108(19)
C(15)-C(16)	1.3876(19)	C(31)-C(32)	1.387(2)
C(15)-H(15)	0.9500	C(31)-H(31)	0.9500
C(16)-C(17)	1.387(2)	C(32)-C(33)	1.387(2)
C(16)-H(16)	0.9500	C(32)-H(32)	0.9500
C(17)-C(18)	1.385(2)	C(33)-C(34)	1.389(2)

C(33)-H(33)	0.9500	C(45)-H(45A)	0.9900
C(34)-C(35)	1.3953(19)	C(45)-H(45B)	0.9900
C(34)-H(34)	0.9500	C(46)-C(47)	1.5078(19)
C(35)-C(36)	1.5245(18)	C(46)-H(46A)	0.9900
C(36)-O(6)	1.4063(15)	C(46)-H(46B)	0.9900
C(36)-O(4)	1.4297(15)	C(47)-F(8)	1.3702(15)
C(36)-C(37)	1.5328(18)	C(47)-F(7)	1.3960(16)
C(37)-C(48)	1.3360(19)	C(47)-C(48)	1.5086(18)
C(37)-C(38)	1.4866(18)	C(49)-O(7)	1.2172(17)
C(38)-C(39)	1.4016(18)	C(49)-C(51)	1.492(2)
C(38)-C(43)	1.402(2)	C(49)-C(50)	1.496(2)
C(39)-C(40)	1.3865(19)	C(50)-H(50A)	0.9800
C(39)-H(39)	0.9500	C(50)-H(50B)	0.9800
C(40)-C(41)	1.385(2)	C(50)-H(50C)	0.9800
C(40)-H(40)	0.9500	C(51)-H(51A)	0.9800
C(41)-C(42)	1.388(2)	C(51)-H(51B)	0.9800
C(41)-H(41)	0.9500	C(51)-H(51C)	0.9800
C(42)-C(43)	1.3950(19)	O(2)-H(2)	0.8400
C(42)-H(42)	0.9500	O(3)-H(3)	0.8400
C(43)-C(44)	1.5053(19)	O(5)-H(5)	0.8400
C(44)-C(45)	1.5428(19)	O(6)-H(6)	0.8400
C(44)-H(44A)	0.9900	O(8)-H(8X)	0.832(5)
C(44)-H(44B)	0.9900	O(8)-H(8Y)	0.827(5)
C(45)-C(46)	1.534(2)		

O(2)-C(1)-O(1)	111.07(10)	C(8)-C(7)-C(6)	122.10(14)
O(2)-C(1)-C(24)	108.33(10)	C(8)-C(7)-H(7)	118.9
O(1)-C(1)-C(24)	104.19(10)	C(6)-C(7)-H(7)	118.9
O(2)-C(1)-C(2)	110.04(10)	C(9)-C(8)-C(7)	119.58(14)
O(1)-C(1)-C(2)	105.02(10)	C(9)-C(8)-H(8)	120.2
C(24)-C(1)-C(2)	117.96(11)	C(7)-C(8)-H(8)	120.2
F(1)-C(2)-F(2)	104.94(10)	C(8)-C(9)-C(10)	119.74(14)
F(1)-C(2)-C(3)	108.27(11)	C(8)-C(9)-H(9)	120.1
F(2)-C(2)-C(3)	108.30(11)	C(10)-C(9)-H(9)	120.1
F(1)-C(2)-C(1)	108.14(11)	C(11)-C(10)-C(9)	120.89(14)
F(2)-C(2)-C(1)	105.01(10)	C(11)-C(10)-H(10)	119.6
C(3)-C(2)-C(1)	121.05(11)	C(9)-C(10)-H(10)	119.6
C(2)-C(3)-C(4)	116.51(12)	C(10)-C(11)-C(6)	119.79(12)
C(2)-C(3)-H(3A)	108.2	C(10)-C(11)-C(12)	119.39(12)
C(4)-C(3)-H(3A)	108.2	C(6)-C(11)-C(12)	120.82(12)
C(2)-C(3)-H(3B)	108.2	O(3)-C(12)-O(1)	110.55(10)
C(4)-C(3)-H(3B)	108.2	O(3)-C(12)-C(11)	108.53(10)
H(3A)-C(3)-H(3B)	107.3	O(1)-C(12)-C(11)	107.60(10)
C(3)-C(4)-C(5)	112.93(12)	O(3)-C(12)-C(13)	108.48(10)
C(3)-C(4)-H(4A)	109.0	O(1)-C(12)-C(13)	103.63(10)
C(5)-C(4)-H(4A)	109.0	C(11)-C(12)-C(13)	117.90(10)
C(3)-C(4)-H(4B)	109.0	C(24)-C(13)-C(14)	130.27(12)
C(5)-C(4)-H(4B)	109.0	C(24)-C(13)-C(12)	108.75(11)
H(4A)-C(4)-H(4B)	107.8	C(14)-C(13)-C(12)	120.85(11)
C(6)-C(5)-C(4)	116.48(12)	C(15)-C(14)-C(19)	119.41(12)
C(6)-C(5)-H(5A)	108.2	C(15)-C(14)-C(13)	119.47(12)
C(4)-C(5)-H(5A)	108.2	C(19)-C(14)-C(13)	121.10(12)
C(6)-C(5)-H(5B)	108.2	C(16)-C(15)-C(14)	120.69(13)
C(4)-C(5)-H(5B)	108.2	C(16)-C(15)-H(15)	119.7
H(5A)-C(5)-H(5B)	107.3	C(14)-C(15)-H(15)	119.7
C(7)-C(6)-C(11)	117.88(13)	C(17)-C(16)-C(15)	119.93(13)
C(7)-C(6)-C(5)	118.35(13)	C(17)-C(16)-H(16)	120.0
C(11)-C(6)-C(5)	123.71(12)	C(15)-C(16)-H(16)	120.0

C(18)-C(17)-C(16)	119.67(13)	C(29)-C(28)-H(28B)	109.0
C(18)-C(17)-H(17)	120.2	H(28A)-C(28)-H(28B)	107.8
C(16)-C(17)-H(17)	120.2	C(30)-C(29)-C(28)	115.96(11)
C(17)-C(18)-C(19)	121.37(13)	C(30)-C(29)-H(29A)	108.3
C(17)-C(18)-H(18)	119.3	C(28)-C(29)-H(29A)	108.3
C(19)-C(18)-H(18)	119.3	C(30)-C(29)-H(29B)	108.3
C(18)-C(19)-C(14)	118.90(13)	C(28)-C(29)-H(29B)	108.3
C(18)-C(19)-C(20)	119.88(12)	H(29A)-C(29)-H(29B)	107.4
C(14)-C(19)-C(20)	120.88(12)	C(31)-C(30)-C(35)	118.19(12)
C(19)-C(20)-C(21)	109.64(11)	C(31)-C(30)-C(29)	118.34(12)
C(19)-C(20)-H(20A)	109.7	C(35)-C(30)-C(29)	123.42(12)
C(21)-C(20)-H(20A)	109.7	C(32)-C(31)-C(30)	121.92(13)
C(19)-C(20)-H(20B)	109.7	C(32)-C(31)-H(31)	119.0
C(21)-C(20)-H(20B)	109.7	C(30)-C(31)-H(31)	119.0
H(20A)-C(20)-H(20B)	108.2	C(31)-C(32)-C(33)	119.68(12)
C(22)-C(21)-C(20)	114.52(11)	C(31)-C(32)-H(32)	120.2
C(22)-C(21)-H(21A)	108.6	C(33)-C(32)-H(32)	120.2
C(20)-C(21)-H(21A)	108.6	C(32)-C(33)-C(34)	119.42(12)
C(22)-C(21)-H(21B)	108.6	C(32)-C(33)-H(33)	120.3
C(20)-C(21)-H(21B)	108.6	C(34)-C(33)-H(33)	120.3
H(21A)-C(21)-H(21B)	107.6	C(33)-C(34)-C(35)	121.34(13)
C(23)-C(22)-C(21)	116.16(12)	C(33)-C(34)-H(34)	119.3
C(23)-C(22)-H(22A)	108.2	C(35)-C(34)-H(34)	119.3
C(21)-C(22)-H(22A)	108.2	C(34)-C(35)-C(30)	119.44(12)
C(23)-C(22)-H(22B)	108.2	C(34)-C(35)-C(36)	118.99(12)
C(21)-C(22)-H(22B)	108.2	C(30)-C(35)-C(36)	121.57(11)
H(22A)-C(22)-H(22B)	107.4	O(6)-C(36)-O(4)	111.05(10)
F(3)-C(23)-F(4)	104.41(10)	O(6)-C(36)-C(35)	107.52(10)
F(3)-C(23)-C(22)	108.26(11)	O(4)-C(36)-C(35)	107.58(10)
F(4)-C(23)-C(22)	106.98(11)	O(6)-C(36)-C(37)	108.82(10)
F(3)-C(23)-C(24)	108.70(10)	O(4)-C(36)-C(37)	103.78(10)
F(4)-C(23)-C(24)	107.03(11)	C(35)-C(36)-C(37)	118.04(10)
C(22)-C(23)-C(24)	120.34(11)	C(48)-C(37)-C(38)	131.06(12)
C(13)-C(24)-C(23)	128.76(12)	C(48)-C(37)-C(36)	108.96(11)
C(13)-C(24)-C(1)	109.41(11)	C(38)-C(37)-C(36)	119.68(11)
C(23)-C(24)-C(1)	121.83(11)	C(39)-C(38)-C(43)	119.51(12)
O(5)-C(25)-O(4)	111.59(10)	C(39)-C(38)-C(37)	119.46(12)
O(5)-C(25)-C(48)	108.71(10)	C(43)-C(38)-C(37)	121.03(12)
O(4)-C(25)-C(48)	104.03(10)	C(40)-C(39)-C(38)	120.76(12)
O(5)-C(25)-C(26)	109.49(10)	C(40)-C(39)-H(39)	119.6
O(4)-C(25)-C(26)	104.31(10)	C(38)-C(39)-H(39)	119.6
C(48)-C(25)-C(26)	118.51(11)	C(41)-C(40)-C(39)	119.77(13)
F(5)-C(26)-F(6)	104.58(10)	C(41)-C(40)-H(40)	120.1
F(5)-C(26)-C(27)	107.84(10)	C(39)-C(40)-H(40)	120.1
F(6)-C(26)-C(27)	108.51(11)	C(40)-C(41)-C(42)	119.87(13)
F(5)-C(26)-C(25)	108.02(10)	C(40)-C(41)-H(41)	120.1
F(6)-C(26)-C(25)	105.00(10)	C(42)-C(41)-H(41)	120.1
C(27)-C(26)-C(25)	121.65(11)	C(41)-C(42)-C(43)	121.24(13)
C(26)-C(27)-C(28)	116.61(11)	C(41)-C(42)-H(42)	119.4
C(26)-C(27)-H(27A)	108.1	C(43)-C(42)-H(42)	119.4
C(28)-C(27)-H(27A)	108.1	C(42)-C(43)-C(38)	118.83(12)
C(26)-C(27)-H(27B)	108.1	C(42)-C(43)-C(44)	119.64(12)
C(28)-C(27)-H(27B)	108.1	C(38)-C(43)-C(44)	121.24(12)
H(27A)-C(27)-H(27B)	107.3	C(43)-C(44)-C(45)	110.51(11)
C(27)-C(28)-C(29)	113.04(11)	C(43)-C(44)-H(44A)	109.5
C(27)-C(28)-H(28A)	109.0	C(45)-C(44)-H(44A)	109.5
C(29)-C(28)-H(28A)	109.0	C(43)-C(44)-H(44B)	109.5
C(27)-C(28)-H(28B)	109.0	C(45)-C(44)-H(44B)	109.5

H(44A)-C(44)-H(44B)	108.1
C(46)-C(45)-C(44)	114.66(11)
C(46)-C(45)-H(45A)	108.6
C(44)-C(45)-H(45A)	108.6
C(46)-C(45)-H(45B)	108.6
C(44)-C(45)-H(45B)	108.6
H(45A)-C(45)-H(45B)	107.6
C(47)-C(46)-C(45)	115.66(11)
C(47)-C(46)-H(46A)	108.4
C(45)-C(46)-H(46A)	108.4
C(47)-C(46)-H(46B)	108.4
C(45)-C(46)-H(46B)	108.4
H(46A)-C(46)-H(46B)	107.4
F(8)-C(47)-F(7)	104.33(10)
F(8)-C(47)-C(46)	108.23(11)
F(7)-C(47)-C(46)	106.98(11)
F(8)-C(47)-C(48)	109.01(10)
F(7)-C(47)-C(48)	106.54(11)
C(46)-C(47)-C(48)	120.57(11)
C(37)-C(48)-C(47)	128.25(12)
C(37)-C(48)-C(25)	109.48(11)
C(47)-C(48)-C(25)	122.26(11)
O(7)-C(49)-C(51)	120.45(14)
O(7)-C(49)-C(50)	122.53(13)
C(51)-C(49)-C(50)	117.02(13)
C(49)-C(50)-H(50A)	109.5
C(49)-C(50)-H(50B)	109.5
H(50A)-C(50)-H(50B)	109.5
C(49)-C(50)-H(50C)	109.5
H(50A)-C(50)-H(50C)	109.5
H(50B)-C(50)-H(50C)	109.5
C(49)-C(51)-H(51A)	109.5
C(49)-C(51)-H(51B)	109.5
H(51A)-C(51)-H(51B)	109.5
C(49)-C(51)-H(51C)	109.5
H(51A)-C(51)-H(51C)	109.5
H(51B)-C(51)-H(51C)	109.5
C(1)-O(1)-C(12)	110.73(9)
C(1)-O(2)-H(2)	109.5
C(12)-O(3)-H(3)	109.5
C(25)-O(4)-C(36)	110.81(9)
C(25)-O(5)-H(5)	109.5
C(36)-O(6)-H(6)	109.5
H(8X)-O(8)-H(8Y)	95.4(18)

Table S14. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for dimer **13**. The anisotropic displacement factor exponent takes the form: $-2p^2[\ h^2 a^{*2} U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	15(1)	15(1)	17(1)	-8(1)	-3(1)	0(1)
C(2)	15(1)	23(1)	24(1)	-13(1)	-4(1)	-3(1)
C(3)	20(1)	24(1)	33(1)	-20(1)	-3(1)	-4(1)
C(4)	22(1)	16(1)	35(1)	-14(1)	-3(1)	-3(1)
C(5)	18(1)	15(1)	30(1)	-13(1)	-3(1)	1(1)
C(6)	13(1)	14(1)	23(1)	-5(1)	0(1)	-3(1)
C(7)	19(1)	14(1)	32(1)	-5(1)	-1(1)	0(1)
C(8)	24(1)	20(1)	27(1)	2(1)	-6(1)	3(1)
C(9)	27(1)	29(1)	19(1)	-3(1)	-7(1)	1(1)
C(10)	20(1)	20(1)	18(1)	-6(1)	-2(1)	0(1)
C(11)	13(1)	14(1)	16(1)	-4(1)	0(1)	-2(1)
C(12)	13(1)	13(1)	17(1)	-8(1)	-2(1)	-1(1)
C(13)	16(1)	10(1)	16(1)	-6(1)	-2(1)	0(1)
C(14)	15(1)	18(1)	13(1)	-8(1)	-3(1)	-3(1)
C(15)	18(1)	16(1)	17(1)	-8(1)	-4(1)	-2(1)
C(16)	16(1)	24(1)	20(1)	-12(1)	-5(1)	1(1)
C(17)	16(1)	29(1)	21(1)	-13(1)	-2(1)	-6(1)
C(18)	22(1)	20(1)	21(1)	-9(1)	-2(1)	-7(1)
C(19)	19(1)	17(1)	16(1)	-9(1)	-3(1)	-3(1)
C(20)	21(1)	14(1)	23(1)	-7(1)	-4(1)	-2(1)
C(21)	25(1)	18(1)	21(1)	-2(1)	-5(1)	-3(1)
C(22)	22(1)	23(1)	14(1)	-6(1)	-3(1)	-2(1)
C(23)	18(1)	19(1)	18(1)	-10(1)	-7(1)	2(1)
C(24)	14(1)	11(1)	18(1)	-8(1)	-3(1)	0(1)
C(25)	16(1)	15(1)	16(1)	-8(1)	-4(1)	-1(1)
C(26)	17(1)	17(1)	18(1)	-7(1)	-6(1)	-1(1)
C(27)	20(1)	16(1)	26(1)	-11(1)	-9(1)	4(1)
C(28)	22(1)	13(1)	24(1)	-9(1)	-7(1)	1(1)
C(29)	18(1)	16(1)	21(1)	-11(1)	-4(1)	0(1)
C(30)	18(1)	15(1)	14(1)	-5(1)	-3(1)	-2(1)
C(31)	24(1)	14(1)	15(1)	-5(1)	-4(1)	-3(1)
C(32)	23(1)	20(1)	14(1)	-4(1)	-5(1)	-9(1)
C(33)	17(1)	23(1)	16(1)	-5(1)	-5(1)	-3(1)
C(34)	17(1)	17(1)	14(1)	-4(1)	-3(1)	-1(1)
C(35)	17(1)	14(1)	12(1)	-3(1)	-3(1)	-3(1)
C(36)	16(1)	13(1)	15(1)	-6(1)	-4(1)	1(1)
C(37)	16(1)	11(1)	17(1)	-8(1)	-2(1)	0(1)
C(38)	12(1)	16(1)	16(1)	-7(1)	-2(1)	0(1)
C(39)	15(1)	14(1)	19(1)	-7(1)	-3(1)	-1(1)
C(40)	19(1)	23(1)	20(1)	-11(1)	-6(1)	0(1)
C(41)	23(1)	21(1)	16(1)	-4(1)	-6(1)	1(1)
C(42)	21(1)	14(1)	21(1)	-4(1)	-3(1)	-1(1)
C(43)	14(1)	17(1)	18(1)	-8(1)	-1(1)	0(1)
C(44)	24(1)	14(1)	21(1)	-8(1)	-3(1)	-2(1)
C(45)	24(1)	19(1)	25(1)	-9(1)	-4(1)	-7(1)
C(46)	17(1)	23(1)	21(1)	-11(1)	-1(1)	-5(1)
C(47)	16(1)	20(1)	21(1)	-11(1)	-5(1)	-1(1)
C(48)	18(1)	11(1)	16(1)	-8(1)	-3(1)	0(1)
C(49)	22(1)	22(1)	20(1)	-13(1)	-5(1)	0(1)
C(50)	33(1)	21(1)	29(1)	-12(1)	-10(1)	0(1)
C(51)	28(1)	29(1)	47(1)	-16(1)	9(1)	-1(1)
O(1)	13(1)	14(1)	17(1)	-7(1)	-2(1)	-2(1)

O(2)	14(1)	17(1)	22(1)	-10(1)	-3(1)	2(1)
O(3)	19(1)	15(1)	17(1)	-9(1)	-6(1)	3(1)
O(4)	16(1)	16(1)	15(1)	-6(1)	-4(1)	-2(1)
O(5)	24(1)	14(1)	15(1)	-8(1)	-6(1)	1(1)
O(6)	16(1)	18(1)	24(1)	-13(1)	-5(1)	2(1)
O(7)	20(1)	23(1)	28(1)	-14(1)	-4(1)	1(1)
O(8)	43(1)	21(1)	19(1)	-7(1)	-4(1)	9(1)
F(1)	23(1)	31(1)	29(1)	-17(1)	-11(1)	-1(1)
F(2)	14(1)	27(1)	34(1)	-18(1)	0(1)	-6(1)
F(3)	21(1)	27(1)	20(1)	-8(1)	-10(1)	4(1)
F(4)	28(1)	28(1)	21(1)	-16(1)	-5(1)	-3(1)
F(5)	19(1)	18(1)	26(1)	-10(1)	-10(1)	1(1)
F(6)	26(1)	16(1)	17(1)	-4(1)	-6(1)	-1(1)
F(7)	17(1)	24(1)	28(1)	-9(1)	-3(1)	3(1)
F(8)	27(1)	35(1)	23(1)	-13(1)	-7(1)	-12(1)

Table S15. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for dimer **13**.

	x	y	z	U(eq)
H(3A)	3753	10668	-701	27
H(3B)	2498	10059	-235	27
H(4A)	3259	10936	-2327	28
H(4B)	2645	11675	-1822	28
H(5A)	1159	10047	-1112	25
H(5B)	718	11210	-1530	25
H(7)	338	12109	-3135	30
H(8)	-132	12140	-4529	35
H(9)	313	10712	-4826	33
H(10)	1290	9276	-3745	25
H(15)	-737	9482	-1384	20
H(16)	-2836	9051	-936	23
H(17)	-3334	7366	-34	25
H(18)	-1724	6117	360	25
H(20A)	1576	6424	-375	24
H(20B)	536	5597	109	24
H(21A)	156	5626	1625	28
H(21B)	1660	5643	1281	28
H(22A)	49	7399	1086	25
H(22B)	765	6794	1983	25
H(27A)	5538	3605	7770	23
H(27B)	5592	4506	6690	23
H(28A)	3175	3797	7798	23
H(28B)	4098	3104	7340	23
H(29A)	3967	5073	5906	21
H(29B)	3779	4075	5834	21
H(31)	1758	3568	6066	22
H(32)	-383	3962	6056	23
H(33)	-1348	5396	6247	23
H(34)	-159	6403	6475	20
H(39)	2804	6279	4528	20
H(40)	2574	7258	2893	23
H(41)	2773	8992	2174	26
H(42)	3245	9735	3079	24
H(44A)	3487	8588	5584	23

H(44B)	3488	9647	4629	23
H(45A)	5577	9402	3960	27
H(45B)	5546	9068	5104	27
H(46A)	5609	7684	4284	23
H(46B)	6827	7935	4504	23
H(50A)	6511	6494	8556	40
H(50B)	7999	6420	8511	40
H(50C)	7497	6384	7642	40
H(51A)	8529	8954	6685	57
H(51B)	8947	7989	6466	57
H(51C)	9219	7994	7433	57
H(2)	4943	8013	-1482	27
H(3)	2864	7756	-2259	24
H(5)	3889	6104	8482	25
H(6)	1714	7305	6968	27
H(8X)	4117(18)	6322(7)	9734(14)	32
H(8Y)	3926(18)	5485(13)	10420(4)	32

Dimer 14

A colorless prism 0.12 x 0.10 x 0.05 mm in size was mounted on a Cryoloop with Paratone oil. Data were collected in a nitrogen gas stream at 100(2) K using phi and omega scans. Crystal-to-detector distance was 60 mm and exposure time was 5 seconds per frame using a scan width of 1.0°. Data collection was 97.7% complete to 67.00° in θ. A total of 16000 reflections were collected covering the indices, -9<=*h*<=9, -10<=*k*<=10, -17<=*l*<=16. 3374 reflections were found to be symmetry independent, with an R_{int} of 0.0180. Indexing and unit cell refinement indicated a primitive, triclinic lattice. The space group was found to be P-1 (No. 2). The data were integrated using the Bruker SAINT software program and scaled using the SADABS software program. Solution by direct methods (SIR-2008) produced a complete heavy-atom phasing model consistent with the proposed structure. All non-hydrogen atoms were refined anisotropically by full-matrix least-squares (SHELXL-97). All hydrogen atoms were placed using a riding model. Their positions were constrained relative to their parent atom using the appropriate HFIX command in SHELXL-97.

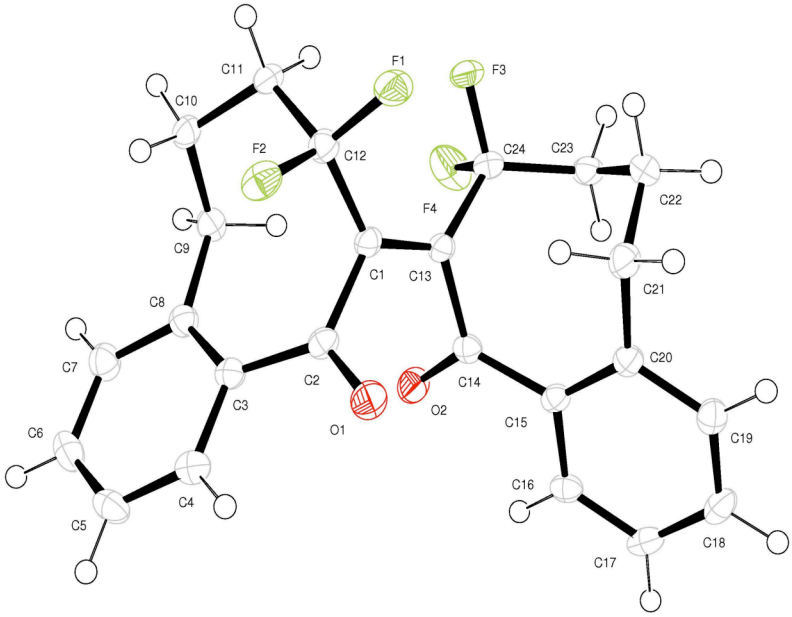


Figure S15. X-ray structure of dimer **14** with carbon, fluorine, and oxygen atoms labeled. ORTEPs are shown for 50% probability.

Table S16. Crystal data and structure refinement for dimer **14**.

Empirical formula	C24 H20 F4 O2	
Formula weight	416.40	
Temperature	100(2) K	
Wavelength	1.54178 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 7.5863(4) Å	α= 91.070(3)°.
	b = 8.9598(5) Å	β= 96.218(2)°.
	c = 14.6268(8) Å	γ = 104.754(3)°.
Volume	954.66(9) $Å^3$	
Z	2	
Density (calculated)	1.449 Mg/m^3	
Absorption coefficient	0.999 mm^{-1}	
F(000)	432	
Crystal size	0.12 x 0.10 x 0.05 mm^3	
Crystal color/habit	colorless prism	
Theta range for data collection	3.04 to 68.25°.	
Index ranges	-9<=h<=9, -10<=k<=10, -17<=l<=16	
Reflections collected	16000	
Independent reflections	3374 [R(int) = 0.0180]	
Completeness to theta = 67.00°	97.7 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.9517 and 0.8895	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3374 / 0 / 271	
Goodness-of-fit on F^2	1.036	
Final R indices [I>2sigma(I)]	R1 = 0.0322, wR2 = 0.0853	
R indices (all data)	R1 = 0.0351, wR2 = 0.0883	
Largest diff. peak and hole	0.272 and -0.204 e.$Å^{-3}$	

Table S17. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for dimer **14**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	12252(2)	2961(1)	6896(1)	18(1)
C(2)	12516(2)	1932(1)	7688(1)	19(1)
C(3)	14068(2)	1181(1)	7779(1)	18(1)
C(4)	14073(2)	193(1)	8511(1)	22(1)
C(5)	15486(2)	-508(2)	8708(1)	26(1)
C(6)	16916(2)	-244(2)	8167(1)	28(1)
C(7)	16912(2)	713(2)	7436(1)	26(1)
C(8)	15515(2)	1449(1)	7221(1)	20(1)
C(9)	15678(2)	2433(2)	6387(1)	21(1)
C(10)	14448(2)	1616(2)	5515(1)	23(1)
C(11)	12760(2)	2208(2)	5238(1)	21(1)
C(12)	11502(2)	2072(1)	5977(1)	21(1)
C(13)	12558(2)	4474(1)	7096(1)	18(1)
C(14)	13326(2)	5058(1)	8084(1)	18(1)
C(15)	12265(2)	5722(1)	8711(1)	18(1)
C(16)	13174(2)	6194(1)	9597(1)	20(1)
C(17)	12371(2)	6865(1)	10240(1)	23(1)
C(18)	10646(2)	7100(2)	10005(1)	25(1)
C(19)	9720(2)	6609(1)	9142(1)	22(1)
C(20)	10479(2)	5894(1)	8482(1)	19(1)
C(21)	9325(2)	5367(2)	7574(1)	24(1)
C(22)	9776(2)	6534(2)	6819(1)	30(1)
C(23)	11806(2)	7061(2)	6697(1)	25(1)
C(24)	12602(2)	5765(2)	6433(1)	22(1)
O(1)	11426(1)	1773(1)	8252(1)	28(1)
O(2)	14870(1)	4949(1)	8333(1)	27(1)
F(1)	9898(1)	2411(1)	5641(1)	29(1)
F(2)	10947(1)	526(1)	6182(1)	26(1)
F(3)	11843(1)	5208(1)	5567(1)	34(1)
F(4)	14446(1)	6370(1)	6350(1)	40(1)

Table S18. Bond lengths [Å] and angles [°] for dimer **14**.

C(1)-C(13)	1.3367(18)	C(13)-C(24)	1.5202(17)
C(1)-C(2)	1.5217(17)	C(13)-C(14)	1.5271(17)
C(1)-C(12)	1.5269(17)	C(14)-O(2)	1.2172(16)
C(2)-O(1)	1.2144(15)	C(14)-C(15)	1.4925(17)
C(2)-C(3)	1.4933(17)	C(15)-C(16)	1.4041(18)
C(3)-C(4)	1.4027(18)	C(15)-C(20)	1.4071(18)
C(3)-C(8)	1.4128(18)	C(16)-C(17)	1.3806(18)
C(4)-C(5)	1.3819(19)	C(16)-H(16)	0.9500
C(4)-H(4)	0.9500	C(17)-C(18)	1.386(2)
C(5)-C(6)	1.386(2)	C(17)-H(17)	0.9500
C(5)-H(5)	0.9500	C(18)-C(19)	1.382(2)
C(6)-C(7)	1.384(2)	C(18)-H(18)	0.9500
C(6)-H(6)	0.9500	C(19)-C(20)	1.3992(18)
C(7)-C(8)	1.3960(18)	C(19)-H(19)	0.9500
C(7)-H(7)	0.9500	C(20)-C(21)	1.5070(18)
C(8)-C(9)	1.5157(17)	C(21)-C(22)	1.539(2)
C(9)-C(10)	1.5462(19)	C(21)-H(21A)	0.9900
C(9)-H(9A)	0.9900	C(21)-H(21B)	0.9900
C(9)-H(9B)	0.9900	C(22)-C(23)	1.523(2)
C(10)-C(11)	1.5247(18)	C(22)-H(22A)	0.9900
C(10)-H(10A)	0.9900	C(22)-H(22B)	0.9900
C(10)-H(10B)	0.9900	C(23)-C(24)	1.5014(18)
C(11)-C(12)	1.5040(17)	C(23)-H(23A)	0.9900
C(11)-H(11A)	0.9900	C(23)-H(23B)	0.9900
C(11)-H(11B)	0.9900	C(24)-F(3)	1.3621(15)
C(12)-F(1)	1.3705(15)	C(24)-F(4)	1.3853(16)
C(12)-F(2)	1.3894(15)		

C(13)-C(1)-C(2)	117.47(11)	C(10)-C(9)-H(9B)	108.9
C(13)-C(1)-C(12)	128.58(12)	H(9A)-C(9)-H(9B)	107.7
C(2)-C(1)-C(12)	113.73(10)	C(11)-C(10)-C(9)	115.11(10)
O(1)-C(2)-C(3)	121.58(11)	C(11)-C(10)-H(10A)	108.5
O(1)-C(2)-C(1)	116.93(11)	C(9)-C(10)-H(10A)	108.5
C(3)-C(2)-C(1)	121.48(10)	C(11)-C(10)-H(10B)	108.5
C(4)-C(3)-C(8)	119.64(12)	C(9)-C(10)-H(10B)	108.5
C(4)-C(3)-C(2)	115.06(11)	H(10A)-C(10)-H(10B)	107.5
C(8)-C(3)-C(2)	125.24(11)	C(12)-C(11)-C(10)	113.26(11)
C(5)-C(4)-C(3)	121.22(12)	C(12)-C(11)-H(11A)	108.9
C(5)-C(4)-H(4)	119.4	C(10)-C(11)-H(11A)	108.9
C(3)-C(4)-H(4)	119.4	C(12)-C(11)-H(11B)	108.9
C(4)-C(5)-C(6)	119.47(12)	C(10)-C(11)-H(11B)	108.9
C(4)-C(5)-H(5)	120.3	H(11A)-C(11)-H(11B)	107.7
C(6)-C(5)-H(5)	120.3	F(1)-C(12)-F(2)	103.67(10)
C(7)-C(6)-C(5)	119.80(12)	F(1)-C(12)-C(11)	110.31(10)
C(7)-C(6)-H(6)	120.1	F(2)-C(12)-C(11)	108.22(10)
C(5)-C(6)-H(6)	120.1	F(1)-C(12)-C(1)	109.52(10)
C(6)-C(7)-C(8)	122.28(13)	F(2)-C(12)-C(1)	105.89(10)
C(6)-C(7)-H(7)	118.9	C(11)-C(12)-C(1)	118.15(11)
C(8)-C(7)-H(7)	118.9	C(1)-C(13)-C(24)	128.07(11)
C(7)-C(8)-C(3)	117.59(12)	C(1)-C(13)-C(14)	117.45(11)
C(7)-C(8)-C(9)	116.26(11)	C(24)-C(13)-C(14)	113.32(10)
C(3)-C(8)-C(9)	126.13(11)	O(2)-C(14)-C(15)	121.57(11)
C(8)-C(9)-C(10)	113.26(10)	O(2)-C(14)-C(13)	115.77(11)
C(8)-C(9)-H(9A)	108.9	C(15)-C(14)-C(13)	122.66(11)
C(10)-C(9)-H(9A)	108.9	C(16)-C(15)-C(20)	119.40(11)
C(8)-C(9)-H(9B)	108.9	C(16)-C(15)-C(14)	115.14(11)

C(20)-C(15)-C(14)	125.46(11)
C(17)-C(16)-C(15)	121.25(12)
C(17)-C(16)-H(16)	119.4
C(15)-C(16)-H(16)	119.4
C(16)-C(17)-C(18)	119.56(12)
C(16)-C(17)-H(17)	120.2
C(18)-C(17)-H(17)	120.2
C(19)-C(18)-C(17)	119.70(12)
C(19)-C(18)-H(18)	120.2
C(17)-C(18)-H(18)	120.2
C(18)-C(19)-C(20)	122.08(12)
C(18)-C(19)-H(19)	119.0
C(20)-C(19)-H(19)	119.0
C(19)-C(20)-C(15)	117.92(12)
C(19)-C(20)-C(21)	117.50(12)
C(15)-C(20)-C(21)	124.58(11)
C(20)-C(21)-C(22)	113.06(11)
C(20)-C(21)-H(21A)	109.0
C(22)-C(21)-H(21A)	109.0
C(20)-C(21)-H(21B)	109.0
C(22)-C(21)-H(21B)	109.0
H(21A)-C(21)-H(21B)	107.8
C(23)-C(22)-C(21)	114.45(11)
C(23)-C(22)-H(22A)	108.6
C(21)-C(22)-H(22A)	108.6
C(23)-C(22)-H(22B)	108.6
C(21)-C(22)-H(22B)	108.6
H(22A)-C(22)-H(22B)	107.6
C(24)-C(23)-C(22)	113.44(11)
C(24)-C(23)-H(23A)	108.9
C(22)-C(23)-H(23A)	108.9
C(24)-C(23)-H(23B)	108.9
C(22)-C(23)-H(23B)	108.9
H(23A)-C(23)-H(23B)	107.7
F(3)-C(24)-F(4)	104.44(10)
F(3)-C(24)-C(23)	108.69(11)
F(4)-C(24)-C(23)	108.52(11)
F(3)-C(24)-C(13)	111.77(11)
F(4)-C(24)-C(13)	104.68(10)
C(23)-C(24)-C(13)	117.80(10)

Symmetry transformations used to generate equivalent atoms:

Table S19. Anisotropic displacement parameters ($Å^2 x\ 10^3$)for dimer **14**. The anisotropic displacement factor exponent takes the form: $-2p^2[\ h^2a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	15(1)	23(1)	16(1)	0(1)	4(1)	7(1)
C(2)	20(1)	18(1)	17(1)	-2(1)	3(1)	2(1)
C(3)	20(1)	16(1)	18(1)	-1(1)	1(1)	2(1)
C(4)	23(1)	20(1)	20(1)	1(1)	2(1)	0(1)
C(5)	31(1)	19(1)	24(1)	5(1)	-3(1)	4(1)
C(6)	27(1)	25(1)	34(1)	3(1)	-1(1)	12(1)
C(7)	24(1)	27(1)	29(1)	3(1)	6(1)	10(1)
C(8)	21(1)	18(1)	21(1)	0(1)	2(1)	4(1)
C(9)	19(1)	23(1)	23(1)	5(1)	8(1)	7(1)
C(10)	30(1)	23(1)	19(1)	3(1)	10(1)	11(1)
C(11)	27(1)	22(1)	15(1)	-2(1)	3(1)	7(1)
C(12)	20(1)	21(1)	21(1)	-1(1)	1(1)	6(1)
C(13)	17(1)	23(1)	16(1)	1(1)	4(1)	7(1)
C(14)	19(1)	16(1)	18(1)	3(1)	3(1)	3(1)
C(15)	22(1)	15(1)	16(1)	2(1)	4(1)	3(1)
C(16)	23(1)	18(1)	18(1)	3(1)	2(1)	2(1)
C(17)	32(1)	20(1)	15(1)	0(1)	4(1)	0(1)
C(18)	33(1)	20(1)	22(1)	1(1)	13(1)	5(1)
C(19)	24(1)	20(1)	25(1)	4(1)	8(1)	6(1)
C(20)	22(1)	16(1)	19(1)	2(1)	5(1)	4(1)
C(21)	19(1)	32(1)	23(1)	-3(1)	1(1)	10(1)
C(22)	37(1)	41(1)	20(1)	1(1)	-1(1)	24(1)
C(23)	39(1)	20(1)	17(1)	3(1)	2(1)	12(1)
C(24)	27(1)	26(1)	15(1)	3(1)	4(1)	9(1)
O(1)	29(1)	31(1)	26(1)	8(1)	13(1)	10(1)
O(2)	21(1)	36(1)	25(1)	-5(1)	0(1)	10(1)
F(1)	22(1)	44(1)	22(1)	-4(1)	-2(1)	13(1)
F(2)	30(1)	21(1)	23(1)	-2(1)	5(1)	-2(1)
F(3)	65(1)	32(1)	14(1)	1(1)	1(1)	26(1)
F(4)	32(1)	45(1)	48(1)	27(1)	18(1)	13(1)

Table S20. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for dimer **14**.

	x	y	z	U(eq)
H(4)	13087	3	8878	26
H(5)	15478	-1165	9211	31
H(6)	17895	-719	8299	34
H(7)	17895	874	7067	31
H(9A)	16972	2714	6258	26
H(9B)	15341	3401	6531	26
H(10A)	15199	1735	4995	27
H(10B)	14037	498	5618	27
H(11A)	13165	3305	5087	25
H(11B)	12069	1617	4676	25
H(16)	14365	6049	9757	24
H(17)	12997	7163	10840	28
H(18)	10102	7596	10435	29
H(19)	8531	6762	8992	27
H(21A)	8014	5194	7662	29
H(21B)	9516	4368	7366	29
H(22A)	9085	6061	6227	36
H(22B)	9343	7453	6969	36
H(23A)	11971	7831	6216	30
H(23B)	12494	7578	7280	30