Supplementary Information

Fluorinated liquid crystals: Evaluation of selectively fluorinated facially polarised cyclohexyl motifs for liquid crystal applications

Nawaf Al-Maharik[a], Peer Kirsch*[b], Alexandra M. Z. Slawin,[a] David B. Cordes and David O'Hagan*[a]

[a]*EaStCHEM School of Chemistry, University of St. Andrews, St. Andrews, Fife KY16 9ST, UK. E. mail do1@st-andrews.ac.uk*

[b]*Merck KGaA, Liquid Crystals R&D Chemistry, Frankfurter Str. 250, D-64293 Darmstadt, Germany. E-mail: peer.kirsch@merckgroup.com*

Experimental Protocols

General experimental procedures: Reagents were purchased from commercial suppliers and used without purification, unless otherwise stated. Reactions were carried out in oven-dried glassware or teflon under argon atmosphere. Anhydrous CH_2Cl_2 and THF were obtained from a MBraun MB SPS-800 solvent purification system, where the solvent was dried by passage through activated filter columns and dispensed under an atmosphere of argon gas. All NMR spectra were recorded using Bruker Avance 400 usig $CDCl_3$. ^{13}C NMR spectra were recorded using the DEPTQ pulse sequence and broadband proton decoupling at 75 MHz. ^{19}F NMR spectra were recorded at 376 MHz. Chemical shifts are given in parts per million relative to residual solvent peaks. Melting points were determined using a Griffin MPA350 melting point apparatus and are uncorrected. High and low resolution mass spectra were carried out at the University of St Andrews and were obtained using a Waters Micromass LCT (ES) or GCT (EI/CI). Thin layer chromatography was carried out on aluminium backed Merck TLC silica gel 60 F254 plates. These plates were visualised using UV light at a wavelength of 254 nm followed by staining with potassium permanganate or phosphomolybdic acid dip. Chromatography was carried out on 230−400 mesh silica gel.

4'-(*trans*-4-Propylcyclohexyl)biphenyl 11: 2-Propanol (30 mL) was added to a mixture of phenylboronic ester **10** (2.3 g, 19.6 mmol), 1-bromo-4-(trans-4-propylcyclohexyl)benzene **9** (5.0 g, 17.8 mmol) and [Pd(IPr)(cin)Cl] (115 mg, 0.178 mmol) under Ar. The mixture was evacuated and fluhed with Ar (3 times). After 15 h stirring at room temperature, the resulting mixture was diluted with dichloromethane (100 mL) and filtered through celite. The filtrate was washed with water (50 mL) and

brine (50 mL), dried over $MgSO_4$, filtered, and concentrated in *vacuo*. The crude residue was purified by SiO_2 column chromatography (petroleum ether) to give 4-(*trans*-4-propylcyclohexyl)biphenyl **11** (3.92 g, 80%) as a white solid; M.p. = 90-91 ^oC; ^1H NMR (400 MHz, $CDCl_3$) δ 7.59 (d, J = 8.2 Hz, 2H), 7.53 (d, J = 8.2 Hz, 2H), 7.43 (t, J = 8.2 Hz, 2H), 7.32 (t, J = 8.2 Hz, 1H), 7.29 (d, J = 8.2 Hz, 2H), 2.52 (tt, J = 3.2, 12.6 Hz, 1H), 1.87-1.96 (m, 4H), 1.18-1.55 (m, 7H), 1.00-1.12 (m, 2H), 0.91 (q, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.0, 141.2,138.7, 128.7, 127.2, 127.0, 126.9, 44.3, 39.7, 37.0, 34.3, 33.6, 20.0, 14.4; **MS** (CI, +ve) *m*/*z* 278 (M+, 100%); **HRMS** (CI, MH +ve) $C_{21}H_{29}$ requires *m*/*z* 279.2113, found 279.2121.

4'-(trans-4-Propylcyclohexyl)-1,4-dihydro-1,1'-biphenyl 12: Lithium (0.69 g, 96.0 mmol) was added portionwise to a solution of biphenyl **11** (12 g, 43.1 mmol) in ammonia (400 mL) and THF (200 mL) at -78 ^oC. The reaction was allowed to warm to -40 ^oC and stirred for 3 h. The reaction mixture was quenched with solid NH_4Cl, the mixture was allowed to warm up, and water (200 mL) and ether (200 mL) were carefully added and the layers were separated. The aqueous layer was extracted with ether (200 mL). The combined organic layers were dried over $MgSO_4$, filtered, and concentrated under reduced pressure. The filtrate was washed with water and brine, dried over $MgSO_4$, filtered, and concentrated under reduced pressure to afford diene **12** (12 g, 92%) as a white solid, M.p. = 72-73 ^oC ; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.22-7.23 (m, 4H), 5.77-5.90 (m, 4H), 3.95-4.04 (m, 1H), 2.77-2.84 (m, 2H), 2.51 (tt, J = 3.2, 12.2 Hz, 1H), 1.89-1.97 (m, 4H), 1.23-1.56 (m, 7H), 1.03-1.17 (m, 2H), 0.97 (t, J = 7.3 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 145.9, 142.5, 128.7, 127.8, 127.0, 123.5, 44.2, 41.6, 39.7, 37.0, 34.4, 33.6, 25.8, 20.0, 14.4; **MS** (CI, +ve) *m*/*z* 280 (M+, 46%), 155 (100%); **HRMS** (CI, M + H, +ve) $C_{21}H_{28}$ requires *m*/*z* 281.2269, found 281.2267.

4'-(trans-4-propylcyclohexyl)phenyl-cis-2,5-diepoxycyclohexane 13. *meta*-Chloroperbenzoic acid (5.42 g, 31.4 mmol) was added to an ice-cold solution of diene **12** (4 g, 14.3 mmol) in dichloromethane (50 mL) under argon, and the resulting mixture was stirred at 0 °C for 5 h. The white precipitate was removed by filtration (cold dichloromethane wash), and the filtrate was washed with a solution of sodium hydroxide in brine (10%, 100 mL). The organic layer was dried ($MgSO_4$), filtered, and concentrated under reduced pressure. The crude product was subjected to flash chromatography (petroleum ether/ethyl acetate 8:2) to afford cis diepoxide **13** (3.97 g, 89%) as a white solid; M.p. = 168-169 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.22-7.23 (m, 4H), 3.93 (d, J = 1.4 Hz, 1H), 3.17-3.26 (m, 2H), 3.00-3.13 (m, 2H), 2.88-2.94 (dm, 1H), 2.38-2.52 (m, 2H), 1.73-1.92 (m, 4H), 1.02-1.45 (m, 9H), 0.90 (t, J = 7.3 Hz, 3H); **^{13}C {1H} NMR** (75 MHz, $CDCl_3$) δ 147.6, 135.4, 128.4, 127.6, 53.6, 49.3, 44.2, 39.7, 39.2, 37.0, 34.3, 33.5, 23.7, 20.0, 14.4; **MS** (CI, +ve) *m/z* 312 (MH+, 100%); **HRMS** (CI, +ve) $C_{21}H_{28}O_2$ + requires *m/z* 312.2089, found 312.2085.

Fluorohydrin 14 and 15: A mixture of the diepoxide **13** (3.7 g, 11.9 mmol) and triethylamine trihydrofluoride (9.7 mL, 59.9 mmol) in a Teflon round-bottom flask was stirred at 145 °C for 24 h under Argon. The mixture was cooled down to room temperature, diluted with dichloromethane (100 mL), washed with ice-cold aq. $NaHCO_3$ (100 mL), and the aqueous phase was back-extracted with dichloromethane (100 mL). The combined organic layers were dried ($MgSO_4$), filtered, and concentrated to afford a light yellow solid (3.89 g) as a mixture of 2 difluorodiols, 4,6-difluoro-2-(4-(trans-4-propylcyclohexyl)phenyl)cyclohexane-1,3-diol **14** and 2,5-difluoro-6-(4-(trans-4-propylcyclohexyl)phenyl)cyclohexane-1,4-diol **15** in 2:3 ratio. Part of the crude product was subjected to triflation without purification.

Triflation of difluorodiols 14 and 15: Triflic anhydride (3.44 mL, 20.5 mmol) and dry pyridine (2.36 mL, 20.5 mmol) were consequently and slowly added to a solution of difluorodiols **14** and **15** (2.4 g, 6.82 mmol) in dry CH_2Cl_2 (60 mL) at 0 °C under

Ar. A thick yellow precipitate was formed, and the mixture was stirred at 0 ^{o}C for 5 h. The mixture was poured into water (100 mL), extracted with CH_2Cl_2 (50 mL x 3), the combined organic layers were washed with saturated aqueous $CuSO_4$ solution, dried ($MgSO_4$), filtered and the concentrated in *vacuo*. The light yellow residue was purified by SiO_2 column chromatography (petroleum ether/CH_2Cl_2 8:2 to 7:3) to give asymmetric 2,5-difluoro-6-(4-(trans)-4-propylcyclohexyl)phenyl)cyclohexane-1,4-diyl bis(triflate) **17** (2.35 g, 56%) as a white solid; M.p. = 153 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.23-7.29 (m, 4H), 5.36 (dd, *J* = 8.9, 11.3 Hz, 1H), 5.28-5.30 (m, 1H), 5.05 (dm, *J* = 47.5 Hz, 1H), 4.93 (dm, *J* = 47.8 Hz, 1H), 3.27 (dd, *J* = 11.8, 37.9 Hz, 1H), 2.74-2.80 (dm, 1H), 2.43-2.53 (m, 2H), 1.85-1.90 (m, 4H), 1.18-1.48 (m, 7H), 1.00-1.10 (m, 2H), 0.90 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 149.1, 129.3, 129.2, 127.6, 118.4 (d, *J* = 319.4 Hz), 117.9 (d, *J* = 320.2 Hz), 89.9 (d, *J* = 181.6 Hz), 87.1 (d, *J* = 186.0 Hz), 85.3 (dd, *J* = 2.4, 19.8 Hz), 79.9 (dd, *J* = 34.4, 13.3 Hz), 45.7 (dd, *J* = 17.6, 5.5 Hz), 44.3, 39.7, 37.0, 34.2 (d, *J* = 2.1 Hz), 33.5, 31.1, 30.9, 20.0, 14.4; ^{19}F-NMR (376MHz, $CDCl_3$) δ -75.0 (s), -75.2 (d, *J* = 7.9 Hz), -187.9 (dm, *J* = 48.4 Hz), -194.0 (tbs, *J* = 47.5 Hz); ^{19}F-NMR (decoupled) δ -75.0 (s), -75.2 (d, *J* = 7.9 Hz), -187.9 (q, *J* = 8.3 Hz), -194.0 (s); MS (ESI) *m/z* 639 (M+Na, 100%); HRMS (ESI) $C_{23}H_{28}O_6F_8NaS_2$ requires *m/z* 639.1092, found 639.1089. Followed by the symmetric 4,6-difluoro-2-(4-(trans-4-propylcyclohexyl)phenyl)cyclohexane-1,3-diyl ditriflate **17**(1.73 g, 41%) as a white solid; M.p. = 156-167 ^{o}C (decompose); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.27 (d, *J* = 8.3 Hz, 2H), 7.17 (d, *J* = 8.3 Hz, 2H), 5.05 (dt, *J* = 1.6, 10.7 Hz, 2H), 4.79 (dm, *J* = 48.5, 2H), 3.08 (t, *J* = 11.4 Hz, 1H), 2.90-2.98 (m, 1H), 2.46-2.49 (m, 1H), 1.83-1.88 (dm, 4H), 1.18-1.47 (m, 8H), 0.99-1.09 (m, 2H), 0.90 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 149.8, 128.5, 128.0, 127.6, 117.9 (d, *J* = 319.8 Hz), 86.9 (dd, *J* = 188.5, 15.1 Hz), 86.3 (d, *J* = 18.8 Hz), 47.3 (t, *J* = 6.0 Hz), 45.9, 44.3, 39.7, 37.0,

34.2, 33.4, 31.8 (t, *J* = 21.6 Hz), 20.0, 14.4; ^{19}F-NMR (376MHz, $CDCl_3$) δ -75.2 (d, *J* = 7.9 Hz), -185.9 (dm, *J* = 48.9 Hz); ^{19}F-NMR (decoupled) -75.2 (d, *J* = 7.9 Hz), -185.9 (m); MS (ESI) *m*/*z* 639 (M+Na, 100%); HRMS (ESI) $C_{23}H_{28}O_6F_8NaS_2$ requires *m*/*z* 639.1092, found 639.1091.

1-(trans-4-Propylcyclohexyl)-4-(2,3,5,6-tetrafluorocyclohexyl)benzene 3. A mixture of the ditriflate **16** (2.0 g, 3.25 mmol) and triethylamine trihydrofluoride (10.9 mL, 66.9 mmol) in a Teflon round-bottom flask was stirred at 100 ^{o}C for 56 h under Argon. The mixture was cooled down to room temperature, diluted with dichloromethane (50 mL), washed with ice-cold aq. $NaHCO_3$ (50 mL), and the aqueous phase was back-extracted with dichloromethane (100 mL). The combined organic layers were dried ($MgSO_4$), filtered, and concentrated. Purification by flash chromatography on silica gel (petroleum ether/CH_2Cl_2 8:2 to 7:3) to give the tetrafluoro compound **3** (1.03 g, 89%) as a white solid, M.p. = 219 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) *δ* 7.39 (d, *J* = 8.3 Hz, 2H), 7.22 (d, *J* = 8.3 Hz, 2H), 4.91-5.11 (m, 2H), 4.52-4.73 (m, *J* = 48.5, 2H), 2.70-2.83 (m, 1H), 2.57 (tt, *J* = 1.7, 37.4 Hz, 1H), 2.43-2.52 (m, 2H), 1.84-1.93 (m, 4H), 1.19-1.54 (m, 8H), 1.00-1.10 (m, 2H), 0.90 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) *δ* 147.8, 132.9, 129.1, 127.3, 86.05-90.17 (m), 44.2, 43.5-43.9 (m), 39.7, 37.0, 34.3, 33.5, 27.2 (t, *J* = 22.4 Hz), 20.0, 14.4; ^{19}F-NMR δ -190.7 (dm, *J* = 48.4 Hz, 2F), -210.1-(-210.5) (m, 2F); ^{19}F-NMR (376MHz, $CDCl_3$) (decoupled) δ -190.7 (dd, *J* = 5.5, 8.0 Hz), -210.3 (dd, *J* = 8.0, 4.8 Hz); MS (CI, +ve) *m*/*z* 356 (M+, 56%), 258 (100%); HRMS (CI, +ve) $C_{21}H_{28}F_4$ requires *m*/*z* 356.2127, found 356.2122.

2,3,6-trifluoro-4'-(trans-4-propylcyclohexyl)-1,2,3,6-tetrahydro-1,1'-biphenyl 18. Following the same procedure as was used to prepare **3**. The asymmetric ditriflate **17** (0.08 g, 0.13 mmol) was converted to the trifluoro **18** in (0.038g, 87%) yield as a white solid; M.p. = 157-158 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.24-7.31 (m, 4H),

6.11-6.16 (m, 2H), 5.20-5.45 (m, 2H), 5.01 (dm, J = 47.9 Hz, 1H), 3.17-3.23 (m, 4H), 2.50 (tt, J =3.2, 12.2 Hz, 1H), 1.86-1.95 (m, 4H), 1.21-1.52 (m, 8H), 1.02-1.13 (m, 2H), 0.93 (t, J = 7.2 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 147.5, 132.1, 130.7 (dt, J = 9.2, 228 Hz), 128.9, 127.1, 127.0-127.3 (m), 91.1 (ddd, J = 3.6, 22.4, 175.1 Hz), 89.3 (dd, J = 18.7, 182.8 Hz), 87.4 (dd, J = 7.8, 172.8 Hz), 48.1 (td, J = 7.0, 19.6 Hz),44.2, 39.7, 37.0, 34.3 (d, J = 2.3 Hz), 33.6, 20.0, 14.4; ^{19}F-NMR (376MHz, $CDCl_3$) δ -176.2-(-176.4) (m, 1F), -185.4 (dm, J = 47.5 Hz, 1F), -195.1 (dm, J = 47.5 Hz, 1F); ^{19}F-NMR (decoupled) -176.2 (d, J = 11.2 Hz, 1F), -185.4 (dd, J = 4.2, 14.3 Hz, 1F), -195.1 (d, J = 14.3 Hz, 1F); **MS** (CI, +ve) m/z 336 (M, 52%), 246 (100%); **HRMS** (CI, +ve) $C_{21}H_{27}F_3$ requires m/z 336.2065, found 336.2059.

Controlled elimination:

3,6-Difluoro-4'-(trans-4-propylcyclohexyl)-1,2,3,6-tetrahydro-[1,1'-biphenyl]-2-yl trifluoromethanesulfonate 19. DBU (0.393 mL, 2.63 mmol) was added dropwise to a solution of the asymmetric ditriflate **17** (1.5 g, 2.43 mmol) in dry THF (40 mL) under argon at 0 ^{o}C. After the solution was stirred for 1 h, the resulting mixture was warmed to rt and the solution was then concentrated in vacuo. Purification by flash chromatography on silica gel (petroleum ether/CH_2Cl_2 8:2) provided alkene **19** (1.11 g, 98%) as a white solid; M.p. = 138 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.23-7.31 (m, 4H), 6.17-6.24 (m, 1H), 6.14 (ddd, J = 1.9, 4.7, 10.0 Hz, 1H), 5.61(ddd, J = 4.7, 10.0, 13.2 Hz, 1H), 5.35 (dm, J = 48.5 Hz, 1H), 5.09 (dm, J = 48.8, 1H), 3.19 (dddd, J = 1.0, 3.1, 12.5, 30.5 Hz, 1H), 2.50 (tt, J = 3.1, 12.5 Hz, 1H), 1.85-1.94 (m, 4H), 1.19-1.52 (m, 8H), 0.97-1.13 (m, 2H), 0.92 (t, J = 7.2 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 148.5, 130.4, 130.1 (dd, J = 9.7, 22.7 Hz), 129.7, 127.6 (dd, J = 8.8, 17.0 Hz), 127.2, 118.0 (q, J = 321.0 Hz), 90.0 (dd, J = 3,3, 178.6 Hz), 86.7 (d, J = 174.9Hz), 84.6 (d, J = 18.6 Hz), 48.3 (dd, J = 5.7, 19.3 Hz), 44.3, 39.7, 37.0, 34.22, 34.20, 33.5, 20.1, 14.4; ^{19}F-NMR (376MHz, $CDCl_3$) δ -75.2 (d, J = 9.1 Hz, 3F), -175.1-(-175.4)

(m, 1F), -183.3 (dm, J = 49.2 Hz, 1F); ^{19}F-NMR (decoupled) δ -75.2 (d, J = 9.1 Hz, 3F), -175. (d, J = 9.7 Hz, 1F), -183.3 (quintet, J = 9.2 Hz, 1F); MS (CI, +ve) m/z 489 (M+Na, 100%); HRMS (CI, +ve) $C_{22}H_{27}O_3F_5SNa$ requires m/z 489.1493, found 489.1487.

3,6-Difluoro-2-(4-(trans-4-propylcyclohexyl)phenyl)cyclohexyl trifluoromethanesulfonate 21. Compound **19** (1.0 g, 2.15 mmol) and Pd/C (10%, 100 mg) were suspended in ethyl acetate (20 mL), evacuated flushed 3 times with hydrogen. The mixture was stirred under hydrogen atmosphere for 3 h, and then filtered through a pad of Celite. The filtrate was concentrated and the residue was subjected to silica column chromatography (petroleum ether/EtOAc 8:2) to afford **21** (1.0 g, 100%) as a white solid; M.p. = 107-108 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.23-7.30 (m, 4H), 5.42 (td, J = 8.8, 11.1 Hz, 1H), 4.89 (dbs, J = 47.7 Hz, 1H), 4.74 (dm, J = 49.9 Hz, 1H), 2.91 (dd, J = 11.8, 34.8 Hz, 1H), 2.46 (tt, J = 3.2, 12.1 Hz, 1H), 2.15-2.30 (m, 3H), 1.84-1.88 (m, 4H), 1.65-1.79 (m, 1H), 1.18-1.47 (m, 7H), 1.00-1.08 (m, 2H), 0.86 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 148.2, 131.7, 129.1, 127.2, 118.1 (q, J = 319.7 Hz), 91.5 (d, J = 177.8 Hz), 91.3 (d, J = 184.6 Hz), 87.9 (dd, J = 1.8, 18.8 Hz), 53.5, 51.0 (dd, J = 4.7, 17.8 Hz), 44.3, 39.6, 37.1, 34.2 (d, J = 3.6 Hz), 33.5, 27.8 (dd, J = 11.3, 21.7 Hz), 24.9 (d, J = 19.1 Hz), 20.1, 14.4; ^{19}F-NMR δ -75.2 (d, J = 9.1 Hz, 3F), -179.4 (dm, J = 47.7 Hz, 1F), -194.1-(-194.6) (dm, J = 47.7 Hz, 1F); ^{19}F-NMR (376MHz, $CDCl_3$) (decoupled) δ -75.5 (d, J = 7.9 Hz, 3F), -179.4 (q, J = 7.9 Hz, 1F), -194.3 (s, 1F); MS (CI, +ve) m/z 491 (M+Na, 100%); HRMS (CI, +ve) $C_{22}H_{29}O_3F_5SNa$ requires m/z 491.1655, found 491.1640.

1-(trans-4-Propylcyclohexyl)-4-(2,3,6-trifluorocyclohexyl)benzene 4. A mixture of the triflate **21** (0.9 g, 1.95 mmol) and triethylamine trihydrofluoride (3.2 mL, 19.5 mmol) in a Teflon round-bottom flask was stirred at 100 ^{o}C for 56 h under Argon.

The mixture was cooled down to room temperature, diluted with dichloromethane (50 mL), washed with ice-cold aq. $NaHCO_3$ (50 mL), and the aqueous phase was back-extracted with dichloromethane (100 mL). The combined organic layers were dried ($MgSO_4$), filtered, and concentrated. Purification by flash chromatography on silica gel (petroleum ether/CH_2Cl_2 8:2 to 7:3) to give the all-syn trifluoro compound **4** (0.547 g, 84%) as a white solid; M.p. = 186-187 ^oC. ^1H NMR (400 MHz, $CDCl_3$) δ 7.39 (d, J = 8.2 Hz, 2H), 7.20 (d, J = 8.2 Hz, 2H), 5.09 (ddm, J = 8.6, 51.7 Hz, 1H), 4.87 (dm, J = 47.6 Hz, 1H), 4.62 (ddddd, J = 2.1, 4.7, 11.8, 25.4, 45.5 Hz, 1H), 2.66 (tbs, J = 38.6 Hz, 1H), 2.30-2.57 (m, 3H), 1.84-1.99 (m, 5H),), 1.18-1.70 (m, 7H), 1.00-1.10 (m, 2H), 0.90 (t, J = 7.2 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 147.3, 134.8, 129.1, 127.1, 90.7 (dd, J = 17.8, 187.7 Hz), 90.2 (dd, J = 19.2, 184.7 Hz), 89.1 (d, J = 181.6 Hz), 48.1 (td, J = 5.4, 17.3 Hz), 44.2, 39.7, 37.0, 34.3, 33.6, 28.6 (dd, J = 11.7, 22.4 Hz), 20.4 (dd, J = 3.7, 20.2 Hz), 20.0, 14.4; ^{19}F-NMR (376MHz, $CDCl_3$) δ -183.2 (dm, J = 45.5 Hz, 1F), -191.2-(-191.7) (m, 1F), -209.9-(-210.3) (m, 1F); ^{19}F-NMR (decoupled) -183.2 (d, J = 14.8 Hz, 1F), -191.5 (d, J = 26.3Hz, 1F), -210.1 (dd, J = 14.8, 26.3 Hz, 1F); MS (CI, +ve) m/z 361 (M+Na, 100%); HRMS (CI, +ve) $C_{21}H_{29}F_3Na$ requires m/z 361.2119, found 361.2112.

4,4,5,5-Tetramethyl-2-[4-(trans-4-propylcyclohexyl)phenyl]-1,3,2-dioxaborolane 22. Bromo-4-(trans-4-propylcyclohexyl)benzene **9** (2.6 g, 9.24 mmol), $Pd_2(dba)_3$ (0.211 g, 0.23 mmol), XPhos (0.220 g, 0.462 mmol), bis(pinocolate)diborane (3.52 g, 13.88 mmol) and potassium acetate (2.72 g 27.7 mmol) were placed in a flask equipped with a condenser and flushed with argon. 1,4-Dioxane (50 mL) was added and the reaction mixture was heated to 90^{o}C. After 5h stirring at maintain temperature, the mixture was cooled down to room temperature, passed through a short pad of silica, washed with dichloromethane (50 mL) and solvent were removed

under reduced pressure. The brown residue was subjected to flash column chromatography (petroleum ether/dichloromethane 9:1 to 8:2) to give the title compound **22** (2.86 g, 89%) as a white solid, M.p. = 123-124 ^{o}C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.74 (d, J = 8.1 Hz, 2H), 7.23 (d, J = 8.1 Hz, 2H), 2.48 (dd, J = 3.3, 12.2 Hz, 1H), 1.84-1.90 (m, 4H), 1.57 (d, J = 0.8 Hz, 1H), 1.47 (dq, J = 3.3, 12.6 Hz, 2H), 1.18-1.39 (m, 17H), 0.99-1.09 (m, 2H), 0.90 (t, J = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 151.3, 134.9, 126 (C), 126.4, 44.9, 39.7, 37.0, 34.2, 33.5, 24.9, 20.0, 14.4; HRMS (ESI, +ve) *m/z* calcd for C21H24O5Na [M+Na+] 379.1521, found 379.1515.

4'-(trans-4-Propylcyclohexyl)-1,2,3,4-tetrahydro-1,1'-biphenyl 23: Bis(dibenzylidenacetone)palladium (9 mg, 0.01 eq, 15 μmol) and KF (0.23 g, 2.6 eq., 3.96 mmol) were subsequently added to a solution of cyclohex-2-enyl acetate (0.213g, 1.524 mmol) and the aryl boronate **22** (0.5 g, 1.524 mmol) in degased anhydrous MeOH (10 mL) at room temperature under argon. The reaction was stirred for 20 h at reflux temperature, diluted with brine (50 mL) and extracted with ether (50 mL x 3). The combined organic phase was washed with water (50 mL x 3), dried ($MgSO_4$), filtered and concentrated under reduced pressure. Chromatography of the residue on silica gel (petroleum ether) afforded the desired cyclohexene **23** (0.62 g, 58%) as a white solid, M.p. = 72-73 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.22-7.43 (m, 4H), 5.96-6.02 (m, 1H), 5.81-5.87 (m, 1H), 3.45-3.53 (m, 1H), 2.56 (tt, J = 3.3, 12.2 Hz, 1H), 1.09-2.23 (m, 19H), 1.03 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 145.9, 144.4, 130.9, 128.6, 128.0, 127.1, 44.2, 41.4, 39.7, 37.0, 34.4, 33.6, 32.6, 25.0, 21.2, 20.0, 14.4; MS (CI, +ve) *m/z* 280 (M+, 46%), 155 (100%); HRMS (CI, M + H, +ve) $C_{21}H_{28}$ requires *m/z* 281.2269, found 281.2267.

2-[4-(trans-4-Propylcyclohexyl)phenyl]-cyclohexene epoxide 24. Epoxidation of **23** (2.9 g, 10.28 mmol) with *m*CPBA (3.46 g, 15.4 mmol) in dichloromethane (50 mL) according to the procedure described for **13** gave the epoxide **24** (petroleum ether/dicholromethane 75:25) (2.53g, 83%) as a white solid; M.p. = 168-169 ºC; ^{1}H

NMR (400 MHz, $CDCl_3$) δ 7.18 (brs, 4H), 3.30-3.82 (m, 1H), 3.19 (d, J = 3.8 Hz, 1H), 3.11 (dd, J = 6.0, 9.7 Hz, 1H), 2.45 (tt, J = 3.4, 12.1 Hz, 1H), 2.14 (dt, J = 4.7, 15.0Hz, 1H), 1.80-191 (m, 6H), 1.19-1.49 (m, 10H), 1.00-1.09 (m, 2H), 0.90 (t, J = 7.3 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 146.0, 141.6, 127.7, 127.0, 56.4, 52.9, 44.2, 40.8, 39.7, 37.0, 34.4, 33.6, 29.7, 24.6, 20.1, 16.9, 14.4; MS (ESI, +ve) *m*/*z* 321 (MNa+, 100%); HRMS (ESI, +ve) $C_{21}H_{30}ONa$ + requires 321.2194 *m*/*z* found 321.2189,

Preparation of 27 and 28.

Following the general procedure to prepare **14** and **15** but with epoxide **24** (1 g, 3.36 mmol) and triethylamine trihydrofluoride (1.64 mL, 10.07 mmol) to give a 1:0.8 mixture of fluorohydrine **25** and **26** (1.07 g, 98%) as a white solid.

The product was dissolved in anhydrous pyridine (5 mL) at -0 ^{o}C under argon, and triflic anhydride (0.85 mL, 5.03 mmol) was then slowly added. A thick yellow precipitate was formed, and the mixture was stirred at room temperature for 5 h. The mixture was poured into water (100 mL), extracted with CH_2Cl_2 (50 mL x 3), the combined organic layers were washed with saturated aqueous $CuSO_4$ solution (3 mL), dried ($MgSO_4$), filtered and the concentrated in *vacuo*. The light yellow residue was purified by SiO_2 column chromatography (petroleum ether/CH_2Cl_2 9:1 to 8:2) to give 6-fluoro-4'-(trans-4-propylcyclohexyl)-1,2,3,6-tetrahydro-1,1'-biphenyl **28** (0.456 g, 56%) as a white solid; M.p. = 153 ^{o}C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.23 (d, J = 8.5 Hz, 2H), 7.17 (d, J = 8.5 Hz, 2H), 6.12-6.20 (m, 1H), 5.97-6.03 (m, 1H), 4.91 (dm, J = 49.9 Hz, 1H), 2.84 (tt, J = 2.8, 13.0, 32.4 Hz, 1H), 2.45 (dd, J = 3.2, 12.2 Hz, 1H), 2.08-2.39 (m, 3H), 1.78-1.92 (m, 4H), 0.97-1.11 (m, 2H), 1.17-1.51 (m, 7H), 0.90 (t, J = 7.0 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 146.2, 139.3, 135.1 (d, J = 9.9 Hz), 128.0, 126.8, 124.6 (d, J = 16.2 Hz), 86.7 (d, J = 170.2 Hz), 44.6 (d, J = 19.9 Hz), 39.8, 37.0, 34.4 (d, J = 2.4 Hz), 33.6, 26.6 (d, J = 3.4 Hz), 22.2, 20.1, 14.5; ^{19}F-NMR

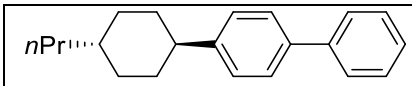
δ −175.4-(−174.8) (m); ^{19}F-NMR (decoupled) −175.0 (s); **MS** (ESI) *m*/*z* 323 (M+Na, 100%); **HRMS** (ESI) $C_{21}H_{29}FNa$ requires *m*/*z* 323.2151, found 323.2138.

Followed by the 2-fluoro-6-[4-((trans-4-propylcyclohexyl)phenyl]cyclohexyl trifluoromethanesulfonate **27** (0.653 g, 43%) as a white solid; M.p. = 156-167 (decompose) ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.18 (d, *J* = 8.2 Hz, 2H), 7.13 (d, *J* = 8.2 Hz, 2H), 4.90 (dt, *J* = 9.0, 10.4 Hz, 1H), 4.64 (dddd, *J* = 5.3, 8.6, 11.6, 50.3 Hz, 1H), 2.81 (dt, *J* = 11.9, 3.8 Hz, 1H), 2.45 (tt, *J* = 11.3, 3.1 Hz, 1H), 2.34-2.40 (m, 1H), 1.84-2.01(m, 6H), 1.63-1.78 (m, 2H), 1.19-1.51 (m, 8H), 0.99-1.08 (m, 2H), 0.90 (t, *J* = 6.7 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 147.7, 135.6, 127.6, 127.3, 118.0 (d, *J* = 319.6Hz), 92.8 (d, *J* = 183.8 Hz), 92.4 (d, *J* = 16.8 Hz), 47.7 (t, *J* = 1.7 Hz), 44.3, 39.7, 37.0, 34.3, 33.5, 32.6, 33.0 (d, *J* = 17.7 Hz), 22.2 (d, *J* = 11.6 Hz), 20.0, 14.4; ^{19}F-NMR δ −75.2 (d, J = 7.9 Hz, 3F), −185.9 (dm, *J* = 48.9 Hz, 1F); ^{19}F-NMR (decoupled) δ −75.2 (d, J = 7.9 Hz, 3F), −185.9 (m, 1F); MS (ESI) m/z 473 (M+Na, 100); HRMS (ESI) $C_{22}H_{30}O_3F_4SNa$ requires m/z 473.1744, found 473.1740.

1-(2,3-Difluorocyclohexyl)-4-(trans-4-propylcyclohexyl)benzene 5. Following the general procedure to prepare **3** with fluorotriflate **27** (0.64 g, 1.42 mmol) and triethylamine trihydrofluoride (0.55 mL, 5.69 mmol). The white residue was purified by SiO_2 column chromatography (petroleum ether/CH_2Cl_2 9:1) to afford the difluorocyclohexane **5** (0.43 g, 94%) as a white solid; M.p. = 128 ^{o}C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.21 (d, *J* = 8.2 Hz, 2H), 7.17 (d, *J* = 8.2 Hz, 2H), 4.96 (dd, *J* = 9.8, 53.1 Hz, 1H), 4.48-4.66 (m, 1H), 2.62 (ddd, *J* = 3.7, 13.0, 35.6 Hz, 1H), 2.45 (tt, *J* = 3.1, 12.1 Hz, 1H), 1.85-2.02 (m, 8H), 1.67-1.71 (m, 1H), 1.19-1.48 (m, 8H), 1.00-1.08 (m, 2H), 0.90 (t, *J* = 7.3 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 146.6, 138.6, 127.8, 126.9, 91.7 (dd, *J* = 16.5, 182.1 Hz), 91.65(dd, *J* = 19.4, 182.4 Hz), 45.6 (dd, *J* = 5.6, 19.3 Hz), 44.2, 39.7, 37.0, 34.3, 33.6, 25.7 (dd, *J* = 3.1, 18.7 Hz), 25.3 (d, *J* = 3.3 Hz), 22.6 (d, *J* = 12.2 Hz), 20.1, 14.4; ^{19}F-NMR δ −185.9 (m, 1F), −213.7 (m, 1F); ^{19}F-

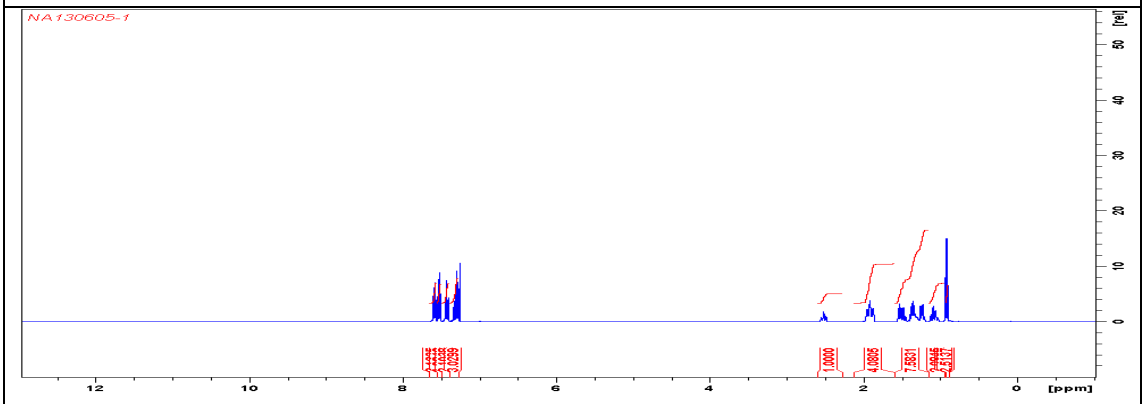
NMR (decoupled) δ −185.9 (d, J = 15.5 Hz, 1F), −213.7 (d, J = 15.5 Hz, 1F); MS (ESI) m/z 343 (M+Na, 100%); HRMS (ESI) $C_{21}H_{30}F_2Na$ requires m/z 343.2213, found 343.2211.

1-(2-Fluorocyclohexyl)-4-(trans-4-propylcyclohexyl]benzene 6. Compound **28** (0.45 g, 2.15 mmol) and Pd/C (10%, 50 mg) were suspended in ethyl acetate (20 mL), evacuated flushed 3 times with hydrogen. The mixture was stirred under hydrogen atmosphere for 3 h, and then filtered through a pad of Celite. The filtrate was concentrated and the residue was subjected to silica column chromatography (petroleum ether) to afford **5** (0.43 g, 99%) as a white solid; M.p. = 94 ^{o}C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.21 (d, J = 8.1 Hz, 2H), 7.15 (d, J = 8.1 Hz, 2H), 4.85 (dd, J = 1.7, 48.9 Hz, 1H), 2.63 (ddd, J = 2.8, 13.0,37.0, Hz, 1H), 2.44 (tt, J = 3.2, 12.1 Hz, 1H), 2.10-2.18 (m, 1H), 2.01(dq, J = 3.6, 13.0 Hz, 1H), 1.84-1.91 (m, 5H), 1.51-1.74 (m, 5H), 1.18-1.48 (m, 8H), 1.00-1.08 (m, 2H), 0.90 (t, J = 7.0 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 146.0, 140.9, 127.8, 126.7, 92.2 (d, J = 173.6 Hz), 47.2 (d, J = 19.7 Hz), 44.2, 39.8, 37.0, 34.4 (d, J = 2.5 Hz), 33.6, 31.7 (d, J = 22.0 Hz), 26.5 (d, J = 2.6 Hz), 25.9, 20.1, 19.8, 14.5; ^{19}F-NMR δ −175.4 (m); ^{19}F-NMR (decoupled) −197.1 (s); MS (ESI) m/z 325 (M+Na, 100%); HRMS (ESI) $C_{21}H_{32}FNa$ requires m/z 325.2307, found 325.2305.

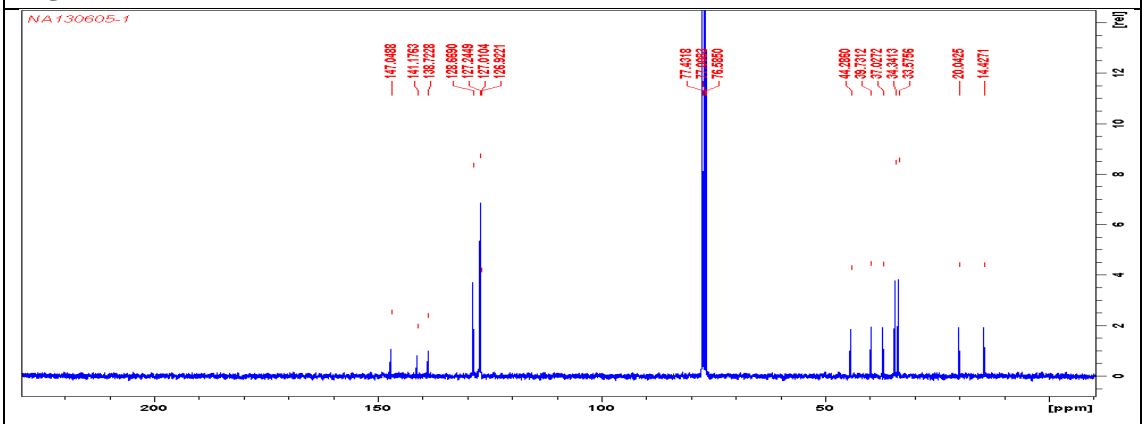


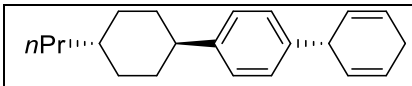
11

^{1}H NMR



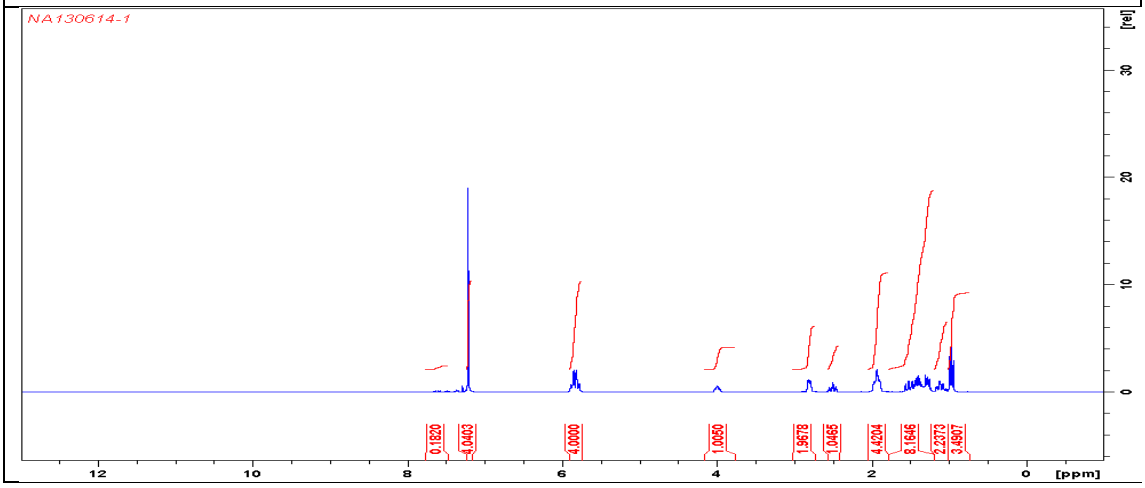
^{13}C NMR



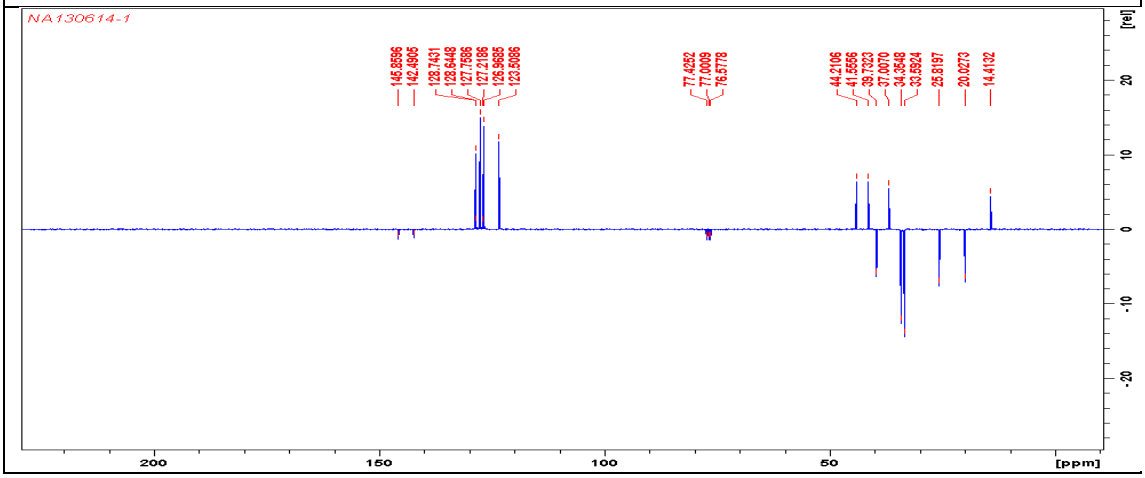


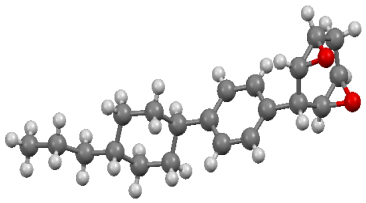
12

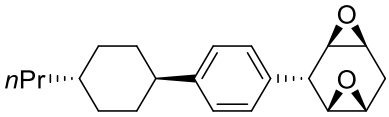
^{1}H NMR



^{13}C NMR

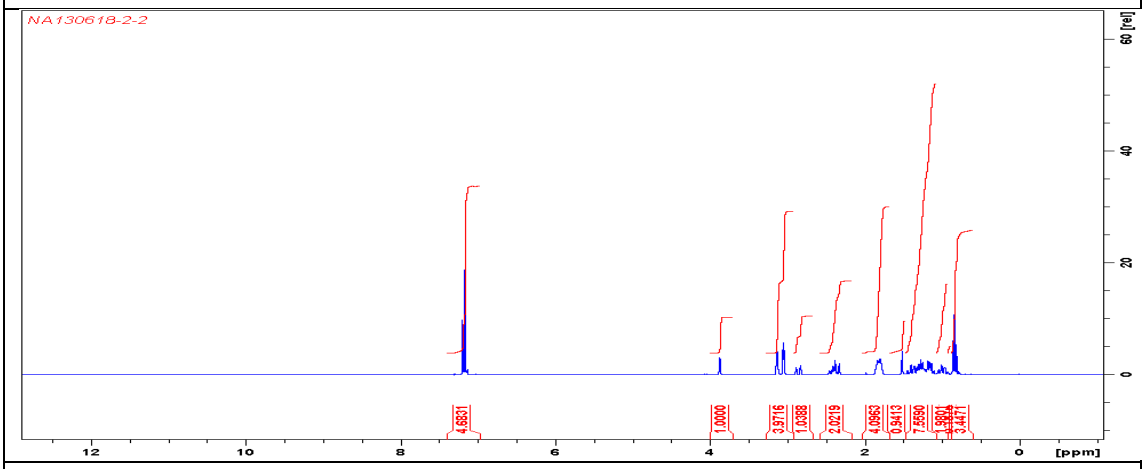




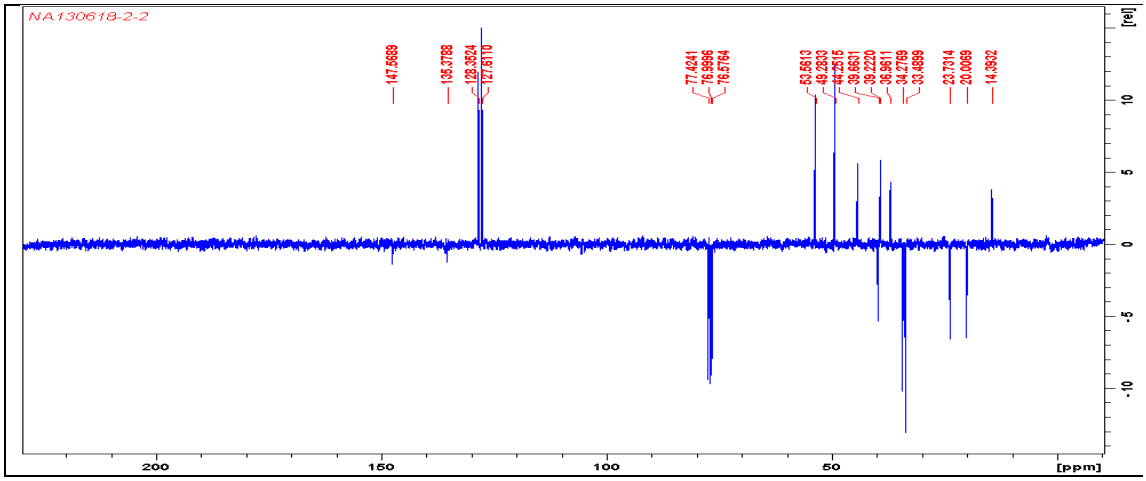


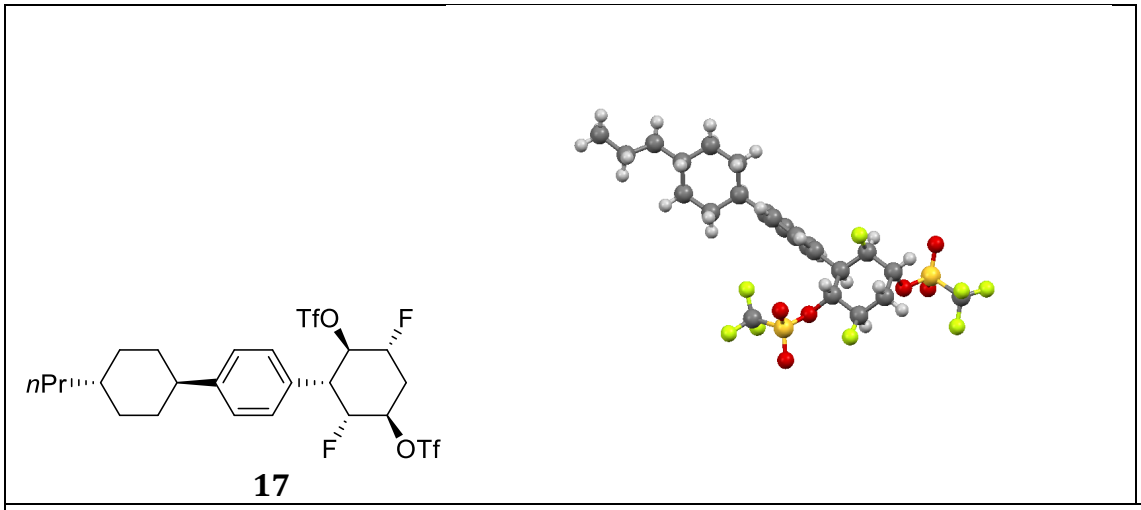
13

^{1}H NMR



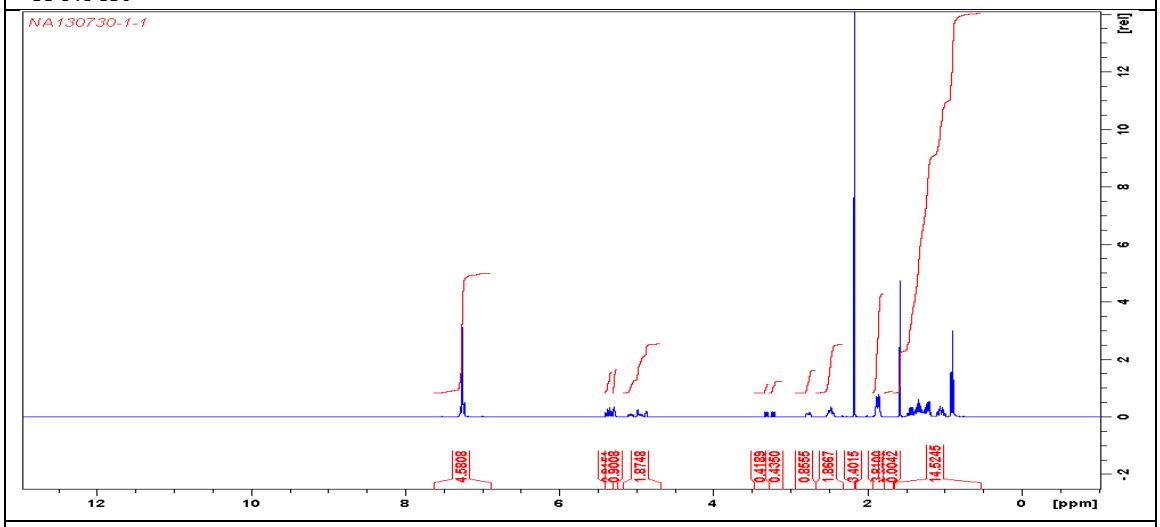
^{13}C NMR



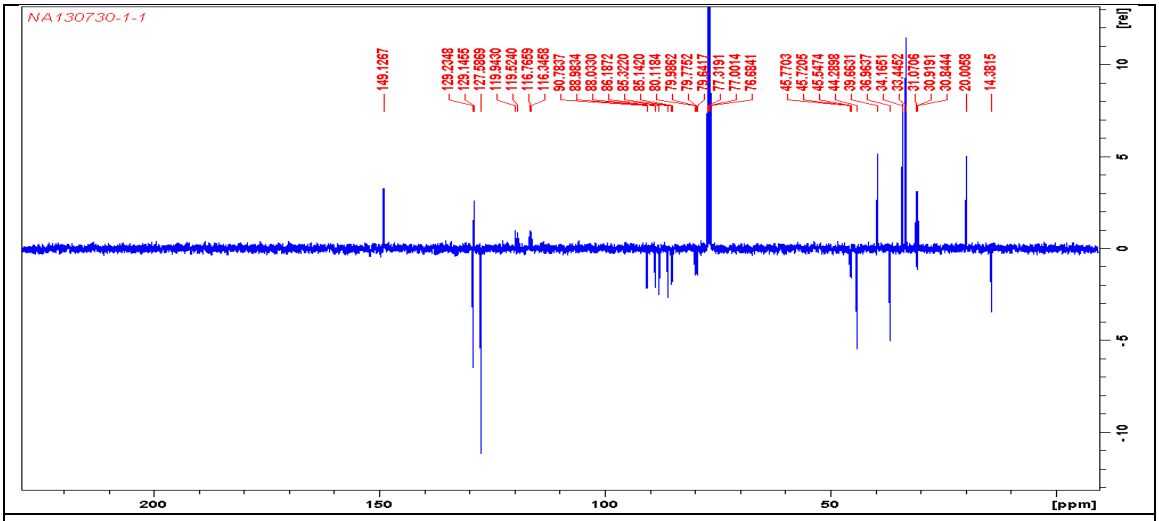


17

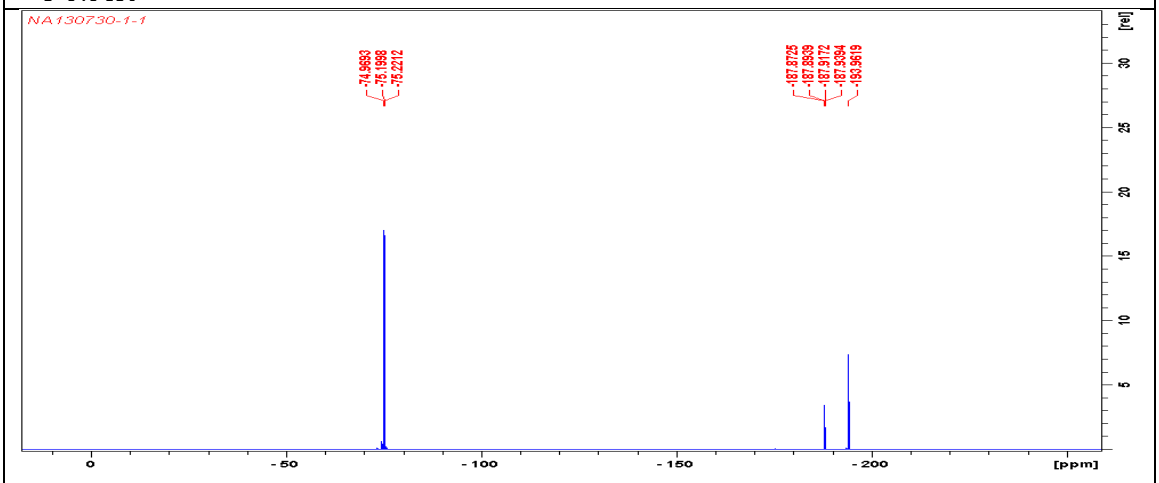
^{1}H NMR

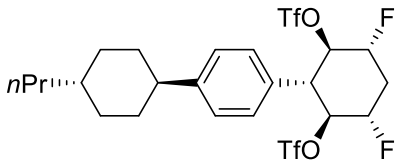


^{13}C NMR



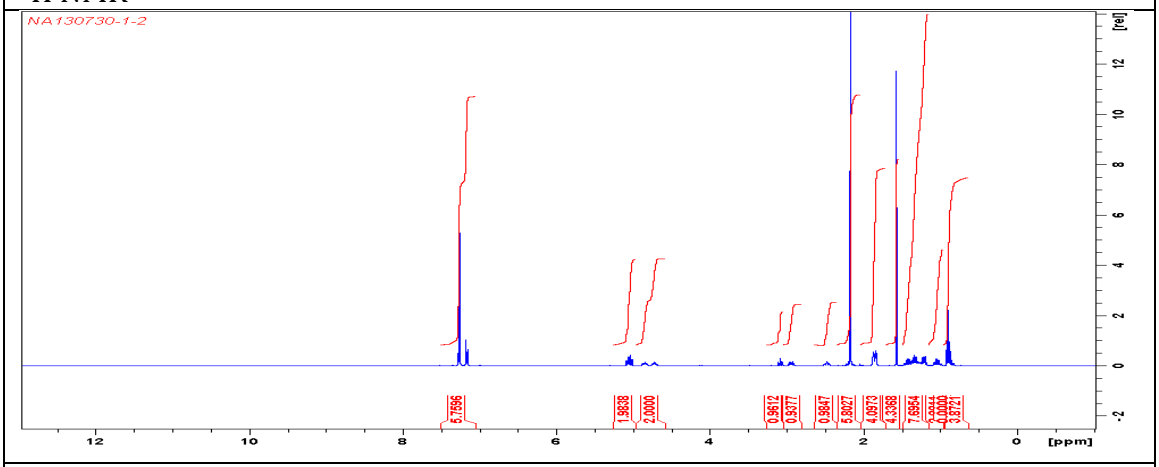
^{19}F NMR



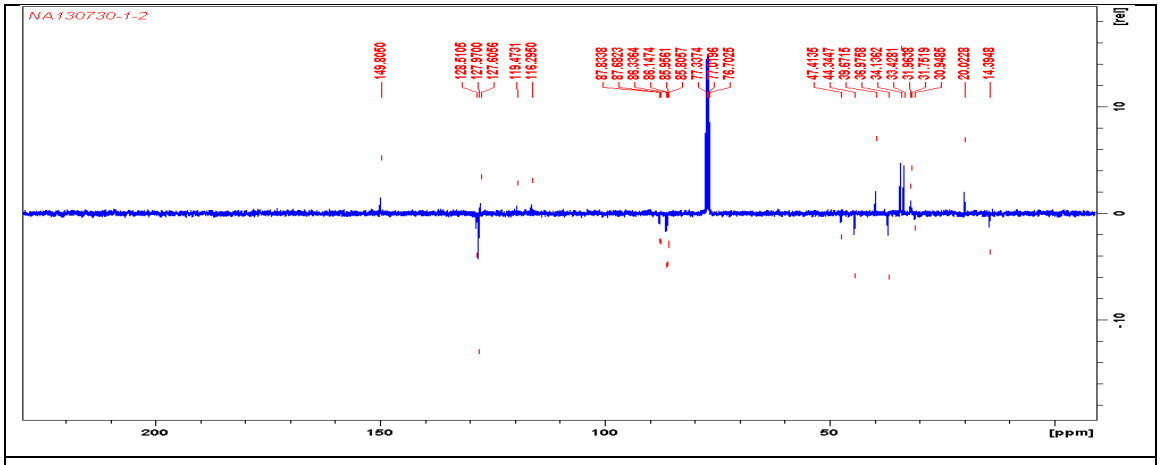


16

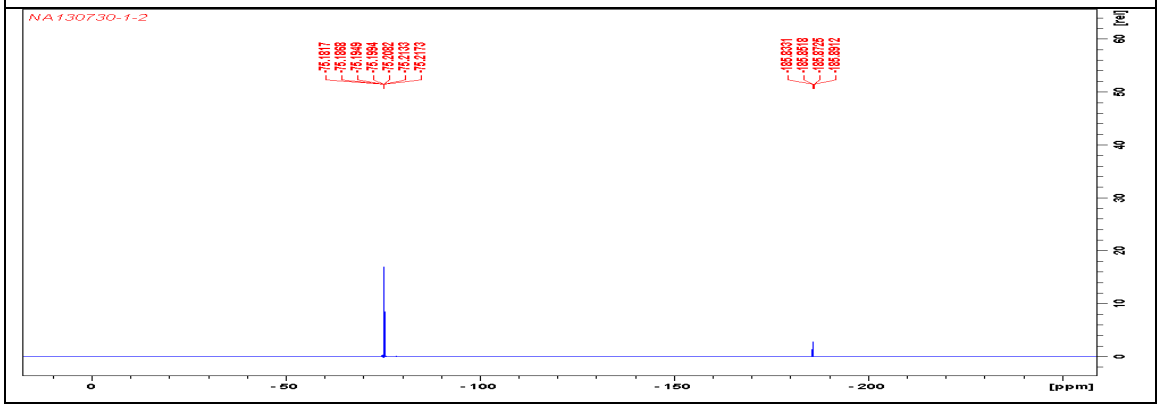
^{1}H NMR

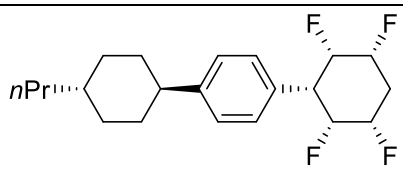


^{13}C NMR



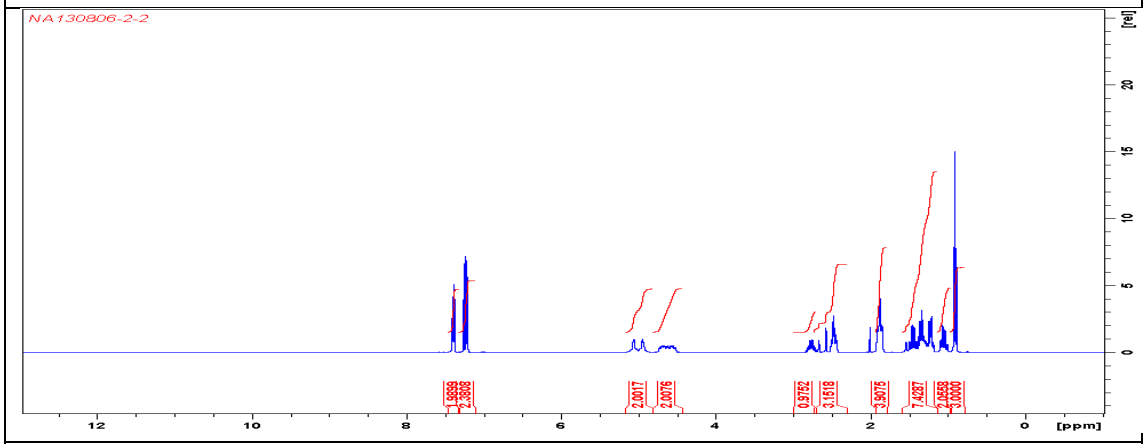
^{19}F NMR



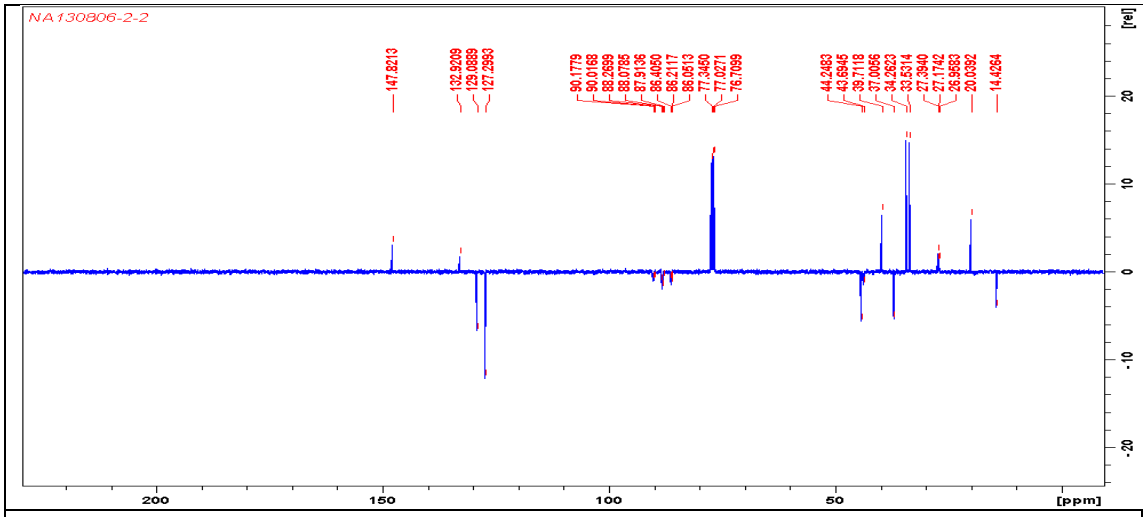


3

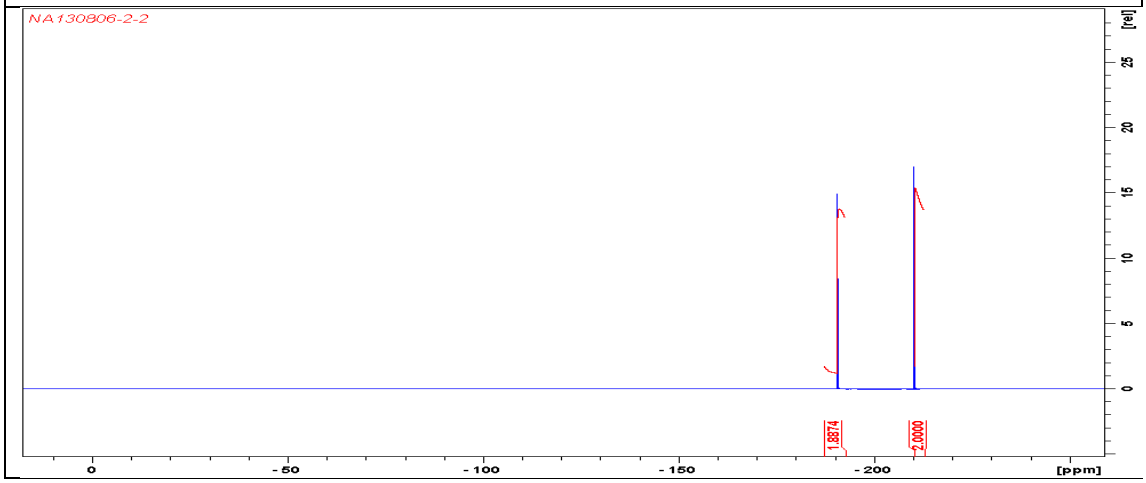
^{1}H NMR

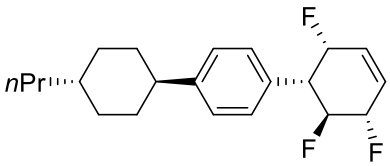


^{13}C NMR

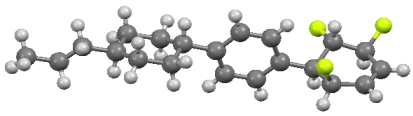


^{19}F NMR

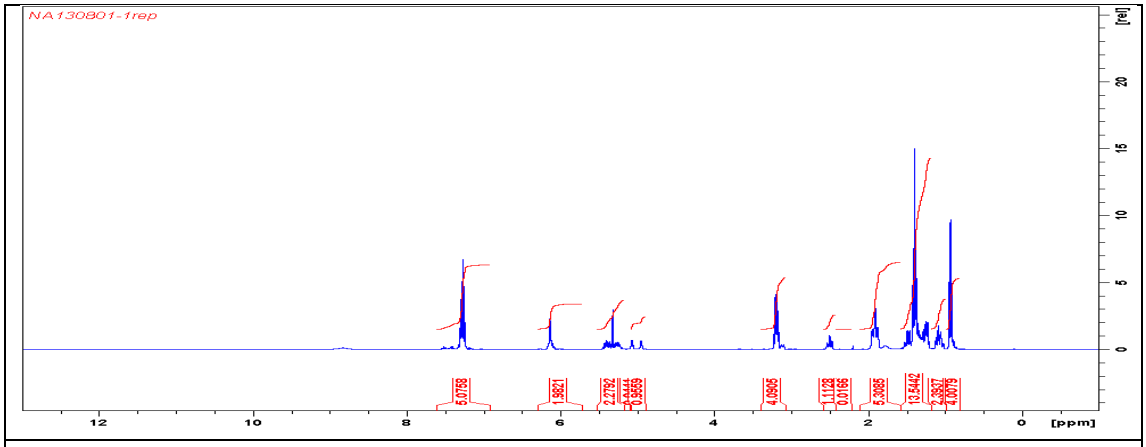




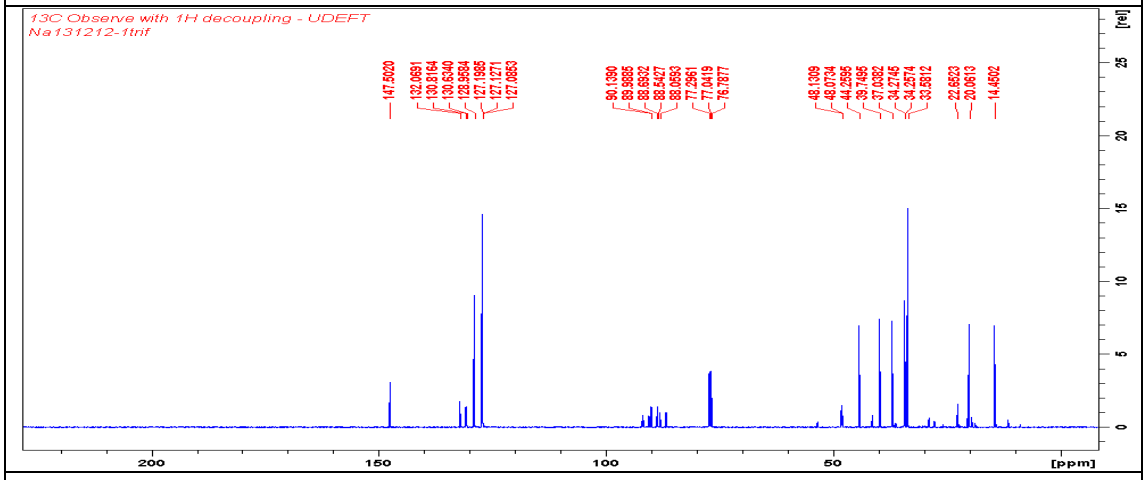
18



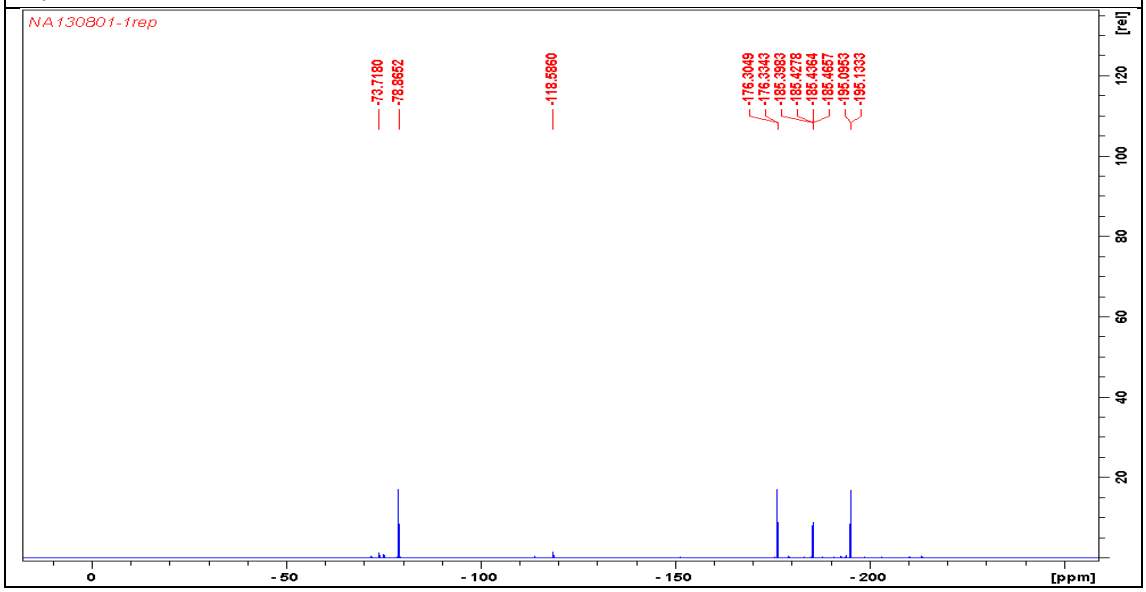
^{1}H NMR

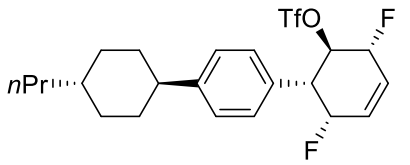


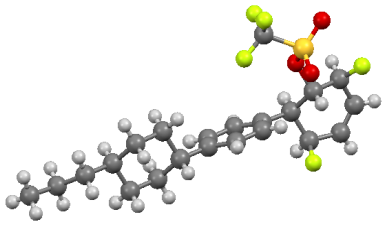
^{13}C NMR



19F NMR

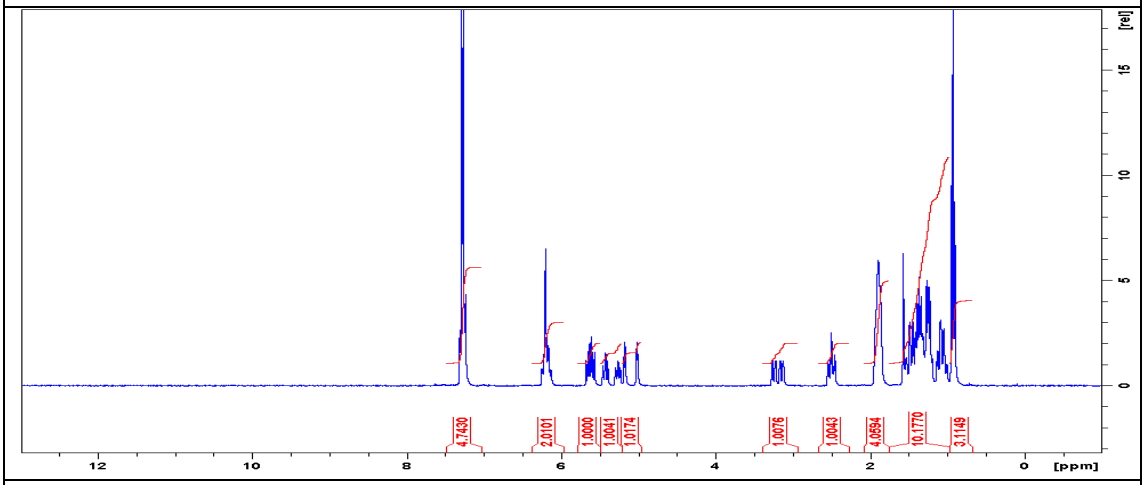




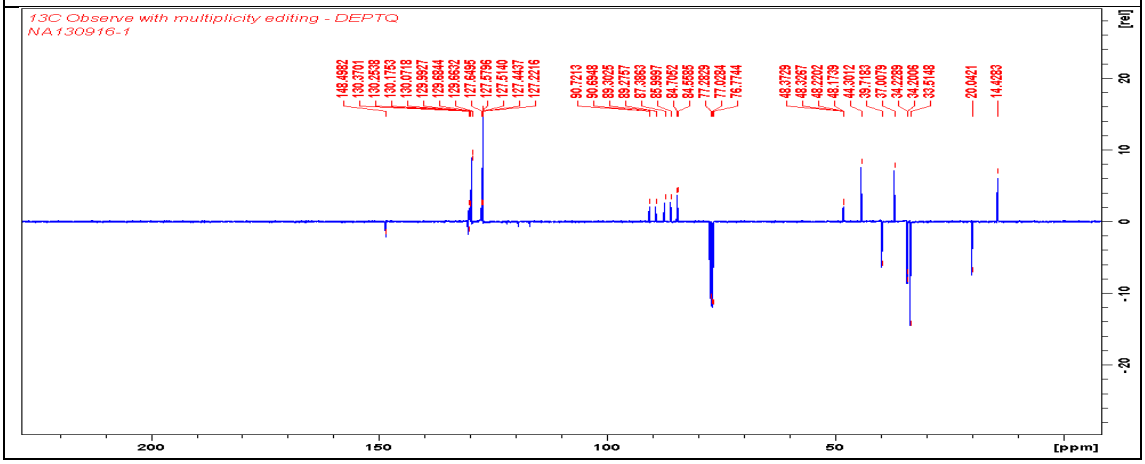


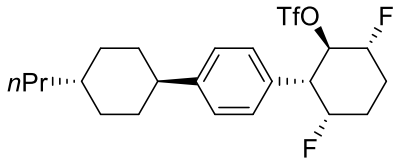
19

^{1}H NMR



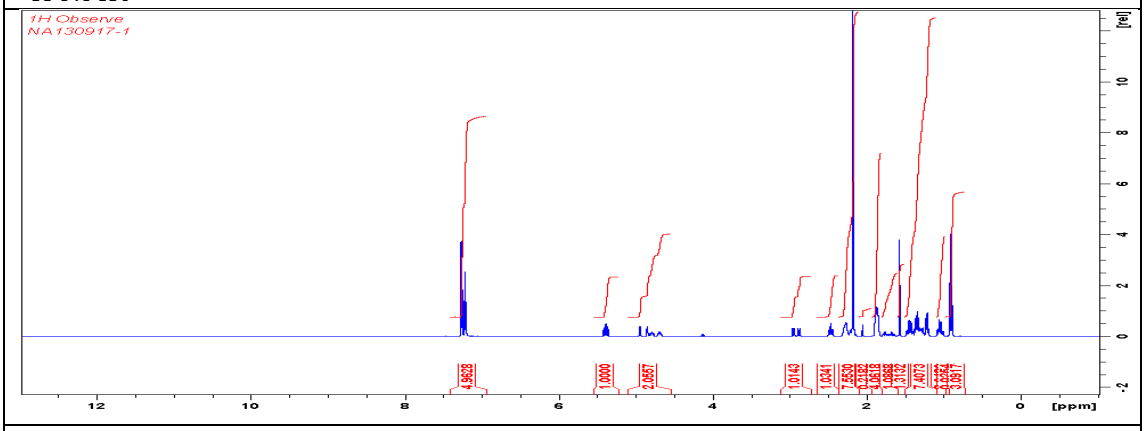
^{13}C NMR



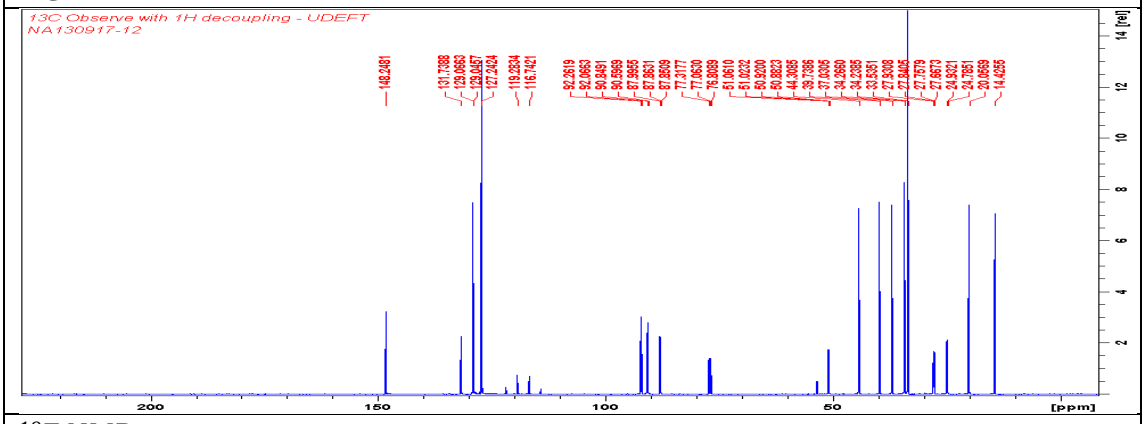


21

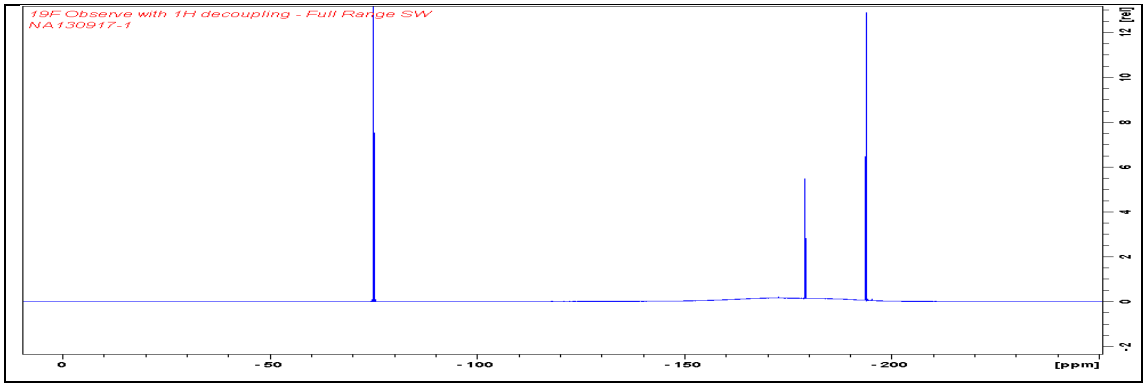
^{1}H NMR

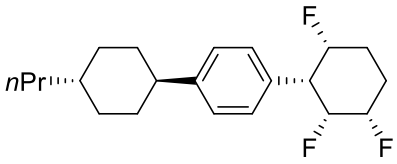


^{13}C NMR



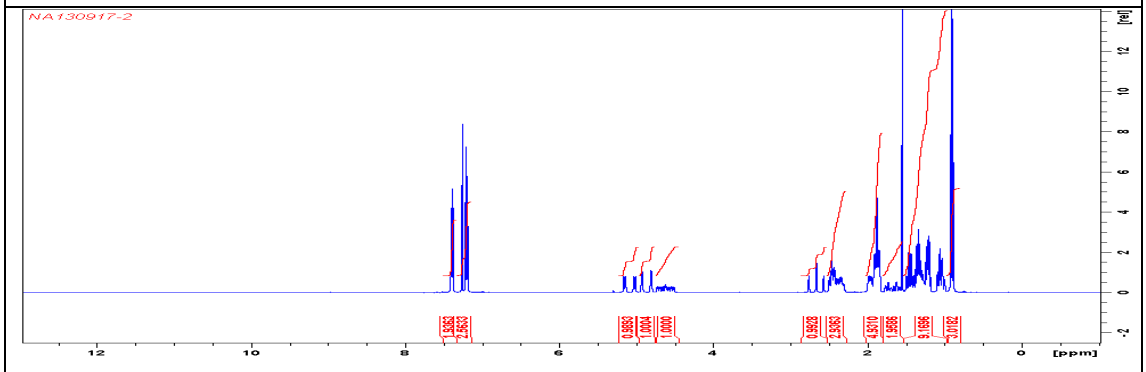
^{19}F NMR



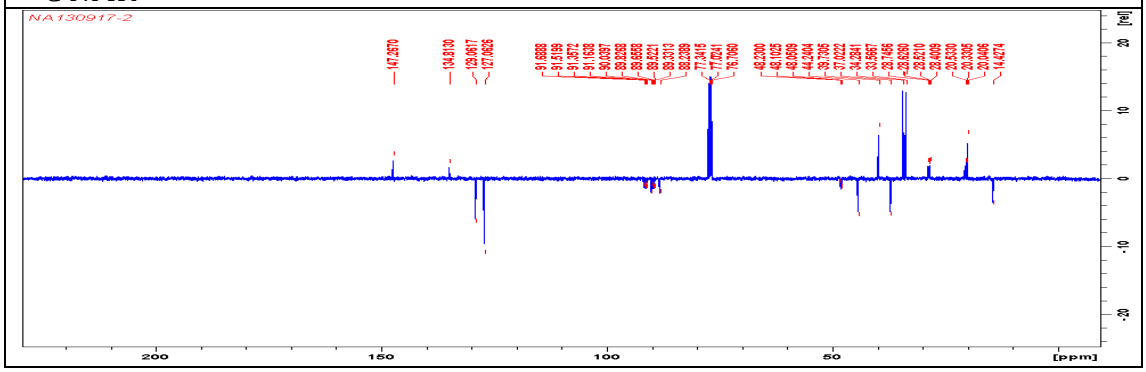


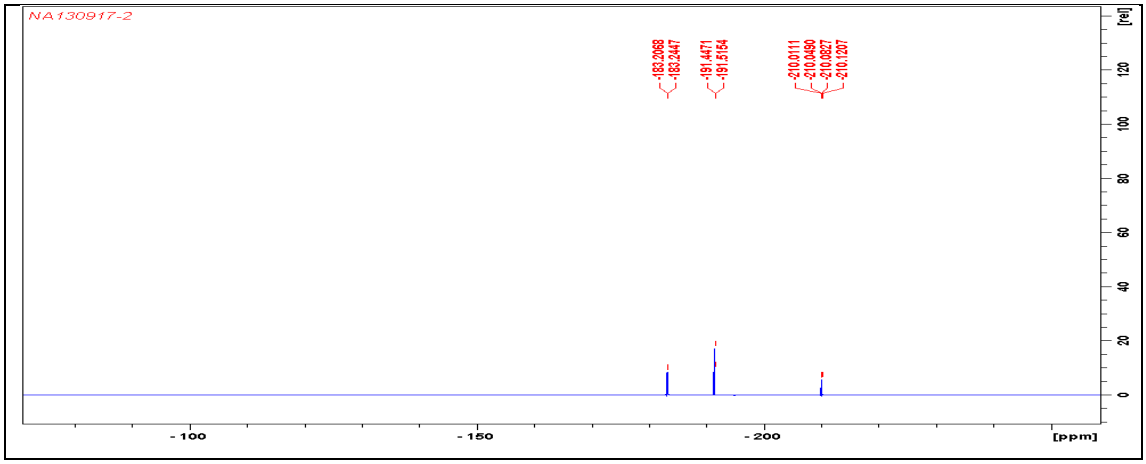
4

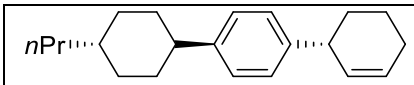
^{1}H NMR



^{13}C NMR

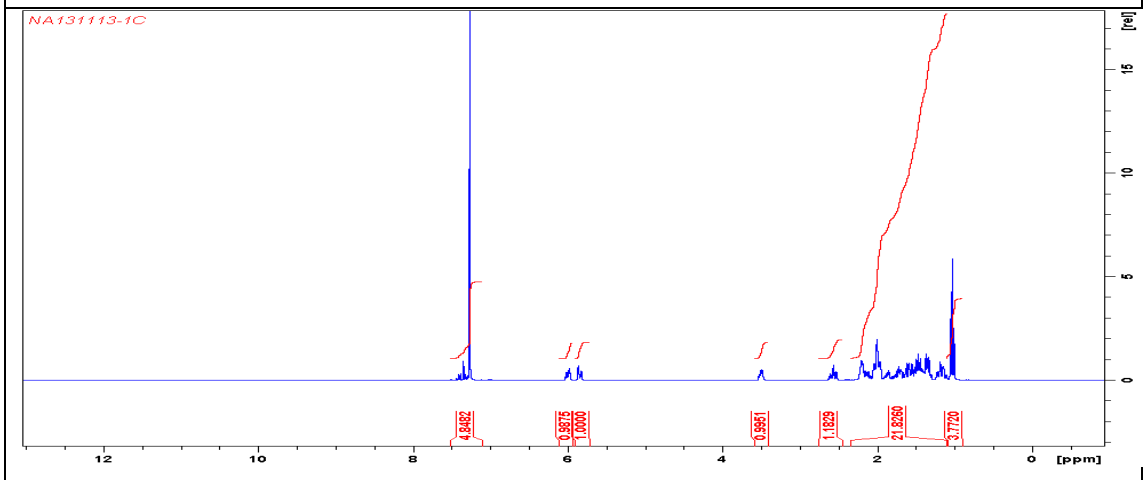




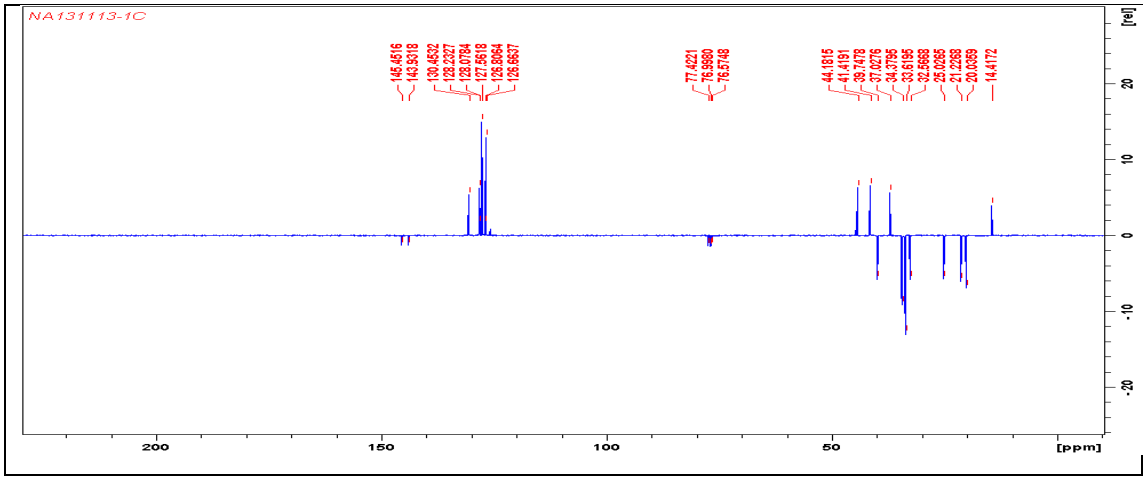


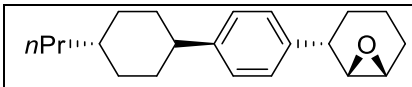
23

^{1}H NMR



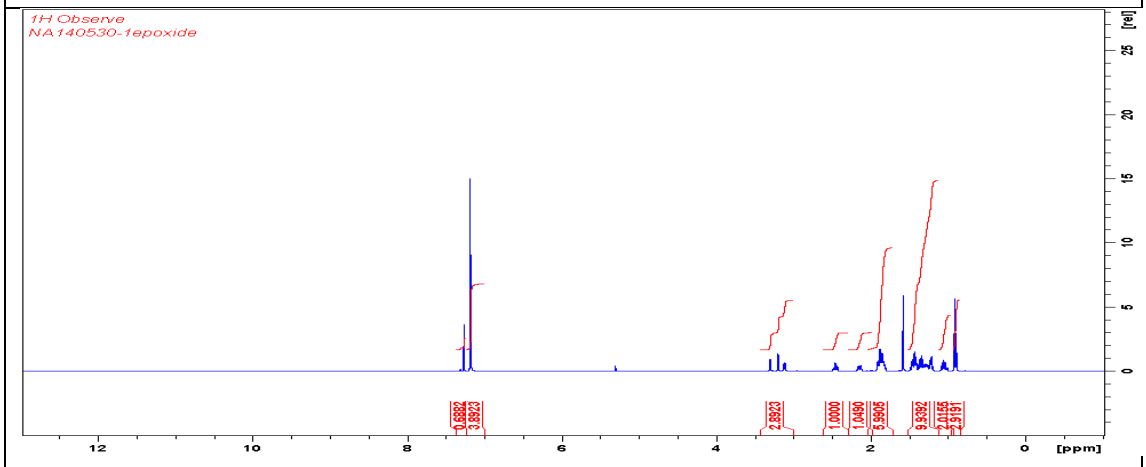
^{13}C NMR



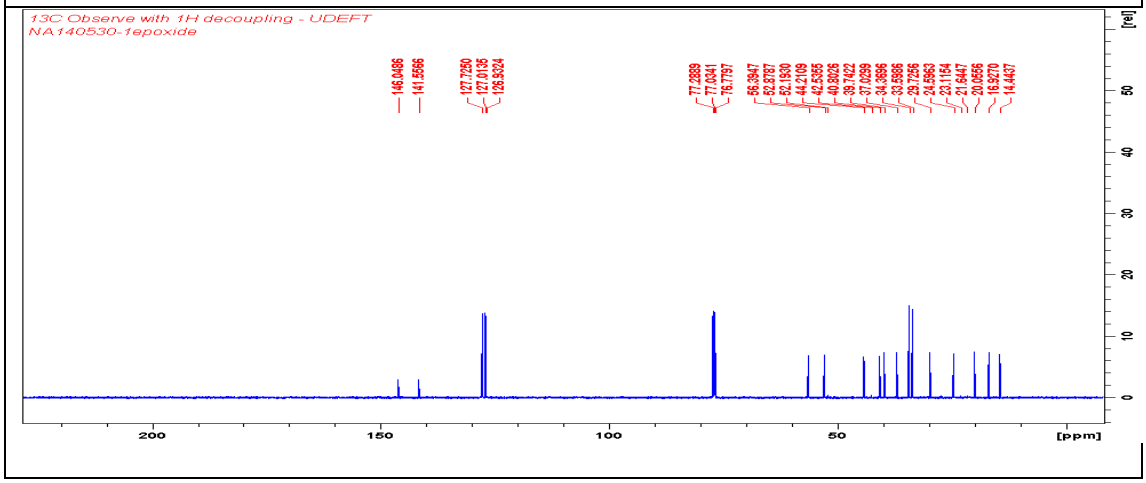


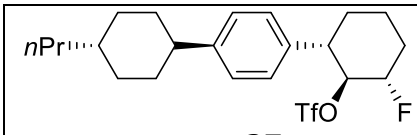
24

^{1}H NMR



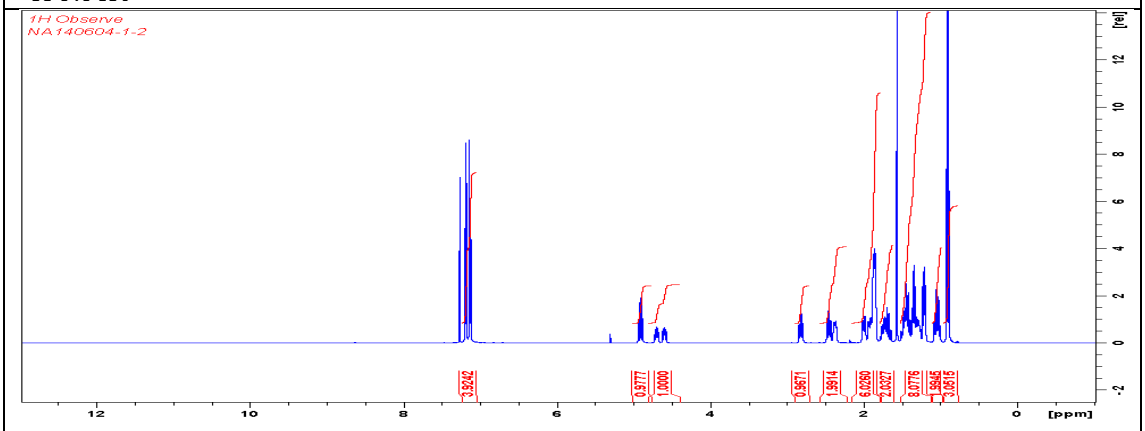
^{13}C NMR



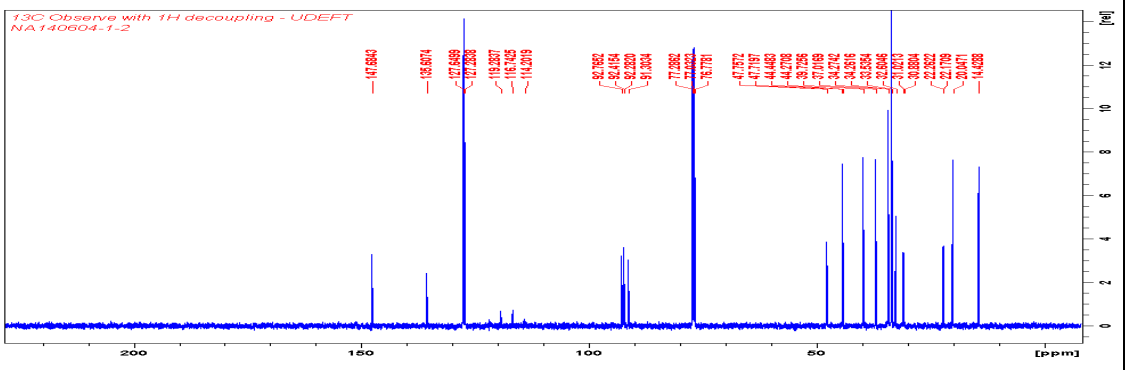


27

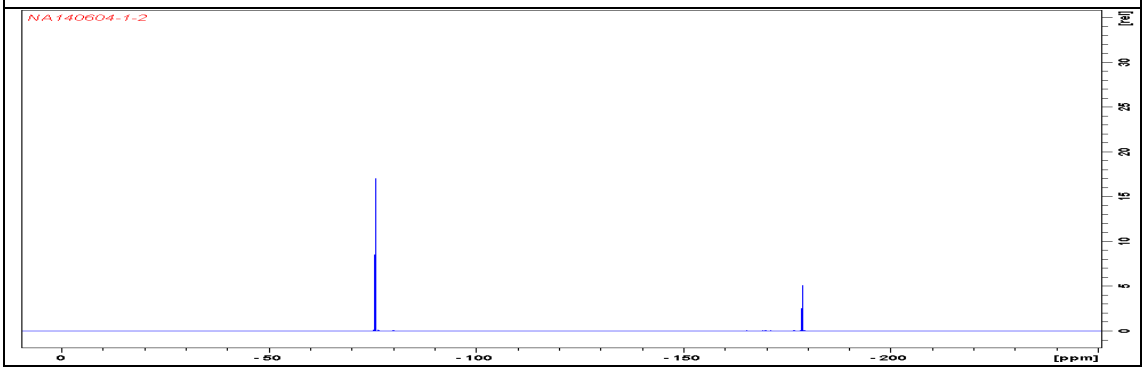
^{1}H NMR

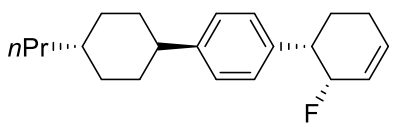


^{13}C NMR



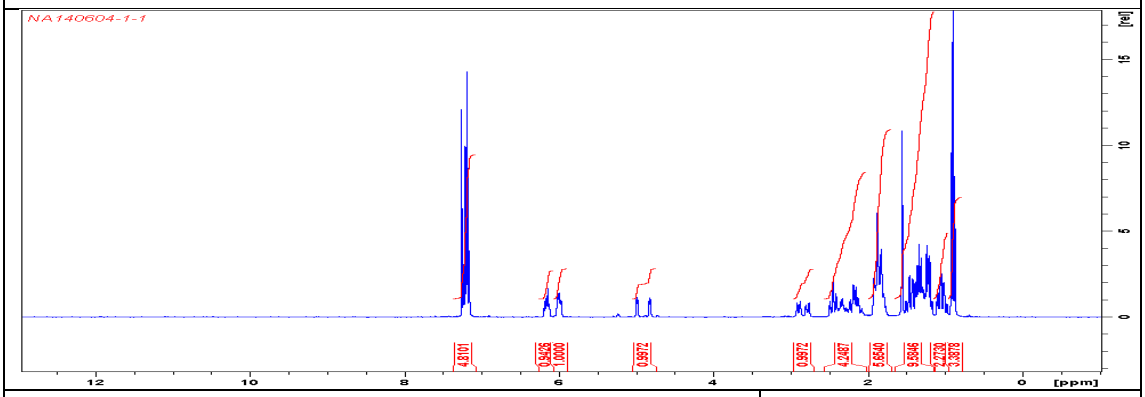
^{19}F NMR



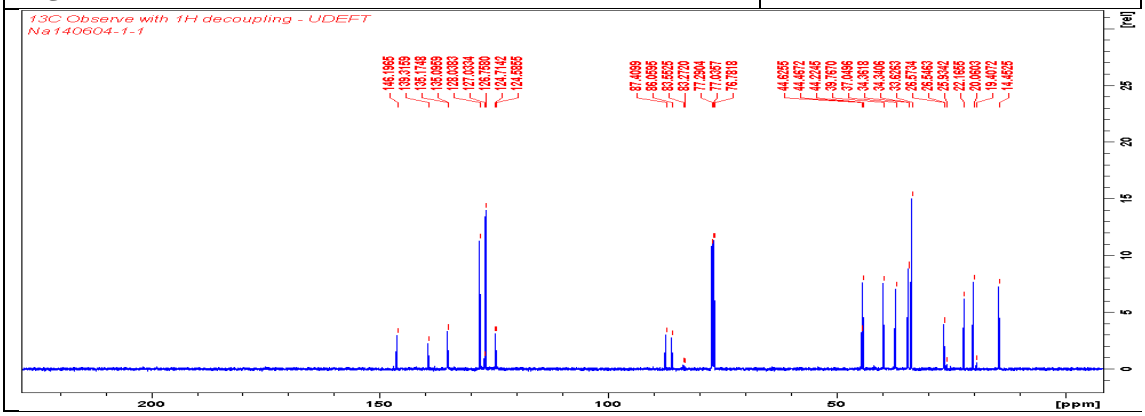


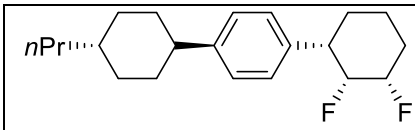
28

^{1}H NMR



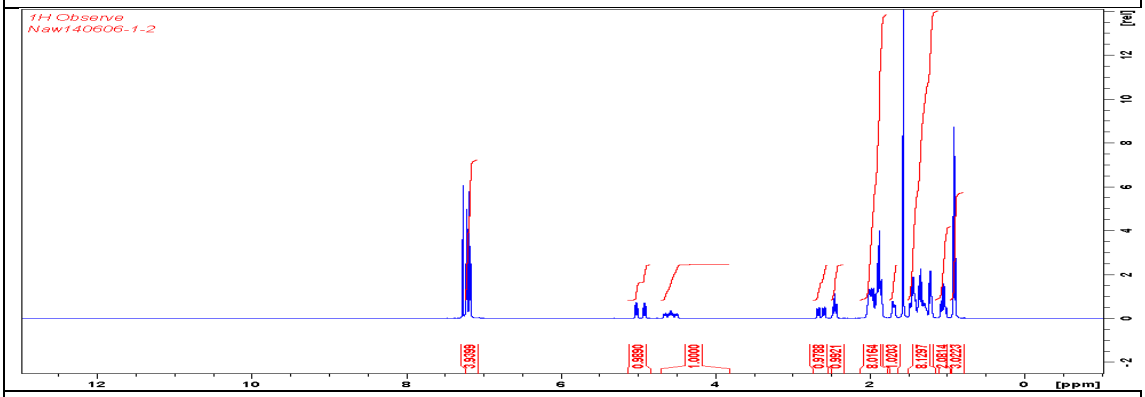
^{13}C NMR



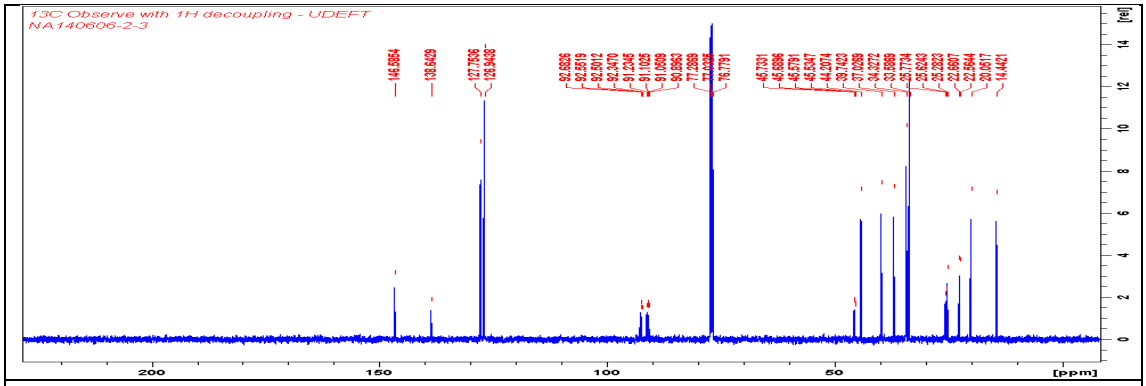


5

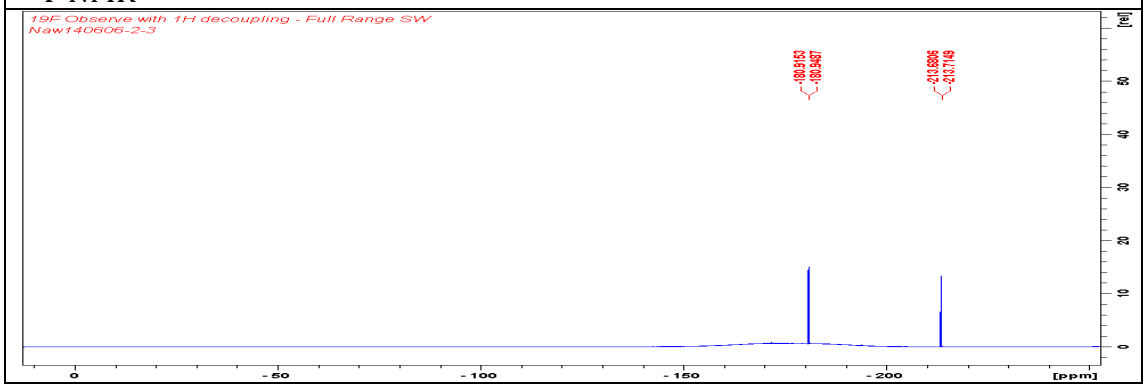
^{1}H NMR

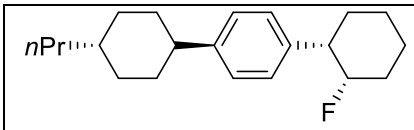


^{13}C NMR



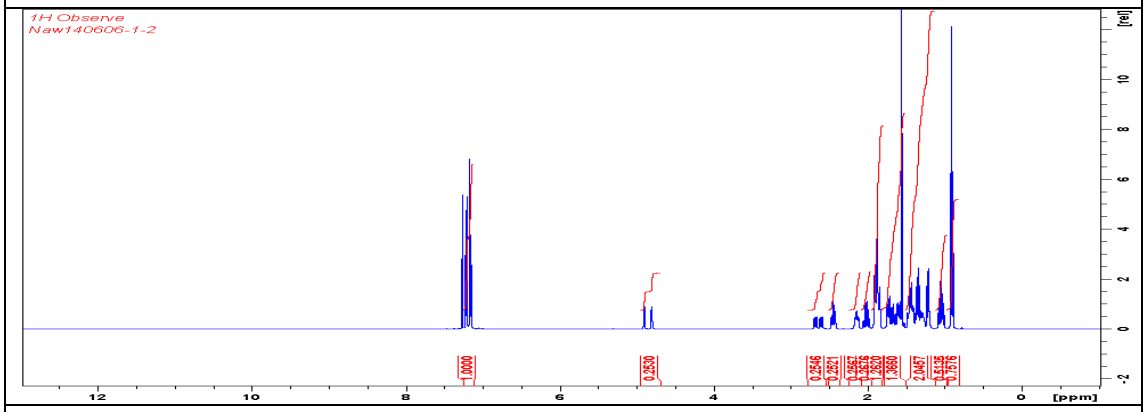
^{19}F NMR



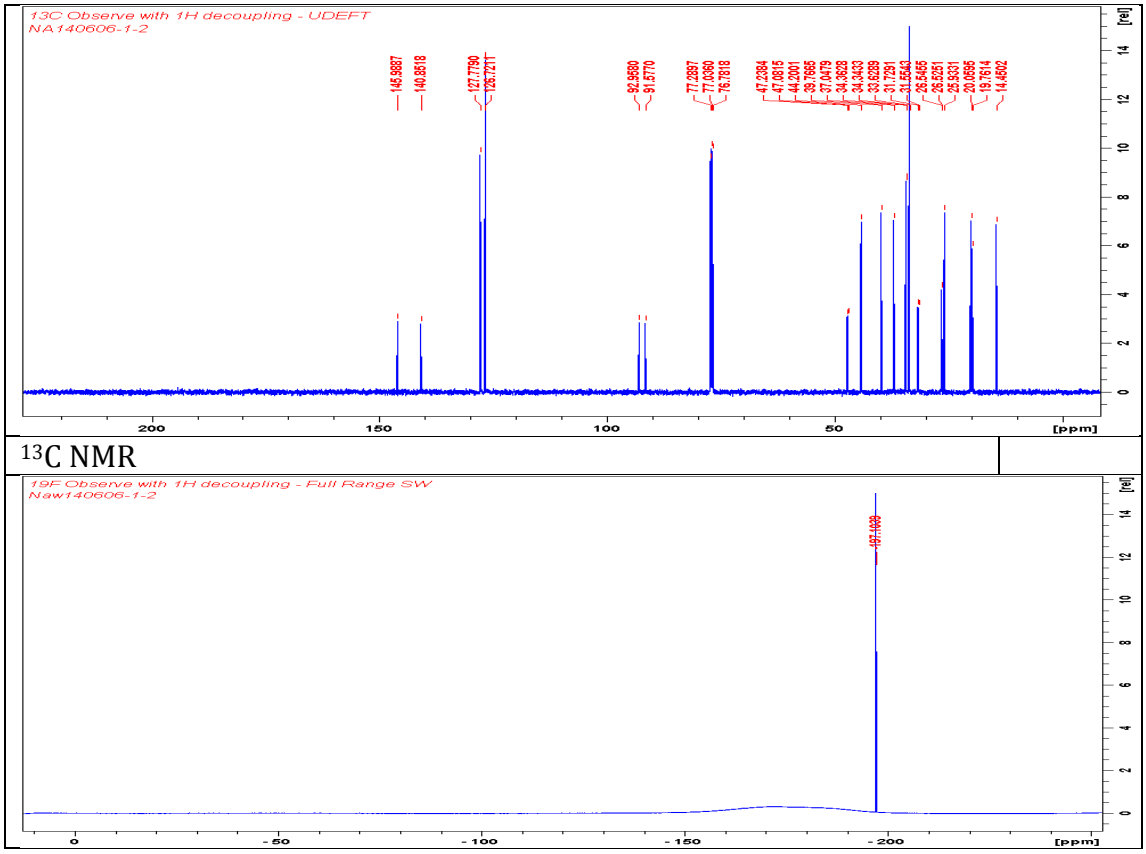


6

^{1}H NMR



^{13}C NMR


13C Observe with 1H decoupling - UDEFT
NA140606-1-2
145.9887
140.8818
127.7790
126.7211
92.9590
91.9770
77.2897
77.0360
76.7818
47.2084
47.0815
44.2001
39.7665
37.0479
34.9828
34.3433
33.8289
31.7291
31.1543
26.6455
26.6251
25.9331
20.9595
19.7614
14.4902
200
150
100
50
[ppm]
[rel]
19F Observe with 1H decoupling - Full Range SW
Naw140606-1-2
-197.1003
0
-50
-100
-150
-200
[ppm]
[rel]

^{13}C NMR

X_Ray Data

X-Ray structure data has been deposited in the Cambridge Crystallographic Data Centre (CCDC) with the following numbers are 1503420-1503427 for compounds **3-6, 13, 16, 17, 19** respectively**.**

X-Ray Data Collection for **3**

A colorless platelet crystal of $C_{21}H_{28}F_4$ having approximate dimensions of 0.200 x 0.200 x 0.020 mm was mounted in a loop. All measurements were made on a Rigaku XtaLAB P200 diffractometer using multi-layer mirror monochromated Cu-Kα radiation.

Cell constants and an orientation matrix for data collection corresponded to a primitive monoclinic cell with dimensions:

a = 11.943(2) Å
b = 5.5757(11) Å β = 91.791(7)°
c = 27.176(6) Å
V = 1808.7(6) $Å^3$

For Z = 4 and F.W. = 356.45, the calculated density is 1.309 g/cm^3. Based on the reflection conditions of:

h0l: l = 2n

packing considerations, a statistical analysis of intensity distribution, and the successful solution and refinement of the structure, the space group was determined to be:

P2/c (#13)

The data were collected at a temperature of -148 ± 1°C to a maximum 2θ value of 136.5°. Readout was performed in the 0.172 mm pixel mode.

Data Reduction

Of the 23264 reflections were collected, where 3315 were unique (R_{int} = 0.0500); equivalent reflections were merged. Data were collected and processed using CrystalClear (Rigaku). [1]

The linear absorption coefficient, μ, for Cu-Kα radiation is 8.627 cm^{-1}. An empirical absorption correction was applied which resulted in transmission

factors ranging from 0.871 to 0.983. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by direct methods[2] and expanded using Fourier techniques. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined using the riding model. The final cycle of full-matrix least-squares refinement[3] on F^2 was based on 3315 observed reflections and 227 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R1 = \Sigma \, ||Fo| - |Fc|| \, / \, \Sigma \, |Fo| = 0.0516$$

$$wR2 = [\Sigma (w (Fo^2 - Fc^2)^2) / \Sigma \, w(Fo^2)^2]^{1/2} = 0.1727$$

The goodness of fit[4] was 1.06. Unit weights were used. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.44 and -0.37 $e^-/Å^3$, respectively.

Neutral atom scattering factors were taken from International Tables for Crystallography (IT), Vol. C, Table 6.1.1.4 [5]. Anomalous dispersion effects were included in Fcalc[6]; the values for Δf' and Δf'' were those of Creagh and McAuley[7]. The values for the mass attenuation coefficients are those of Creagh and Hubbell[8]. All calculations were performed using the CrystalStructure[9] crystallographic software package except for refinement, which was performed using SHELXL2013[10].

References

(1) CrystalClear: Data Collection and Processing Software, Rigaku Corporation (1998-2014). Tokyo 196-8666, Japan.

(2) SIR2011: Burla, M. C., Caliandro, R., Camalli, M., Carrozzini, B., Cascarano, G. L., Giacovazzo, C., Mallamo, M., Mazzone, A., Polidori, G. and Spagna, R. (2012). J. Appl. Cryst. 45, 357-361.

(3) Least Squares function minimized: (SHELXL2013)

$\Sigma w(F_o^2 - F_c^2)^2$ where w = Least Squares weights.

(4) Goodness of fit is defined as:

$$[\Sigma w(F_o^2\text{-}F_c^2)^2/(N_o\text{-}N_v)]^{1/2}$$

where: N_o = number of observations
N_v = number of variables

(5) International Tables for Crystallography, Vol.C (1992). Ed. A.J.C. Wilson, Kluwer Academic Publishers, Dordrecht, Netherlands, Table 6.1.1.4, pp. 572.

(6) Ibers, J. A. & Hamilton, W. C.; Acta Crystallogr., 17, 781 (1964).

(7) Creagh, D. C. & McAuley, W.J .; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.6.8, pages 219-222 (1992).

(8) Creagh, D. C. & Hubbell, J.H..; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.4.3, pages 200-206 (1992).

(9) CrystalStructure 4.1: Crystal Structure Analysis Package, Rigaku Corporation (2000-2014). Tokyo 196-8666, Japan.

(10) SHELXL2013: Sheldrick, G. M. (2008). Acta Cryst. A64, 112-122.

A. Crystal Data

Empirical Formula	$C_{21}H_{28}F_4$
Formula Weight	356.45
Crystal Color, Habit	colorless, platelet
Crystal Dimensions	0.200 X 0.200 X 0.020 mm
Crystal System	monoclinic
Lattice Type	Primitive
Lattice Parameters	a = 11.943(2) Å b = 5.5757(11) Å c = 27.176(6) Å β = 91.791(7) ° V = 1808.7(6) $Å^3$
Space Group	P2/c (#13)
Z value	4
D_{calc}	1.309 g/cm^3
F_{000}	760.00
μ(CuKα)	8.627 cm^{-1}

B. Intensity Measurements

Diffractometer	XtaLAB P200
Radiation monochromated	CuKα (λ = 1.54187 Å) multi-layer mirror
Voltage, Current	40kV, 30mA
Temperature	-148.0°C
Detector Aperture	83.8 x 70.0 mm
Pixel Size	0.172 mm
$2\theta_{max}$	136.5°
No. of Reflections Measured	Total: 23264 Unique: 3315 (R_{int} = 0.0500)
Corrections	Lorentz-polarization Absorption (trans. factors: 0.871 - 0.983)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SIR2011)
Refinement	Full-matrix least-squares on F^2
Function Minimized	Σ w $(Fo^2 - Fc^2)^2$
Least Squares Weights	w = 1/ [$\sigma^2(Fo^2)$ + $(0.1323 \cdot P)^2$ + 0.3158 · P] where P = (Max(Fo^2,0) + $2Fc^2$)/3
$2\theta_{max}$ cutoff	136.5º
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (All reflections)	3315
No. Variables	227
Reflection/Parameter Ratio	14.60
Residuals: R1 (I>2.00σ(I))	0.0516
Residuals: R (All reflections)	0.0537
Residuals: wR2 (All reflections)	0.1727
Goodness of Fit Indicator	1.059
Max Shift/Error in Final Cycle	0.001
Maximum peak in Final Diff. Map	0.44 $e^-/Å^3$
Minimum peak in Final Diff. Map	-0.37 $e^-/Å^3$

Table 1. Atomic coordinates and B_{iso}/B_{eq}

atom	x	y	z	B_{eq}
F1	0.43852(8)	0.13875(14)	0.33544(4)	2.83(2)
F2	0.65333(8)	0.13964(17)	0.36736(3)	2.83(2)
F3	0.43928(9)	0.6803(2)	0.46102(3)	3.86(3)
F4	0.31097(7)	0.45189(18)	0.39089(3)	2.57(2)
C1	0.50295(13)	0.3428(3)	0.32672(5)	1.90(3)
C2	0.59347(12)	0.3559(3)	0.36672(5)	1.88(3)
C3	0.54730(13)	0.3993(3)	0.41714(5)	2.03(3)
C4	0.48291(13)	0.6339(3)	0.41502(5)	2.20(3)
C5	0.38734(12)	0.6272(3)	0.37701(5)	1.80(3)
C6	0.43050(11)	0.5689(2)	0.32605(5)	1.46(3)
C7	0.33780(11)	0.5732(2)	0.28620(5)	1.52(3)
C8	0.25458(12)	0.3983(3)	0.28187(5)	1.83(3)
C9	0.16939(12)	0.4166(3)	0.24558(5)	1.80(3)
C10	0.16275(11)	0.6100(3)	0.21329(5)	1.64(3)
C11	0.24641(11)	0.7844(3)	0.21775(5)	1.78(3)
C12	0.33186(11)	0.7665(3)	0.25359(5)	1.65(3)
C13	0.07032(11)	0.6261(3)	0.17361(5)	1.72(3)
C14	0.11568(12)	0.5729(3)	0.12267(5)	1.90(3)
C15	0.02322(13)	0.5864(3)	0.08243(5)	2.06(3)
C16	-0.03631(12)	0.8296(3)	0.08148(5)	1.73(3)
C17	-0.08203(12)	0.8803(3)	0.13229(5)	1.96(3)
C18	0.01013(12)	0.8692(3)	0.17294(5)	2.00(3)
C19	-0.12715(12)	0.8371(3)	0.04013(5)	1.91(3)
C20	-0.17840(15)	1.0820(3)	0.03095(6)	2.69(3)
C21	-0.26365(14)	1.0872(3)	-0.01213(6)	2.86(3)

$B_{eq} = 8/3\ \pi^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$

Table 2. Atomic coordinates and B_{iso} involving hydrogen atoms

atom	x	y	z	B_{iso}
H1	0.53877	0.32571	0.29413	2.280
H2	0.64585	0.48956	0.35891	2.254
H3A	0.60921	0.40908	0.44215	2.441
H3B	0.49705	0.26619	0.42615	2.441
H4	0.53537	0.76634	0.40653	2.636
H5	0.34920	0.78685	0.37596	2.156
H6	0.48207	0.70344	0.31788	1.758
H8	0.25578	0.26554	0.30378	2.197
H9	0.11452	0.29350	0.24292	2.162
H11	0.24497	0.91755	0.19593	2.138
H12	0.38732	0.88848	0.25590	1.985
H13	0.01349	0.50015	0.18080	2.067
H14A	0.14943	0.41077	0.12274	2.276
H14B	0.17518	0.69010	0.11535	2.276
H15A	0.05635	0.55735	0.05002	2.478
H15B	-0.03239	0.45829	0.08800	2.478
H16	0.02054	0.95558	0.07440	2.081
H17A	-0.14087	0.76137	0.13943	2.355
H17B	-0.11686	1.04146	0.13224	2.355
H18A	0.06533	0.99836	0.16758	2.400
H18B	-0.02355	0.89693	0.20528	2.400
H19A	-0.09405	0.78030	0.00928	2.289
H19B	-0.18757	0.72382	0.04841	2.289
H20A	-0.11777	1.19770	0.02439	3.225
H20B	-0.21562	1.13514	0.06112	3.225
H21A	-0.32696	0.98266	-0.00482	3.434
H21B	-0.22811	1.03071	-0.04201	3.434
H21C	-0.29058	1.25170	-0.01719	3.434

Table 3. Anisotropic displacement parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
F1	0.0471(6)	0.0185(5)	0.0407(6)	-0.0012(4)	-0.0185(4)	-0.0009(4)
F2	0.0412(6)	0.0372(6)	0.0288(5)	0.0209(4)	-0.0034(4)	-0.0027(4)
F3	0.0592(7)	0.0688(8)	0.0182(5)	0.0299(6)	-0.0052(4)	-0.0133(5)
F4	0.0283(5)	0.0448(6)	0.0249(5)	-0.0031(4)	0.0050(3)	0.0085(4)
C1	0.0314(8)	0.0231(7)	0.0177(7)	0.0047(6)	-0.0008(6)	-0.0020(5)
C2	0.0245(7)	0.0240(7)	0.0228(8)	0.0069(5)	-0.0011(6)	0.0002(5)
C3	0.0301(8)	0.0293(8)	0.0175(7)	0.0058(6)	-0.0046(5)	-0.0010(5)
C4	0.0338(8)	0.0316(8)	0.0176(8)	0.0080(6)	-0.0051(6)	-0.0081(6)
C5	0.0238(7)	0.0250(7)	0.0194(8)	0.0018(5)	-0.0003(5)	-0.0010(5)
C6	0.0210(7)	0.0188(7)	0.0157(7)	-0.0009(5)	-0.0005(5)	0.0010(5)
C7	0.0228(7)	0.0211(7)	0.0140(7)	0.0008(5)	0.0001(5)	-0.0015(5)
C8	0.0279(7)	0.0220(7)	0.0195(7)	-0.0015(5)	-0.0020(5)	0.0041(5)
C9	0.0248(7)	0.0217(7)	0.0217(7)	-0.0039(5)	-0.0019(5)	0.0001(5)
C10	0.0232(7)	0.0243(7)	0.0150(7)	-0.0004(5)	0.0003(5)	-0.0020(5)
C11	0.0270(7)	0.0236(7)	0.0170(7)	-0.0012(5)	-0.0008(5)	0.0038(5)
C12	0.0233(7)	0.0210(7)	0.0185(7)	-0.0024(5)	-0.0006(5)	-0.0002(5)
C13	0.0227(7)	0.0255(7)	0.0171(7)	-0.0031(5)	-0.0023(5)	0.0013(5)
C14	0.0266(7)	0.0268(7)	0.0184(7)	0.0035(5)	-0.0033(5)	-0.0033(5)
C15	0.0314(8)	0.0300(8)	0.0167(7)	0.0047(6)	-0.0054(6)	-0.0060(5)
C16	0.0244(7)	0.0268(7)	0.0145(7)	-0.0006(5)	-0.0026(5)	-0.0017(5)
C17	0.0238(7)	0.0335(8)	0.0171(7)	0.0048(6)	-0.0028(5)	-0.0026(5)
C18	0.0291(8)	0.0317(8)	0.0150(7)	0.0041(6)	-0.0027(6)	-0.0045(5)

C19	0.0275(7)	0.0290(8)	0.0158(7)	-0.0008(5)	-0.0037(5)	-0.0024(5)
C20	0.0396(9)	0.0330(9)	0.0286(8)	0.0040(7)	-0.0134(7)	-0.0018(6)
C21	0.0349(9)	0.0410(9)	0.0320(9)	-0.0029(7)	-0.0122(7)	0.0090(7)

The general temperature factor expression: $\exp(-2\pi^2(a^{*2}U_{11}h^2 + b^{*2}U_{22}k^2 + c^{*2}U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$

Table 4. Bond lengths (Å)

atom	atom	distance	atom	atom	distance
F1	C1	1.3979(17)	F2	C2	1.4017(17)
F3	C4	1.3935(17)	F4	C5	1.3968(17)
C1	C2	1.511(2)	C1	C6	1.529(2)
C2	C3	1.512(2)	C3	C4	1.518(2)
C4	C5	1.516(2)	C5	C6	1.528(2)
C6	C7	1.5245(19)	C7	C8	1.3946(19)
C7	C12	1.3958(19)	C8	C9	1.398(2)
C9	C10	1.391(2)	C10	C11	1.3968(19)
C10	C13	1.5215(19)	C11	C12	1.3921(19)
C13	C14	1.531(2)	C13	C18	1.534(2)
C14	C15	1.531(2)	C15	C16	1.531(2)
C16	C17	1.527(2)	C16	C19	1.538(2)
C17	C18	1.536(2)	C19	C20	1.514(2)
C20	C21	1.528(2)			

Table 5. Bond lengths involving hydrogens (Å)

atom	atom	distance	atom	atom	distance
C1	H1	1.000	C2	H2	1.000
C3	H3A	0.990	C3	H3B	0.990
C4	H4	1.000	C5	H5	1.000
C6	H6	1.000	C8	H8	0.950
C9	H9	0.950	C11	H11	0.950
C12	H12	0.950	C13	H13	1.000
C14	H14A	0.990	C14	H14B	0.990
C15	H15A	0.990	C15	H15B	0.990
C16	H16	1.000	C17	H17A	0.990
C17	H17B	0.990	C18	H18A	0.990
C18	H18B	0.990	C19	H19A	0.990
C19	H19B	0.990	C20	H20A	0.990
C20	H20B	0.990	C21	H21A	0.980
C21	H21B	0.980	C21	H21C	0.980

Table 6. Bond angles (°)

atom	atom	atom	angle	atom	atom	atom	angle
F1	C1	C2	107.62(11)	F1	C1	C6	111.03(12)
C2	C1	C6	111.14(11)	F2	C2	C1	108.69(11)
F2	C2	C3	109.06(11)	C1	C2	C3	112.75(12)
C2	C3	C4	107.58(11)	F3	C4	C3	109.21(12)
F3	C4	C5	108.74(12)	C3	C4	C5	111.98(12)
F4	C5	C4	108.51(11)	F4	C5	C6	109.73(11)
C4	C5	C6	110.78(12)	C1	C6	C5	111.81(11)
C1	C6	C7	114.81(11)	C5	C6	C7	112.49(11)
C6	C7	C8	123.28(12)	C6	C7	C12	118.99(12)
C8	C7	C12	117.67(12)	C7	C8	C9	120.63(13)
C8	C9	C10	121.82(13)	C9	C10	C11	117.29(12)
C9	C10	C13	121.24(12)	C11	C10	C13	121.44(12)
C10	C11	C12	121.17(13)	C7	C12	C11	121.40(13)
C10	C13	C14	111.10(11)	C10	C13	C18	112.94(11)
C14	C13	C18	109.82(11)	C13	C14	C15	111.63(12)
C14	C15	C16	112.30(12)	C15	C16	C17	109.14(11)
C15	C16	C19	110.67(11)	C17	C16	C19	113.06(12)
C16	C17	C18	111.99(12)	C13	C18	C17	111.65(11)
C16	C19	C20	114.66(12)	C19	C20	C21	113.56(13)

Table 7. Bond angles involving hydrogens (°)

atom	atom	atom	angle	atom	atom	atom	angle
F1	C1	H1	109.0	C2	C1	H1	109.0
C6	C1	H1	109.0	F2	C2	H2	108.8
C1	C2	H2	108.8	C3	C2	H2	108.7
C2	C3	H3A	110.2	C2	C3	H3B	110.2
C4	C3	H3A	110.2	C4	C3	H3B	110.2
H3A	C3	H3B	108.5	F3	C4	H4	108.9
C3	C4	H4	109.0	C5	C4	H4	109.0
F4	C5	H5	109.3	C4	C5	H5	109.3
C6	C5	H5	109.3	C1	C6	H6	105.6
C5	C6	H6	105.6	C7	C6	H6	105.6
C7	C8	H8	119.7	C9	C8	H8	119.7
C8	C9	H9	119.1	C10	C9	H9	119.1
C10	C11	H11	119.4	C12	C11	H11	119.4
C7	C12	H12	119.3	C11	C12	H12	119.3
C10	C13	H13	107.6	C14	C13	H13	107.6
C18	C13	H13	107.6	C13	C14	H14A	109.3
C13	C14	H14B	109.3	C15	C14	H14A	109.3
C15	C14	H14B	109.3	H14A	C14	H14B	108.0
C14	C15	H15A	109.1	C14	C15	H15B	109.1
C16	C15	H15A	109.1	C16	C15	H15B	109.1
H15A	C15	H15B	107.9	C15	C16	H16	107.9
C17	C16	H16	107.9	C19	C16	H16	107.9

C16	C17	H17A	109.2	C16	C17	H17B
	109.2					
C18	C17	H17A	109.2	C18	C17	H17B
	109.2					
H17A	C17	H17B	107.9	C13	C18	H18A
	109.3					
C13	C18	H18B	109.3	C17	C18	H18A
	109.3					
C17	C18	H18B	109.3	H18A	C18	H18B
	107.9					
C16	C19	H19A	108.6	C16	C19	H19B
	108.6					
C20	C19	H19A	108.6	C20	C19	H19B
	108.6					
H19A	C19	H19B	107.6	C19	C20	H20A
	108.9					
C19	C20	H20B	108.8	C21	C20	H20A
	108.9					
C21	C20	H20B	108.9	H20A	C20	H20B
	107.7					
C20	C21	H21A	109.5	C20	C21	H21B
	109.5					
C20	C21	H21C	109.5	H21A	C21	H21B
	109.5					
H21A	C21	H21C	109.5	H21B	C21	H21C
	109.5					

Table 8. Torsion Angles(º)
(Those having bond angles > 160 or < 20 degrees are excluded.)

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
F1	C1	C2	F2	55.13(14)	F1	C1	C2	C3	-65.91(13)
F1	C1	C6	C5	68.55(13)	F1	C1	C6	C7	-61.16(14)
C2	C1	C6	C5	-51.21(14)	C2	C1	C6	C7	179.08(10)
C6	C1	C2	F2	176.90(10)	C6	C1	C2	C3	55.85(14)
F2	C2	C3	C4	-179.84(9)	C1	C2	C3	C4	-59.00(14)
C2	C3	C4	F3	-179.83(11)	C2	C3	C4	C5	59.67(14)
F3	C4	C5	F4	-57.69(14)	F3	C4	C5	C6	-178.20(10)
C3	C4	C5	F4	63.09(14)	C3	C4	C5	C6	-57.42(14)
F4	C5	C6	C1	-67.71(13)	F4	C5	C6	C7	63.21(13)
C4	C5	C6	C1	52.08(14)	C4	C5	C6	C7	-177.00(10)
C1	C6	C7	C8	57.82(16)	C1	C6	C7	C12	-125.27(12)
C5	C6	C7	C8	-71.56(15)	C5	C6	C7	C12	105.35(13)
C6	C7	C8	C9	177.72(11)	C6	C7	C12	C11	-177.44(11)
C8	C7	C12	C11	-0.36(19)	C12	C7	C8	C9	0.77(19)
C7	C8	C9	C10	-1.3(2)	C8	C9	C10	C11	1.4(2)
C8	C9	C10	C13	179.31(11)	C9	C10	C11	C12	-0.94(19)
C9	C10	C13	C14	-105.93(14)	C9	C10	C13	C18	130.16(13)
C11	C10	C13	C14	71.93(16)	C11	C10	C13	C18	-51.99(16)
C13	C10	C11	C12	-178.88(11)	C10	C11	C12	C7	0.5(2)
C10	C13	C14	C15	179.49(10)	C10	C13	C18	C17	179.60(10)
C14	C13	C18	C17	54.99(14)	C18	C13	C14	C15	-54.84(14)

C13	C14	C15	C16	56.62(15)	C14	C15	C16	C17	-55.84(14)
C14	C15	C16	C19	179.13(10)	C15	C16	C17	C18	55.79(14)
C15	C16	C19	C20	-170.89(10)	C17	C16	C19	C20	66.33(15)
C19	C16	C17	C18	179.42(10)	C16	C17	C18	C13	-56.81(15)
C16	C19	C20	C21	176.81(11)					

Table 9. Intramolecular contacts less than 3.60 Å

atom	atom	distance	atom	atom	distance
F1	F2	2.6823(14)	F1	F4	2.7898(14)
F1	C3	2.9236(18)	F1	C4	3.5369(18)
F1	C5	3.0182(17)	F1	C7	3.0007(16)
F1	C8	2.9732(17)	F3	F4	2.7239(13)
F4	C1	2.9862(18)	F4	C2	3.4984(18)
F4	C3	2.9043(18)	F4	C7	2.9515(17)
F4	C8	3.0324(17)	C1	C4	2.913(2)
C1	C8	3.186(2)	C2	C5	2.910(2)
C3	C6	2.958(2)	C5	C8	3.251(2)
C5	C12	3.486(2)	C7	C10	2.8424(19)
C8	C11	2.769(2)	C9	C12	2.756(2)
C9	C14	3.492(2)	C11	C14	3.202(2)
C11	C18	3.075(2)	C13	C16	2.996(2)
C14	C17	2.936(2)	C15	C18	2.930(2)
C17	C20	3.158(2)			

Table 10. Intramolecular contacts less than 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
F1	H2	3.204	F1	H3B	2.639
F1	H6	3.229	F1	H8	2.426
F2	H1	2.596	F2	H3A	2.595
F2	H3B	2.592	F3	H3A	2.595
F3	H3B	2.597	F3	H5	2.588
F4	H3B	2.607	F4	H4	3.220
F4	H6	3.214	F4	H8	2.650
C1	H3A	3.367	C1	H3B	2.739
C1	H4	3.222	C1	H5	3.383
C1	H8	3.029	C2	H4	2.634
C2	H6	2.679	C3	H1	3.366
C3	H5	3.370	C3	H6	3.260
C4	H2	2.635	C4	H6	2.668
C5	H1	3.380	C5	H2	3.234
C5	H3A	3.368	C5	H3B	2.728
C5	H8	3.209	C5	H12	3.599
C6	H2	2.732	C6	H3B	3.278
C6	H4	2.719	C6	H8	2.739
C6	H12	2.648	C7	H1	2.771
C7	H5	2.714	C7	H9	3.276
C7	H11	3.281	C8	H1	3.424
C8	H5	3.511	C8	H6	3.327

C8	H12	3.248	C9	H11
	3.243			
C9	H13	2.565	C9	H14A
	3.339			
C10	H8	3.286	C10	H12
	3.279			
C10	H14A	2.700	C10	H14B
	2.707			
C10	H18A	2.738	C10	H18B
	2.744			
C11	H9	3.241	C11	H13
	3.329			
C11	H14A	3.487	C11	H14B
	2.932			
C11	H18A	2.788	C11	H18B
	3.292			
C12	H5	3.327	C12	H6
	2.490			
C12	H8	3.250	C13	H9
	2.684			
C13	H11	2.698	C13	H15A
	3.379			
C13	H15B	2.759	C13	H16
	3.301			
C13	H17A	2.765	C13	H17B
	3.386			
C14	H11	3.138	C14	H16
	2.733			
C14	H17A	3.284	C14	H18A
	2.743			

Table 10. Intramolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
C14	H18B	3.362	C15	H13	2.722
C15	H17A	2.717	C15	H17B	3.348
C15	H18A	3.288	C15	H19A	2.628
C15	H19B	2.763	C16	H13	3.303
C16	H14A	3.387	C16	H14B	2.772
C16	H18A	2.767	C16	H18B	3.384
C16	H20A	2.734	C16	H20B	2.779
C17	H13	2.727	C17	H14B	3.296
C17	H15A	3.347	C17	H15B	2.717
C17	H19A	3.388	C17	H19B	2.714
C17	H20A	3.440	C17	H20B	2.848
C18	H11	2.867	C18	H14A	3.362
C18	H14B	2.743	C18	H15B	3.281
C18	H16	2.727	C19	H15A	2.696
C19	H15B	2.709	C19	H17A	2.741
C19	H17B	2.750	C19	H21A	2.768
C19	H21B	2.727	C19	H21C	3.375
C20	H16	2.713	C20	H17A	3.465
C20	H17B	2.836	C21	H19A	2.701
C21	H19B	2.747	H1	H2	2.330
H1	H6	2.310	H1	H8	3.414

H2	H3A	2.360	H2	H3B
	2.872			
H2	H4	2.430	H2	H6
	2.521			
H3A	H4	2.373	H3B	H4
	2.878			
H4	H5	2.352	H4	H6
	2.497			
H5	H6	2.320	H5	H12
	3.357			
H6	H12	2.250	H8	H9
	2.331			
H9	H13	2.346	H9	H14A
	3.369			
H11	H12	2.324	H11	H14B
	2.643			
H11	H18A	2.301	H11	H18B
	3.227			
H13	H14A	2.352	H13	H14B
	2.869			
H13	H15B	2.575	H13	H17A
	2.580			
H13	H18A	2.871	H13	H18B
	2.356			
H14A	H15A	2.382	H14A	H15B
	2.355			
H14B	H15A	2.357	H14B	H15B
	2.873			
H14B	H16	2.591	H14B	H18A
	2.609			
H15A	H16	2.360	H15A	H19A
	2.423			

Table 10. Intramolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H15A	H19B	3.056	H15B	H16	2.870
H15B	H17A	2.569	H15B	H19A	2.871
H15B	H19B	2.581	H16	H17A	2.868
H16	H17B	2.357	H16	H18A	2.583
H16	H19A	2.410	H16	H19B	2.870
H16	H20A	2.502	H16	H20B	3.004
H17A	H18A	2.877	H17A	H18B	2.362
H17A	H19A	3.600	H17A	H19B	2.527
H17A	H20B	3.090	H17B	H18A	2.363
H17B	H18B	2.386	H17B	H19B	2.987
H17B	H20A	3.057	H17B	H20B	2.293
H19A	H20A	2.382	H19A	H20B	2.852
H19A	H21A	3.015	H19A	H21B	2.513
H19A	H21C	3.582	H19B	H20A	2.852
H19B	H20B	2.345	H19B	H21A	2.607
H19B	H21B	3.021	H20A	H21A	2.861
H20A	H21B	2.390	H20A	H21C	2.341
H20B	H21A	2.356	H20B	H21B	2.862
H20B	H21C	2.374			

Table 11. Intermolecular contacts less than 3.60 Å

atom	atom	distance	atom	atom	distance
F1	C4[1]	3.5789(18)	F1	C5[1]	3.1351(17)
F1	C6[1]	3.1889(16)	F1	C12[1]	3.2712(17)
F2	C11[2]	3.2985(17)	F3	F3[3]	3.2315(15)
F3	C3[3]	3.3396(17)	F3	C21[4]	3.414(2)
F4	C20[5]	3.388(2)	C3	F3[3]	3.3396(17)
C4	F1[6]	3.5789(18)	C5	F1[6]	3.1351(17)
C6	F1[6]	3.1889(16)	C11	F2[7]	3.2985(17)
C12	F1[6]	3.2712(17)	C20	F4[8]	3.388(2)
C21	F3[4]	3.414(2)			

Symmetry Operators:

(1) X,Y-1,Z
(2) -X+1,Y-1,-Z+1/2
(3) -X+1,-Y+1,-Z+1
(4) -X,Y,-Z+1/2
(5) -X,Y-1,-Z+1/2
(6) X,Y+1,Z
(7) -X+1,Y+1,-Z+1/2
(8) -X,Y+1,-Z+1/2

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
F1	H4[1]	3.040	F1	H5[1]	2.505
F1	H6[1]	2.531	F1	H12[1]	2.629
F1	H12[2]	3.572	F2	H4[1]	2.747
F2	H6[1]	3.426	F2	H11[2]	2.469
F2	H14A[3]	2.805	F2	H14B[2]	3.262
F2	H21A[4]	3.540	F2	H21B[4]	2.959
F3	H3A[5]	2.757	F3	H3B[6]	3.477
F3	H3B[5]	3.150	F3	H19B[7]	3.019
F3	H21A[7]	2.482	F3	H21A[8]	3.468
F3	H21C[9]	3.371	F3	H21C[8]	3.284
F4	H15B[7]	3.394	F4	H17A[7]	2.771
F4	H17B[9]	3.304	F4	H19B[7]	2.710
F4	H20B[9]	2.492	C1	H1[3]	3.308
C1	H6[1]	3.581	C1	H12[1]	3.444
C2	H4[1]	3.536	C2	H11[2]	3.578
C2	H14A[3]	3.090	C2	H14B[3]	3.355
C3	H4[1]	3.543	C3	H21A[4]	3.330
C3	H21C[8]	3.241	C4	H3B[6]	3.542
C4	H21C[8]	3.287	C5	H17A[7]	3.056
C5	H19B[7]	3.224	C6	H1[3]	3.566
C7	H1[3]	3.007	C7	H17A[7]	3.319
C8	H1[3]	3.292	C8	H11[1]	3.555

C8	H12[1]	3.341	C8	H13[7]
	3.436			
C8	H17A[7]	3.271	C8	H17B[9]
	3.515			
C9	H9[7]	3.483	C9	H11[1]
	3.233			
C9	H13[7]	3.044	C9	H18A[1]
	3.363			
C9	H18B[7]	3.484	C10	H2[3]
	3.131			
C10	H18B[7]	3.233	C11	H2[3]
	2.977			
C11	H6[3]	3.443	C11	H8[6]
	3.558			
C11	H9[6]	3.328	C11	H18B[7]
	3.493			
C12	H1[3]	3.199	C12	H2[3]
	3.443			
C12	H6[3]	3.017	C12	H8[6]
	3.241			
C12	H12[3]	3.440	C13	H18A[1]
	3.504			
C14	H2[3]	2.913	C14	H18A[1]
	3.487			
C14	H21B[10]	3.417	C15	H16[1]
	3.524			
C15	H19A[11]	3.352	C15	H20A[1]
	3.139			
C15	H20A[10]	3.370	C15	H21B[10]
	3.453			

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
C16	H15B[6]	3.510	C16	H20A[10]	3.467
C16	H21B[10]	3.456	C17	H5[7]	3.234
C17	H8[12]	3.487	C17	H15B[6]	3.497
C18	H9[6]	3.259	C18	H13[6]	3.524
C18	H18B[7]	3.312	C19	H5[7]	3.562
C19	H15A[11]	3.418	C19	H16[10]	3.591
C19	H20A[1]	3.593	C19	H20A[10]	3.462
C20	H15A[10]	3.348	C20	H15B[6]	3.111
C20	H16[10]	3.483	C20	H19A[10]	3.549
C21	H3A[13]	3.375	C21	H3A[14]	3.409
C21	H4[14]	3.314	C21	H14B[10]	3.273
C21	H15A[10]	3.358	C21	H16[10]	3.414
H1	C1[3]	3.308	H1	C6[3]	3.566
H1	C7[3]	3.007	H1	C8[3]	3.292
H1	C12[3]	3.199	H1	H1[3]	2.544
H1	H6[1]	3.597	H1	H11[2]	3.447
H1	H12[1]	3.190	H1	H12[2]	2.940
H1	H12[3]	3.542	H2	C10[3]	3.131
H2	C11[3]	2.977	H2	C12[3]	3.443
H2	C14[3]	2.913	H2	H11[3]	3.120
H2	H14A[3]	2.519	H2	H14B[3]	2.493

H3A	F3[5]	2.757	H3A	C21[4]
	3.375			
H3A	C21[8]	3.409	H3A	H14A[3]
	3.426			
H3A	H14B[3]	3.432	H3A	H21A[4]
	2.713			
H3A	H21B[4]	3.150	H3A	H21C[8]
	2.479			
H3B	F3[1]	3.477	H3B	F3[5]
	3.150			
H3B	C4[1]	3.542	H3B	H4[1]
	2.877			
H3B	H5[1]	3.460	H3B	H20B[9]
	3.467			
H3B	H21A[9]	3.386	H3B	H21A[4]
	3.101			
H3B	H21C[9]	3.548	H4	F1[6]
	3.040			
H4	F2[6]	2.747	H4	C2[6]
	3.536			
H4	C3[6]	3.543	H4	C21[8]
	3.314			
H4	H3B[6]	2.877	H4	H14B[3]
	3.551			
H4	H21A[8]	3.197	H4	H21B[8]
	3.312			
H4	H21C[8]	2.891	H5	F1[6]
	2.505			
H5	C17[7]	3.234	H5	C19[7]
	3.562			
H5	H3B[6]	3.460	H5	H8[6]
	3.476			

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H5	H17A^{7}	2.514	H5	H17B^{7}	3.119
H5	H19B^{7}	2.885	H5	H20B^{7}	3.068
H6	F1^{6}	2.531	H6	F2^{6}	3.426
H6	C1^{6}	3.581	H6	C11^{3}	3.443
H6	C12^{3}	3.017	H6	H1^{6}	3.597
H6	H11^{3}	3.503	H6	H12^{3}	2.777
H8	C11^{1}	3.558	H8	C12^{1}	3.241
H8	C17^{9}	3.487	H8	H5^{1}	3.476
H8	H11^{1}	3.514	H8	H12^{1}	2.951
H8	H13^{7}	3.509	H8	H17A^{9}	3.507
H8	H17A^{7}	3.470	H8	H17B^{9}	2.742
H8	H18B^{9}	3.455	H9	C9^{7}	3.483
H9	C11^{1}	3.328	H9	C18^{1}	3.259
H9	H9^{7}	2.774	H9	H11^{1}	2.928
H9	H13^{7}	2.857	H9	H18A^{1}	2.678
H9	H18B^{1}	2.924	H9	H18B^{9}	2.854
H11	F2^{15}	2.469	H11	C2^{15}	3.578
H11	C8^{6}	3.555	H11	C9^{6}	3.233
H11	H1^{15}	3.447	H11	H2^{3}	3.120
H11	H6^{3}	3.503	H11	H8^{6}	3.514
H11	H9^{6}	2.928	H11	H14A^{6}	3.561

H12	F1[6]	2.629	H12	F1[15]
	3.572			
H12	C1[6]	3.444	H12	C8[6]
	3.341			
H12	C12[3]	3.440	H12	H1[6]
	3.190			
H12	H1[3]	3.542	H12	H1[15]
	2.940			
H12	H6[3]	2.777	H12	H8[6]
	2.951			
H12	H12[3]	2.720	H13	C8[7]
	3.436			
H13	C9[7]	3.044	H13	C18[1]
	3.524			
H13	H8[7]	3.509	H13	H9[7]
	2.857			
H13	H17B[1]	3.252	H13	H18A[1]
	2.890			
H13	H18B[1]	3.460	H14A	F2[3]
	2.805			
H14A	C2[3]	3.090	H14A	H2[3]
	2.519			
H14A	H3A[3]	3.426	H14A	H11[1]
	3.561			
H14A	H16[1]	3.226	H14A	H18A[1]
	2.803			
H14A	H21B[11]	3.447	H14B	F2[15]
	3.262			
H14B	C2[3]	3.355	H14B	C21[10]
	3.273			
H14B	H2[3]	2.493	H14B	H3A[3]
	3.432			

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H14B	H4[3]	3.551	H14B	H21B[10]	2.622
H14B	H21C[10]	3.058	H15A	C19[11]	3.418
H15A	C20[10]	3.348	H15A	C21[10]	3.358
H15A	H15A[11]	3.063	H15A	H16[1]	3.449
H15A	H19A[11]	2.528	H15A	H19B[11]	3.511
H15A	H20A[1]	2.956	H15A	H20A[10]	2.566
H15A	H21B[10]	3.092	H15A	H21C[10]	3.148
H15B	F4[7]	3.394	H15B	C16[1]	3.510
H15B	C17[1]	3.497	H15B	C20[1]	3.111
H15B	H16[1]	2.900	H15B	H17B[1]	2.818
H15B	H18A[1]	3.530	H15B	H19A[11]	3.360
H15B	H20A[1]	2.455	H15B	H20B[1]	2.910
H16	C15[6]	3.524	H16	C19[10]	3.591
H16	C20[10]	3.483	H16	C21[10]	3.414
H16	H14A[6]	3.226	H16	H15A[6]	3.449
H16	H15B[6]	2.900	H16	H19A[10]	2.870
H16	H20A[10]	3.079	H16	H21B[10]	2.657
H17A	F4[7]	2.771	H17A	C5[7]	3.056
H17A	C7[7]	3.319	H17A	C8[7]	3.271
H17A	H5[7]	2.514	H17A	H8[7]	3.470
H17A	H8[12]	3.507	H17B	F4[12]	3.304

H17B	C8[12]	3.515	H17B	H5[7]
	3.119			
H17B	H8[12]	2.742	H17B	H13[6]
	3.252			
H17B	H15B[6]	2.818	H18A	C9[6]
	3.363			
H18A	C13[6]	3.504	H18A	C14[6]
	3.487			
H18A	H9[6]	2.678	H18A	H13[6]
	2.890			
H18A	H14A[6]	2.803	H18A	H15B[6]
	3.530			
H18A	H18B[7]	3.552	H18B	C9[7]
	3.484			
H18B	C10[7]	3.233	H18B	C11[7]
	3.493			
H18B	C18[7]	3.312	H18B	H8[12]
	3.455			
H18B	H9[6]	2.924	H18B	H9[12]
	2.854			
H18B	H13[6]	3.460	H18B	H18A[7]
	3.552			
H18B	H18B[7]	2.478	H19A	C15[11]
	3.352			
H19A	C20[10]	3.549	H19A	H15A[11]
	2.528			
H19A	H15B[11]	3.360	H19A	H16[10]
	2.870			
H19A	H19A[10]	3.373	H19A	H20A[1]
	3.288			
H19A	H20A[10]	2.720	H19B	F3[7]
	3.019			

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H19B	F4[7]	2.710	H19B	C5[7]	3.224
H19B	H5[7]	2.885	H19B	H15A[11]	3.511
H19B	H20A[1]	3.124	H19B	H20B[1]	3.318
H19B	H21C[1]	3.389	H20A	C15[6]	3.139
H20A	C15[10]	3.370	H20A	C16[10]	3.467
H20A	C19[6]	3.593	H20A	C19[10]	3.462
H20A	H15A[6]	2.956	H20A	H15A[10]	2.566
H20A	H15B[6]	2.455	H20A	H16[10]	3.079
H20A	H19A[6]	3.288	H20A	H19A[10]	2.720
H20A	H19B[6]	3.124	H20B	F4[12]	2.492
H20B	H3B[12]	3.467	H20B	H5[7]	3.068
H20B	H15B[6]	2.910	H20B	H19B[6]	3.318
H21A	F2[13]	3.540	H21A	F3[7]	2.482
H21A	F3[14]	3.468	H21A	C3[13]	3.330
H21A	H3A[13]	2.713	H21A	H3B[12]	3.386
H21A	H3B[13]	3.101	H21A	H4[14]	3.197
H21B	F2[13]	2.959	H21B	C14[10]	3.417
H21B	C15[10]	3.453	H21B	C16[10]	3.456
H21B	H3A[13]	3.150	H21B	H4[14]	3.312
H21B	H14A[11]	3.447	H21B	H14B[10]	2.622
H21B	H15A[10]	3.092	H21B	H16[10]	2.657

H21C	F3[12]	3.371		H21C	F3[14]
	3.284				
H21C	C3[14]	3.241		H21C	C4[14]
	3.287				
H21C	H3A[14]	2.479		H21C	H3B[12]
	3.548				
H21C	H4[14]	2.891		H21C	H14B[10]
	3.058				
H21C	H15A[10]	3.148		H21C	H19B[6]
	3.389				

Symmetry Operators:

(1) X,Y-1,Z
(2) -X+1,Y-1,-Z+1/2
(3) -X+1,Y,-Z+1/2
(4) X+1,-Y+1,Z+1
(5) -X+1,-Y+1,-Z+1
(6) X,Y+1,Z
(7) -X,Y,-Z+1/2
(8) X+1,-Y+2,Z+1
(9) -X,Y-1,-Z+1/2
(10) -X,-Y+2,-Z
(11) -X,-Y+1,-Z
(12) -X,Y+1,-Z+1/2
(13) X-1,-Y+1,Z
(14) X-1,-Y+2,Z
(15) -X+1,Y+1,-Z+1/2

X-Ray Data Collection for **4**

Data Collection

A colorless platelet crystal of $C_{21}H_{29}F_3$ having approximate dimensions of 0.240 x 0.050 x 0.010 mm was mounted in a loop. All measurements were made on a Rigaku XtaLAB P200 diffractometer using multi-layer mirror monochromated Cu-Kα radiation.

Cell constants and an orientation matrix for data collection corresponded to a primitive monoclinic cell with dimensions:

a = 27.34(2) Å
b = 5.534(4) Å β = 102.294(19)°
c = 12.092(11) Å
V = 1787(3) Å^3

For Z = 4 and F.W. = 338.46, the calculated density is 1.258 g/cm^3. The reflection conditions of:

h0l: l = 2n
0k0: k = 2n

uniquely determine the space group to be:

$P2_1/c$ (#14)

The data were collected at a temperature of -148 ± 1°C to a maximum 2θ value of 136.8°. Readout was performed in the 0.172 mm pixel mode.

Data Reduction

Of the 17873 reflections were collected, where 3249 were unique (R_{int} = 0.1377); equivalent reflections were merged. Data were collected and processed using CrystalClear (Rigaku). [1]

The linear absorption coefficient, μ, for Cu-Kα radiation is 7.615 cm^{-1}. An empirical absorption correction was applied which resulted in transmission factors ranging from 0.766 to 0.992. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by charge flipping method[2] and expanded using Fourier techniques. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined using the riding model. The final cycle of full-matrix least-squares refinement[3] on F^2 was based on 3249 observed reflections and 218 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R1 = \Sigma \, ||Fo| - |Fc|| \, / \, \Sigma \, |Fo| = 0.0909$$

$$wR2 = [\Sigma (w (Fo^2 - Fc^2)^2) / \Sigma \, w(Fo^2)^2]^{1/2} = 0.2943$$

The goodness of fit[4] was 1.11. Unit weights were used. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.80 and -0.35 $e^-/Å^3$, respectively.

Neutral atom scattering factors were taken from International Tables for Crystallography (IT), Vol. C, Table 6.1.1.4 [5]. Anomalous dispersion effects were included in Fcalc[6]; the values for Δf' and Δf'' were those of Creagh and McAuley[7]. The values for the mass attenuation coefficients are those of Creagh and Hubbell[8]. All calculations were performed using the CrystalStructure[9] crystallographic software package except for refinement, which was performed using SHELXL2013[10].

References

(1) CrystalClear: Data Collection and Processing Software, Rigaku Corporation (1998-2014). Tokyo 196-8666, Japan.

(2) Superflip: Palatinus L., Chapuis G. (2007), J. Appl. Cryst. 40, 786-790.

(3) Least Squares function minimized: (SHELXL2013)

$\Sigma w(F_o^2 - F_c^2)^2$ where w = Least Squares weights.

(4) Goodness of fit is defined as:

$[\Sigma w(F_o^2 - F_c^2)^2 / (N_o - N_v)]^{1/2}$

where: N_o = number of observations

N_V = number of variables

(5) International Tables for Crystallography, Vol.C (1992). Ed. A.J.C. Wilson, Kluwer Academic Publishers, Dordrecht, Netherlands, Table 6.1.1.4, pp. 572.

(6) Ibers, J. A. & Hamilton, W. C.; Acta Crystallogr., 17, 781 (1964).

(7) Creagh, D. C. & McAuley, W.J .; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.6.8, pages 219-222 (1992).

(8) Creagh, D. C. & Hubbell, J.H..; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.4.3, pages 200-206 (1992).

(9) CrystalStructure 4.1: Crystal Structure Analysis Package, Rigaku Corporation (2000-2014). Tokyo 196-8666, Japan.

(10) SHELXL2013: Sheldrick, G. M. (2008). Acta Cryst. A64, 112-122.

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$C_{21}H_{29}F_3$
Formula Weight	338.46
Crystal Color, Habit	colorless, platelet
Crystal Dimensions	0.240 X 0.050 X 0.010 mm
Crystal System	monoclinic
Lattice Type	Primitive
Lattice Parameters	a = 27.34(2) Å b = 5.534(4) Å c = 12.092(11) Å β = 102.294(19) ° V = 1787(3) Å^3
Space Group	$P2_1/c$ (#14)
Z value	4
D_{calc}	1.258 g/cm^3
F_{000}	728.00
μ(CuKα)	7.615 cm^{-1}

B. Intensity Measurements

Diffractometer	XtaLAB P200
Radiation monochromated	CuKα (λ = 1.54187 Å) multi-layer mirror
Voltage, Current	40kV, 30mA
Temperature	-148.0°C
Detector Aperture	83.8 x 70.0 mm
Pixel Size	0.172 mm
$2\theta_{max}$	136.8°
No. of Reflections Measured	Total: 17873 Unique: 3249 (R_{int} = 0.1377)
Corrections	Lorentz-polarization Absorption (trans. factors: 0.766 - 0.992)

C. Structure Solution and Refinement

Structure Solution	Charge Flipping (Superflip)
Refinement	Full-matrix least-squares on F^2
Function Minimized	Σ w $(Fo^2 - Fc^2)^2$
Least Squares Weights	w = 1/ [$\sigma^2(Fo^2)$ + $(0.1122 \cdot P)^2$ + 6.1854 · P] where P = (Max(Fo^2,0) + $2Fc^2$)/3
$2\theta_{max}$ cutoff	136.8º
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (All reflections)	3249
No. Variables	218
Reflection/Parameter Ratio	14.90
Residuals: R1 (I>2.00σ(I))	0.0909
Residuals: R (All reflections)	0.1506
Residuals: wR2 (All reflections)	0.2943
Goodness of Fit Indicator	1.108
Max Shift/Error in Final Cycle	0.000
Maximum peak in Final Diff. Map	0.80 $e^-/Å^3$
Minimum peak in Final Diff. Map	-0.35 $e^-/Å^3$

Table 1. Atomic coordinates and B_{iso}/B_{eq}

atom	x	y	z	B_{eq}
F1	0.87716(12)	0.6903(5)	0.3765(3)	3.05(7)
F2	0.90698(13)	0.6741(6)	0.1782(3)	3.62(7)
F3	0.93388(11)	0.3765(6)	0.5380(2)	2.90(6)
C1	0.86773(18)	0.4811(9)	0.3110(4)	2.11(9)
C2	0.90755(19)	0.4654(9)	0.2410(4)	2.24(9)
C3	0.95910(19)	0.4258(10)	0.3135(4)	2.33(9)
C4	0.9590(2)	0.1957(11)	0.3820(5)	2.69(10)
C5	0.91975(19)	0.2001(9)	0.4532(4)	2.20(9)
C6	0.86764(18)	0.2581(9)	0.3831(4)	1.81(8)
C7	0.82725(17)	0.2554(9)	0.4533(4)	1.69(8)
C8	0.82357(18)	0.4340(9)	0.5314(4)	1.97(9)
C9	0.78707(18)	0.4148(9)	0.5970(4)	1.92(8)
C10	0.75442(18)	0.2217(9)	0.5874(4)	1.89(8)
C11	0.75820(18)	0.0441(9)	0.5074(4)	1.98(9)
C12	0.79409(18)	0.0618(9)	0.4418(4)	1.94(9)
C13	0.71549(18)	0.2054(9)	0.6594(4)	2.02(9)
C14	0.71610(19)	-0.0381(10)	0.7211(4)	2.41(10)
C15	0.67680(18)	-0.0490(10)	0.7934(4)	2.26(9)
C16	0.62394(18)	0.0009(10)	0.7259(4)	2.13(9)
C17	0.62337(19)	0.2404(10)	0.6629(5)	2.61(10)
C18	0.66250(18)	0.2533(10)	0.5897(4)	2.30(9)
C19	0.58474(19)	-0.0002(10)	0.7986(4)	2.46(10)
C20	0.5775(2)	-0.2406(11)	0.8527(5)	3.26(11)
C21	0.5393(2)	-0.2285(13)	0.9268(5)	3.58(12)

$B_{eq} = 8/3\ \pi^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$

Table 2. Atomic coordinates and B_{iso} involving hydrogen atoms

atom	x	y	z	B_{iso}
H1	0.83423	0.49664	0.25867	2.531
H2	0.89937	0.32592	0.18769	2.682
H3A	0.96856	0.56501	0.36509	2.800
H3B	0.98390	0.41194	0.26498	2.800
H4A	0.95266	0.05590	0.32980	3.229
H4B	0.99238	0.17362	0.43208	3.229
H5	0.91886	0.03866	0.48987	2.636
H6	0.85913	0.12115	0.32846	2.172
H8	0.84562	0.56863	0.54062	2.364
H9	0.78474	0.53911	0.64979	2.304
H11	0.73601	-0.09008	0.49775	2.378
H12	0.79603	-0.06104	0.38803	2.328
H13	0.72323	0.33441	0.71848	2.422
H14A	0.70987	-0.17006	0.66453	2.893
H14B	0.74965	-0.06394	0.76995	2.893
H15A	0.68530	0.07120	0.85518	2.715
H15B	0.67765	-0.21105	0.82840	2.715
H16	0.61506	-0.12998	0.66807	2.561
H17A	0.62943	0.37378	0.71873	3.135
H17B	0.58974	0.26465	0.61412	3.135
H18A	0.66142	0.41525	0.55447	2.757
H18B	0.65437	0.13224	0.52819	2.757
H19A	0.59433	0.12176	0.85927	2.955
H19B	0.55228	0.05058	0.75100	2.955
H20A	0.60996	-0.29461	0.89910	3.914
H20B	0.56653	-0.36217	0.79249	3.914
H21A	0.50752	-0.16656	0.88248	4.296
H21B	0.55148	-0.12048	0.99103	4.296
H21C	0.53409	-0.39053	0.95497	4.296

Table 3. Anisotropic displacement parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
F1	0.061(2)	0.0197(16)	0.0431(18)	-0.0003(14)	0.0287(15)	-0.0029(13)
F2	0.059(2)	0.046(2)	0.0352(17)	0.0053(16)	0.0158(15)	0.0204(15)
F3	0.0351(16)	0.051(2)	0.0229(14)	-0.0072(14)	0.0037(12)	-0.0033(14)
C1	0.030(3)	0.023(3)	0.027(3)	0.001(2)	0.006(2)	0.007(2)
C2	0.041(3)	0.023(3)	0.023(3)	-0.002(2)	0.012(2)	0.005(2)
C3	0.031(3)	0.027(3)	0.034(3)	0.000(2)	0.015(2)	-0.000(2)
C4	0.036(3)	0.036(3)	0.034(3)	0.007(2)	0.016(2)	0.007(2)
C5	0.035(3)	0.021(3)	0.029(3)	0.001(2)	0.008(2)	0.002(2)
C6	0.030(3)	0.023(3)	0.017(2)	-0.003(2)	0.0077(19)	-0.0061(19)
C7	0.027(2)	0.021(3)	0.014(2)	0.002(2)	0.0014(18)	0.0067(18)
C8	0.028(3)	0.022(3)	0.024(2)	-0.000(2)	0.005(2)	0.002(2)
C9	0.035(3)	0.020(2)	0.018(2)	0.001(2)	0.0045(19)	-0.000(2)
C10	0.028(3)	0.024(3)	0.019(2)	0.003(2)	0.0037(19)	0.003(2)
C11	0.033(3)	0.024(3)	0.020(2)	-0.004(2)	0.007(2)	-0.001(2)
C12	0.035(3)	0.023(3)	0.016(2)	-0.000(2)	0.0057(19)	-0.004(2)
C13	0.028(3)	0.027(3)	0.021(2)	-0.001(2)	0.0049(19)	-0.004(2)
C14	0.029(3)	0.036(3)	0.027(3)	0.004(2)	0.007(2)	0.012(2)
C15	0.032(3)	0.033(3)	0.022(2)	0.001(2)	0.007(2)	0.007(2)
C16	0.027(3)	0.033(3)	0.022(2)	0.002(2)	0.0062(19)	0.002(2)
C17	0.031(3)	0.037(3)	0.031(3)	0.008(2)	0.008(2)	0.009(2)
C18	0.034(3)	0.029(3)	0.025(3)	0.006(2)	0.006(2)	0.008(2)
C19	0.032(3)	0.037(3)	0.027(3)	0.001(2)	0.012(2)	0.002(2)

C20	0.046(3)	0.040(3)	0.044(3)	-0.001(3)	0.024(3)
	0.006(3)				
C21	0.044(3)	0.057(4)	0.039(3)	-0.010(3)	0.017(3)
	0.003(3)				

The general temperature factor expression: $\exp(-2\pi^2(a^{*2}U_{11}h^2 + b^{*2}U_{22}k^2 + c^{*2}U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$

Table 4. Bond lengths (Å)

atom	atom	distance	atom	atom	distance
F1	C1	1.396(6)	F2	C2	1.381(6)
F3	C5	1.409(6)	C1	C2	1.517(8)
C1	C6	1.512(7)	C2	C3	1.509(7)
C3	C4	1.520(8)	C4	C5	1.512(8)
C5	C6	1.529(7)	C6	C7	1.529(7)
C7	C8	1.385(7)	C7	C12	1.391(7)
C8	C9	1.404(8)	C9	C10	1.381(7)
C10	C11	1.398(7)	C10	C13	1.515(8)
C11	C12	1.390(8)	C13	C14	1.538(8)
C13	C18	1.536(6)	C14	C15	1.524(8)
C15	C16	1.526(7)	C16	C17	1.527(8)
C16	C19	1.524(8)	C17	C18	1.529(8)
C19	C20	1.514(9)	C20	C21	1.516(10)

Table 5. Bond lengths involving hydrogens (Å)

atom	atom	distance	atom	atom	distance
C1	H1	1.000	C2	H2	1.000
C3	H3A	0.990	C3	H3B	0.990
C4	H4A	0.990	C4	H4B	0.990
C5	H5	1.000	C6	H6	1.000
C8	H8	0.950	C9	H9	0.950
C11	H11	0.950	C12	H12	0.950
C13	H13	1.000	C14	H14A	0.990
C14	H14B	0.990	C15	H15A	0.990
C15	H15B	0.990	C16	H16	1.000
C17	H17A	0.990	C17	H17B	0.990
C18	H18A	0.990	C18	H18B	0.990
C19	H19A	0.990	C19	H19B	0.990
C20	H20A	0.990	C20	H20B	0.990
C21	H21A	0.980	C21	H21B	0.980
C21	H21C	0.980			

Table 6. Bond angles (°)

atom	atom	atom	angle	atom	atom	atom	angle
F1	C1	C2	107.3(4)	F1	C1	C6	111.9(4)
C2	C1	C6	111.4(4)	F2	C2	C1	109.5(4)
F2	C2	C3	109.9(4)	C1	C2	C3	112.1(4)
C2	C3	C4	109.1(4)	C3	C4	C5	112.4(5)
F3	C5	C4	107.9(4)	F3	C5	C6	109.7(4)
C4	C5	C6	112.2(4)	C1	C6	C5	112.0(4)
C1	C6	C7	114.7(4)	C5	C6	C7	112.8(4)
C6	C7	C8	122.5(4)	C6	C7	C12	119.0(4)
C8	C7	C12	118.5(5)	C7	C8	C9	119.6(5)
C8	C9	C10	122.4(5)	C9	C10	C11	117.4(5)
C9	C10	C13	121.2(4)	C11	C10	C13	121.4(4)
C10	C11	C12	120.7(5)	C7	C12	C11	121.4(5)
C10	C13	C14	113.2(4)	C10	C13	C18	111.7(4)
C14	C13	C18	109.2(4)	C13	C14	C15	112.1(4)
C14	C15	C16	112.9(4)	C15	C16	C17	109.4(4)
C15	C16	C19	113.1(4)	C17	C16	C19	110.9(4)
C16	C17	C18	113.3(5)	C13	C18	C17	111.6(4)
C16	C19	C20	115.2(5)	C19	C20	C21	112.8(5)

Table 7. Bond angles involving hydrogens (°)

atom	atom	atom	angle	atom	atom	atom	angle
F1	C1	H1	108.8	C2	C1	H1	108.8
C6	C1	H1	108.8	F2	C2	H2	108.4
C1	C2	H2	108.4	C3	C2	H2	108.4
C2	C3	H3A	109.8	C2	C3	H3B	109.8
C4	C3	H3A	109.9	C4	C3	H3B	109.9
H3A	C3	H3B	108.3	C3	C4	H4A	109.1
C3	C4	H4B	109.1	C5	C4	H4A	109.1
C5	C4	H4B	109.1	H4A	C4	H4B	107.8
F3	C5	H5	109.0	C4	C5	H5	109.0
C6	C5	H5	109.0	C1	C6	H6	105.4
C5	C6	H6	105.4	C7	C6	H6	105.4
C7	C8	H8	120.2	C9	C8	H8	120.2
C8	C9	H9	118.8	C10	C9	H9	118.8
C10	C11	H11	119.7	C12	C11	H11	119.7
C7	C12	H12	119.3	C11	C12	H12	119.3
C10	C13	H13	107.5	C14	C13	H13	107.5
C18	C13	H13	107.5	C13	C14	H14A	109.2
C13	C14	H14B	109.2	C15	C14	H14A	109.2
C15	C14	H14B	109.2	H14A	C14	H14B	107.9
C14	C15	H15A	109.0	C14	C15	H15B	109.0
C16	C15	H15A	109.0	C16	C15	H15B	109.0
H15A	C15	H15B	107.8	C15	C16	H16	107.7

C17	C16	H16	107.7	C19	C16	H16
	107.7					
C16	C17	H17A	108.9	C16	C17	H17B
	108.9					
C18	C17	H17A	108.9	C18	C17	H17B
	108.9					
H17A	C17	H17B	107.7	C13	C18	H18A
	109.3					
C13	C18	H18B	109.3	C17	C18	H18A
	109.3					
C17	C18	H18B	109.3	H18A	C18	H18B
	108.0					
C16	C19	H19A	108.5	C16	C19	H19B
	108.5					
C20	C19	H19A	108.5	C20	C19	H19B
	108.5					
H19A	C19	H19B	107.5	C19	C20	H20A
	109.0					
C19	C20	H20B	109.0	C21	C20	H20A
	109.0					
C21	C20	H20B	109.0	H20A	C20	H20B
	107.8					
C20	C21	H21A	109.5	C20	C21	H21B
	109.5					
C20	C21	H21C	109.5	H21A	C21	H21B
	109.5					
H21A	C21	H21C	109.5	H21B	C21	H21C
	109.5					

Table 8. Torsion Angles(°)
(Those having bond angles > 160 or < 20 degrees are excluded.)

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
F1	C1	C2	F2	-56.2(4)	F1	C1	C2	C3	66.1(5)
F1	C1	C6	C5	-68.3(5)	F1	C1	C6	C7	62.1(5)
C2	C1	C6	C5	51.7(5)	C2	C1	C6	C7	-177.9(3)
C6	C1	C2	F2	-178.9(3)	C6	C1	C2	C3	-56.6(5)
F2	C2	C3	C4	180.0(4)	C1	C2	C3	C4	57.9(5)
C2	C3	C4	C5	-56.3(5)	C3	C4	C5	F3	-67.7(5)
C3	C4	C5	C6	53.3(5)	F3	C5	C6	C1	69.5(5)
F3	C5	C6	C7	-61.8(5)	C4	C5	C6	C1	-50.5(5)
C4	C5	C6	C7	178.2(4)	C1	C6	C7	C8	-58.2(5)
C1	C6	C7	C12	124.5(4)	C5	C6	C7	C8	71.8(5)
C5	C6	C7	C12	-105.6(4)	C6	C7	C8	C9	-177.0(3)
C6	C7	C12	C11	176.9(3)	C8	C7	C12	C11	-0.6(6)
C12	C7	C8	C9	0.4(6)	C7	C8	C9	C10	0.5(6)
C8	C9	C10	C11	-1.2(6)	C8	C9	C10	C13	179.8(4)
C9	C10	C11	C12	0.9(6)	C9	C10	C13	C14	-128.8(4)
C9	C10	C13	C18	107.4(4)	C11	C10	C13	C14	52.2(5)
C11	C10	C13	C18	-71.5(5)	C13	C10	C11	C12	179.9(4)
C10	C11	C12	C7	-0.1(6)	C10	C13	C14	C15	179.7(3)
C10	C13	C18	C17	-179.2(4)	C14	C13	C18	C17	54.8(5)
C18	C13	C14	C15	-55.2(5)	C13	C14	C15	C16	56.2(5)
C14	C15	C16	C17	-53.6(5)	C14	C15	C16	C19	-177.8(4)

C15	C16	C17	C18	53.7(5)	C15	C16	C19	C20	-64.3(5)
C17	C16	C19	C20	172.4(3)	C19	C16	C17	C18	179.2(3)
C16	C17	C18	C13	-56.0(5)	C16	C19	C20	C21	178.2(4)

Table 9. Intramolecular contacts less than 3.60 Å

atom	atom	distance	atom	atom	distance
F1	F2	2.694(5)	F1	F3	2.818(4)
F1	C3	2.910(7)	F1	C4	3.527(7)
F1	C5	3.019(6)	F1	C7	3.009(6)
F1	C8	2.972(6)	F3	C1	3.005(6)
F3	C2	3.543(6)	F3	C3	2.952(7)
F3	C7	2.954(6)	F3	C8	3.017(6)
C1	C4	2.923(7)	C1	C8	3.163(8)
C2	C5	2.913(7)	C3	C6	2.953(8)
C5	C8	3.247(8)	C5	C12	3.494(8)
C7	C10	2.829(7)	C8	C11	2.777(7)
C9	C12	2.743(7)	C9	C18	3.504(8)
C11	C14	3.078(8)	C11	C18	3.207(8)
C13	C16	3.008(8)	C14	C17	2.922(8)
C15	C18	2.934(8)	C15	C20	3.137(9)

Table 10. Intramolecular contacts less than 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
F1	H2	3.200	F1	H3A	2.625
F1	H6	3.222	F1	H8	2.421
F2	H1	2.587	F2	H3A	2.586
F2	H3B	2.586	F3	H3A	2.684
F3	H4A	3.209	F3	H4B	2.516
F3	H6	3.223	F3	H8	2.643
C1	H3A	2.734	C1	H3B	3.361
C1	H4A	3.279	C1	H5	3.367
C1	H8	3.004	C2	H4A	2.691
C2	H4B	3.323	C2	H6	2.664
C3	H1	3.359	C3	H5	3.368
C3	H6	3.249	C4	H2	2.658
C4	H6	2.700	C5	H1	3.369
C5	H2	3.216	C5	H3A	2.757
C5	H3B	3.366	C5	H8	3.209
C6	H2	2.712	C6	H3A	3.287
C6	H4A	2.777	C6	H4B	3.368
C6	H8	2.726	C6	H12	2.647
C7	H1	2.747	C7	H5	2.727
C7	H9	3.261	C7	H11	3.277
C8	H1	3.390	C8	H5	3.519
C8	H6	3.314	C8	H12	3.244

C9	H11	3.237	C9	H13
	2.549			
C9	H18A	3.364	C10	H8
	3.290			
C10	H12	3.274	C10	H14A
	2.745			
C10	H14B	2.741	C10	H18A
	2.709			
C10	H18B	2.721	C11	H9
	3.235			
C11	H13	3.324	C11	H14A
	2.798			
C11	H14B	3.287	C11	H18A
	3.491			
C11	H18B	2.943	C12	H1
	3.597			
C12	H5	3.339	C12	H6
	2.488			
C12	H8	3.250	C13	H9
	2.665			
C13	H11	2.698	C13	H15A
	2.769			
C13	H15B	3.385	C13	H16
	3.334			
C13	H17A	2.764	C13	H17B
	3.380			
C14	H11	2.881	C14	H16
	2.747			
C14	H17A	3.284	C14	H18A
	3.362			

Table 10. Intramolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
C14	H18B	2.739	C15	H13	2.724
C15	H17A	2.733	C15	H17B	3.344
C15	H18B	3.291	C15	H19A	2.716
C15	H19B	3.378	C15	H20A	2.796
C15	H20B	3.476	C16	H13	3.300
C16	H14A	2.776	C16	H14B	3.383
C16	H18A	3.392	C16	H18B	2.790
C16	H20A	2.747	C16	H20B	2.772
C17	H13	2.719	C17	H14A	3.276
C17	H15A	2.734	C17	H15B	3.344
C17	H19A	2.739	C17	H19B	2.627
C18	H11	3.136	C18	H14A	2.736
C18	H14B	3.361	C18	H15A	3.295
C18	H16	2.757	C19	H15A	2.717
C19	H15B	2.749	C19	H17A	2.685
C19	H17B	2.697	C19	H21A	2.692
C19	H21B	2.754	C19	H21C	3.358
C20	H15A	3.410	C20	H15B	2.820
C20	H16	2.719	C21	H19A	2.686
C21	H19B	2.712	H1	H2	2.335
H1	H6	2.291	H1	H8	3.380

H2	H3A	2.864	H2	H3B
	2.350			
H2	H4A	2.498	H2	H4B
	3.565			
H2	H6	2.488	H3A	H4A
	2.869			
H3A	H4B	2.355	H3B	H4A
	2.347			
H3B	H4B	2.382	H4A	H5
	2.318			
H4A	H6	2.579	H4B	H5
	2.384			
H5	H6	2.308	H5	H12
	3.367			
H6	H12	2.245	H8	H9
	2.340			
H9	H13	2.323	H9	H18A
	3.393			
H11	H12	2.325	H11	H14A
	2.321			
H11	H14B	3.236	H11	H18B
	2.642			
H13	H14A	2.872	H13	H14B
	2.361			
H13	H15A	2.581	H13	H17A
	2.574			
H13	H18A	2.358	H13	H18B
	2.872			
H14A	H15A	2.865	H14A	H15B
	2.345			
H14A	H16	2.611	H14A	H18B
	2.599			

Table 10. Intramolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H14B	H15A	2.343	H14B	H15B	2.373
H15A	H16	2.863	H15A	H17A	2.605
H15A	H19A	2.514	H15A	H19B	3.588
H15A	H20A	3.015	H15B	H16	2.340
H15B	H19A	3.014	H15B	H20A	2.246
H15B	H20B	3.091	H16	H17A	2.863
H16	H17B	2.341	H16	H18B	2.626
H16	H19A	2.857	H16	H19B	2.384
H16	H20A	2.970	H16	H20B	2.554
H17A	H18A	2.346	H17A	H18B	2.869
H17A	H19A	2.539	H17A	H19B	2.855
H17B	H18A	2.379	H17B	H18B	2.350
H17B	H19A	3.045	H17B	H19B	2.432
H19A	H20A	2.374	H19A	H20B	2.853
H19A	H21A	2.923	H19A	H21B	2.550
H19A	H21C	3.592	H19B	H20A	2.853
H19B	H20B	2.353	H19B	H21A	2.510
H19B	H21B	3.057	H19B	H21C	3.579
H20A	H21A	2.854	H20A	H21B	2.340
H20A	H21C	2.374	H20B	H21A	2.389
H20B	H21B	2.853	H20B	H21C	2.327

Table 11. Intermolecular contacts less than 3.60 Å

atom	atom	distance	atom	atom
	distance			
F1	C4[1]	3.573(7)	F1	C5[1]
	3.117(6)			
F1	C6[1]	3.155(6)	F1	C12[1]
	3.282(6)			
F2	F3[2]	3.183(5)	F2	C5[3]
	3.495(7)			
F2	C8[2]	3.366(6)	F3	F2[4]
	3.183(5)			
F3	C2[5]	3.298(6)	F3	C3[6]
	3.278(6)			
C2	F3[3]	3.298(6)	C3	F3[6]
	3.278(6)			
C4	F1[7]	3.573(7)	C5	F1[7]
	3.117(6)			
C5	F2[5]	3.495(7)	C6	F1[7]
	3.155(6)			
C8	F2[4]	3.366(6)	C12	F1[7]
	3.282(6)			

Symmetry Operators:

(1) X,Y+1,Z
(2) X,-Y+2,Z
(3) X,-Y+1,Z
(4) X,-Y+2,Z+1
(5) X,-Y+1,Z+1
(6) -X+2,-Y+1,-Z+1
(7) X,Y-1,Z

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
F1	H4A[1]	3.028	F1	H5[1]	2.496
F1	H6[1]	2.478	F1	H12[1]	2.639
F1	H14B[2]	3.520	F2	H3B[3]	3.199
F2	H4A[1]	2.900	F2	H4B[3]	3.303
F2	H5[2]	2.644	F2	H6[1]	3.485
F2	H8[4]	2.534	F3	H2[5]	2.479
F3	H3A[6]	2.695	F3	H3B[6]	3.134
F3	H3B[5]	3.217	F3	H4A[5]	3.476
F3	H4B[6]	3.175	C1	H6[1]	3.559
C1	H12[1]	3.451	C1	H14B[2]	3.193
C2	H4A[1]	3.576	C2	H5[2]	3.118
C3	H3B[7]	3.471	C3	H3B[3]	3.346
C3	H4A[1]	3.499	C3	H4A[3]	3.335
C4	H3A[8]	3.509	C4	H3A[6]	3.529
C4	H3B[7]	3.041	C4	H4B[9]	3.118
C5	H2[5]	3.007	C5	H4B[9]	3.249
C7	H2[5]	3.121	C7	H13[2]	3.600
C7	H14B[2]	3.214	C8	H2[5]	2.875
C8	H6[5]	3.529	C8	H11[1]	3.524
C8	H12[1]	3.290	C8	H14B[2]	3.446
C9	H1[5]	3.098	C9	H2[5]	3.312
C9	H6[5]	3.067	C9	H11[1]	3.191

C9	H12[5]	3.569	C9	H14A[1]
	3.337			
C9	H15A[2]	3.582	C10	H1[5]
	2.930			
C10	H15A[2]	3.242	C11	H1[5]
	3.304			
C11	H8[8]	3.519	C11	H9[8]
	3.281			
C11	H13[2]	3.486	C11	H15A[2]
	3.214			
C11	H15B[10]	3.302	C12	H8[8]
	3.185			
C12	H9[2]	3.530	C12	H13[2]
	3.025			
C12	H14B[10]	3.509	C12	H14B[2]
	3.527			
C12	H15A[2]	3.570	C13	H1[5]
	3.402			
C13	H14A[1]	3.461	C14	H1[5]
	3.173			
C14	H9[8]	3.229	C14	H12[11]
	3.448			
C14	H13[8]	3.478	C15	H11[11]
	3.319			
C15	H17A[8]	3.493	C15	H18A[5]
	3.355			
C16	H17A[8]	3.475	C17	H16[1]
	3.493			
C17	H20B[1]	3.278	C17	H21A[12]
	3.541			

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
C17	H21B[2]	3.296	C18	H14A[1]	3.490
C18	H15A[2]	3.182	C18	H19A[2]	3.083
C18	H20A[10]	3.522	C19	H18A[5]	3.380
C19	H20B[1]	3.564	C19	H21A[12]	3.495
C20	H17A[8]	3.190	C20	H18A[5]	3.475
C20	H18B[11]	3.423	C20	H19A[8]	3.557
C21	H16[11]	3.293	C21	H17B[13]	3.456
C21	H17B[5]	3.506	C21	H19B[13]	3.177
C21	H21B[14]	3.452	C21	H21C[15]	3.425
H1	C9[2]	3.098	H1	C10[2]	2.930
H1	C11[2]	3.304	H1	C13[2]	3.402
H1	C14[2]	3.173	H1	H6[1]	3.588
H1	H9[2]	3.405	H1	H9[4]	3.067
H1	H12[1]	3.198	H1	H13[2]	3.489
H1	H14A[2]	3.483	H1	H14B[2]	2.375
H2	F3[2]	2.479	H2	C5[2]	3.007
H2	C7[2]	3.121	H2	C8[2]	2.875
H2	C9[2]	3.312	H2	H5[2]	2.667
H2	H8[2]	2.996	H3A	F3[6]	2.695
H3A	C4[1]	3.509	H3A	C4[6]	3.529
H3A	H3A[6]	3.425	H3A	H3B[3]	2.953

H3A	H4A[1]	2.770	H3A	H4A[3]
		3.515		
H3A	H4B[1]	3.493	H3A	H4B[6]
		2.852		
H3A	H5[1]	3.445	H3B	F2[7]
		3.199		
H3B	F3[6]	3.134	H3B	F3[2]
		3.217		
H3B	C3[7]	3.346	H3B	C3[3]
		3.471		
H3B	C4[3]	3.041	H3B	H3A[7]
		2.953		
H3B	H3B[7]	2.949	H3B	H3B[3]
		2.949		
H3B	H4A[3]	2.409	H3B	H4B[3]
		2.975		
H3B	H5[2]	3.433	H4A	F1[8]
		3.028		
H4A	F2[8]	2.900	H4A	F3[2]
		3.476		
H4A	C2[8]	3.576	H4A	C3[8]
		3.499		
H4A	C3[7]	3.335	H4A	H3A[8]
		2.770		
H4A	H3A[7]	3.515	H4A	H3B[7]
		2.409		
H4A	H4B[9]	3.212	H4B	F2[7]
		3.303		
H4B	F3[6]	3.175	H4B	C4[9]
		3.118		
H4B	C5[9]	3.249	H4B	H3A[8]
		3.493		

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H4B	H3A^{6}	2.852	H4B	H3B^{7}	2.975
H4B	H4A^{9}	3.212	H4B	H4B^{9}	2.504
H4B	H5^{9}	2.680	H5	F1^{8}	2.496
H5	F2^{5}	2.644	H5	C2^{5}	3.118
H5	H2^{5}	2.667	H5	H3A^{8}	3.445
H5	H3B^{5}	3.433	H5	H4B^{9}	2.680
H5	H8^{8}	3.417	H6	F1^{8}	2.478
H6	F2^{8}	3.485	H6	C1^{8}	3.559
H6	C8^{2}	3.529	H6	C9^{2}	3.067
H6	H1^{8}	3.588	H6	H8^{2}	3.579
H6	H9^{2}	2.778	H8	F2^{16}	2.534
H8	C11^{1}	3.519	H8	C12^{1}	3.185
H8	H2^{5}	2.996	H8	H5^{1}	3.417
H8	H6^{5}	3.579	H8	H11^{1}	3.486
H8	H12^{1}	2.894	H9	C11^{1}	3.281
H9	C12^{5}	3.530	H9	C14^{1}	3.229
H9	H1^{5}	3.405	H9	H1^{16}	3.067
H9	H6^{5}	2.778	H9	H11^{1}	2.885
H9	H12^{5}	2.834	H9	H14A^{1}	2.640
H9	H14B^{1}	2.907	H11	C8^{8}	3.524
H11	C9^{8}	3.191	H11	C15^{10}	3.319

H11	H8[8]	3.486	H11	H9[8]
	2.885			
H11	H14B[10]	3.439	H11	H15A[10]
	3.311			
H11	H15A[2]	3.481	H11	H15B[10]
	2.561			
H11	H18A[8]	3.566	H11	H20A[10]
	3.458			
H12	F1[8]	2.639	H12	C1[8]
	3.451			
H12	C8[8]	3.290	H12	C9[2]
	3.569			
H12	C14[10]	3.448	H12	H1[8]
	3.198			
H12	H8[8]	2.894	H12	H9[2]
	2.834			
H12	H13[2]	2.828	H12	H14A[10]
	3.515			
H12	H14B[10]	2.680	H12	H15B[10]
	3.405			
H13	C7[5]	3.600	H13	C11[5]
	3.486			
H13	C12[5]	3.025	H13	C14[1]
	3.478			
H13	H1[5]	3.489	H13	H12[5]
	2.828			
H13	H14A[1]	2.824	H13	H14B[1]
	3.435			
H13	H15B[1]	3.217	H14A	C9[8]
	3.337			
H14A	C13[8]	3.461	H14A	C18[8]
	3.490			

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H14A	$H1^{5}$	3.483	H14A	$H9^{8}$	2.640
H14A	$H12^{11}$	3.515	H14A	$H13^{8}$	2.824
H14A	$H17A^{8}$	3.500	H14A	$H18A^{8}$	2.836
H14B	$F1^{5}$	3.520	H14B	$C1^{5}$	3.193
H14B	$C7^{5}$	3.214	H14B	$C8^{5}$	3.446
H14B	$C12^{11}$	3.509	H14B	$C12^{5}$	3.527
H14B	$H1^{5}$	2.375	H14B	$H9^{8}$	2.907
H14B	$H11^{11}$	3.439	H14B	$H12^{11}$	2.680
H14B	$H13^{8}$	3.435	H15A	$C9^{5}$	3.582
H15A	$C10^{5}$	3.242	H15A	$C11^{5}$	3.214
H15A	$C12^{5}$	3.570	H15A	$C18^{5}$	3.182
H15A	$H11^{11}$	3.311	H15A	$H11^{5}$	3.481
H15A	$H18A^{5}$	2.629	H15A	$H18B^{5}$	2.922
H15B	$C11^{11}$	3.302	H15B	$H11^{11}$	2.561
H15B	$H12^{11}$	3.405	H15B	$H13^{8}$	3.217
H15B	$H17A^{8}$	2.834	H15B	$H18A^{5}$	3.296
H15B	$H18B^{11}$	3.511	H16	$C17^{8}$	3.493
H16	$C21^{10}$	3.293	H16	$H17A^{8}$	2.823
H16	$H17B^{8}$	3.455	H16	$H18A^{8}$	3.251
H16	$H20A^{10}$	3.252	H16	$H21B^{10}$	2.815
H16	$H21C^{10}$	3.021	H17A	$C15^{1}$	3.493

H17A	C16[1]	3.475	H17A	C20[1]
	3.190			
H17A	H14A[1]	3.500	H17A	H15B[1]
	2.834			
H17A	H16[1]	2.823	H17A	H20A[1]
	2.984			
H17A	H20B[1]	2.558	H17A	H21B[2]
	3.389			
H17B	C21[12]	3.456	H17B	C21[2]
	3.506			
H17B	H16[1]	3.455	H17B	H19A[2]
	3.174			
H17B	H20B[1]	3.147	H17B	H21A[12]
	2.695			
H17B	H21B[2]	2.554	H17B	H21C[12]
	3.420			
H17B	H21C[10]	3.010	H18A	C15[2]
	3.355			
H18A	C19[2]	3.380	H18A	C20[2]
	3.475			
H18A	H11[1]	3.566	H18A	H14A[1]
	2.836			
H18A	H15A[2]	2.629	H18A	H15B[2]
	3.296			
H18A	H16[1]	3.251	H18A	H19A[2]
	2.671			
H18A	H20A[2]	2.965	H18A	H21B[2]
	3.151			
H18B	C20[10]	3.423	H18B	H15A[2]
	2.922			
H18B	H15B[10]	3.511	H18B	H19A[2]
	2.700			

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H18B	H20A[10]	2.568	H18B	H21C[10]	3.485
H19A	C18[5]	3.083	H19A	C20[1]	3.557
H19A	H17B[5]	3.174	H19A	H18A[5]	2.671
H19A	H18B[5]	2.700	H19A	H20A[1]	3.280
H19A	H20B[1]	3.021	H19A	H21C[1]	3.485
H19B	C21[12]	3.177	H19B	H20B[1]	3.299
H19B	H20B[12]	3.215	H19B	H21A[12]	2.570
H19B	H21C[12]	3.063	H20A	C18[11]	3.522
H20A	H11[11]	3.458	H20A	H16[11]	3.252
H20A	H17A[8]	2.984	H20A	H18A[5]	2.965
H20A	H18B[11]	2.568	H20A	H19A[8]	3.280
H20B	C17[8]	3.278	H20B	C19[8]	3.564
H20B	H17A[8]	2.558	H20B	H17B[8]	3.147
H20B	H19A[8]	3.021	H20B	H19B[8]	3.299
H20B	H19B[13]	3.215	H20B	H21A[13]	3.096
H20B	H21B[10]	3.582	H21A	C17[13]	3.541
H21A	C19[13]	3.495	H21A	H17B[13]	2.695
H21A	H19B[13]	2.570	H21A	H20B[12]	3.096
H21A	H21A[14]	3.485	H21A	H21B[14]	2.918
H21A	H21C[15]	3.481	H21B	C17[5]	3.296
H21B	C21[14]	3.452	H21B	H16[11]	2.815

H21B	H17A[5]	3.389	H21B	H17B[5]
	2.554			
H21B	H18A[5]	3.151	H21B	H20B[11]
	3.582			
H21B	H21A[14]	2.918	H21B	H21B[14]
	3.164			
H21C	C21[15]	3.425	H21C	H16[11]
	3.021			
H21C	H17B[13]	3.420	H21C	H17B[11]
	3.010			
H21C	H18B[11]	3.485	H21C	H19A[8]
	3.485			
H21C	H19B[13]	3.063	H21C	H21A[15]
	3.481			
H21C	H21C[15]	2.644		

Symmetry Operators:

(1) X,Y+1,Z
(2) X,-Y+1,Z
(3) -X+2,Y+1/2,-Z+1/2
(4) X,-Y+2,Z
(5) X,-Y+1,Z+1
(6) -X+2,-Y+1,-Z+1
(7) -X+2,Y+1/2-1,-Z+1/2
(8) X,Y-1,Z
(9) -X+2,-Y,-Z+1
(10) X,-Y,Z
(11) X,-Y,Z+1
(12) -X+1,Y+1/2,-Z+1/2+1
(13) -X+1,Y+1/2-1,-Z+1/2+1
(14) -X+1,-Y,-Z+2
(15) -X+1,-Y-1,-Z+2
(16) X,-Y+2,Z+1

X-Ray Data Collection for **5**

Data Collection

A colorless platelet crystal of $C_{21}H_{30}F_2$ having approximate dimensions of 0.100 x 0.040 x 0.010 mm was mounted in a loop. All measurements were made on a Rigaku XtaLAB P200 diffractometer using multi-layer mirror monochromated Mo-Kα radiation.

The crystal-to-detector distance was 45.01 mm.

Cell constants and an orientation matrix for data collection corresponded to a primitive monoclinic cell with dimensions:

a = 11.934(3) Å
b = 5.5818(15) Å β = 90.656(6)°
c = 26.703(7) Å
V = 1778.7(8) $Å^3$

For Z = 4 and F.W. = 320.46, the calculated density is 1.197 g/cm^3. Based on the reflection conditions of:

h0l: l = 2n

packing considerations, a statistical analysis of intensity distribution, and the successful solution and refinement of the structure, the space group was determined to be:

P2/c (#13)

The data were collected at a temperature of -100 ± 1°C to a maximum 2θ value of 50.8°. A total of 1080 oscillation images were collected. A sweep of data was done using ω scans from -100.0 to 80.0° in 0.50° step, at χ=45.0° and ϕ = 0.0°. The exposure rate was 24.0 [sec./°]. The detector swing angle was -10.42°. A second sweep was performed using ω scans from -100.0 to 80.0° in 0.50° step, at χ=45.0° and ϕ = 90.0°. The exposure rate was 24.0 [sec./°]. The detector swing angle was -10.42°. Another sweep was performed using ω scans from -100.0 to 80.0° in 0.50° step, at χ=45.0° and ϕ = 180.0°. The exposure rate was 24.0 [sec./°]. The detector swing angle was -10.42°. The crystal-to-detector distance was 45.01 mm. Readout was performed in the 0.172 mm pixel mode.

Data Reduction

Of the 19791 reflections were collected, where 3250 were unique (R_{int} = 0.1637); equivalent reflections were merged. Data were collected and processed using CrystalClear (Rigaku). [1]

The linear absorption coefficient, μ, for Mo-Kα radiation is 0.816 cm^{-1}. An empirical absorption correction was applied which resulted in transmission factors ranging from 0.827 to 0.999. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by direct methods[2] and expanded using Fourier techniques. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined using the riding model. The final cycle of full-matrix least-squares refinement[3] on F^2 was based on 3250 observed reflections and 228 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R1 = \Sigma \, ||Fo| - |Fc|| \, / \, \Sigma \, |Fo| = 0.0810$$

$$wR2 = [\Sigma (w (Fo^2 - Fc^2)^2) / \Sigma \, w(Fo^2)^2]^{1/2} = 0.1732$$

The goodness of fit[4] was 1.02. Unit weights were used. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.17 and -0.16 $e^-/Å^3$, respectively.

Neutral atom scattering factors were taken from International Tables for Crystallography (IT), Vol. C, Table 6.1.1.4 [5]. Anomalous dispersion effects were included in Fcalc[6]; the values for Δf' and Δf'' were those of Creagh and McAuley[7]. The values for the mass attenuation coefficients are those of Creagh and Hubbell[8]. All calculations were performed using the CrystalStructure[9] crystallographic software package except for refinement, which was performed using SHELXL2013[10].

References

(1) CrystalClear: Data Collection and Processing Software, Rigaku Corporation (1998-2014). Tokyo 196-8666, Japan.

(2) SIR2011: Burla, M. C., Caliandro, R., Camalli, M., Carrozzini, B., Cascarano, G. L., Giacovazzo, C., Mallamo, M., Mazzone, A., Polidori, G. and Spagna, R. (2012). J. Appl. Cryst. 45, 357-361.

(3) Least Squares function minimized: (SHELXL2013)

$$\Sigma w(F_o^2\text{-}F_c^2)^2$$ where w = Least Squares weights.

(4) Goodness of fit is defined as:

$$[\Sigma w(F_o^2\text{-}F_c^2)^2/(N_o\text{-}N_v)]^{1/2}$$

where: N_o = number of observations
N_v = number of variables

(5) International Tables for Crystallography, Vol.C (1992). Ed. A.J.C. Wilson, Kluwer Academic Publishers, Dordrecht, Netherlands, Table 6.1.1.4, pp. 572.

(6) Ibers, J. A. & Hamilton, W. C.; Acta Crystallogr., 17, 781 (1964).

(7) Creagh, D. C. & McAuley, W.J .; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.6.8, pages 219-222 (1992).

(8) Creagh, D. C. & Hubbell, J.H..; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.4.3, pages 200-206 (1992).

(9) CrystalStructure 4.1: Crystal Structure Analysis Package, Rigaku Corporation (2000-2014). Tokyo 196-8666, Japan.

(10) SHELXL2013: Sheldrick, G. M. (2008). Acta Cryst. A64, 112-122.

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$C_{21}H_{30}F_2$
Formula Weight	320.46
Crystal Color, Habit	colorless, platelet
Crystal Dimensions	0.100 X 0.040 X 0.010 mm
Crystal System	monoclinic
Lattice Type	Primitive
Lattice Parameters	a = 11.934(3) Å b = 5.5818(15) Å c = 26.703(7) Å β = 90.656(6) ° V = 1778.7(8) $Å^3$
Space Group	P2/c (#13)
Z value	4
D_{calc}	1.197 g/cm^3
F_{000}	696.00
μ(MoKα)	0.816 cm^{-1}

B. Intensity Measurements

Diffractometer	XtaLAB P200
Radiation monochromated	MoKα (λ = 0.71075 Å) multi-layer mirror
Voltage, Current	45kV, 66mA
Temperature	-100.0°C
Detector Aperture	83.8 x 70.0 mm
Data Images	1080 exposures
ω oscillation Range (χ=45.0, ϕ=0.0)	-100.0 - 80.0°
Exposure Rate	24.0 sec./°
Detector Swing Angle	-10.42°
ω oscillation Range (χ=45.0, ϕ=90.0)	-100.0 - 80.0°
Exposure Rate	24.0 sec./°
Detector Swing Angle	-10.42°
ω oscillation Range (χ=45.0, ϕ=180.0)	-100.0 - 80.0°
Exposure Rate	24.0 sec./°
Detector Swing Angle	-10.42°
Detector Position	45.01 mm
Pixel Size	0.172 mm
$2\theta_{max}$	50.8°
No. of Reflections Measured	Total: 19791 Unique: 3250 (R_{int} = 0.1637)
Corrections	Lorentz-polarization

Absorption
(trans. factors: 0.827 - 0.999)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SIR2011)
Refinement	Full-matrix least-squares on F^2
Function Minimized	Σ w $(Fo^2 - Fc^2)^2$
Least Squares Weights	w = 1/ [$\sigma^2(Fo^2)$ + $(0.0462 \cdot P)^2$ + 1.2477 · P] where P = (Max(Fo^2,0) + $2Fc^2$)/3
$2\theta_{max}$ cutoff	50.8^o
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (All reflections)	3250
No. Variables	228
Reflection/Parameter Ratio	14.25
Residuals: R1 (I>2.00σ(I))	0.0810
Residuals: R (All reflections)	0.2025
Residuals: wR2 (All reflections)	0.1732
Goodness of Fit Indicator	1.019
Max Shift/Error in Final Cycle	0.000
Maximum peak in Final Diff. Map	0.17 $e^-/Å^3$
Minimum peak in Final Diff. Map	-0.16 $e^-/Å^3$

Table 1. Atomic coordinates and B_{iso}/B_{eq} and occupancy

atom	x	y	z	B_{eq}	occ
F1	0.9313(3)	-0.3605(5)	0.34185(12)	5.14(9)	
	0.742(4)				
F2	1.1524(3)	-0.3612(6)	0.36773(11)	5.05(9)	
	0.742(4)				
F1A	0.8168(7)	-0.0626(16)	0.3923(3)	4.2(2)	
	0.258(4)				
F2A	0.9569(9)	0.1699(19)	0.4588(4)	5.9(3)	
	0.258(4)				
C1	0.9965(3)	-0.1729(8)	0.32871(15)	3.78(9)	1
C2	1.0884(3)	-0.1613(8)	0.36712(15)	4.18(9)	1
C3	1.0477(3)	-0.1147(7)	0.41880(14)	3.87(9)	1
C4	0.9790(4)	0.1147(8)	0.41868(18)	4.67(10)	1
C5	0.8853(3)	0.1076(7)	0.38025(14)	3.67(9)	1
C6	0.9283(3)	0.0577(6)	0.32800(14)	2.88(8)	1
C7	0.8381(3)	0.0678(6)	0.28784(13)	2.69(7)	1
C8	0.7545(3)	-0.1040(7)	0.28260(14)	3.11(8)	1
C9	0.6719(3)	-0.0834(7)	0.24659(14)	3.18(8)	1
C10	0.6657(3)	0.1097(6)	0.21377(13)	2.64(7)	1
C11	0.7494(3)	0.2829(6)	0.21954(13)	2.92(8)	1
C12	0.8326(3)	0.2612(6)	0.25523(14)	2.94(8)	1
C13	0.5758(3)	0.1297(7)	0.17432(13)	2.76(7)	1
C14	0.6196(3)	0.0809(7)	0.12207(13)	3.39(8)	1
C15	0.5274(3)	0.0958(7)	0.08224(14)	3.44(8)	1
C16	0.4656(3)	0.3359(6)	0.08250(13)	2.88(8)	1
C17	0.4229(3)	0.3843(7)	0.13485(13)	3.43(8)	1
C18	0.5156(3)	0.3729(7)	0.17400(13)	3.41(8)	1
C19	0.3744(3)	0.3433(7)	0.04208(13)	3.35(8)	1
C20	0.3148(3)	0.5811(7)	0.03574(16)	4.18(9)	1
C21	0.2268(3)	0.5828(8)	-0.00557(15)	4.84(10)	1

$B_{eq} = 8/3\ \pi^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$

Table 2. Atomic coordinates and B_{iso} and occupancy involving hydrogen atoms

atom	x	y	z	B_{iso}	occ
H3A	1.11224	-0.09855	0.44218	4.649	1
H3B	1.00100	-0.25047	0.43012	4.649	1
H6	0.98212	0.18947	0.32026	3.453	1
H8	0.75444	-0.23853	0.30439	3.734	1
H9	0.61683	-0.20589	0.24400	3.821	1
H11	0.74880	0.41915	0.19823	3.500	1
H12	0.88822	0.38263	0.25768	3.532	1
H13	0.51835	0.00437	0.18164	3.316	1
H14A	0.65364	-0.08075	0.12129	4.064	1
H14B	0.67883	0.19882	0.11426	4.064	1
H15A	0.47277	-0.03469	0.08784	4.133	1
H15B	0.56078	0.07099	0.04887	4.133	1
H16	0.52139	0.46356	0.07450	3.459	1
H17A	0.38790	0.54501	0.13568	4.120	1
H17B	0.36463	0.26491	0.14306	4.120	1
H18A	0.48337	0.40229	0.20745	4.087	1
H18B	0.57075	0.50111	0.16738	4.087	1
H19A	0.40845	0.29967	0.00969	4.017	1
H19B	0.31785	0.21946	0.04988	4.017	1
H20A	0.37113	0.70627	0.02854	5.013	1
H20B	0.27863	0.62332	0.06773	5.013	1
H21A	0.16651	0.47105	0.00286	5.811	1
H21B	0.26089	0.53383	-0.03716	5.811	1
H21C	0.19589	0.74470	-0.00913	5.811	1
H1A	1.02894	-0.20191	0.29488	4.530	
	0.742(4)				
H1B	0.94670	-0.30974	0.33621	4.530	
	0.258(4)				
H1C	1.02916	-0.19964	0.29526	4.530	
	0.258(4)				
H2A	1.13818	-0.02444	0.35786	5.016	
	0.742(4)				
H2B	1.13003	-0.31470	0.36696	5.016	
	0.258(4)				
H2C	1.14139	-0.03274	0.35775	5.016	
	0.258(4)				
H4A	0.94695	0.13926	0.45232	5.600	
	0.742(4)				
H4B	1.02874	0.25214	0.41149	5.600	
	0.742(4)				
H4C	1.03130	0.24178	0.40666	5.600	
	0.258(4)				
H5A	0.84546	0.26317	0.38027	4.407	
	0.742(4)				

H5B	0.83108	-0.01844	0.38957	4.407
	0.742(4)			
H5C	0.84461	0.26421	0.38034	4.407
	0.258(4)			

Table 3. Anisotropic displacement parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
F1	0.079(2)	0.0299(18)	0.085(3)	-0.0041(17)	-0.037(2)	0.0015(18)
F2	0.071(2)	0.063(2)	0.058(2)	0.0389(19)	-0.0040(17)	-0.0069(18)
F1A	0.044(5)	0.063(6)	0.053(6)	-0.009(5)	0.013(4)	0.015(5)
F2A	0.101(8)	0.087(8)	0.037(6)	0.033(6)	-0.011(5)	-0.024(6)
C1	0.050(3)	0.051(3)	0.042(3)	0.007(2)	0.005(2)	0.003(2)
C2	0.044(3)	0.055(3)	0.060(3)	0.013(2)	-0.000(2)	0.015(2)
C3	0.051(3)	0.052(3)	0.045(3)	0.009(2)	-0.008(2)	-0.000(2)
C4	0.067(3)	0.058(3)	0.052(3)	0.012(2)	-0.023(3)	-0.008(3)
C5	0.056(3)	0.038(3)	0.046(3)	0.007(2)	-0.003(2)	-0.002(2)
C6	0.032(2)	0.030(2)	0.047(2)	-0.0029(18)	-0.0016(19)	0.0116(19)
C7	0.034(2)	0.029(2)	0.040(2)	0.0022(18)	-0.0004(19)	-0.0005(19)
C8	0.041(2)	0.033(2)	0.044(2)	-0.0034(19)	-0.002(2)	0.0080(19)
C9	0.036(2)	0.032(2)	0.052(3)	-0.0048(18)	-0.001(2)	-0.001(2)
C10	0.037(2)	0.029(2)	0.035(2)	-0.0024(18)	0.0018(18)	-0.0041(18)
C11	0.041(2)	0.031(2)	0.039(2)	-0.0023(18)	0.001(2)	0.0070(18)
C12	0.036(2)	0.030(2)	0.046(2)	-0.0026(18)	0.002(2)	0.0043(19)
C13	0.035(2)	0.037(2)	0.033(2)	-0.0044(18)	0.0003(18)	0.0017(19)
C14	0.047(2)	0.036(2)	0.045(3)	0.0037(19)	-0.005(2)	-0.004(2)
C15	0.051(3)	0.044(2)	0.036(2)	0.006(2)	-0.002(2)	-0.006(2)
C16	0.044(2)	0.030(2)	0.035(2)	-0.0056(18)	-0.0033(19)	-0.0016(19)
C17	0.044(2)	0.047(3)	0.040(2)	0.007(2)	-0.0003(19)	-0.002(2)
C18	0.047(3)	0.049(3)	0.034(2)	0.011(2)	-0.000(2)	-0.002(2)

C19	0.046(3)	0.043(2)	0.039(2)	-0.003(2)	-0.003(2)	0.000(2)
C20	0.054(3)	0.046(3)	0.058(3)	0.001(2)	-0.015(2)	-0.002(2)
C21	0.058(3)	0.057(3)	0.068(3)	-0.003(2)	-0.020(3)	0.011(3)

The general temperature factor expression: $\exp(-2\pi^2(a^{*2}U_{11}h^2 + b^{*2}U_{22}k^2 + c^{*2}U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$

Table 4. Bond lengths (Å)

atom	atom	distance	atom	atom	distance
F1	C1	1.354(5)	F2	C2	1.352(5)
F1A	C5	1.297(9)	F2A	C4	1.148(11)
C1	C2	1.495(6)	C1	C6	1.523(5)
C2	C3	1.491(6)	C3	C4	1.520(6)
C4	C5	1.510(6)	C5	C6	1.518(5)
C6	C7	1.511(5)	C7	C8	1.390(5)
C7	C12	1.388(5)	C8	C9	1.374(5)
C9	C10	1.391(5)	C10	C11	1.397(5)
C10	C13	1.500(5)	C11	C12	1.374(5)
C13	C14	1.520(5)	C13	C18	1.536(5)
C14	C15	1.524(5)	C15	C16	1.530(5)
C16	C17	1.517(5)	C16	C19	1.524(5)
C17	C18	1.514(5)	C19	C20	1.514(5)
C20	C21	1.515(6)			

Table 5. Bond lengths involving hydrogens (Å)

atom	atom	distance	atom	atom	distance
C1	H1A	1.000	C1	H1B	0.990
C1	H1C	0.990	C2	H2A	1.000
C2	H2B	0.990	C2	H2C	0.990
C3	H3A	0.990	C3	H3B	0.990
C4	H4A	0.990	C4	H4B	0.990
C4	H4C	1.000	C5	H5A	0.990
C5	H5B	0.990	C5	H5C	1.000
C6	H6	1.000	C8	H8	0.950
C9	H9	0.950	C11	H11	0.950
C12	H12	0.950	C13	H13	1.000
C14	H14A	0.990	C14	H14B	0.990
C15	H15A	0.990	C15	H15B	0.990
C16	H16	1.000	C17	H17A	0.990
C17	H17B	0.990	C18	H18A	0.990
C18	H18B	0.990	C19	H19A	0.990
C19	H19B	0.990	C20	H20A	0.990
C20	H20B	0.990	C21	H21A	0.980
C21	H21B	0.980	C21	H21C	0.980

Table 6. Bond angles (°)

atom	atom	atom	angle	atom	atom	atom	angle
F1	C1	C2	106.0(3)	F1	C1	C6	110.4(3)
C2	C1	C6	111.1(3)	F2	C2	C1	112.5(4)
F2	C2	C3	108.8(3)	C1	C2	C3	113.5(3)
C2	C3	C4	109.0(3)	F2A	C4	C3	110.7(6)
F2A	C4	C5	117.7(6)	C3	C4	C5	112.0(4)
F1A	C5	C4	108.3(5)	F1A	C5	C6	108.3(5)
C4	C5	C6	112.0(3)	C1	C6	C5	109.3(3)
C1	C6	C7	114.6(3)	C5	C6	C7	113.6(3)
C6	C7	C8	123.4(3)	C6	C7	C12	120.2(3)
C8	C7	C12	116.4(3)	C7	C8	C9	121.4(3)
C8	C9	C10	122.5(3)	C9	C10	C11	115.7(3)
C9	C10	C13	122.2(3)	C11	C10	C13	122.1(3)
C10	C11	C12	121.7(3)	C7	C12	C11	122.2(3)
C10	C13	C14	112.4(3)	C10	C13	C18	113.6(3)
C14	C13	C18	108.6(3)	C13	C14	C15	112.2(3)
C14	C15	C16	112.9(3)	C15	C16	C17	109.1(3)
C15	C16	C19	111.1(3)	C17	C16	C19	113.8(3)
C16	C17	C18	112.2(3)	C13	C18	C17	112.3(3)
C16	C19	C20	115.7(3)	C19	C20	C21	114.1(3)

Table 7. Bond angles involving hydrogens (°)

atom	atom	atom	angle	atom	atom	atom	angle
F1	C1	H1A	109.8	C2	C1	H1A	109.8
C2	C1	H1B	109.4	C2	C1	H1C	109.4
C6	C1	H1A	109.8	C6	C1	H1B	109.4
C6	C1	H1C	109.4	H1B	C1	H1C	108.0
F2	C2	H2A	107.2	C1	C2	H2A	107.2
C1	C2	H2B	108.9	C1	C2	H2C	108.9
C3	C2	H2A	107.2	C3	C2	H2B	108.9
C3	C2	H2C	108.9	H2B	C2	H2C	107.7
C2	C3	H3A	109.9	C2	C3	H3B	109.9
C4	C3	H3A	109.9	C4	C3	H3B	109.9
H3A	C3	H3B	108.3	F2A	C4	H4C	105.1
C3	C4	H4A	109.2	C3	C4	H4B	109.2
C3	C4	H4C	105.1	C5	C4	H4A	109.2
C5	C4	H4B	109.2	C5	C4	H4C	105.1
H4A	C4	H4B	107.9	F1A	C5	H5C	109.4
C4	C5	H5A	109.2	C4	C5	H5B	109.2
C4	C5	H5C	109.4	C6	C5	H5A	109.2
C6	C5	H5B	109.2	C6	C5	H5C	109.4
H5A	C5	H5B	107.9	C1	C6	H6	106.2
C5	C6	H6	106.2	C7	C6	H6	106.2
C7	C8	H8	119.3	C9	C8	H8	119.3
C8	C9	H9	118.7	C10	C9	H9	118.7

C10	C11	H11	119.1	C12	C11	H11
	119.1					
C7	C12	H12	118.9	C11	C12	H12
	118.9					
C10	C13	H13	107.3	C14	C13	H13
	107.3					
C18	C13	H13	107.3	C13	C14	H14A
	109.2					
C13	C14	H14B	109.2	C15	C14	H14A
	109.2					
C15	C14	H14B	109.2	H14A	C14	H14B
	107.9					
C14	C15	H15A	109.0	C14	C15	H15B
	109.0					
C16	C15	H15A	109.0	C16	C15	H15B
	109.0					
H15A	C15	H15B	107.8	C15	C16	H16
	107.5					
C17	C16	H16	107.5	C19	C16	H16
	107.5					
C16	C17	H17A	109.2	C16	C17	H17B
	109.2					
C18	C17	H17A	109.2	C18	C17	H17B
	109.2					
H17A	C17	H17B	107.9	C13	C18	H18A
	109.1					
C13	C18	H18B	109.2	C17	C18	H18A
	109.1					
C17	C18	H18B	109.1	H18A	C18	H18B
	107.9					

Table 7. Bond angles involving hydrogens (°) (continued)

atom	atom	atom	angle	atom	atom	atom	angle
C16	C19	H19A	108.4	C16	C19	H19B	108.4
C20	C19	H19A	108.4	C20	C19	H19B	108.4
H19A	C19	H19B	107.4	C19	C20	H20A	108.7
C19	C20	H20B	108.7	C21	C20	H20A	108.7
C21	C20	H20B	108.7	H20A	C20	H20B	107.7
C20	C21	H21A	109.5	C20	C21	H21B	109.5
C20	C21	H21C	109.5	H21A	C21	H21B	109.5
H21A	C21	H21C	109.5	H21B	C21	H21C	109.5

Table 8. Torsion Angles(O)
(Those having bond angles > 160 or < 20 degrees are excluded.)

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
F1	C1	C2	F2	61.7(4)	F1	C1	C2	C3	-62.3(4)
F1	C1	C6	C5	62.2(4)	F1	C1	C6	C7	-66.6(4)
C2	C1	C6	C5	-55.0(4)	C2	C1	C6	C7	176.2(3)
C6	C1	C2	F2	-178.4(3)	C6	C1	C2	C3	57.6(4)
F2	C2	C3	C4	178.1(3)	C1	C2	C3	C4	-55.9(4)
C2	C3	C4	F2A	-172.0(3)	C2	C3	C4	C5	54.5(4)
F2A	C4	C5	F1A	-66.6(8)	F2A	C4	C5	C6	174.0(7)
C3	C4	C5	F1A	63.4(4)	C3	C4	C5	C6	-56.0(4)
F1A	C5	C6	C1	-64.4(5)	F1A	C5	C6	C7	65.0(5)
C4	C5	C6	C1	55.0(4)	C4	C5	C6	C7	-175.6(3)
C1	C6	C7	C8	55.7(4)	C1	C6	C7	C12	-127.4(3)
C5	C6	C7	C8	-70.9(4)	C5	C6	C7	C12	106.0(3)
C6	C7	C8	C9	177.8(3)	C6	C7	C12	C11	-177.2(3)
C8	C7	C12	C11	-0.1(5)	C12	C7	C8	C9	0.7(5)
C7	C8	C9	C10	-0.8(6)	C8	C9	C10	C11	0.2(5)
C8	C9	C10	C13	179.5(3)	C9	C10	C11	C12	0.5(5)
C9	C10	C13	C14	-106.4(4)	C9	C10	C13	C18	129.8(3)
C11	C10	C13	C14	72.9(4)	C11	C10	C13	C18	-50.9(4)
C13	C10	C11	C12	-178.9(3)	C10	C11	C12	C7	-0.5(5)
C10	C13	C14	C15	178.7(3)	C10	C13	C18	C17	-178.2(3)
C14	C13	C18	C17	56.0(3)	C18	C13	C14	C15	-54.7(4)

C13	C14	C15	C16	55.9(4)	C14	C15	C16	C17	-53.9(4)
C14	C15	C16	C19	179.9(3)	C15	C16	C17	C18	54.7(4)
C15	C16	C19	C20	-174.6(3)	C17	C16	C19	C20	61.8(4)
C19	C16	C17	C18	179.4(3)	C16	C17	C18	C13	-57.7(4)
C16	C19	C20	C21	178.6(3)					

Table 9. Intramolecular contacts less than 3.60 Å

atom	atom	distance	atom	atom	distance
F1	F2	2.721(5)	F1	C3	2.823(5)
F1	C4	3.397(6)	F1	C5	2.862(5)
F1	C7	2.999(5)	F1	C8	2.988(5)
F1A	F2A	2.750(13)	F1A	C1	2.819(9)
F1A	C2	3.364(9)	F1A	C3	2.852(9)
F1A	C7	2.897(9)	F1A	C8	3.023(9)
F2A	C2	3.459(11)	F2A	C6	3.561(10)
C1	C4	2.899(6)	C1	C8	3.150(5)
C2	C5	2.876(6)	C3	C6	2.959(5)
C5	C8	3.247(5)	C5	C12	3.497(5)
C7	C10	2.847(5)	C8	C11	2.739(5)
C9	C12	2.725(5)	C9	C14	3.498(5)
C11	C14	3.218(5)	C11	C18	3.073(5)
C13	C16	2.999(5)	C14	C17	2.917(5)
C15	C18	2.902(5)	C17	C20	3.130(6)

Table 10. Intramolecular contacts less than 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
F1	H3B	2.565	F1	H6	3.183
F1	H8	2.422	F1	H2A	3.126
F1	H5B	2.595	F2	H3A	2.521
F2	H3B	2.548	F2	H1A	2.585
F1A	H3B	2.627	F1A	H6	3.109
F1A	H8	2.644	F1A	H1B	2.570
F1A	H4C	3.093	F2A	H3A	2.429
F2A	H3B	2.526	F2A	H5C	2.529
C1	H3A	3.341	C1	H3B	2.742
C1	H8	2.976	C1	H4B	3.262
C1	H4C	3.137	C1	H5A	3.335
C1	H5B	2.712	C1	H5C	3.346
C2	H6	2.640	C2	H4A	3.306
C2	H4B	2.694	C2	H4C	2.580
C2	H5B	3.236	C3	H6	3.221
C3	H1A	3.350	C3	H1B	2.728
C3	H1C	3.338	C3	H5A	3.358
C3	H5B	2.745	C3	H5C	3.368
C4	H6	2.662	C4	H1B	3.254
C4	H2A	2.630	C4	H2B	3.310
C4	H2C	2.674	C5	H3A	3.360
C5	H3B	2.763	C5	H8	3.194

C5	H1A	3.348	C5	H1B
	2.714			
C5	H1C	3.336	C5	H2A
	3.170			
C5	H2C	3.218	C6	H3B
	3.331			
C6	H8	2.721	C6	H12
	2.650			
C6	H2A	2.660	C6	H2B
	3.338			
C6	H2C	2.703	C6	H4A
	3.356			
C6	H4B	2.743	C6	H4C
	2.631			
C7	H9	3.257	C7	H11
	3.263			
C7	H1A	2.735	C7	H1B
	2.784			
C7	H1C	2.731	C7	H5A
	2.699			
C7	H5B	2.761	C7	H5C
	2.703			
C8	H6	3.318	C8	H12
	3.224			
C8	H1A	3.333	C8	H1B
	2.925			
C8	H1C	3.335	C8	H5A
	3.481			
C8	H5B	3.027	C8	H5C
	3.483			

Table 10. Intramolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
C9	H11	3.226	C9	H13	2.556
C9	H14A	3.351	C10	H8	3.271
C10	H12	3.267	C10	H14A	2.691
C10	H14B	2.710	C10	H18A	2.724
C10	H18B	2.749	C11	H9	3.225
C11	H13	3.314	C11	H14A	3.499
C11	H14B	2.963	C11	H18A	3.257
C11	H18B	2.811	C12	H6	2.508
C12	H8	3.226	C12	H5A	3.341
C12	H5C	3.342	C13	H9	2.681
C13	H11	2.693	C13	H15A	2.761
C13	H15B	3.369	C13	H16	3.311
C13	H17A	3.378	C13	H17B	2.751
C14	H11	3.163	C14	H16	2.741
C14	H17B	3.266	C14	H18A	3.338
C14	H18B	2.706	C15	H13	2.706
C15	H17A	3.339	C15	H17B	2.715
C15	H18B	3.245	C15	H19A	2.646
C15	H19B	2.725	C16	H13	3.285
C16	H14A	3.386	C16	H14B	2.781
C16	H18A	3.361	C16	H18B	2.738

C16	H20A	2.754	C16	H20B
	2.773			
C17	H13	2.706	C17	H14B
	3.277			
C17	H15A	2.723	C17	H15B
	3.335			
C17	H19A	3.378	C17	H19B
	2.739			
C17	H20A	3.410	C17	H20B
	2.808			
C18	H11	2.862	C18	H14A
	3.341			
C18	H14B	2.712	C18	H15A
	3.271			
C18	H16	2.706	C19	H15A
	2.700			
C19	H15B	2.698	C19	H17A
	2.744			
C19	H17B	2.736	C19	H21A
	2.775			
C19	H21B	2.716	C19	H21C
	3.371			
C20	H16	2.743	C20	H17A
	2.805			
C20	H17B	3.412	C21	H19A
	2.710			
C21	H19B	2.730	H3A	H2A
	2.314			
H3A	H2B	2.355	H3A	H2C
	2.314			
H3A	H4A	2.396	H3A	H4B
	2.340			

Table 10. Intramolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H3A	H4C	2.328	H3B	H1B	2.604
H3B	H2A	2.840	H3B	H2B	2.325
H3B	H2C	2.845	H3B	H4A	2.347
H3B	H4B	2.869	H3B	H4C	2.842
H3B	H5B	2.629	H6	H12	2.273
H6	H1A	2.356	H6	H1B	2.851
H6	H1C	2.342	H6	H2A	2.421
H6	H2B	3.541	H6	H2C	2.472
H6	H4A	3.567	H6	H4B	2.517
H6	H4C	2.392	H6	H5A	2.336
H6	H5B	2.846	H6	H5C	2.345
H8	H9	2.296	H8	H1A	3.295
H8	H1B	2.470	H8	H1C	3.297
H8	H5B	2.733	H9	H13	2.342
H9	H14A	3.384	H11	H12	2.296
H11	H14B	2.682	H11	H18A	3.181
H11	H18B	2.316	H12	H5A	3.385
H12	H5C	3.387	H13	H14A	2.343
H13	H14B	2.857	H13	H15A	2.566
H13	H17B	2.549	H13	H18A	2.364
H13	H18B	2.869	H14A	H15A	2.341

H14A	H15B	2.374	H14B	H15A
	2.864			
H14B	H15B	2.342	H14B	H16
	2.607			
H14B	H18B	2.562	H15A	H16
	2.864			
H15A	H17B	2.585	H15A	H19A
	2.897			
H15A	H19B	2.533	H15B	H16
	2.345			
H15B	H19A	2.446	H15B	H19B
	3.016			
H16	H17A	2.339	H16	H17B
	2.856			
H16	H18B	2.551	H16	H19A
	2.365			
H16	H19B	2.855	H16	H20A
	2.551			
H16	H20B	3.035	H17A	H18A
	2.357			
H17A	H18B	2.344	H17A	H19B
	3.034			
H17A	H20A	3.004	H17A	H20B
	2.265			
H17B	H18A	2.344	H17B	H18B
	2.859			
H17B	H19B	2.556	H17B	H20B
	3.009			
H19A	H20A	2.368	H19A	H20B
	2.850			
H19A	H21A	3.045	H19A	H21B
	2.515			

Table 10. Intramolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom
	distance			
H19A	H21C	3.582	H19B	H20A
	2.850			
H19B	H20B	2.352	H19B	H21A
	2.600			
H19B	H21B	2.984	H20A	H21A
	2.849			
H20A	H21B	2.384	H20A	H21C
	2.320			
H20B	H21A	2.337	H20B	H21B
	2.851			
H20B	H21C	2.366	H1A	H2A
	2.336			
H1B	H2B	2.328	H1B	H2C
	2.844			
H1C	H2B	2.340	H1C	H2C
	2.323			
H2A	H4A	3.541	H2A	H4B
	2.487			
H2B	H4C	3.491	H2C	H4C
	2.412			
H4A	H5A	2.365	H4A	H5B
	2.333			
H4B	H5A	2.333	H4B	H5B
	2.856			
H4C	H5C	2.332		

Table 11. Intermolecular contacts less than 3.60 Å

atom	atom	distance	atom	atom	distance
F1	C5[1]	3.190(5)	F1	C6[1]	3.269(5)
F1	C12[1]	3.336(5)	F2	C11[2]	3.288(5)
F1A	C20[3]	3.191(9)	F2A	F2A[4]	3.074(14)
F2A	C3[4]	3.284(10)	F2A	C21[5]	3.426(11)
C3	F2A[4]	3.284(10)	C5	F1[6]	3.190(5)
C6	F1[6]	3.269(5)	C11	F2[7]	3.288(5)
C12	F1[6]	3.336(5)	C20	F1A[8]	3.191(9)
C21	F2A[5]	3.426(11)			

Symmetry Operators:

(1) X,Y-1,Z
(2) -X+2,Y-1,-Z+1/2
(3) -X+1,Y-1,-Z+1/2
(4) -X+2,-Y,-Z+1
(5) -X+1,Y,-Z+1/2
(6) X,Y+1,Z
(7) -X+2,Y+1,-Z+1/2
(8) -X+1,Y+1,-Z+1/2

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
F1	H6[1]	2.649	F1	H12[1]	2.710
F1	H20B[2]	3.500	F1	H4B[1]	3.072
F1	H5A[1]	2.557	F2	H6[1]	3.460
F2	H11[3]	2.458	F2	H14A[4]	2.807
F2	H14B[3]	3.209	F2	H18B[3]	3.529
F2	H21B[5]	2.997	F2	H4B[1]	2.871
F1A	H15A[6]	3.505	F1A	H17A[2]	3.358
F1A	H17B[6]	2.979	F1A	H19B[6]	2.739
F1A	H20A[2]	3.357	F1A	H20B[2]	2.353
F1A	H21C[2]	3.305	F2A	H3A[7]	2.807
F2A	H3B[8]	3.367	F2A	H3B[7]	3.037
F2A	H19B[6]	3.296	F2A	H21A[6]	2.465
F2A	H21A[9]	3.405	F2A	H21C[2]	3.289
F2A	H21C[9]	3.007	C1	H6[1]	3.571
C1	H12[1]	3.372	C1	H1A[4]	3.315
C1	H1C[4]	3.324	C2	H11[3]	3.519
C2	H14A[4]	3.123	C2	H14B[4]	3.460
C2	H4B[1]	3.557	C2	H4C[1]	3.564
C3	H21A[5]	3.306	C3	H21C[9]	3.319
C3	H4A[7]	3.444	C3	H4B[1]	3.547
C4	H3B[8]	3.566	C4	H21A[6]	3.383
C4	H21C[9]	3.304	C5	H17B[6]	3.164

C5	H19B[6]	3.139	C5	H1B[8]
	3.538			
C6	H1B[8]	3.545	C7	H17B[6]
	3.251			
C7	H1A[4]	3.121	C7	H1C[4]
	3.121			
C8	H11[1]	3.487	C8	H12[1]
	3.351			
C8	H13[6]	3.457	C8	H17A[2]
	3.402			
C8	H17B[6]	3.204	C8	H1A[4]
	3.374			
C8	H1C[4]	3.376	C9	H9[6]
	3.524			
C9	H11[1]	3.201	C9	H13[6]
	3.028			
C9	H17B[6]	3.561	C9	H18A[6]
	3.513			
C9	H18B[1]	3.354	C9	H2C[4]
	3.599			
C10	H18A[6]	3.216	C10	H2A[4]
	3.130			
C10	H2C[4]	3.111	C11	H6[4]
	3.427			
C11	H8[8]	3.503	C11	H9[8]
	3.331			
C11	H18A[6]	3.477	C11	H2A[4]
	3.013			
C11	H2B[10]	3.539	C11	H2C[4]
	3.020			

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
C12	H6[4]	3.035	C12	H8[8]	3.228
C12	H12[4]	3.421	C12	H1A[4]	3.355
C12	H1B[8]	3.492	C12	H1C[4]	3.348
C12	H2A[4]	3.436	C12	H2C[4]	3.451
C13	H18A[6]	3.582	C13	H18B[1]	3.514
C14	H18B[1]	3.506	C14	H21B[11]	3.446
C14	H2A[4]	2.992	C14	H2C[4]	2.966
C15	H16[1]	3.536	C15	H19A[12]	3.395
C15	H20A[1]	3.194	C15	H20A[11]	3.394
C15	H21B[11]	3.490	C16	H15A[8]	3.517
C16	H19A[11]	3.542	C16	H20A[11]	3.574
C16	H21B[11]	3.569	C17	H8[13]	3.409
C17	H15A[8]	3.530	C17	H5A[6]	3.294
C17	H5C[6]	3.283	C18	H9[8]	3.230
C18	H9[13]	3.592	C18	H13[8]	3.531
C18	H18A[6]	3.170	C19	H15B[12]	3.448
C19	H16[11]	3.534	C19	H19A[11]	3.561
C19	H20A[1]	3.574	C19	H5A[6]	3.392
C19	H5C[6]	3.383	C20	H15A[8]	3.167
C20	H15B[11]	3.340	C20	H16[11]	3.561
C20	H19A[11]	3.594	C20	H19B[8]	3.583

C20	H5A[6]	3.455	C20	H5B[13]
	3.476			
C20	H5C[6]	3.445	C21	H3A[14]
	3.328			
C21	H3A[15]	3.472	C21	H3B[13]
	3.527			
C21	H14B[11]	3.355	C21	H15B[11]
	3.401			
C21	H16[11]	3.552	C21	H4A[6]
	3.538			
C21	H4B[15]	3.349	C21	H4C[15]
	3.430			
H3A	F2A[7]	2.807	H3A	C21[5]
	3.328			
H3A	C21[9]	3.472	H3A	H14A[4]
	3.286			
H3A	H14B[4]	3.366	H3A	H21A[5]
	2.710			
H3A	H21B[5]	3.055	H3A	H21C[9]
	2.561			
H3A	H4A[7]	2.921	H3B	F2A[1]
	3.367			
H3B	F2A[7]	3.037	H3B	C4[1]
	3.566			
H3B	C21[2]	3.527	H3B	H20B[2]
	3.412			
H3B	H21A[2]	3.114	H3B	H21A[5]
	3.017			
H3B	H21B[5]	3.581	H3B	H21C[2]
	3.176			
H3B	H4A[1]	3.518	H3B	H4A[7]
	3.253			

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H3B	H4B[1]	2.841	H3B	H4C[1]	2.926
H3B	H5A[1]	3.540	H3B	H5C[1]	3.540
H6	F1[8]	2.649	H6	F2[8]	3.460
H6	C1[8]	3.571	H6	C11[4]	3.427
H6	C12[4]	3.035	H6	H11[4]	3.498
H6	H12[4]	2.822	H6	H1A[8]	3.510
H6	H1B[8]	2.860	H6	H1C[8]	3.521
H6	H2B[8]	3.504	H8	C11[1]	3.503
H8	C12[1]	3.228	H8	C17[2]	3.409
H8	H11[1]	3.419	H8	H12[1]	2.936
H8	H13[6]	3.550	H8	H17A[2]	2.640
H8	H17B[2]	3.423	H8	H17B[6]	3.454
H8	H18A[2]	3.486	H8	H20B[2]	3.527
H8	H5C[1]	3.595	H9	C9[6]	3.524
H9	C11[1]	3.331	H9	C18[1]	3.230
H9	C18[2]	3.592	H9	H9[6]	2.810
H9	H11[1]	2.898	H9	H13[6]	2.828
H9	H17A[2]	3.502	H9	H18A[1]	2.870
H9	H18A[2]	2.816	H9	H18B[1]	2.672
H11	F2[10]	2.458	H11	C2[10]	3.519
H11	C8[8]	3.487	H11	C9[8]	3.201

H11	H6[4]	3.498	H11	H8[8]
	3.419			
H11	H9[8]	2.898	H11	H1A[10]
	3.396			
H11	H1C[10]	3.401	H11	H2A[4]
	3.200			
H11	H2B[10]	2.718	H11	H2C[4]
	3.219			
H12	F1[8]	2.710	H12	C1[8]
	3.372			
H12	C8[8]	3.351	H12	C12[4]
	3.421			
H12	H6[4]	2.822	H12	H8[8]
	2.936			
H12	H12[4]	2.704	H12	H1A[8]
	3.024			
H12	H1A[10]	2.891	H12	H1B[8]
	2.793			
H12	H1C[8]	3.039	H12	H1C[10]
	2.905			
H13	C8[6]	3.457	H13	C9[6]
	3.028			
H13	C18[1]	3.531	H13	H8[6]
	3.550			
H13	H9[6]	2.828	H13	H17A[1]
	3.234			
H13	H18A[1]	3.457	H13	H18B[1]
	2.904			
H14A	F2[4]	2.807	H14A	C2[4]
	3.123			
H14A	H3A[4]	3.286	H14A	H16[1]
	3.237			

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H14A	H18B[1]	2.823	H14A	H21B[12]	3.541
H14A	H2A[4]	2.559	H14A	H2B[4]	2.907
H14A	H2C[4]	2.517	H14B	F2[10]	3.209
H14B	C2[4]	3.460	H14B	C21[11]	3.355
H14B	H3A[4]	3.366	H14B	H21B[11]	2.649
H14B	H21C[11]	3.211	H14B	H2A[4]	2.615
H14B	H2B[10]	3.578	H14B	H2C[4]	2.607
H14B	H4B[4]	3.577	H14B	H4C[4]	3.519
H15A	F1A[6]	3.505	H15A	C16[1]	3.517
H15A	C17[1]	3.530	H15A	C20[1]	3.167
H15A	H16[1]	2.883	H15A	H17A[1]	2.862
H15A	H18B[1]	3.541	H15A	H19A[12]	3.326
H15A	H20A[1]	2.455	H15A	H20B[1]	3.045
H15B	C19[12]	3.448	H15B	C20[11]	3.340
H15B	C21[11]	3.401	H15B	H15B[12]	3.075
H15B	H16[1]	3.492	H15B	H19A[12]	2.622
H15B	H19B[12]	3.430	H15B	H20A[1]	3.088
H15B	H20A[11]	2.553	H15B	H21B[11]	3.083
H15B	H21C[11]	3.269	H16	C15[8]	3.536
H16	C19[11]	3.534	H16	C20[11]	3.561
H16	C21[11]	3.552	H16	H14A[8]	3.237

H16	H15A[8]	2.883	H16	H15B[8]
	3.492			
H16	H19A[11]	2.747	H16	H20A[11]
	3.193			
H16	H21B[11]	2.794	H17A	F1A[13]
	3.358			
H17A	C8[13]	3.402	H17A	H8[13]
	2.640			
H17A	H9[13]	3.502	H17A	H13[8]
	3.234			
H17A	H15A[8]	2.862	H17A	H5A[6]
	3.223			
H17A	H5C[6]	3.211	H17B	F1A[6]
	2.979			
H17B	C5[6]	3.164	H17B	C7[6]
	3.251			
H17B	C8[6]	3.204	H17B	C9[6]
	3.561			
H17B	H8[6]	3.454	H17B	H8[13]
	3.423			
H17B	H5A[6]	2.577	H17B	H5B[6]
	2.944			
H17B	H5C[6]	2.567	H18A	C9[6]
	3.513			
H18A	C10[6]	3.216	H18A	C11[6]
	3.477			
H18A	C13[6]	3.582	H18A	C18[6]
	3.170			
H18A	H8[13]	3.486	H18A	H9[8]
	2.870			
H18A	H9[13]	2.816	H18A	H13[8]
	3.457			

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H18A	H18A^{6}	2.302	H18A	H18B^{6}	3.456
H18B	F2^{10}	3.529	H18B	C9^{8}	3.354
H18B	C13^{8}	3.514	H18B	C14^{8}	3.506
H18B	H9^{8}	2.672	H18B	H13^{8}	2.904
H18B	H14A^{8}	2.823	H18B	H15A^{8}	3.541
H18B	H18A^{6}	3.456	H19A	C15^{12}	3.395
H19A	C16^{11}	3.542	H19A	C19^{11}	3.561
H19A	C20^{11}	3.594	H19A	H15A^{12}	3.326
H19A	H15B^{12}	2.622	H19A	H16^{11}	2.747
H19A	H19A^{11}	3.173	H19A	H20A^{1}	3.381
H19A	H20A^{11}	2.833	H19B	F1A^{6}	2.739
H19B	F2A^{6}	3.296	H19B	C5^{6}	3.139
H19B	C20^{1}	3.583	H19B	H15B^{12}	3.430
H19B	H20A^{1}	2.990	H19B	H20B^{1}	3.395
H19B	H21C^{1}	3.402	H19B	H4A^{6}	3.192
H19B	H5A^{6}	2.724	H19B	H5B^{6}	2.757
H19B	H5C^{6}	2.716	H20A	F1A^{13}	3.357
H20A	C15^{8}	3.194	H20A	C15^{11}	3.394
H20A	C16^{11}	3.574	H20A	C19^{8}	3.574
H20A	H15A^{8}	2.455	H20A	H15B^{8}	3.088
H20A	H15B^{11}	2.553	H20A	H16^{11}	3.193

H20A	H19A[8]	3.381	H20A	H19A[11]
	2.833			
H20A	H19B[8]	2.990	H20B	F1[13]
	3.500			
H20B	F1A[13]	2.353	H20B	H3B[13]
	3.412			
H20B	H8[13]	3.527	H20B	H15A[8]
	3.045			
H20B	H19B[8]	3.395	H20B	H5A[6]
	2.865			
H20B	H5B[13]	2.655	H20B	H5C[6]
	2.855			
H21A	F2A[6]	2.465	H21A	F2A[15]
	3.405			
H21A	C3[14]	3.306	H21A	C4[6]
	3.383			
H21A	H3A[14]	2.710	H21A	H3B[13]
	3.114			
H21A	H3B[14]	3.017	H21A	H4A[6]
	2.595			
H21A	H4B[6]	3.504	H21A	H4B[15]
	3.309			
H21A	H4C[15]	3.417	H21A	H5A[6]
	3.334			
H21A	H5C[6]	3.330	H21B	F2[14]
	2.997			
H21B	C14[11]	3.446	H21B	C15[11]
	3.490			
H21B	C16[11]	3.569	H21B	H3A[14]
	3.055			
H21B	H3B[14]	3.581	H21B	H14A[12]
	3.541			

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H21B	H14B[11]	2.649	H21B	H15B[11]	3.083
H21B	H16[11]	2.794	H21B	H2B[14]	3.225
H21B	H4B[15]	3.301	H21B	H4C[15]	3.351
H21C	F1A[13]	3.305	H21C	F2A[13]	3.289
H21C	F2A[15]	3.007	H21C	C3[15]	3.319
H21C	C4[15]	3.304	H21C	H3A[15]	2.561
H21C	H3B[13]	3.176	H21C	H14B[11]	3.211
H21C	H15B[11]	3.269	H21C	H19B[8]	3.402
H21C	H4A[13]	3.181	H21C	H4A[15]	3.199
H21C	H4B[15]	2.894	H21C	H4C[15]	2.970
H21C	H5B[13]	3.474	H1A	C1[4]	3.315
H1A	C7[4]	3.121	H1A	C8[4]	3.374
H1A	C12[4]	3.355	H1A	H6[1]	3.510
H1A	H11[3]	3.396	H1A	H12[1]	3.024
H1A	H12[3]	2.891	H1A	H1A[4]	2.487
H1B	C5[1]	3.538	H1B	C6[1]	3.545
H1B	C12[1]	3.492	H1B	H6[1]	2.860
H1B	H12[1]	2.793	H1B	H1C[4]	3.579
H1B	H4C[1]	3.284	H1B	H5C[1]	2.926
H1C	C1[4]	3.324	H1C	C7[4]	3.121
H1C	C8[4]	3.376	H1C	C12[4]	3.348

H1C	H6[1]	3.521	H1C	H11[3]
	3.401			
H1C	H12[1]	3.039	H1C	H12[3]
	2.905			
H1C	H1B[4]	3.579	H1C	H1C[4]
	2.508			
H2A	C10[4]	3.130	H2A	C11[4]
	3.013			
H2A	C12[4]	3.436	H2A	C14[4]
	2.992			
H2A	H11[4]	3.200	H2A	H14A[4]
	2.559			
H2A	H14B[4]	2.615	H2B	C11[3]
	3.539			
H2B	H6[1]	3.504	H2B	H11[3]
	2.718			
H2B	H14A[4]	2.907	H2B	H14B[3]
	3.578			
H2B	H21B[5]	3.225	H2B	H4C[1]
	2.944			
H2C	C9[4]	3.599	H2C	C10[4]
	3.111			
H2C	C11[4]	3.020	H2C	C12[4]
	3.451			
H2C	C14[4]	2.966	H2C	H11[4]
	3.219			
H2C	H14A[4]	2.517	H2C	H14B[4]
	2.607			
H4A	C3[7]	3.444	H4A	C21[6]
	3.538			
H4A	H3A[7]	2.921	H4A	H3B[8]
	3.518			

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H4A	H3B^{7}	3.253	H4A	H19B^{6}	3.192
H4A	H21A^{6}	2.595	H4A	H21C^{2}	3.181
H4A	H21C^{9}	3.199	H4A	H4A^{7}	3.230
H4B	F1^{8}	3.072	H4B	F2^{8}	2.871
H4B	C2^{8}	3.557	H4B	C3^{8}	3.547
H4B	C21^{9}	3.349	H4B	H3B^{8}	2.841
H4B	H14B^{4}	3.577	H4B	H21A^{6}	3.504
H4B	H21A^{9}	3.309	H4B	H21B^{9}	3.301
H4B	H21C^{9}	2.894	H4C	C2^{8}	3.564
H4C	C21^{9}	3.430	H4C	H3B^{8}	2.926
H4C	H14B^{4}	3.519	H4C	H21A^{9}	3.417
H4C	H21B^{9}	3.351	H4C	H21C^{9}	2.970
H4C	H1B^{8}	3.284	H4C	H2B^{8}	2.944
H5A	F1^{8}	2.557	H5A	C17^{6}	3.294
H5A	C19^{6}	3.392	H5A	C20^{6}	3.455
H5A	H3B^{8}	3.540	H5A	H17A^{6}	3.223
H5A	H17B^{6}	2.577	H5A	H19B^{6}	2.724
H5A	H20B^{6}	2.865	H5A	H21A^{6}	3.334
H5B	C20^{2}	3.476	H5B	H17B^{6}	2.944
H5B	H19B^{6}	2.757	H5B	H20B^{2}	2.655
H5B	H21C^{2}	3.474	H5C	C17^{6}	3.283

H5C	C19[6]	3.383	H5C	C20[6]
	3.445			
H5C	H3B[8]	3.540	H5C	H8[8]
	3.595			
H5C	H17A[6]	3.211	H5C	H17B[6]
	2.567			
H5C	H19B[6]	2.716	H5C	H20B[6]
	2.855			
H5C	H21A[6]	3.330	H5C	H1B[8]
	2.926			

Symmetry Operators:

(1) X,Y-1,Z
(2) -X+1,Y-1,-Z+1/2
(3) -X+2,Y-1,-Z+1/2
(4) -X+2,Y,-Z+1/2
(5) X+1,-Y,Z+1
(6) -X+1,Y,-Z+1/2
(7) -X+2,-Y,-Z+1
(8) X,Y+1,Z
(9) X+1,-Y+1,Z+1
(10) -X+2,Y+1,-Z+1/2
(11) -X+1,-Y+1,-Z
(12) -X+1,-Y,-Z
(13) -X+1,Y+1,-Z+1/2
(14) X-1,-Y,Z
(15) X-1,-Y+1,Z

X-Ray Data Collection for **6**

Data Collection

A colorless prism crystal of $C_{21}H_{31}F$ having approximate dimensions of 0.130 x 0.130 x 0.100 mm was mounted in a loop. All measurements were made on a Rigaku XtaLAB P200 diffractometer using multi-layer mirror monochromated Mo-Kα radiation.

The crystal-to-detector distance was 45.05 mm.

Cell constants and an orientation matrix for data collection corresponded to a primitive monoclinic cell with dimensions:

a = 5.3032(6) Å
b = 25.832(3) Å β = 97.667(3)°
c = 13.1164(17) Å
V = 1780.8(4) $Å^3$

For Z = 4 and F.W. = 302.47, the calculated density is 1.128 g/cm^3. The reflection conditions of:

h0l: l = 2n
0k0: k = 2n

uniquely determine the space group to be:

$P2_1/c$ (#14)

The data were collected at a temperature of -100 ± 1°C to a maximum 2θ value of 50.7°. A total of 1080 oscillation images were collected. A sweep of data was done using ω scans from -100.0 to 80.0° in 0.50° step, at χ=45.0° and ϕ = 0.0°. The exposure rate was 10.0 [sec./°]. The detector swing angle was -10.42°. A second sweep was performed using ω scans from -100.0 to 80.0° in 0.50° step, at χ=45.0° and ϕ = 90.0°. The exposure rate was 10.0 [sec./°]. The detector swing angle was -10.42°. Another sweep was performed using ω scans from -100.0 to 80.0° in 0.50° step, at χ=45.0° and ϕ = 180.0°. The exposure rate was 10.0 [sec./°]. The detector swing angle was -10.42°. The crystal-to-detector distance was 45.05 mm. Readout was performed in the 0.172 mm pixel mode.

Data Reduction

Of the 21702 reflections were collected, where 3277 were unique (R_{int} = 0.0437); equivalent reflections were merged. Data were collected and processed using CrystalClear (Rigaku). [1]

The linear absorption coefficient, μ, for Mo-Kα radiation is 0.701 cm^{-1}. An empirical absorption correction was applied which resulted in transmission factors ranging from 0.878 to 0.993. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by direct methods[2] and expanded using Fourier techniques. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined using the riding model. The final cycle of full-matrix least-squares refinement[3] on F^2 was based on 3277 observed reflections and 210 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R1 = \Sigma \, ||Fo| - |Fc|| \, / \, \Sigma \, |Fo| = 0.0532$$

$$wR2 = [\Sigma (w (Fo^2 - Fc^2)^2) / \Sigma \, w(Fo^2)^2]^{1/2} = 0.1545$$

The goodness of fit[4] was 1.07. Unit weights were used. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.43 and -0.24 $e^-/Å^3$, respectively.

Neutral atom scattering factors were taken from International Tables for Crystallography (IT), Vol. C, Table 6.1.1.4 [5]. Anomalous dispersion effects were included in Fcalc[6]; the values for Δf' and Δf'' were those of Creagh and McAuley[7]. The values for the mass attenuation coefficients are those of Creagh and Hubbell[8]. All calculations were performed using the CrystalStructure[9] crystallographic software package except for refinement, which was performed using SHELXL2013[10].

References

(1) CrystalClear: Data Collection and Processing Software, Rigaku Corporation (1998-2014). Tokyo 196-8666, Japan.

(2) SIR2011: Burla, M. C., Caliandro, R., Camalli, M., Carrozzini, B., Cascarano, G. L., Giacovazzo, C., Mallamo, M., Mazzone, A., Polidori, G. and Spagna, R. (2012). J. Appl. Cryst. 45, 357-361.

(3) Least Squares function minimized: (SHELXL2013)

$\Sigma w(F_o^2\text{-}F_c^2)^2$ where w = Least Squares weights.

(4) Goodness of fit is defined as:

$[\Sigma w(F_o^2\text{-}F_c^2)^2/(N_o\text{-}N_v)]^{1/2}$

where: N_o = number of observations
N_v = number of variables

(5) International Tables for Crystallography, Vol.C (1992). Ed. A.J.C. Wilson, Kluwer Academic Publishers, Dordrecht, Netherlands, Table 6.1.1.4, pp. 572.

(6) Ibers, J. A. & Hamilton, W. C.; Acta Crystallogr., 17, 781 (1964).

(7) Creagh, D. C. & McAuley, W.J .; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.6.8, pages 219-222 (1992).

(8) Creagh, D. C. & Hubbell, J.H..; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.4.3, pages 200-206 (1992).

(9) CrystalStructure 4.1: Crystal Structure Analysis Package, Rigaku Corporation (2000-2014). Tokyo 196-8666, Japan.

(10) SHELXL2013: Sheldrick, G. M. (2008). Acta Cryst. A64, 112-122.

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$C_{21}H_{31}F$
Formula Weight	302.47
Crystal Color, Habit	colorless, prism
Crystal Dimensions	0.130 X 0.130 X 0.100 mm
Crystal System	monoclinic
Lattice Type	Primitive
Lattice Parameters	a = 5.3032(6) Å b = 25.832(3) Å c = 13.1164(17) Å β = 97.667(3) ° V = 1780.8(4) $Å^3$
Space Group	$P2_1/c$ (#14)
Z value	4
D_{calc}	1.128 g/cm^3
F_{000}	664.00
μ(MoKα)	0.701 cm^{-1}

B. Intensity Measurements

Diffractometer	XtaLAB P200
Radiation monochromated	MoKα (λ = 0.71075 Å) multi-layer mirror
Voltage, Current	45kV, 66mA
Temperature	-100.0°C
Detector Aperture	83.8 x 70.0 mm
Data Images	1080 exposures
ω oscillation Range (χ=45.0, ϕ=0.0)	-100.0 - 80.0°
Exposure Rate	10.0 sec./°
Detector Swing Angle	-10.42°
ω oscillation Range (χ=45.0, ϕ=90.0)	-100.0 - 80.0°
Exposure Rate	10.0 sec./°
Detector Swing Angle	-10.42°
ω oscillation Range (χ=45.0, ϕ=180.0)	-100.0 - 80.0°
Exposure Rate	10.0 sec./°
Detector Swing Angle	-10.42°
Detector Position	45.05 mm
Pixel Size	0.172 mm
$2\theta_{max}$	50.7°
No. of Reflections Measured	Total: 21702 Unique: 3277 (R_{int} = 0.0437)
Corrections	Lorentz-polarization

Absorption
(trans. factors: 0.878 - 0.993)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SIR2011)
Refinement	Full-matrix least-squares on F^2
Function Minimized	Σ w $(Fo^2 - Fc^2)^2$
Least Squares Weights	w = 1/ [$\sigma^2(Fo^2)$ + $(0.0790 \cdot P)^2$ + 0.5173 · P] where P = (Max(Fo^2,0) + $2Fc^2$)/3
$2\theta_{max}$ cutoff	50.7º
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (All reflections)	3277
No. Variables	210
Reflection/Parameter Ratio	15.60
Residuals: R1 (I>2.00σ(I))	0.0532
Residuals: R (All reflections)	0.0650
Residuals: wR2 (All reflections)	0.1545
Goodness of Fit Indicator	1.067
Max Shift/Error in Final Cycle	0.000
Maximum peak in Final Diff. Map	0.43 $e^-/Å^3$
Minimum peak in Final Diff. Map	-0.24 $e^-/Å^3$

Table 1. Atomic coordinates and B_{iso}/B_{eq} and occupancy

atom	x	y	z	B_{eq}	occ
F1	1.0514(4)	0.24672(7)	0.60659(15)	6.20(6)	
	0.735(4)				
F1A	1.1384(9)	0.1828(2)	0.7331(4)	6.83(18)	
	0.265(4)				
C1	0.7980(5)	0.25144(8)	0.62893(16)	4.78(5)	1
C2	0.7134(3)	0.19845(7)	0.65605(13)	3.32(3)	1
C3	0.8729(5)	0.17961(8)	0.75577(14)	4.91(5)	1
C4	0.8518(6)	0.21836(9)	0.84230(16)	5.68(5)	1
C5	0.9348(4)	0.27142(8)	0.81629(17)	4.63(4)	1
C6	0.7907(6)	0.28981(9)	0.71587(17)	5.74(5)	1
C7	0.6977(3)	0.16083(7)	0.56674(12)	2.93(3)	1
C8	0.8825(3)	0.12386(7)	0.55594(12)	3.17(3)	1
C9	0.8547(3)	0.08924(7)	0.47488(13)	3.19(3)	1
C10	0.6416(3)	0.09001(6)	0.40037(12)	2.79(3)	1
C11	0.4597(3)	0.12777(7)	0.40998(13)	3.18(3)	1
C12	0.4865(3)	0.16220(7)	0.49128(13)	3.36(3)	1
C13	0.6018(3)	0.05050(6)	0.31459(12)	2.82(3)	1
C14	0.8167(3)	0.04823(7)	0.24805(13)	3.24(3)	1
C15	0.7579(3)	0.01017(7)	0.15924(13)	3.24(3)	1
C16	0.7056(3)	-0.04443(6)	0.19577(12)	2.83(3)	1
C17	0.5035(4)	-0.04284(7)	0.26802(13)	3.53(3)	1
C18	0.5587(3)	-0.00385(7)	0.35531(13)	3.40(3)	1
C19	0.6254(3)	-0.08164(7)	0.10653(13)	3.37(3)	1
C20	0.8103(4)	-0.08884(8)	0.02918(15)	4.10(4)	1
C21	0.7165(5)	-0.12858(9)	-0.05293(17)	5.45(5)	1

$B_{eq} = 8/3\ \pi^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$

Table 2. Atomic coordinates and B_{iso} and occupancy involving hydrogen atoms

atom	x	y	z	B_{iso}	occ
H2	0.53563	0.20247	0.67246	3.983	1
H4A	0.67306	0.21974	0.85632	6.812	1
H4B	0.95802	0.20633	0.90569	6.812	1
H5A	1.11933	0.27112	0.81111	5.551	1
H5B	0.90542	0.29569	0.87192	5.551	1
H6A	0.86413	0.32308	0.69661	6.889	1
H6B	0.61126	0.29619	0.72541	6.889	1
H8	1.03085	0.12235	0.60522	3.801	1
H9	0.98422	0.06431	0.46989	3.827	1
H11	0.31379	0.12995	0.35958	3.818	1
H12	0.35795	0.18739	0.49583	4.031	1
H13	0.44355	0.06054	0.26863	3.381	1
H14A	0.84341	0.08313	0.22021	3.891	1
H14B	0.97608	0.03767	0.29109	3.891	1
H15A	0.90371	0.00909	0.11938	3.889	1
H15B	0.60749	0.02259	0.11282	3.889	1
H16	0.86630	-0.05796	0.23535	3.401	1
H17A	0.48851	-0.07773	0.29788	4.242	1
H17B	0.33756	-0.03424	0.22761	4.242	1
H18A	0.71186	-0.01492	0.40171	4.075	1
H18B	0.41381	-0.00307	0.39571	4.075	1
H19A	0.59321	-0.11596	0.13581	4.039	1
H19B	0.46200	-0.06923	0.06919	4.039	1
H20A	0.97670	-0.10015	0.06560	4.920	1
H20B	0.83572	-0.05524	-0.00429	4.920	1
H21A	0.55164	-0.11756	-0.08916	6.539	1
H21B	0.69778	-0.16227	-0.02041	6.539	1
H21C	0.83972	-0.13148	-0.10206	6.539	1
H1A	0.68655	0.26412	0.56640	5.738	
	0.735(4)				
H1B	0.68649	0.26398	0.56733	5.738	
	0.265(4)				
H1C	0.97369	0.24942	0.61149	5.738	
	0.265(4)				
H3A	0.81106	0.14528	0.77506	5.893	
	0.735(4)				
H3B	1.05311	0.17602	0.74475	5.893	
	0.735(4)				
H3C	0.82584	0.14372	0.77471	5.893	
	0.265(4)				

Table 3. Anisotropic displacement parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
F1	0.0951(14)	0.0563(12)	0.0952(15)	-0.0299(9)	0.0534(11)	-0.0219(9)
F1A	0.054(3)	0.099(4)	0.100(4)	0.012(3)	-0.015(3)	-0.040(3)
C1	0.0962(17)	0.0413(12)	0.0461(11)	0.0075(10)	0.0166(11)	0.0019(8)
C2	0.0391(9)	0.0507(11)	0.0362(9)	-0.0027(8)	0.0047(7)	-0.0047(8)
C3	0.0939(17)	0.0517(13)	0.0370(10)	-0.0068(11)	-0.0059(10)	-0.0028(9)
C4	0.115(2)	0.0578(14)	0.0385(11)	-0.0057(13)	-0.0063(11)	-0.0021(10)
C5	0.0593(12)	0.0527(12)	0.0620(13)	-0.0046(10)	0.0014(10)	-0.0225(10)
C6	0.122(2)	0.0403(12)	0.0594(13)	0.0037(12)	0.0240(13)	-0.0029(10)
C7	0.0367(8)	0.0420(10)	0.0328(8)	-0.0063(7)	0.0048(7)	0.0011(7)
C8	0.0361(8)	0.0480(10)	0.0342(8)	-0.0018(7)	-0.0029(7)	0.0015(7)
C9	0.0377(9)	0.0422(10)	0.0399(9)	0.0048(7)	0.0003(7)	0.0021(7)
C10	0.0360(8)	0.0367(9)	0.0330(8)	-0.0033(7)	0.0037(6)	0.0035(7)
C11	0.0340(8)	0.0465(10)	0.0380(9)	0.0014(7)	-0.0041(7)	-0.0016(8)
C12	0.0365(8)	0.0466(10)	0.0435(10)	0.0043(7)	0.0012(7)	-0.0050(8)
C13	0.0335(8)	0.0386(9)	0.0339(8)	-0.0004(7)	0.0005(6)	0.0021(7)
C14	0.0457(9)	0.0350(9)	0.0440(9)	-0.0078(7)	0.0115(8)	0.0027(7)
C15	0.0482(10)	0.0385(10)	0.0384(9)	-0.0022(7)	0.0131(7)	0.0044(7)
C16	0.0355(8)	0.0358(9)	0.0361(8)	-0.0011(7)	0.0039(7)	0.0037(7)
C17	0.0497(10)	0.0431(10)	0.0434(10)	-0.0143(8)	0.0133(8)	-0.0010(8)
C18	0.0495(10)	0.0426(10)	0.0385(9)	-0.0094(8)	0.0120(8)	0.0006(8)
C19	0.0449(9)	0.0398(10)	0.0434(10)	-0.0029(8)	0.0065(7)	0.0007(8)
C20	0.0587(11)	0.0512(12)	0.0481(11)	0.0032(9)	0.0149(9)	-0.0028(9)

C21 0.0911(17) 0.0624(14) 0.0547(12) 0.0108(12) 0.0143(12) -0.0112(11)

The general temperature factor expression: $\exp(-2\pi^2(a^{*2}U_{11}h^2 + b^{*2}U_{22}k^2 + c^{*2}U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$

Table 4. Bond lengths (Å)

atom	atom	distance	atom	atom	distance
F1	C1	1.418(3)	F1A	C3	1.480(6)
C1	C2	1.498(3)	C1	C6	1.515(3)
C2	C3	1.539(3)	C2	C7	1.516(2)
C3	C4	1.529(3)	C4	C5	1.493(3)
C5	C6	1.509(3)	C7	C8	1.389(2)
C7	C12	1.393(2)	C8	C9	1.382(2)
C9	C10	1.392(2)	C10	C11	1.389(2)
C10	C13	1.513(2)	C11	C12	1.381(2)
C13	C14	1.526(2)	C13	C18	1.530(2)
C14	C15	1.524(2)	C15	C16	1.527(2)
C16	C17	1.523(3)	C16	C19	1.530(2)
C17	C18	1.524(2)	C19	C20	1.514(3)
C20	C21	1.523(3)			

Table 5. Bond lengths involving hydrogens (Å)

atom	atom	distance	atom	atom	distance
C1	H1A	1.000	C1	H1B	0.990
C1	H1C	0.990	C2	H2	1.000
C3	H3A	0.990	C3	H3B	0.990
C3	H3C	1.000	C4	H4A	0.990
C4	H4B	0.990	C5	H5A	0.990
C5	H5B	0.990	C6	H6A	0.990
C6	H6B	0.990	C8	H8	0.950
C9	H9	0.950	C11	H11	0.950
C12	H12	0.950	C13	H13	1.000
C14	H14A	0.990	C14	H14B	0.990
C15	H15A	0.990	C15	H15B	0.990
C16	H16	1.000	C17	H17A	0.990
C17	H17B	0.990	C18	H18A	0.990
C18	H18B	0.990	C19	H19A	0.990
C19	H19B	0.990	C20	H20A	0.990
C20	H20B	0.990	C21	H21A	0.980
C21	H21B	0.980	C21	H21C	0.980

Table 6. Bond angles (°)

atom	atom	atom	angle	atom	atom	atom	angle
F1	C1	C2	107.01(17)	F1	C1	C6	109.4(2)
C2	C1	C6	112.36(18)	C1	C2	C3	110.05(15)
C1	C2	C7	112.93(15)	C3	C2	C7	114.92(15)
F1A	C3	C2	104.0(3)	F1A	C3	C4	106.1(3)
C2	C3	C4	109.74(18)	C3	C4	C5	112.25(19)
C4	C5	C6	110.99(18)	C1	C6	C5	113.0(2)
C2	C7	C8	123.57(14)	C2	C7	C12	119.26(15)
C8	C7	C12	117.16(15)	C7	C8	C9	121.28(14)
C8	C9	C10	121.60(16)	C9	C10	C11	117.06(15)
C9	C10	C13	122.15(14)	C11	C10	C13	120.76(13)
C10	C11	C12	121.39(15)	C7	C12	C11	121.50(16)
C10	C13	C14	114.23(13)	C10	C13	C18	111.84(13)
C14	C13	C18	108.79(14)	C13	C14	C15	111.68(14)
C14	C15	C16	112.53(14)	C15	C16	C17	110.04(14)
C15	C16	C19	112.57(13)	C17	C16	C19	110.24(14)
C16	C17	C18	113.88(15)	C13	C18	C17	111.53(14)
C16	C19	C20	116.81(15)	C19	C20	C21	112.17(18)

Table 7. Bond angles involving hydrogens (°)

atom	atom	atom	angle	atom	atom	atom	angle
F1	C1	H1A	109.3	C2	C1	H1A	109.3
C2	C1	H1B	109.1	C2	C1	H1C	109.1
C6	C1	H1A	109.3	C6	C1	H1B	109.1
C6	C1	H1C	109.1	H1B	C1	H1C	107.9
C1	C2	H2	106.1	C3	C2	H2	106.1
C7	C2	H2	106.1	F1A	C3	H3C	112.2
C2	C3	H3A	109.7	C2	C3	H3B	109.7
C2	C3	H3C	112.2	C4	C3	H3A	109.7
C4	C3	H3B	109.7	C4	C3	H3C	112.2
H3A	C3	H3B	108.2	C3	C4	H4A	109.2
C3	C4	H4B	109.2	C5	C4	H4A	109.2
C5	C4	H4B	109.1	H4A	C4	H4B	107.9
C4	C5	H5A	109.4	C4	C5	H5B	109.4
C6	C5	H5A	109.4	C6	C5	H5B	109.4
H5A	C5	H5B	108.0	C1	C6	H6A	109.0
C1	C6	H6B	109.0	C5	C6	H6A	109.0
C5	C6	H6B	109.0	H6A	C6	H6B	107.8
C7	C8	H8	119.4	C9	C8	H8	119.4
C8	C9	H9	119.2	C10	C9	H9	119.2
C10	C11	H11	119.3	C12	C11	H11	119.3
C7	C12	H12	119.3	C11	C12	H12	119.3
C10	C13	H13	107.2	C14	C13	H13	107.2

C18	C13	H13	107.2	C13	C14	H14A
	109.3					
C13	C14	H14B	109.3	C15	C14	H14A
	109.3					
C15	C14	H14B	109.3	H14A	C14	H14B
	107.9					
C14	C15	H15A	109.1	C14	C15	H15B
	109.1					
C16	C15	H15A	109.1	C16	C15	H15B
	109.1					
H15A	C15	H15B	107.8	C15	C16	H16
	107.9					
C17	C16	H16	107.9	C19	C16	H16
	108.0					
C16	C17	H17A	108.8	C16	C17	H17B
	108.8					
C18	C17	H17A	108.8	C18	C17	H17B
	108.8					
H17A	C17	H17B	107.7	C13	C18	H18A
	109.3					
C13	C18	H18B	109.3	C17	C18	H18A
	109.3					
C17	C18	H18B	109.3	H18A	C18	H18B
	108.0					
C16	C19	H19A	108.1	C16	C19	H19B
	108.1					
C20	C19	H19A	108.1	C20	C19	H19B
	108.1					
H19A	C19	H19B	107.3	C19	C20	H20A
	109.2					

Table 7. Bond angles involving hydrogens (°) (continued)

atom	atom	atom	angle	atom	atom	atom	angle
C19	C20	H20B	109.2	C21	C20	H20A	109.2
C21	C20	H20B	109.2	H20A	C20	H20B	107.9
C20	C21	H21A	109.5	C20	C21	H21B	109.5
C20	C21	H21C	109.5	H21A	C21	H21B	109.5
H21A	C21	H21C	109.5	H21B	C21	H21C	109.5

Table 8. Torsion Angles(º)
(Those having bond angles > 160 or < 20 degrees are excluded.)

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
F1	C1	C2	C3	64.96(18)	F1	C1	C2	C7	-64.94(18)
F1	C1	C6	C5	-65.5(2)	C2	C1	C6	C5	53.1(3)
C6	C1	C2	C3	-55.1(2)	C6	C1	C2	C7	174.99(17)
C1	C2	C3	F1A	-56.36(19)	C1	C2	C3	C4	56.8(2)
C1	C2	C7	C8	101.34(19)	C1	C2	C7	C12	-79.6(2)
C3	C2	C7	C8	-26.0(2)	C3	C2	C7	C12	152.98(15)
C7	C2	C3	F1A	72.45(18)	C7	C2	C3	C4	-174.37(14)
F1A	C3	C4	C5	54.2(3)	C2	C3	C4	C5	-57.6(3)
C3	C4	C5	C6	54.7(3)	C4	C5	C6	C1	-51.9(3)
C2	C7	C8	C9	177.68(14)	C2	C7	C12	C11	-178.10(14)
C8	C7	C12	C11	1.0(2)	C12	C7	C8	C9	-1.4(2)
C7	C8	C9	C10	0.3(3)	C8	C9	C10	C11	1.1(2)
C8	C9	C10	C13	-176.54(14)	C9	C10	C11	C12	-1.5(2)
C9	C10	C13	C14	-57.4(2)	C9	C10	C13	C18	66.70(19)
C11	C10	C13	C14	125.00(15)	C11	C10	C13	C18	-110.88(16)
C13	C10	C11	C12	176.19(13)	C10	C11	C12	C7	0.5(3)
C10	C13	C14	C15	-176.48(11)	C10	C13	C18	C17	176.70(12)
C14	C13	C18	C17	-56.19(16)	C18	C13	C14	C15	57.79(15)
C13	C14	C15	C16	-57.10(17)	C14	C15	C16	C17	51.94(17)
C14	C15	C16	C19	175.33(12)	C15	C16	C17	C18	-51.20(16)
C15	C16	C19	C20	58.47(18)	C17	C16	C19	C20	-178.24(12)

C19	C16	C17	C18	-175.94(12)	C16	C17	C18	C13
	54.72(18)							
C16	C19	C20	C21	177.19(12)				

Table 9. Intramolecular contacts less than 3.60 Å

atom	atom	distance	atom	atom	distance
F1	C3	2.868(3)	F1	C4	3.477(3)
F1	C5	2.968(3)	F1	C7	2.907(3)
F1	C8	3.340(3)	F1A	C1	2.759(6)
F1A	C5	2.812(6)	F1A	C6	3.313(6)
F1A	C7	3.031(5)	F1A	C8	2.953(6)
C1	C4	2.904(3)	C1	C8	3.478(3)
C1	C12	3.241(3)	C2	C5	2.948(3)
C3	C6	2.917(3)	C3	C8	2.997(3)
C7	C10	2.833(2)	C8	C11	2.749(2)
C9	C12	2.743(2)	C9	C14	3.139(2)
C9	C18	3.170(2)	C11	C18	3.528(3)
C13	C16	2.996(2)	C14	C17	2.912(3)
C15	C18	2.930(3)	C15	C20	3.107(3)

Table 10. Intramolecular contacts less than 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
F1	H2	3.188	F1	H5A	2.732
F1	H6A	2.566	F1	H6B	3.237
F1	H8	3.215	F1	H3B	2.572
F1A	H2	3.230	F1A	H4A	3.267
F1A	H4B	2.643	F1A	H5A	2.507
F1A	H8	2.308	F1A	H1C	2.428
C1	H4A	3.245	C1	H5A	2.790
C1	H5B	3.362	C1	H8	3.584
C1	H12	3.185	C1	H3A	3.341
C1	H3B	2.718	C1	H3C	3.368
C2	H4A	2.720	C2	H4B	3.363
C2	H5A	3.336	C2	H6A	3.343
C2	H6B	2.762	C2	H8	2.728
C2	H12	2.645	C3	H5A	2.751
C3	H5B	3.357	C3	H6B	3.317
C3	H8	2.689	C3	H1A	3.355
C3	H1B	3.344	C3	H1C	2.718
C4	H2	2.635	C4	H6A	3.318
C4	H6B	2.738	C4	H1C	3.279
C5	H2	3.185	C5	H1A	3.371
C5	H1B	3.360	C5	H1C	2.780
C5	H3A	3.353	C5	H3B	2.739

C5	H3C	3.381	C6	H2
	2.653			
C6	H4A	2.714	C6	H4B
	3.324			
C6	H3B	3.253	C7	H9
	3.264			
C7	H11	3.269	C7	H1A
	2.669			
C7	H1B	2.665	C7	H1C
	2.737			
C7	H3A	2.748	C7	H3B
	2.825			
C7	H3C	2.757	C8	H2
	3.255			
C8	H12	3.235	C8	H1C
	3.345			
C8	H3A	3.000	C8	H3B
	2.858			
C8	H3C	2.969	C9	H11
	3.235			
C9	H13	3.324	C9	H14A
	3.337			
C9	H14B	2.901	C9	H18A
	2.922			
C9	H18B	3.404	C10	H8
	3.271			
C10	H12	3.265	C10	H14A
	2.727			
C10	H14B	2.776	C10	H18A
	2.736			

Table 10. Intramolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
C10	H18B	2.688	C11	H9	3.234
C11	H13	2.533	C11	H18B	3.392
C12	H2	2.575	C12	H8	3.237
C12	H1A	2.958	C12	H1B	2.958
C13	H9	2.699	C13	H11	2.671
C13	H15A	3.370	C13	H15B	2.747
C13	H16	3.360	C13	H17A	3.368
C13	H17B	2.762	C14	H9	2.956
C14	H16	2.763	C14	H17B	3.299
C14	H18A	2.708	C14	H18B	3.343
C15	H13	2.677	C15	H17A	3.346
C15	H17B	2.761	C15	H18A	3.287
C15	H19A	3.377	C15	H19B	2.751
C15	H20A	3.370	C15	H20B	2.804
C16	H13	3.248	C16	H14A	3.381
C16	H14B	2.764	C16	H18A	2.803
C16	H18B	3.391	C16	H20A	2.776
C16	H20B	2.815	C17	H13	2.690
C17	H14B	3.240	C17	H15A	3.348
C17	H15B	2.758	C17	H19A	2.650
C17	H19B	2.676	C18	H9	3.090

C18	H14A	3.344	C18	H14B
	2.694			
C18	H15B	3.297	C18	H16
	2.789			
C19	H15A	2.763	C19	H15B
	2.696			
C19	H17A	2.706	C19	H17B
	2.645			
C19	H21A	2.708	C19	H21B
	2.726			
C19	H21C	3.356	C20	H15A
	2.809			
C20	H15B	3.311	C20	H16
	2.797			
C21	H19A	2.663	C21	H19B
	2.705			
H2	H4A	2.465	H2	H4B
	3.544			
H2	H6A	3.563	H2	H6B
	2.536			
H2	H8	3.546	H2	H12
	2.413			
H2	H1A	2.326	H2	H1B
	2.315			
H2	H1C	2.829	H2	H3A
	2.367			
H2	H3B	2.864	H2	H3C
	2.433			
H4A	H5A	2.844	H4A	H5B
	2.311			
H4A	H6B	2.609	H4A	H3A
	2.362			

Table 10. Intramolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H4A	H3B	2.875	H4A	H3C	2.427
H4B	H5A	2.314	H4B	H5B	2.360
H4B	H3A	2.382	H4B	H3B	2.368
H4B	H3C	2.395	H5A	H6A	2.312
H5A	H6B	2.852	H5A	H1C	2.688
H5A	H3B	2.614	H5B	H6A	2.388
H5B	H6B	2.308	H6A	H1A	2.386
H6A	H1B	2.381	H6A	H1C	2.319
H6B	H1A	2.328	H6B	H1B	2.318
H6B	H1C	2.855	H8	H9	2.311
H8	H1C	3.298	H8	H3A	2.714
H8	H3B	2.286	H8	H3C	2.660
H9	H14A	3.296	H9	H14B	2.439
H9	H18A	2.594	H9	H18B	3.513
H11	H12	2.311	H11	H13	2.309
H11	H18B	3.499	H12	H1A	2.719
H12	H1B	2.719	H13	H14A	2.366
H13	H14B	2.862	H13	H15A	3.582
H13	H15B	2.523	H13	H17A	3.597
H13	H17B	2.553	H13	H18A	2.865
H13	H18B	2.361	H14A	H15A	2.371

H14A	H15B	2.350	H14B	H15A
	2.352			
H14B	H15B	2.867	H14B	H16
	2.620			
H14B	H18A	2.541	H15A	H16
	2.331			
H15A	H19B	3.099	H15A	H20A
	2.947			
H15A	H20B	2.317	H15B	H16
	2.865			
H15B	H17B	2.654	H15B	H19A
	3.593			
H15B	H19B	2.536	H15B	H20B
	2.892			
H16	H17A	2.321	H16	H17B
	2.859			
H16	H18A	2.673	H16	H19A
	2.354			
H16	H19B	2.861	H16	H20A
	2.615			
H16	H20B	3.127	H17A	H18A
	2.337			
H17A	H18B	2.379	H17A	H19A
	2.473			
H17A	H19B	2.992	H17B	H18A
	2.863			
H17B	H18B	2.331	H17B	H19A
	2.860			
H17B	H19B	2.436	H19A	H20A
	2.377			
H19A	H20B	2.850	H19A	H21A
	2.930			

Table 10. Intramolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom
	distance			
H19A	H21B	2.498	H19A	H21C
	3.563			
H19B	H20A	2.850	H19B	H20B
	2.345			
H19B	H21A	2.522	H19B	H21B
	3.019			
H19B	H21C	3.584	H20A	H21A
	2.861			
H20A	H21B	2.364	H20A	H21C
	2.365			
H20B	H21A	2.378	H20B	H21B
	2.861			
H20B	H21C	2.352		

Table 11. Intermolecular contacts less than 3.60 Å

atom	atom	distance	atom	atom
	distance			
F1	C21[1]	3.552(3)	F1A	C2[2]
	3.362(6)			
F1A	C19[3]	3.482(6)	C2	F1A[4]
	3.362(6)			
C8	C12[2]	3.562(2)	C9	C11[2]
	3.569(2)			
C11	C9[4]	3.569(2)	C12	C8[4]
	3.562(2)			
C19	F1A[3]	3.482(6)	C21	F1[5]
	3.552(3)			

Symmetry Operators:

(1) -X+2,Y+1/2,-Z+1/2
(2) X+1,Y,Z
(3) -X+2,-Y,-Z+1
(4) X-1,Y,Z
(5) -X+2,Y+1/2-1,-Z+1/2

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
F1	H2[1]	2.838	F1	H4B[2]	2.883
F1	H5B[2]	3.262	F1	H6B[1]	3.412
F1	H12[1]	2.781	F1	H21B[3]	2.996
F1	H21C[3]	3.201	F1	H1A[1]	3.507
F1A	H2[1]	2.403	F1A	H4A[1]	3.215
F1A	H12[1]	3.468	F1A	H16[4]	3.253
F1A	H17A[4]	3.416	F1A	H19A[4]	2.704
F1A	H20A[4]	3.512	C1	H4B[2]	3.339
C1	H21B[5]	3.593	C2	H17A[6]	3.378
C3	H16[4]	3.429	C3	H17A[6]	3.276
C3	H19A[6]	3.431	C3	H19A[4]	3.416
C3	H20A[4]	3.137	C4	H19A[6]	3.582
C4	H20A[4]	3.364	C4	H21B[4]	3.431
C4	H1A[7]	3.209	C4	H1B[7]	3.222
C5	H11[8]	3.248	C5	H12[8]	3.210
C6	H14A[7]	3.294	C6	H21A[5]	3.313
C6	H21C[3]	3.314	C7	H5B[2]	3.123
C7	H17A[6]	3.034	C8	H5B[2]	3.200
C8	H12[1]	3.194	C8	H16[4]	3.346
C8	H17A[6]	3.158	C8	H18A[4]	3.541
C8	H18B[6]	3.588	C9	H5B[2]	3.291
C9	H11[1]	3.210	C9	H18A[4]	3.255

C9	H18B[6]	3.242	C10	H5B[2]
	3.310			
C10	H18B[6]	3.535	C11	H5A[9]
	3.337			
C11	H5B[9]	3.522	C11	H5B[2]
	3.172			
C11	H6B[2]	3.299	C11	H9[10]
	3.192			
C12	H5A[9]	3.334	C12	H5B[9]
	3.441			
C12	H5B[2]	3.082	C12	H8[10]
	3.177			
C12	H17A[6]	3.511	C13	H14B[10]
	3.308			
C13	H21A[11]	3.430	C14	H6A[2]
	3.408			
C14	H11[1]	3.538	C14	H13[1]
	3.314			
C14	H17B[1]	3.527	C14	H21A[11]
	3.206			
C14	H21C[12]	3.541	C15	H17B[1]
	3.292			
C15	H19B[11]	3.425	C15	H20B[12]
	3.361			
C15	H21A[11]	3.291	C16	H8[4]
	3.442			
C16	H17B[1]	3.332	C16	H3C[4]
	3.556			
C17	H8[4]	3.462	C17	H14B[10]
	3.531			

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
C17	H16[10]	3.372	C17	H3A[6]	3.139
C17	H3C[6]	3.145	C18	H9[4]	3.477
C18	H14B[10]	3.273	C18	H18B[6]	3.254
C19	H15B[11]	3.344	C19	H20A[10]	3.444
C19	H3A[6]	3.381	C19	H3A[4]	3.579
C19	H3B[4]	3.431	C19	H3C[6]	3.423
C19	H3C[4]	3.501	C20	H4B[4]	3.342
C20	H15A[12]	3.336	C20	H15B[11]	3.195
C20	H19B[1]	3.463	C20	H3A[4]	3.373
C20	H3C[4]	3.321	C21	H4B[4]	3.139
C21	H6A[13]	3.343	C21	H6B[14]	3.296
C21	H13[11]	3.343	C21	H15B[11]	3.272
C21	H1A[14]	3.490	C21	H1B[14]	3.493
H2	F1[10]	2.838	H2	F1A[10]	2.403
H2	H5A[10]	3.521	H2	H8[10]	3.405
H2	H17A[6]	3.250	H2	H19A[6]	3.499
H2	H1C[10]	3.218	H2	H3B[10]	2.926
H4A	F1A[10]	3.215	H4A	H5A[10]	3.205
H4A	H12[7]	3.565	H4A	H19A[6]	3.038
H4A	H21B[6]	3.440	H4A	H1A[7]	2.778
H4A	H1B[7]	2.791	H4A	H1C[7]	3.599

H4A	H3B[10]	3.598	H4B	F1[7]
	2.883			
H4B	C1[7]	3.339	H4B	C20[4]
	3.342			
H4B	C21[4]	3.139	H4B	H12[8]
	3.573			
H4B	H19A[4]	3.429	H4B	H20A[4]
	2.784			
H4B	H21B[4]	2.482	H4B	H21C[4]
	3.284			
H4B	H1A[7]	2.812	H4B	H1B[7]
	2.823			
H4B	H1C[7]	2.922	H5A	C11[8]
	3.337			
H5A	C12[8]	3.334	H5A	H2[1]
	3.521			
H5A	H4A[1]	3.205	H5A	H6B[1]
	3.045			
H5A	H11[8]	2.797	H5A	H12[8]
	2.793			
H5B	F1[7]	3.262	H5B	C7[7]
	3.123			
H5B	C8[7]	3.200	H5B	C9[7]
	3.291			
H5B	C10[7]	3.310	H5B	C11[7]
	3.172			
H5B	C11[8]	3.522	H5B	C12[7]
	3.082			
H5B	C12[8]	3.441	H5B	H11[8]
	2.915			
H5B	H12[7]	3.538	H5B	H12[8]
	2.748			

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H5B	H1A[7]	3.321	H5B	H1B[7]	3.330
H5B	H1C[7]	3.326	H6A	C14[7]	3.408
H6A	C21[3]	3.343	H6A	H11[8]	3.220
H6A	H14A[7]	2.447	H6A	H21A[5]	2.893
H6A	H21B[5]	3.539	H6A	H21B[3]	3.509
H6A	H21C[3]	2.429	H6B	F1[10]	3.412
H6B	C11[7]	3.299	H6B	C21[5]	3.296
H6B	H5A[10]	3.045	H6B	H11[7]	3.157
H6B	H14A[7]	3.356	H6B	H21A[5]	2.914
H6B	H21B[5]	3.145	H6B	H21C[5]	3.286
H8	C12[1]	3.177	H8	C16[4]	3.442
H8	C17[4]	3.462	H8	H2[1]	3.405
H8	H12[1]	2.925	H8	H16[4]	2.671
H8	H17A[6]	3.386	H8	H17A[4]	2.929
H8	H18A[4]	3.099	H9	C11[1]	3.192
H9	C18[4]	3.477	H9	H9[4]	3.414
H9	H11[1]	2.949	H9	H18A[4]	2.517
H9	H18B[1]	3.124	H9	H18B[6]	3.325
H11	C5[9]	3.248	H11	C9[10]	3.210
H11	C14[10]	3.538	H11	H5A[9]	2.797
H11	H5B[9]	2.915	H11	H6A[9]	3.220

H11	H6B[2]	3.157	H11	H9[10]
	2.949			
H11	H14A[10]	3.132	H11	H14B[10]
	3.044			
H11	H21C[11]	3.367	H12	F1[10]
	2.781			
H12	F1A[10]	3.468	H12	C5[9]
	3.210			
H12	C8[10]	3.194	H12	H4A[2]
	3.565			
H12	H4B[9]	3.573	H12	H5A[9]
	2.793			
H12	H5B[9]	2.748	H12	H5B[2]
	3.538			
H12	H8[10]	2.925	H12	H1C[10]
	3.138			
H13	C14[10]	3.314	H13	C21[11]
	3.343			
H13	H14A[10]	3.215	H13	H14B[10]
	2.603			
H13	H15A[10]	3.507	H13	H20B[11]
	3.587			
H13	H21A[11]	2.780	H13	H21C[11]
	3.084			
H14A	C6[2]	3.294	H14A	H6A[2]
	2.447			
H14A	H6B[2]	3.356	H14A	H11[1]
	3.132			
H14A	H13[1]	3.215	H14A	H20B[12]
	3.567			
H14A	H21A[11]	2.678	H14A	H21C[12]
	2.735			

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H14B	C13[1]	3.308	H14B	C17[1]	3.531
H14B	C18[1]	3.273	H14B	H11[1]	3.044
H14B	H13[1]	2.603	H14B	H17B[1]	2.871
H14B	H18B[1]	2.744	H15A	C20[12]	3.336
H15A	H13[1]	3.507	H15A	H15A[12]	3.453
H15A	H17B[1]	2.773	H15A	H19B[11]	3.318
H15A	H20A[12]	3.498	H15A	H20B[12]	2.483
H15A	H21C[12]	3.461	H15B	C19[11]	3.344
H15B	C20[11]	3.195	H15B	C21[11]	3.272
H15B	H15B[11]	3.244	H15B	H19B[11]	2.656
H15B	H20B[11]	2.715	H15B	H20B[12]	3.547
H15B	H21A[11]	2.599	H16	F1A[4]	3.253
H16	C3[4]	3.429	H16	C8[4]	3.346
H16	C17[1]	3.372	H16	H8[4]	2.671
H16	H17A[1]	3.332	H16	H17B[1]	2.588
H16	H3A[4]	2.845	H16	H3B[4]	3.086
H16	H3C[4]	2.766	H17A	F1A[4]	3.416
H17A	C2[6]	3.378	H17A	C3[6]	3.276
H17A	C7[6]	3.034	H17A	C8[6]	3.158
H17A	C12[6]	3.511	H17A	H2[6]	3.250
H17A	H8[6]	3.386	H17A	H8[4]	2.929

H17A	H16[10]	3.332	H17A	H3A[6]
	2.465			
H17A	H3C[6]	2.484	H17B	C14[10]
	3.527			
H17B	C15[10]	3.292	H17B	C16[10]
	3.332			
H17B	H14B[10]	2.871	H17B	H15A[10]
	2.773			
H17B	H16[10]	2.588	H17B	H20A[10]
	3.161			
H17B	H3A[6]	2.974	H17B	H3C[6]
	2.957			
H18A	C8[4]	3.541	H18A	C9[4]
	3.255			
H18A	H8[4]	3.099	H18A	H9[4]
	2.517			
H18A	H18B[6]	2.862	H18B	C8[6]
	3.588			
H18B	C9[6]	3.242	H18B	C10[6]
	3.535			
H18B	C18[6]	3.254	H18B	H9[10]
	3.124			
H18B	H9[6]	3.325	H18B	H14B[10]
	2.744			
H18B	H18A[6]	2.862	H18B	H18B[6]
	2.771			
H19A	F1A[4]	2.704	H19A	C3[6]
	3.431			
H19A	C3[4]	3.416	H19A	C4[6]
	3.582			
H19A	H2[6]	3.499	H19A	H4A[6]
	3.038			

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H19A	H4B[4]	3.429	H19A	H20A[10]	3.302
H19A	H3A[6]	2.684	H19A	H3A[4]	3.307
H19A	H3B[4]	2.756	H19A	H3C[6]	2.743
H19A	H3C[4]	3.228	H19B	C15[11]	3.425
H19B	C20[10]	3.463	H19B	H15A[11]	3.318
H19B	H15B[11]	2.656	H19B	H20A[10]	2.689
H19B	H20B[10]	3.352	H19B	H3A[6]	3.304
H19B	H3C[6]	3.325	H20A	F1A[4]	3.512
H20A	C3[4]	3.137	H20A	C4[4]	3.364
H20A	C19[1]	3.444	H20A	H4B[4]	2.784
H20A	H15A[12]	3.498	H20A	H17B[1]	3.161
H20A	H19A[1]	3.302	H20A	H19B[1]	2.689
H20A	H3A[4]	2.523	H20A	H3B[4]	3.187
H20A	H3C[4]	2.483	H20B	C15[12]	3.361
H20B	H13[11]	3.587	H20B	H14A[12]	3.567
H20B	H15A[12]	2.483	H20B	H15B[11]	2.715
H20B	H15B[12]	3.547	H20B	H19B[1]	3.352
H20B	H20B[12]	3.338	H21A	C6[14]	3.313
H21A	C13[11]	3.430	H21A	C14[11]	3.206
H21A	C15[11]	3.291	H21A	H6A[14]	2.893
H21A	H6B[14]	2.914	H21A	H13[11]	2.780

H21A	H14A^{11}	2.678	H21A	H15B^{11}
	2.599			
H21A	H1A^{14}	3.336	H21A	H1B^{14}
	3.337			
H21B	F1^{13}	2.996	H21B	C1^{14}
	3.593			
H21B	C4^{4}	3.431	H21B	H4A^{6}
	3.440			
H21B	H4B^{4}	2.482	H21B	H6A^{14}
	3.539			
H21B	H6A^{13}	3.509	H21B	H6B^{14}
	3.145			
H21B	H1A^{14}	2.794	H21B	H1B^{14}
	2.798			
H21B	H1C^{13}	3.197	H21C	F1^{13}
	3.201			
H21C	C6^{13}	3.314	H21C	C14^{12}
	3.541			
H21C	H4B^{4}	3.284	H21C	H6A^{13}
	2.429			
H21C	H6B^{14}	3.286	H21C	H11^{11}
	3.367			
H21C	H13^{11}	3.084	H21C	H14A^{12}
	2.735			
H21C	H15A^{12}	3.461	H21C	H1C^{13}
	3.239			
H1A	F1^{10}	3.507	H1A	C4^{2}
	3.209			
H1A	C21^{5}	3.490	H1A	H4A^{2}
	2.778			
H1A	H4B^{2}	2.812	H1A	H5B^{2}
	3.321			

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H1A	H21A^{5}	3.336	H1A	H21B^{5}	2.794
H1B	C4^{2}	3.222	H1B	C21^{5}	3.493
H1B	H4A^{2}	2.791	H1B	H4B^{2}	2.823
H1B	H5B^{2}	3.330	H1B	H21A^{5}	3.337
H1B	H21B^{5}	2.798	H1C	H2^{1}	3.218
H1C	H4A^{2}	3.599	H1C	H4B^{2}	2.922
H1C	H5B^{2}	3.326	H1C	H12^{1}	3.138
H1C	H21B^{3}	3.197	H1C	H21C^{3}	3.239
H3A	C17^{6}	3.139	H3A	C19^{6}	3.381
H3A	C19^{4}	3.579	H3A	C20^{4}	3.373
H3A	H16^{4}	2.845	H3A	H17A^{6}	2.465
H3A	H17B^{6}	2.974	H3A	H19A^{6}	2.684
H3A	H19A^{4}	3.307	H3A	H19B^{6}	3.304
H3A	H20A^{4}	2.523	H3B	C19^{4}	3.431
H3B	H2^{1}	2.926	H3B	H4A^{1}	3.598
H3B	H16^{4}	3.086	H3B	H19A^{4}	2.756
H3B	H20A^{4}	3.187	H3C	C16^{4}	3.556
H3C	C17^{6}	3.145	H3C	C19^{6}	3.423
H3C	C19^{4}	3.501	H3C	C20^{4}	3.321
H3C	H16^{4}	2.766	H3C	H17A^{6}	2.484
H3C	H17B^{6}	2.957	H3C	H19A^{6}	2.743

H3C	H19A[4]	3.228	H3C	H19B[6]
		3.325		
H3C	H20A[4]	2.483		

Symmetry Operators:

(1) X+1,Y,Z
(2) X,-Y+1,Z
(3) -X+2,Y+1/2,-Z+1/2
(4) -X+2,-Y,-Z+1
(5) -X+1,Y+1/2,-Z+1/2
(6) -X+1,-Y,-Z+1
(7) X,-Y+1,Z+1
(8) X+1,-Y+1,Z+1
(9) X-1,-Y+1,Z
(10) X-1,Y,Z
(11) -X+1,-Y,-Z
(12) -X+2,-Y,-Z
(13) -X+2,Y+1/2-1,-Z+1/2
(14) -X+1,Y+1/2-1,-Z+1/2

X-Ray Data Collection for **13**

Data Collection

A colorless platelet crystal of $C_{21}H_{28}O_2$ having approximate dimensions of 0.270 x 0.060 x 0.010 mm was mounted in a loop. All measurements were made on a Rigaku XtaLAB P200 diffractometer using multi-layer mirror monochromated Cu-Kα radiation.

Cell constants and an orientation matrix for data collection corresponded to a primitive triclinic cell with dimensions:

a = 5.4875(6) Å α = 79.139(11)°
b = 9.8366(9) Å β = 84.202(15)°
c = 16.6611(15) Å γ = 79.462(15)°
V = 866.27(16) $Å^3$

For Z = 2 and F.W. = 312.45, the calculated density is 1.198 g/cm^3. Based on a statistical analysis of intensity distribution, and the successful solution and refinement of the structure, the space group was determined to be:

P-1 (#2)

The data were collected at a temperature of -148 ± 1°C to a maximum 2θ value of 136.6°. Readout was performed in the 0.172 mm pixel mode.

Data Reduction

Of the 10865 reflections were collected, where 3048 were unique (R_{int} = 0.0359); equivalent reflections were merged. Data were collected and processed using CrystalClear (Rigaku). [1]

The linear absorption coefficient, μ, for Cu-Kα radiation is 5.814 cm^{-1}. An empirical absorption correction was applied which resulted in transmission factors ranging from 0.902 to 0.994. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by direct methods[2] and expanded using Fourier techniques.

The crystal is a non-merohedral twin with twin law:

-1.00000 0.00000 0.00000
0.00000 -1.00000 0.00000
-0.41800 -0.59600 1.00000

Twin component #1 comprises 23.10% of the crystal.

The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined using the riding model. The final cycle of full-matrix least-squares refinement[3] on F^2 was based on 3048 observed reflections and 210 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R1 = \Sigma \, ||Fo| - |Fc|| \, / \, \Sigma \, |Fo| = 0.0535$$

$$wR2 = [\Sigma (w (Fo^2 - Fc^2)^2) / \Sigma \, w(Fo^2)^2]^{1/2} = 0.1621$$

The goodness of fit[4] was 1.15. Unit weights were used. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.27 and -0.27 $e^-/Å^3$, respectively.

Neutral atom scattering factors were taken from International Tables for Crystallography (IT), Vol. C, Table 6.1.1.4 [5]. Anomalous dispersion effects were included in Fcalc[6]; the values for Δf' and Δf'' were those of Creagh and McAuley[7]. The values for the mass attenuation coefficients are those of Creagh and Hubbell[8]. All calculations were performed using the CrystalStructure[9] crystallographic software package except for refinement, which was performed using SHELXL2013[10].

References

(1) CrystalClear: Data Collection and Processing Software, Rigaku Corporation (1998-2014). Tokyo 196-8666, Japan.

(2) SHELXS2013: Sheldrick, G. M. (2008). Acta Cryst. A64, 112-122.

(3) Least Squares function minimized: (SHELXL2013)

$\Sigma w(F_o^2 - F_c^2)^2$ where w = Least Squares weights.

(4) Goodness of fit is defined as:

$[\Sigma w(F_o^2 - F_c^2)^2 / (N_o - N_v)]^{1/2}$

where: N_O = number of observations
N_V = number of variables

(5) International Tables for Crystallography, Vol.C (1992). Ed. A.J.C. Wilson, Kluwer Academic Publishers, Dordrecht, Netherlands, Table 6.1.1.4, pp. 572.

(6) Ibers, J. A. & Hamilton, W. C.; Acta Crystallogr., 17, 781 (1964).

(7) Creagh, D. C. & McAuley, W.J .; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.6.8, pages 219-222 (1992).

(8) Creagh, D. C. & Hubbell, J.H..; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.4.3, pages 200-206 (1992).

(9) CrystalStructure 4.1: Crystal Structure Analysis Package, Rigaku Corporation (2000-2014). Tokyo 196-8666, Japan.

(10) SHELXL2013: Sheldrick, G. M. (2008). Acta Cryst. A64, 112-122.

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$C_{21}H_{28}O_2$
Formula Weight	312.45
Crystal Color, Habit	colorless, platelet
Crystal Dimensions	0.270 X 0.060 X 0.010 mm
Crystal System	triclinic
Lattice Type	Primitive
Lattice Parameters	a = 5.4875(6) Å b = 9.8366(9) Å c = 16.6611(15) Å α = 79.139(11) ^{o} β = 84.202(15) ^{o} γ = 79.462(15) ^{o} V = 866.27(16) Å^3
Space Group	P-1 (#2)
Z value	2
D_{calc}	1.198 g/cm^3
F_{000}	340.00
μ(CuKα)	5.814 cm^{-1}

B. Intensity Measurements

Diffractometer	XtaLAB P100
Radiation monochromated	CuKα (λ = 1.54187 Å) multi-layer mirror
Voltage, Current	40kV, 30mA
Temperature	-148.1°C
Detector Aperture	83.8 x 33.5 mm
Pixel Size	0.172 mm
$2\theta_{max}$	136.6°
No. of Reflections Measured	Total: 10865 Unique: 3048 (R_{int} = 0.0359)
Corrections	Lorentz-polarization Absorption (trans. factors: 0.902 - 0.994)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SHELXS2013)
Refinement	Full-matrix least-squares on F^2
Function Minimized	Σ w (Fo^2 - Fc^2)^2
Least Squares Weights	w = 1/ [$\sigma^2(Fo^2)$ + (0.0736 · P)^2 + 0.4931 · P] where P = (Max(Fo^2,0) + 2Fc^2)/3
$2\theta_{max}$ cutoff	136.6^o
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (All reflections)	3048
No. Variables	210
Reflection/Parameter Ratio	14.51
Residuals: R1 (I>2.00σ(I))	0.0535
Residuals: R (All reflections)	0.0570
Residuals: wR2 (All reflections)	0.1621
Goodness of Fit Indicator	1.145
Max Shift/Error in Final Cycle	0.000
Maximum peak in Final Diff. Map	0.27 e^-/Å^3
Minimum peak in Final Diff. Map	-0.27 e^-/Å^3

Table 1. Atomic coordinates and B_{iso}/B_{eq}

atom	x	y	z	B_{eq}
O1	1.1956(3)	0.11086(16)	1.14462(9)	2.16(3)
O2	0.8481(3)	0.35967(16)	1.17825(9)	2.50(3)
C1	0.9673(4)	0.0542(2)	1.14991(12)	1.96(4)
C2	0.7518(4)	0.1154(2)	1.20194(13)	2.17(4)
C3	0.6653(4)	0.2705(2)	1.17864(13)	2.17(4)
C4	0.7558(4)	0.3513(2)	1.10177(12)	2.02(4)
C5	0.9394(4)	0.2845(2)	1.04057(12)	1.72(3)
C6	1.0575(4)	0.1350(2)	1.07279(12)	1.78(4)
C7	0.8133(4)	0.2869(2)	0.96304(12)	1.61(3)
C8	0.8852(4)	0.3653(2)	0.88886(12)	1.79(4)
C9	0.7734(4)	0.3655(2)	0.81794(12)	1.82(4)
C10	0.5860(4)	0.2862(2)	0.81836(12)	1.58(3)
C11	0.5133(4)	0.2086(2)	0.89326(12)	1.73(3)
C12	0.6235(4)	0.2090(2)	0.96444(12)	1.75(3)
C13	0.4669(4)	0.2816(2)	0.74107(12)	1.78(4)
C14	0.4029(4)	0.4261(2)	0.68751(12)	2.09(4)
C15	0.2686(4)	0.4156(2)	0.61372(12)	2.14(4)
C16	0.4164(4)	0.3132(2)	0.56131(12)	2.00(4)
C17	0.4969(5)	0.1703(2)	0.61469(13)	2.25(4)
C18	0.6293(4)	0.1811(2)	0.68944(12)	1.93(4)
C19	0.2597(5)	0.3043(2)	0.49196(13)	2.45(4)
C20	0.3851(5)	0.2185(3)	0.42768(13)	2.61(4)
C21	0.2054(5)	0.2132(3)	0.36468(15)	3.10(5)

$$B_{eq} = 8/3\ \pi^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$$

Table 2. Atomic coordinates and B_{iso} involving hydrogen atoms

atom	x	y	z	B_{iso}
H1	0.98832	-0.04951	1.15190	2.350
H2A	0.79796	0.09551	1.25942	2.605
H2B	0.61063	0.06657	1.19971	2.605
H3	0.49321	0.30661	1.19990	2.605
H4	0.63672	0.43530	1.07786	2.419
H5	1.07502	0.34205	1.02567	2.063
H6	1.13150	0.07811	1.02981	2.133
H8	1.01290	0.41961	0.88678	2.148
H9	0.82494	0.42054	0.76807	2.182
H11	0.38549	0.15436	0.89545	2.073
H12	0.56957	0.15586	1.01464	2.102
H13	0.30782	0.24487	0.75807	2.133
H14A	0.29577	0.49099	0.72025	2.508
H14B	0.55727	0.46462	0.66848	2.508
H15A	0.23382	0.50986	0.57939	2.565
H15B	0.10719	0.38535	0.63318	2.565
H16	0.56913	0.35112	0.53633	2.398
H17A	0.60956	0.10894	0.58114	2.704
H17B	0.34848	0.12584	0.63363	2.704
H18A	0.78796	0.21533	0.67090	2.317
H18B	0.66808	0.08687	0.72347	2.317
H19A	0.11092	0.26434	0.51684	2.946
H19B	0.20109	0.40105	0.46374	2.946
H20A	0.44875	0.12175	0.45484	3.134
H20B	0.52838	0.26048	0.39953	3.134
H21A	0.14545	0.30870	0.33674	3.724
H21B	0.06418	0.17084	0.39238	3.724
H21C	0.29102	0.15669	0.32445	3.724

Table 3. Anisotropic displacement parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O1	0.0219(8)	0.0334(8)	0.0271(8)	-0.0076(6)	-0.0087(6)	0.0008(6)
O2	0.0477(10)	0.0305(9)	0.0214(7)	-0.0143(7)	-0.0014(7)	-0.0087(6)
C1	0.0244(11)	0.0230(10)	0.0278(10)	-0.0067(9)	-0.0092(9)	0.0002(8)
C2	0.0281(12)	0.0298(12)	0.0243(10)	-0.0098(9)	-0.0031(9)	0.0016(9)
C3	0.0288(12)	0.0299(12)	0.0244(10)	-0.0056(9)	0.0015(9)	-0.0072(9)
C4	0.0321(12)	0.0219(10)	0.0235(10)	-0.0045(9)	-0.0034(9)	-0.0055(8)
C5	0.0233(11)	0.0220(10)	0.0212(10)	-0.0060(8)	-0.0020(8)	-0.0044(8)
C6	0.0208(11)	0.0233(11)	0.0242(10)	-0.0025(8)	-0.0050(8)	-0.0053(8)
C7	0.0212(10)	0.0187(10)	0.0207(9)	-0.0006(8)	-0.0013(8)	-0.0050(8)
C8	0.0233(11)	0.0194(10)	0.0252(10)	-0.0048(8)	-0.0010(8)	-0.0027(8)
C9	0.0238(11)	0.0235(10)	0.0199(9)	-0.0038(9)	0.0004(8)	-0.0004(8)
C10	0.0182(10)	0.0210(10)	0.0192(9)	0.0007(8)	-0.0002(8)	-0.0037(8)
C11	0.0211(11)	0.0232(10)	0.0214(9)	-0.0054(8)	-0.0011(8)	-0.0025(8)
C12	0.0229(11)	0.0227(10)	0.0196(9)	-0.0041(8)	0.0001(8)	-0.0007(8)
C13	0.0228(11)	0.0247(11)	0.0194(9)	-0.0045(8)	-0.0016(8)	-0.0016(8)
C14	0.0313(12)	0.0231(11)	0.0228(10)	0.0027(9)	-0.0036(9)	-0.0044(8)
C15	0.0307(12)	0.0259(11)	0.0216(10)	0.0019(9)	-0.0058(9)	-0.0010(8)
C16	0.0305(12)	0.0247(11)	0.0198(10)	-0.0036(9)	-0.0039(8)	-0.0014(8)
C17	0.0401(13)	0.0219(11)	0.0240(10)	-0.0020(9)	-0.0080(9)	-0.0049(8)
C18	0.0302(12)	0.0190(10)	0.0227(10)	-0.0000(9)	-0.0053(9)	-0.0020(8)
C19	0.0374(13)	0.0302(12)	0.0244(11)	-0.0006(10)	-0.0071(10)	-0.0037(9)
C20	0.0407(14)	0.0310(12)	0.0267(11)	-0.0012(10)	-0.0049(10)	-0.0062(9)

C21 0.0485(16) 0.0411(14) 0.0291(12) -0.0006(12) -0.0094(11) -0.0113(10)

The general temperature factor expression: $\exp(-2\pi^2(a^{*2}U_{11}h^2 + b^{*2}U_{22}k^2 + c^{*2}U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$

Table 4. Bond lengths (Å)

atom	atom	distance	atom	atom	distance
O1	C1	1.450(3)	O1	C6	1.440(3)
O2	C3	1.447(3)	O2	C4	1.440(3)
C1	C2	1.497(3)	C1	C6	1.469(3)
C2	C3	1.501(3)	C3	C4	1.466(3)
C4	C5	1.513(3)	C5	C6	1.515(3)
C5	C7	1.520(3)	C7	C8	1.390(3)
C7	C12	1.397(3)	C8	C9	1.385(3)
C9	C10	1.399(3)	C10	C11	1.397(3)
C10	C13	1.513(3)	C11	C12	1.386(3)
C13	C14	1.530(3)	C13	C18	1.534(3)
C14	C15	1.524(3)	C15	C16	1.525(3)
C16	C17	1.531(3)	C16	C19	1.533(3)
C17	C18	1.534(3)	C19	C20	1.521(3)
C20	C21	1.525(4)			

Table 5. Bond lengths involving hydrogens (Å)

atom	atom	distance	atom	atom	distance
C1	H1	1.000	C2	H2A	0.990
C2	H2B	0.990	C3	H3	1.000
C4	H4	1.000	C5	H5	1.000
C6	H6	1.000	C8	H8	0.950
C9	H9	0.950	C11	H11	0.950
C12	H12	0.950	C13	H13	1.000
C14	H14A	0.990	C14	H14B	0.990
C15	H15A	0.990	C15	H15B	0.990
C16	H16	1.000	C17	H17A	0.990
C17	H17B	0.990	C18	H18A	0.990
C18	H18B	0.990	C19	H19A	0.990
C19	H19B	0.990	C20	H20A	0.990
C20	H20B	0.990	C21	H21A	0.980
C21	H21B	0.980	C21	H21C	0.980

Table 6. Bond angles (°)

atom	atom	atom	angle	atom	atom	atom	angle
C1	O1	C6	61.11(13)	C3	O2	C4	61.06(14)
O1	C1	C2	117.55(19)	O1	C1	C6	59.09(13)
C2	C1	C6	121.75(17)	C1	C2	C3	115.59(18)
O2	C3	C2	116.32(19)	O2	C3	C4	59.21(14)
C2	C3	C4	121.75(19)	O2	C4	C3	59.72(14)
O2	C4	C5	116.90(18)	C3	C4	C5	122.62(18)
C4	C5	C6	113.85(16)	C4	C5	C7	110.01(17)
C6	C5	C7	109.01(18)	O1	C6	C1	59.80(13)
O1	C6	C5	117.74(19)	C1	C6	C5	122.55(17)
C5	C7	C8	121.0(2)	C5	C7	C12	120.74(17)
C8	C7	C12	118.3(2)	C7	C8	C9	121.0(2)
C8	C9	C10	121.24(18)	C9	C10	C11	117.4(2)
C9	C10	C13	122.02(17)	C11	C10	C13	120.5(2)
C10	C11	C12	121.5(2)	C7	C12	C11	120.59(18)
C10	C13	C14	113.28(19)	C10	C13	C18	111.98(17)
C14	C13	C18	109.12(16)	C13	C14	C15	110.68(19)
C14	C15	C16	113.15(18)	C15	C16	C17	110.16(16)
C15	C16	C19	108.97(18)	C17	C16	C19	112.8(2)
C16	C17	C18	112.8(2)	C13	C18	C17	110.99(17)
C16	C19	C20	116.9(2)	C19	C20	C21	111.4(2)

Table 7. Bond angles involving hydrogens (°)

atom	atom	atom	angle	atom	atom	atom	angle
O1	C1	H1	115.5	C2	C1	H1	115.5
C6	C1	H1	115.5	C1	C2	H2A	108.4
C1	C2	H2B	108.4	C3	C2	H2A	108.4
C3	C2	H2B	108.4	H2A	C2	H2B	107.5
O2	C3	H3	115.8	C2	C3	H3	115.8
C4	C3	H3	115.8	O2	C4	H4	115.3
C3	C4	H4	115.3	C5	C4	H4	115.3
C4	C5	H5	107.9	C6	C5	H5	107.9
C7	C5	H5	107.9	O1	C6	H6	115.1
C1	C6	H6	115.1	C5	C6	H6	115.1
C7	C8	H8	119.5	C9	C8	H8	119.5
C8	C9	H9	119.4	C10	C9	H9	119.4
C10	C11	H11	119.3	C12	C11	H11	119.3
C7	C12	H12	119.7	C11	C12	H12	119.7
C10	C13	H13	107.4	C14	C13	H13	107.4
C18	C13	H13	107.4	C13	C14	H14A	109.5
C13	C14	H14B	109.5	C15	C14	H14A	109.5
C15	C14	H14B	109.5	H14A	C14	H14B	108.1
C14	C15	H15A	108.9	C14	C15	H15B	108.9
C16	C15	H15A	109.0	C16	C15	H15B	109.0
H15A	C15	H15B	107.8	C15	C16	H16	108.3
C17	C16	H16	108.3	C19	C16	H16	108.3

C16	C17	H17A	109.0	C16	C17	H17B
	109.0					
C18	C17	H17A	109.0	C18	C17	H17B
	109.0					
H17A	C17	H17B	107.8	C13	C18	H18A
	109.4					
C13	C18	H18B	109.4	C17	C18	H18A
	109.4					
C17	C18	H18B	109.4	H18A	C18	H18B
	108.0					
C16	C19	H19A	108.1	C16	C19	H19B
	108.1					
C20	C19	H19A	108.1	C20	C19	H19B
	108.1					
H19A	C19	H19B	107.3	C19	C20	H20A
	109.4					
C19	C20	H20B	109.3	C21	C20	H20A
	109.3					
C21	C20	H20B	109.4	H20A	C20	H20B
	108.0					
C20	C21	H21A	109.5	C20	C21	H21B
	109.5					
C20	C21	H21C	109.5	H21A	C21	H21B
	109.5					
H21A	C21	H21C	109.5	H21B	C21	H21C
	109.5					

Table 8. Torsion Angles(º)
(Those having bond angles > 160 or < 20 degrees are excluded.)

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
C1	O1	C6	C1	0.00(9)	C1	O1	C6	C5	113.41(18)
C6	O1	C1	C2	-112.30(17)	C6	O1	C1	C6	-0.00(9)
C3	O2	C4	C3	-0.00(10)	C3	O2	C4	C5	-113.81(19)
C4	O2	C3	C2	112.87(19)	C4	O2	C3	C4	0.00(9)
O1	C1	C2	C3	58.3(2)	O1	C1	C6	O1	0.00(7)
O1	C1	C6	C5	-105.5(2)	C2	C1	C6	O1	105.3(2)
C2	C1	C6	C5	-0.2(3)	C6	C1	C2	C3	-10.7(3)
C1	C2	C3	O2	-58.0(3)	C1	C2	C3	C4	10.6(3)
O2	C3	C4	O2	-0.00(7)	O2	C3	C4	C5	104.4(2)
C2	C3	C4	O2	-103.8(2)	C2	C3	C4	C5	0.6(4)
O2	C4	C5	C6	58.5(2)	O2	C4	C5	C7	-178.83(15)
C3	C4	C5	C6	-11.2(3)	C3	C4	C5	C7	111.5(2)
C4	C5	C6	O1	-59.1(2)	C4	C5	C6	C1	11.1(3)
C4	C5	C7	C8	114.92(18)	C4	C5	C7	C12	-65.8(2)
C6	C5	C7	C8	-119.59(18)	C6	C5	C7	C12	59.7(2)
C7	C5	C6	O1	177.63(15)	C7	C5	C6	C1	-112.2(2)
C5	C7	C8	C9	178.81(15)	C5	C7	C12	C11	-178.33(15)
C8	C7	C12	C11	1.0(3)	C12	C7	C8	C9	-0.5(3)
C7	C8	C9	C10	-0.5(3)	C8	C9	C10	C11	1.0(3)
C8	C9	C10	C13	-178.14(16)	C9	C10	C11	C12	-0.5(3)
C9	C10	C13	C14	-44.3(2)	C9	C10	C13	C18	79.7(2)

C11	C10	C13	C14	136.62(17)	C11	C10	C13	C18	-99.5(2)
C13	C10	C11	C12	178.64(15)	C10	C11	C12	C7	-0.5(3)
C10	C13	C14	C15	-176.28(14)	C10	C13	C18	C17	175.98(15)
C14	C13	C18	C17	-57.8(2)	C18	C13	C14	C15	58.2(2)
C13	C14	C15	C16	-57.0(2)	C14	C15	C16	C17	52.6(2)
C14	C15	C16	C19	176.82(15)	C15	C16	C17	C18	-51.8(2)
C15	C16	C19	C20	173.96(16)	C17	C16	C19	C20	-63.4(2)
C19	C16	C17	C18	-173.79(16)	C16	C17	C18	C13	55.7(2)
C16	C19	C20	C21	177.52(16)					

Table 9. Intramolecular contacts less than 3.60 Å

atom	atom	distance	atom	atom	distance
O1	O2	2.921(2)	O1	C3	3.097(3)
O1	C4	3.090(2)	O2	C1	3.071(3)
O2	C6	3.069(3)	C1	C4	2.931(3)
C1	C7	3.576(3)	C2	C5	3.072(3)
C3	C6	2.931(3)	C3	C7	3.582(3)
C4	C8	3.528(3)	C4	C12	3.104(3)
C6	C8	3.550(3)	C6	C12	3.033(3)
C7	C10	2.825(3)	C8	C11	2.761(3)
C9	C12	2.765(3)	C9	C14	3.029(3)
C9	C18	3.291(3)	C11	C18	3.443(3)
C13	C16	2.988(3)	C14	C17	2.935(3)
C15	C18	2.928(3)	C17	C20	3.167(3)

Table 10. Intramolecular contacts less than 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
O1	H2A	2.759	O1	H2B	3.332
O1	H5	2.748	O2	H2A	2.742
O2	H2B	3.321	O2	H5	2.737
C1	H3	3.407	C1	H5	3.288
C1	H12	3.192	C2	H4	3.415
C2	H6	3.407	C2	H12	3.303
C3	H1	3.403	C3	H5	3.285
C3	H12	3.268	C4	H2A	3.271
C4	H2B	3.158	C4	H6	3.405
C4	H12	2.986	C5	H1	3.433
C5	H3	3.435	C5	H8	2.683
C5	H12	2.689	C6	H2A	3.271
C6	H2B	3.156	C6	H4	3.409
C6	H12	2.898	C7	H4	2.625
C7	H6	2.593	C7	H9	3.266
C7	H11	3.269	C8	H4	3.443
C8	H5	2.555	C8	H6	3.474
C8	H12	3.253	C9	H11	3.250
C9	H13	3.317	C9	H14A	3.156
C9	H14B	2.796	C9	H18A	3.078
C9	H18B	3.565	C10	H8	3.277
C10	H12	3.279	C10	H14A	2.716

C10	H14B	2.769	C10	H18A
	2.750			
C10	H18B	2.692	C11	H9
	3.250			
C11	H13	2.556	C11	H18B
	3.252			
C12	H4	3.194	C12	H5
	3.330			
C12	H6	3.064	C12	H8
	3.253			
C13	H9	2.706	C13	H11
	2.672			
C13	H15A	3.362	C13	H15B
	2.731			
C13	H16	3.361	C13	H17A
	3.376			
C13	H17B	2.758	C14	H9
	2.779			
C14	H16	2.777	C14	H17B
	3.315			
C14	H18A	2.708	C14	H18B
	3.355			
C15	H13	2.665	C15	H17A
	3.353			
C15	H17B	2.765	C15	H18A
	3.271			
C15	H19A	2.688	C15	H19B
	2.597			
C16	H13	3.237	C16	H14A
	3.388			

Table 10. Intramolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
C16	H14B	2.776	C16	H18A	2.791
C16	H18B	3.395	C16	H20A	2.793
C16	H20B	2.826	C17	H13	2.684
C17	H14B	3.269	C17	H15A	3.355
C17	H15B	2.757	C17	H19A	2.722
C17	H19B	3.382	C17	H20A	2.839
C17	H20B	3.521	C18	H9	3.284
C18	H11	3.536	C18	H14A	3.356
C18	H14B	2.704	C18	H15B	3.294
C18	H16	2.788	C19	H15A	2.682
C19	H15B	2.634	C19	H17A	2.782
C19	H17B	2.692	C19	H21A	2.713
C19	H21B	2.706	C19	H21C	3.356
C20	H16	2.782	C20	H17A	2.886
C20	H17B	3.376	C21	H19A	2.659
C21	H19B	2.695	H1	H2A	2.525
H1	H2B	2.319	H1	H6	2.325
H1	H12	3.495	H2A	H3	2.526
H2B	H3	2.332	H2B	H12	3.062
H3	H4	2.330	H4	H5	2.540
H4	H12	3.216	H5	H6	2.546

H5	H8	2.335	H6	H12
	3.063			
H8	H9	2.319	H9	H14A
	3.012			
H9	H14B	2.255	H9	H18A
	2.861			
H11	H12	2.321	H11	H13
	2.346			
H11	H18B	3.240	H13	H14A
	2.374			
H13	H14B	2.868	H13	H15A
	3.571			
H13	H15B	2.505	H13	H17A
	3.593			
H13	H17B	2.539	H13	H18A
	2.871			
H13	H18B	2.375	H14A	H15A
	2.373			
H14A	H15B	2.349	H14B	H15A
	2.355			
H14B	H15B	2.868	H14B	H16
	2.640			
H14B	H18A	2.541	H15A	H16
	2.333			
H15A	H19A	3.010	H15A	H19B
	2.410			
H15B	H16	2.865	H15B	H17B
	2.651			
H15B	H19A	2.455	H15B	H19B
	2.798			
H16	H17A	2.335	H16	H17B
	2.870			

Table 10. Intramolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H16	H18A	2.661	H16	H19A	2.865
H16	H19B	2.385	H16	H20A	3.042
H16	H20B	2.642	H17A	H18A	2.356
H17A	H18B	2.389	H17A	H19A	3.069
H17A	H20A	2.336	H17A	H20B	3.147
H17B	H18A	2.874	H17B	H18B	2.352
H17B	H19A	2.491	H17B	H19B	3.573
H17B	H20A	2.981	H19A	H20A	2.383
H19A	H20B	2.857	H19A	H21A	2.941
H19A	H21B	2.475	H19A	H21C	3.554
H19B	H20A	2.857	H19B	H20B	2.354
H19B	H21A	2.520	H19B	H21B	2.987
H19B	H21C	3.584	H20A	H21A	2.864
H20A	H21B	2.377	H20A	H21C	2.361
H20B	H21A	2.372	H20B	H21B	2.864
H20B	H21C	2.367			

Table 11. Intermolecular contacts less than 3.60 Å

atom	atom	distance	atom	atom	distance
O1	C2[1]	3.299(3)	O1	C3[1]	3.397(3)
O1	C11[2]	3.396(3)	O2	C8[3]	3.269(3)
O2	C14[4]	3.381(3)	C1	C11[2]	3.594(3)
C2	O1[5]	3.299(3)	C3	O1[5]	3.397(3)
C6	C12[1]	3.554(3)	C8	O2[3]	3.269(3)
C8	C11[1]	3.511(3)	C11	O1[2]	3.396(3)
C11	C1[2]	3.594(3)	C11	C8[5]	3.511(3)
C12	C6[5]	3.554(3)	C14	O2[4]	3.381(3)

Symmetry Operators:

(1) X+1,Y,Z
(2) -X+2,-Y,-Z+2
(3) -X+2,-Y+1,-Z+2
(4) -X+1,-Y+1,-Z+2
(5) X-1,Y,Z

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
O1	H2B[1]	2.475	O1	H3[1]	3.057
O1	H11[2]	3.269	O1	H12[1]	2.858
O1	H18B[2]	2.713	O1	H21C[3]	3.220
O2	H3[1]	3.528	O2	H8[4]	2.454
O2	H9[4]	3.341	O2	H14A[5]	2.426
O2	H21A[3]	3.156	C1	H11[6]	3.295
C1	H13[6]	3.569	C1	H18B[2]	3.019
C2	H11[6]	3.583	C2	H13[6]	3.549
C2	H17B[6]	3.341	C2	H18B[6]	3.313
C2	H21C[7]	3.101	C3	H14A[5]	3.183
C3	H21C[7]	3.191	C4	H5[4]	3.561
C4	H8[4]	2.825	C4	H14A[5]	3.562
C5	H8[4]	3.417	C5	H11[1]	3.507
C5	H12[1]	3.471	C6	H6[2]	3.301
C6	H11[1]	3.293	C6	H12[1]	2.909
C7	H1[2]	3.262	C7	H4[5]	3.346
C7	H6[2]	3.528	C7	H11[1]	3.342
C8	H1[2]	3.241	C8	H4[5]	3.223
C8	H5[4]	3.493	C8	H11[1]	3.122
C8	H13[1]	3.237	C8	H14A[1]	3.595
C9	H1[2]	3.112	C9	H3[5]	3.261
C9	H4[5]	3.286	C9	H13[1]	3.096

C9	H14A[1]	3.462	C9	H15B[1]
	3.411			
C10	H1[2]	2.989	C10	H4[5]
	3.482			
C10	H8[8]	3.353	C11	H1[2]
	2.993			
C11	H2B[6]	3.557	C11	H4[5]
	3.561			
C11	H5[8]	3.353	C11	H6[8]
	3.183			
C11	H6[2]	3.245	C11	H8[8]
	3.123			
C12	H1[2]	3.138	C12	H4[5]
	3.496			
C12	H5[8]	3.202	C12	H6[8]
	3.207			
C12	H6[2]	2.886	C13	H2B[6]
	3.473			
C13	H8[8]	3.568	C13	H9[8]
	3.554			
C14	H9[8]	3.322	C14	H19B[9]
	3.549			
C14	H20B[9]	3.222	C15	H9[8]
	3.359			
C15	H16[9]	3.232	C15	H18A[8]
	3.526			
C15	H19B[10]	3.107	C15	H20B[9]
	3.525			
C15	H21A[10]	3.377	C16	H15A[9]
	3.314			

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
C16	H16[9]	3.406	C17	H2A[6]	3.556
C17	H2B[6]	3.572	C17	H20A[11]	3.249
C17	H21C[11]	3.220	C18	H1[2]	3.450
C18	H2B[6]	3.173	C18	H15B[1]	3.537
C18	H21C[11]	3.320	C19	H14B[9]	3.365
C19	H15A[10]	3.179	C19	H15A[9]	3.585
C20	H14B[9]	3.280	C20	H17A[11]	3.246
C20	H20A[11]	3.555	C21	H2A[12]	3.452
C21	H3[13]	3.095	C21	H14B[9]	3.571
C21	H15A[10]	3.493	C21	H17A[11]	3.133
C21	H18B[11]	3.468	H1	C7[2]	3.262
H1	C8[2]	3.241	H1	C9[2]	3.112
H1	C10[2]	2.989	H1	C11[2]	2.993
H1	C12[2]	3.138	H1	C18[2]	3.450
H1	H6[2]	3.235	H1	H11[6]	2.702
H1	H11[2]	3.458	H1	H13[6]	2.854
H1	H18A[2]	3.327	H1	H18B[2]	2.873
H2A	C17[6]	3.556	H2A	C21[3]	3.452
H2A	H13[6]	3.570	H2A	H17B[6]	2.730
H2A	H18A[2]	3.538	H2A	H18B[6]	3.343
H2A	H18B[2]	3.153	H2A	H20B[7]	3.182

H2A	H21A[3]	3.560	H2A	H21B[3]
	3.043			
H2A	H21C[7]	2.881	H2A	H21C[3]
	3.208			
H2B	O1[8]	2.475	H2B	C11[6]
	3.557			
H2B	C13[6]	3.473	H2B	C17[6]
	3.572			
H2B	C18[6]	3.173	H2B	H11[6]
	2.917			
H2B	H13[6]	2.972	H2B	H17B[6]
	3.055			
H2B	H18B[6]	2.445	H2B	H21C[7]
	2.751			
H3	O1[8]	3.057	H3	O2[8]
	3.528			
H3	C9[5]	3.261	H3	C21[7]
	3.095			
H3	H9[5]	3.029	H3	H14A[5]
	3.038			
H3	H14B[5]	3.385	H3	H20B[7]
	3.292			
H3	H21A[7]	2.822	H3	H21C[7]
	2.587			
H4	C7[5]	3.346	H4	C8[5]
	3.223			
H4	C9[5]	3.286	H4	C10[5]
	3.482			
H4	C11[5]	3.561	H4	C12[5]
	3.496			
H4	H4[5]	3.073	H4	H5[4]
	3.093			

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H4	H8[4]	2.762	H5	C4[4]	3.561
H5	C8[4]	3.493	H5	C11[1]	3.353
H5	C12[1]	3.202	H5	H4[4]	3.093
H5	H5[4]	3.045	H5	H8[4]	2.931
H5	H11[1]	3.260	H5	H12[1]	2.994
H6	C6[2]	3.301	H6	C7[2]	3.528
H6	C11[1]	3.183	H6	C11[2]	3.245
H6	C12[1]	3.207	H6	C12[2]	2.886
H6	H1[2]	3.235	H6	H6[2]	2.666
H6	H11[1]	2.576	H6	H11[2]	3.344
H6	H12[1]	2.628	H6	H12[2]	2.748
H8	O2[4]	2.454	H8	C4[4]	2.825
H8	C5[4]	3.417	H8	C10[1]	3.353
H8	C11[1]	3.123	H8	C13[1]	3.568
H8	H4[4]	2.762	H8	H5[4]	2.931
H8	H11[1]	2.996	H8	H13[1]	3.137
H8	H14A[1]	3.065	H9	O2[4]	3.341
H9	C13[1]	3.554	H9	C14[1]	3.322
H9	C15[1]	3.359	H9	H3[5]	3.029
H9	H13[1]	2.895	H9	H14A[1]	2.797
H9	H15B[1]	2.639	H9	H21A[9]	2.918

H11	O1[2]	3.269	H11	C1[6]
	3.295			
H11	C2[6]	3.583	H11	C5[8]
	3.507			
H11	C6[8]	3.293	H11	C7[8]
	3.342			
H11	C8[8]	3.122	H11	H1[6]
	2.702			
H11	H1[2]	3.458	H11	H2B[6]
	2.917			
H11	H5[8]	3.260	H11	H6[8]
	2.576			
H11	H6[2]	3.344	H11	H8[8]
	2.996			
H11	H12[6]	3.113	H12	O1[8]
	2.858			
H12	C5[8]	3.471	H12	C6[8]
	2.909			
H12	H5[8]	2.994	H12	H6[8]
	2.628			
H12	H6[2]	2.748	H12	H11[6]
	3.113			
H12	H12[6]	3.423	H13	C1[6]
	3.569			
H13	C2[6]	3.549	H13	C8[8]
	3.237			
H13	C9[8]	3.096	H13	H1[6]
	2.854			
H13	H2A[6]	3.570	H13	H2B[6]
	2.972			
H13	H8[8]	3.137	H13	H9[8]
	2.895			

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H13	H18A[8]	3.410	H14A	O2[5]	2.426
H14A	C3[5]	3.183	H14A	C4[5]	3.562
H14A	C8[8]	3.595	H14A	C9[8]	3.462
H14A	H3[5]	3.038	H14A	H8[8]	3.065
H14A	H9[8]	2.797	H14A	H20B[9]	3.086
H14A	H21A[10]	2.943	H14B	C19[9]	3.365
H14B	C20[9]	3.280	H14B	C21[9]	3.571
H14B	H3[5]	3.385	H14B	H15B[1]	3.002
H14B	H19B[9]	2.704	H14B	H20B[9]	2.703
H14B	H21A[9]	2.978	H15A	C16[9]	3.314
H15A	C19[10]	3.179	H15A	C19[9]	3.585
H15A	C21[10]	3.493	H15A	H16[9]	2.425
H15A	H19A[10]	2.969	H15A	H19B[10]	2.515
H15A	H19B[9]	3.348	H15A	H20B[9]	2.898
H15A	H21A[10]	2.909	H15A	H21B[10]	3.359
H15B	C9[8]	3.411	H15B	C18[8]	3.537
H15B	H9[8]	2.639	H15B	H14B[8]	3.002
H15B	H18A[8]	2.591	H15B	H19B[10]	2.803
H15B	H21A[10]	3.183	H16	C15[9]	3.232
H16	C16[9]	3.406	H16	H15A[9]	2.425
H16	H16[9]	2.942	H16	H19A[1]	2.942

H16	H19B[9]	2.942	H17A	C20[11]
		3.246		
H17A	C21[11]	3.133	H17A	H19A[1]
		3.373		
H17A	H20A[11]	2.535	H17A	H21B[11]
		2.979		
H17A	H21C[11]	2.775	H17B	C2[6]
		3.341		
H17B	H2A[6]	2.730	H17B	H2B[6]
		3.055		
H17B	H18A[8]	3.074	H17B	H20A[11]
		3.061		
H17B	H21C[11]	3.108	H18A	C15[1]
		3.526		
H18A	H1[2]	3.327	H18A	H2A[2]
		3.538		
H18A	H13[1]	3.410	H18A	H15B[1]
		2.591		
H18A	H17B[1]	3.074	H18A	H19A[1]
		2.981		
H18B	O1[2]	2.713	H18B	C1[2]
		3.019		
H18B	C2[6]	3.313	H18B	C21[11]
		3.468		
H18B	H1[2]	2.873	H18B	H2A[6]
		3.343		
H18B	H2A[2]	3.153	H18B	H2B[6]
		2.445		
H18B	H21B[11]	3.496	H18B	H21C[11]
		2.630		
H19A	H15A[10]	2.969	H19A	H16[8]
		2.942		

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H19A	H17A[8]	3.373	H19A	H18A[8]	2.981
H19A	H19B[10]	3.478	H19B	C14[9]	3.549
H19B	C15[10]	3.107	H19B	H14B[9]	2.704
H19B	H15A[10]	2.515	H19B	H15A[9]	3.348
H19B	H15B[10]	2.803	H19B	H16[9]	2.942
H19B	H19A[10]	3.478	H19B	H19B[10]	2.983
H20A	C17[11]	3.249	H20A	C20[11]	3.555
H20A	H17A[11]	2.535	H20A	H17B[11]	3.061
H20A	H20A[11]	2.576	H20A	H21B[1]	3.524
H20B	C14[9]	3.222	H20B	C15[9]	3.525
H20B	H2A[13]	3.182	H20B	H3[13]	3.292
H20B	H14A[9]	3.086	H20B	H14B[9]	2.703
H20B	H15A[9]	2.898	H20B	H21A[1]	3.531
H20B	H21B[1]	2.908	H21A	O2[12]	3.156
H21A	C15[10]	3.377	H21A	H2A[12]	3.560
H21A	H3[13]	2.822	H21A	H9[9]	2.918
H21A	H14A[10]	2.943	H21A	H14B[9]	2.978
H21A	H15A[10]	2.909	H21A	H15B[10]	3.183
H21A	H20B[8]	3.531	H21B	H2A[12]	3.043
H21B	H15A[10]	3.359	H21B	H17A[11]	2.979
H21B	H18B[11]	3.496	H21B	H20A[8]	3.524

H21B	H20B[8]	2.908	H21C	O1[12]
	3.220			
H21C	C2[13]	3.101	H21C	C3[13]
	3.191			
H21C	C17[11]	3.220	H21C	C18[11]
	3.320			
H21C	H2A[12]	3.208	H21C	H2A[13]
	2.881			
H21C	H2B[13]	2.751	H21C	H3[13]
	2.587			
H21C	H17A[11]	2.775	H21C	H17B[11]
	3.108			
H21C	H18B[11]	2.630		

Symmetry Operators:

(1) X+1,Y,Z
(2) -X+2,-Y,-Z+2
(3) X+1,Y,Z+1
(4) -X+2,-Y+1,-Z+2
(5) -X+1,-Y+1,-Z+2
(6) -X+1,-Y,-Z+2
(7) X,Y,Z+1
(8) X-1,Y,Z
(9) -X+1,-Y+1,-Z+1
(10) -X,-Y+1,-Z+1
(11) -X+1,-Y,-Z+1
(12) X-1,Y,Z-1
(13) X,Y,Z-1

X-Ray Data Collection for **16**

Data Collection

A colorless platelet crystal of $C_{23}H_{28}F_8O_6S_2$ having approximate dimensions of 0.120 x 0.100 x 0.010 mm was mounted in a loop. All measurements were made on a Rigaku Mercury70 diffractometer using filtered Mo-Kα radiation.

Cell constants and an orientation matrix for data collection corresponded to a primitive triclinic cell with dimensions:

a = 10.070(3) Å α = 94.940(7)°
b = 15.616(4) Å β = 91.272(6)°
c = 17.611(4) Å γ = 95.758(7)°
V = 2744.0(12) Å^3

For Z = 4 and F.W. = 616.58, the calculated density is 1.492 g/cm^3. Based on a statistical analysis of intensity distribution, and the successful solution and refinement of the structure, the space group was determined to be:

P-1 (#2)

The data were collected at a temperature of -100 ± 1°C to a maximum 2θ value of 50.7°. Readout was performed in the 0.136 mm pixel mode.

Data Reduction

Of the 17265 reflections were collected, where 9191 were unique (R_{int} = 0.0780); equivalent reflections were merged. Data were collected and processed using CrystalClear (Rigaku). [1]

The linear absorption coefficient, μ, for Mo-Kα radiation is 2.844 cm^{-1}. An empirical absorption correction was applied which resulted in transmission factors ranging from 0.429 to 0.997. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by direct methods[2] and expanded using Fourier techniques. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined using the riding model. The final cycle of full-matrix least-

squares refinement[3] on F^2 was based on 9191 observed reflections and 705 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R1 = \Sigma \, ||Fo| - |Fc|| \, / \, \Sigma \, |Fo| = 0.0963$$

$$wR2 = [\Sigma (w (Fo^2 - Fc^2)^2) / \Sigma \, w(Fo^2)^2]^{1/2} = 0.2772$$

The goodness of fit[4] was 1.05. Unit weights were used. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.95 and -0.42 $e^-/Å^3$, respectively.

Neutral atom scattering factors were taken from International Tables for Crystallography (IT), Vol. C, Table 6.1.1.4 [5]. Anomalous dispersion effects were included in Fcalc[6]; the values for Δf' and Δf'' were those of Creagh and McAuley[7]. The values for the mass attenuation coefficients are those of Creagh and Hubbell[8]. All calculations were performed using the CrystalStructure[9] crystallographic software package except for refinement, which was performed using SHELXL2013[10].

References

(1) CrystalClear: Data Collection and Processing Software, Rigaku Corporation (1998-2014). Tokyo 196-8666, Japan.

(2) SIR2004: Burla, M. C., Caliandro, R., Camalli, M., Carrozzini, B., Cascarano, G. L., De Caro, L., Giacovazzo, C., Polidori, G. and Spagna R. (2005). J. Appl. Cryst. 38, 381-388.

(3) Least Squares function minimized: (SHELXL2013)

$\Sigma w(F_O^2-F_C^2)^2$ where w = Least Squares weights.

(4) Goodness of fit is defined as:

$[\Sigma w(F_O^2-F_C^2)^2/(N_O-N_V)]^{1/2}$

where: N_O = number of observations
N_V = number of variables

(5) International Tables for Crystallography, Vol.C (1992). Ed. A.J.C. Wilson, Kluwer Academic Publishers, Dordrecht, Netherlands, Table 6.1.1.4, pp. 572.

(6) Ibers, J. A. & Hamilton, W. C.; Acta Crystallogr., 17, 781 (1964).

(7) Creagh, D. C. & McAuley, W.J .; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.6.8, pages 219-222 (1992).

(8) Creagh, D. C. & Hubbell, J.H..; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.4.3, pages 200-206 (1992).

(9) CrystalStructure 4.1: Crystal Structure Analysis Package, Rigaku Corporation (2000-2014). Tokyo 196-8666, Japan.

(10) SHELXL2013: Sheldrick, G. M. (2008). Acta Cryst. A64, 112-122.

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$C_{23}H_{28}F_8O_6S_2$
Formula Weight	616.58
Crystal Color, Habit	colorless, platelet
Crystal Dimensions	0.120 X 0.100 X 0.010 mm
Crystal System	triclinic
Lattice Type	Primitive
Lattice Parameters	a = 10.070(3) Å b = 15.616(4) Å c = 17.611(4) Å α = 94.940(7) ^{o} β = 91.272(6) ^{o} γ = 95.758(7) ^{o} V = 2744.0(12) $Å^3$
Space Group	P-1 (#2)
Z value	4
D_{calc}	1.492 g/cm^3
F_{000}	1272.00
μ(MoKα)	2.844 cm^{-1}

B. Intensity Measurements

Diffractometer	Mercury70
Radiation	MoKα (λ = 0.71075 Å)
Voltage, Current	50kV, 16mA
Temperature	-100.0°C
Detector Aperture	70.0 x 70.0 mm
Pixel Size	0.136 mm
$2\theta_{max}$	50.0°
No. of Reflections Measured	Total: 17265 Unique: 9191 (R_{int} = 0.0780)
Corrections	Lorentz-polarization Absorption (trans. factors: 0.429 - 0.997)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SIR2004)
Refinement	Full-matrix least-squares on F^2
Function Minimized	Σ w $(Fo^2 - Fc^2)^2$
Least Squares Weights	w = 1/ [$\sigma^2(Fo^2)$ + $(0.1222 \cdot P)^2$ + 0.0000 · P] where P = (Max(Fo^2,0) + $2Fc^2$)/3
$2\theta_{max}$ cutoff	50.0^o
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (All reflections)	9191
No. Variables	705
Reflection/Parameter Ratio	13.04
Residuals: R1 (I>2.00σ(I))	0.0963
Residuals: R (All reflections)	0.2105
Residuals: wR2 (All reflections)	0.2772
Goodness of Fit Indicator	1.047
Max Shift/Error in Final Cycle	0.000
Maximum peak in Final Diff. Map	0.95 $e^-/Å^3$
Minimum peak in Final Diff. Map	-0.42 $e^-/Å^3$

Table 1. Atomic coordinates and B_{iso}/B_{eq}

atom	x	y	z	B_{eq}
S2	0.29877(18)	1.03566(13)	0.38005(11)	3.02(4)
S6	0.6379(2)	0.63293(17)	0.40692(15)	5.27(6)
S32	1.4594(2)	0.79914(15)	0.09867(16)	4.71(6)
S36	0.86260(18)	1.02486(13)	0.14261(11)	3.16(4)
F3	0.5997(4)	1.0534(3)	0.3423(3)	4.59(10)
F5	0.8317(4)	0.8076(3)	0.3691(3)	5.22(11)
F21	0.1429(5)	0.9307(3)	0.4536(3)	5.66(12)
F22	0.0698(4)	1.0509(3)	0.4369(3)	5.59(12)
F23	0.0673(4)	0.9538(4)	0.3432(3)	6.95(15)
F24	0.3921(6)	0.5839(4)	0.4426(4)	7.98(16)
F25	0.5140(6)	0.4823(4)	0.4182(4)	8.35(17)
F26	0.4404(7)	0.5467(5)	0.3274(4)	9.6(2)
F33	1.5257(4)	0.9992(3)	0.0861(3)	5.44(12)
F35	1.1576(5)	1.1668(3)	0.1011(3)	5.24(11)
F51	1.2432(6)	0.6943(4)	0.0728(4)	7.66(16)
F52	1.4240(5)	0.6341(3)	0.0737(3)	6.40(13)
F53	1.3415(7)	0.6845(3)	0.1783(4)	8.28(17)
F54	0.8880(6)	1.1908(4)	0.1359(4)	8.88(18)
F55	0.6951(6)	1.1375(4)	0.1626(4)	9.4(2)
F56	0.8475(7)	1.1524(4)	0.2442(4)	9.7(2)
O2	0.3551(4)	0.9515(3)	0.3431(2)	2.53(9)
O6	0.5880(5)	0.7012(4)	0.3554(3)	3.80(11)
O21	0.3672(5)	1.0669(3)	0.4503(3)	3.65(11)
O22	0.2776(5)	1.0902(3)	0.3221(3)	4.40(12)
O23	0.6569(6)	0.6672(4)	0.4834(4)	6.39(15)
O24	0.7374(6)	0.5894(4)	0.3682(4)	6.75(17)
O32	1.3707(5)	0.8626(4)	0.1451(3)	4.59(12)
O36	1.0066(5)	1.0338(3)	0.1728(3)	3.90(11)
O51	1.4607(7)	0.8122(5)	0.0223(4)	8.0(2)
O52	1.5738(6)	0.7972(5)	0.1452(5)	9.4(2)
O53	0.8523(5)	1.0155(3)	0.0625(3)	4.11(11)
O54	0.7844(6)	0.9699(4)	0.1869(3)	5.40(14)
C1	0.4656(6)	0.8241(5)	0.3593(4)	2.85(15)
C2	0.4800(6)	0.9228(5)	0.3731(4)	2.83(15)
C3	0.5939(7)	0.9638(5)	0.3296(4)	3.30(16)
C4	0.7221(7)	0.9331(6)	0.3552(4)	3.93(19)
C5	0.7144(7)	0.8373(6)	0.3407(5)	3.77(18)

Table 1. Atomic coordinates and B_{iso}/B_{eq} (continued)

atom	x	y	z	B_{eq}
C6	0.5985(7)	0.7934(5)	0.3805(4)	3.20(16)
C7	0.3519(6)	0.7844(4)	0.4044(4)	2.32(13)
C8	0.3553(7)	0.7946(5)	0.4839(4)	3.43(16)
C9	0.2565(7)	0.7563(5)	0.5250(4)	3.16(15)
C10	0.1453(7)	0.7087(5)	0.4900(4)	2.71(14)
C11	0.1395(7)	0.7006(5)	0.4121(4)	2.88(15)
C12	0.2415(6)	0.7372(4)	0.3679(4)	2.59(14)
C13	0.0367(7)	0.6650(5)	0.5365(4)	3.29(15)
C14	-0.0211(8)	0.7297(5)	0.5940(5)	4.18(18)
C15	-0.1176(7)	0.6839(5)	0.6487(4)	4.08(18)
C16	-0.0542(7)	0.6151(5)	0.6882(4)	3.72(17)
C17	-0.0044(8)	0.5501(5)	0.6309(4)	4.14(17)
C18	0.0937(8)	0.5931(5)	0.5771(4)	3.84(17)
C19	-0.1446(8)	0.5701(6)	0.7455(4)	5.3(2)
C20	-0.1747(9)	0.6287(6)	0.8164(5)	6.0(2)
C21	-0.2516(9)	0.5788(7)	0.8735(5)	6.8(3)
C22	0.1332(8)	0.9876(6)	0.4043(6)	4.30(19)
C23	0.4863(11)	0.5578(7)	0.3968(7)	6.1(2)
C31	1.1820(7)	0.9420(5)	0.1487(4)	3.43(16)
C32	1.3140(6)	0.9323(5)	0.1086(4)	2.99(15)
C33	1.4077(7)	1.0132(6)	0.1194(5)	4.28(18)
C34	1.3472(7)	1.0873(5)	0.0927(4)	3.69(17)
C35	1.2162(7)	1.1010(5)	0.1300(5)	3.56(16)
C36	1.1226(7)	1.0209(5)	0.1231(4)	2.60(14)
C37	1.0852(7)	0.8601(5)	0.1381(5)	3.73(17)
C38	1.0240(8)	0.8299(5)	0.0677(5)	4.13(18)
C39	0.9384(8)	0.7539(5)	0.0604(5)	3.80(17)
C40	0.9131(7)	0.7049(5)	0.1206(4)	3.29(16)
C41	0.9781(8)	0.7367(5)	0.1915(5)	3.81(17)
C42	1.0622(7)	0.8109(5)	0.1974(5)	3.51(16)
C43	0.8268(8)	0.6188(5)	0.1114(5)	4.00(18)
C44	0.9139(9)	0.5453(5)	0.1078(6)	5.7(2)
C45	0.8285(10)	0.4574(6)	0.1038(7)	8.0(3)
C46	0.7326(9)	0.4494(5)	0.1651(7)	7.0(3)
C47	0.6443(8)	0.5215(5)	0.1672(6)	5.4(2)
C48	0.7273(7)	0.6090(5)	0.1761(5)	4.37(19)
C49	0.6462(13)	0.3621(7)	0.1676(7)	10.7(4)

Table 1. Atomic coordinates and B_{iso}/B_{eq} (continued)

atom	x	y	z	B_{eq}
C50	0.5896(14)	0.3507(9)	0.2476(7)	10.9(4)
C51	0.5090(18)	0.2680(10)	0.2626(11)	16.6(5)
C52	1.3602(10)	0.6978(6)	0.1055(6)	5.1(2)
C53	0.8224(10)	1.1322(6)	0.1722(7)	5.4(2)

$B_{eq} = 8/3\ \pi^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$

Table 2. Atomic coordinates and B_{iso} involving hydrogen atoms

atom	x	y	z	B_{iso}
H1	0.44580	0.80763	0.30374	3.414
H2	0.49238	0.94107	0.42886	3.400
H3	0.57812	0.94652	0.27386	3.961
H4A	0.79696	0.96011	0.32709	4.715
H4B	0.73873	0.95016	0.41027	4.715
H5	0.70446	0.82064	0.28461	4.527
H6	0.61449	0.80231	0.43700	3.841
H8	0.42819	0.82899	0.51000	4.116
H9	0.26428	0.76249	0.57908	3.798
H11	0.06340	0.66899	0.38665	3.458
H12	0.23464	0.72961	0.31384	3.109
H13	-0.03711	0.63808	0.50063	3.947
H14A	-0.06957	0.76994	0.56603	5.019
H14B	0.05294	0.76406	0.62420	5.019
H15A	-0.14581	0.72744	0.68765	4.895
H15B	-0.19841	0.65702	0.61941	4.895
H16	0.02577	0.64469	0.71791	4.460
H17A	-0.08111	0.51875	0.60075	4.974
H17B	0.04042	0.50737	0.65797	4.974
H18A	0.11941	0.54853	0.53824	4.608
H18B	0.17557	0.61761	0.60662	4.608
H19A	-0.23012	0.54698	0.71908	6.415
H19B	-0.10130	0.52048	0.76233	6.415
H20A	-0.08968	0.65650	0.84054	7.209
H20B	-0.22711	0.67493	0.80063	7.209
H21A	-0.19351	0.54076	0.89658	8.207
H21B	-0.32861	0.54399	0.84779	8.207
H21C	-0.28254	0.61920	0.91334	8.207
H31	1.20372	0.95360	0.20457	4.117
H32	1.29636	0.91505	0.05296	3.593
H33	1.42688	1.02590	0.17545	5.139
H34A	1.33192	1.07802	0.03677	4.424
H34B	1.41014	1.14007	0.10379	4.424
H35	1.23581	1.11759	0.18550	4.276
H36	1.08955	1.00848	0.06887	3.118
H38	1.04086	0.86143	0.02455	4.950
H39	0.89615	0.73523	0.01219	4.565

Table 2. Atomic coordinates and B_{iso} involving hydrogens/B_{eq} (continued)

atom	x	y	z	B_{eq}
H41	0.96269	0.70576	0.23513	4.576
H42	1.10668	0.82921	0.24506	4.209
H43	0.77463	0.61537	0.06204	4.803
H44A	0.97419	0.55145	0.15356	6.829
H44B	0.96975	0.54775	0.06233	6.829
H45A	0.77886	0.44806	0.05411	9.619
H45B	0.88866	0.41119	0.10571	9.619
H46	0.78688	0.45838	0.21390	8.418
H47A	0.58344	0.51690	0.21040	6.490
H47B	0.58911	0.51653	0.11951	6.490
H48A	0.77702	0.61559	0.22572	5.247
H48B	0.66708	0.65535	0.17608	5.247
H49A	0.70101	0.31438	0.15331	12.894
H49B	0.57125	0.35862	0.12979	12.894
H50A	0.66613	0.35951	0.28469	13.061
H50B	0.53340	0.39829	0.25966	13.061
H51A	0.56922	0.22530	0.27495	19.918
H51B	0.45420	0.24588	0.21711	19.918
H51C	0.45095	0.27891	0.30557	19.918

Table 3. Anisotropic displacement parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
S2	0.0403(11)	0.0461(14)	0.0282(12)	0.0017(9)	0.0032(9)	0.0049(10)
S6	0.0707(16)	0.0781(19)	0.0573(18)	0.0275(14)	-0.0088(13)	0.0188(14)
S32	0.0469(13)	0.0588(17)	0.0770(19)	0.0149(11)	0.0076(13)	0.0147(13)
S36	0.0435(12)	0.0494(14)	0.0268(12)	0.0005(10)	0.0040(9)	0.0044(10)
F3	0.052(3)	0.062(4)	0.059(3)	-0.010(2)	0.012(2)	0.016(3)
F5	0.032(2)	0.108(4)	0.064(3)	0.020(2)	0.004(2)	0.026(3)
F21	0.067(3)	0.065(4)	0.088(4)	0.005(3)	0.040(3)	0.025(3)
F22	0.064(3)	0.069(4)	0.083(4)	0.019(3)	0.034(3)	0.003(3)
F23	0.035(3)	0.137(5)	0.080(4)	-0.008(3)	-0.006(3)	-0.038(4)
F24	0.090(4)	0.081(4)	0.139(6)	0.014(3)	0.017(4)	0.036(4)
F25	0.145(6)	0.062(4)	0.118(5)	0.033(4)	0.026(4)	0.017(4)
F26	0.147(6)	0.120(6)	0.088(5)	-0.037(5)	-0.036(4)	0.020(4)
F33	0.036(3)	0.072(4)	0.094(4)	-0.007(2)	0.003(3)	-0.000(3)
F35	0.075(3)	0.048(3)	0.080(4)	0.015(3)	0.008(3)	0.015(3)
F51	0.065(4)	0.081(4)	0.138(6)	0.010(3)	-0.014(4)	-0.028(4)
F52	0.082(4)	0.061(4)	0.099(4)	0.017(3)	0.007(3)	-0.009(3)
F53	0.179(6)	0.061(4)	0.077(5)	0.014(4)	0.031(4)	0.017(3)
F54	0.092(4)	0.053(4)	0.196(7)	0.020(3)	0.042(5)	0.008(4)
F55	0.062(4)	0.078(5)	0.208(7)	0.020(3)	-0.002(4)	-0.058(4)
F56	0.156(6)	0.110(6)	0.097(5)	0.050(5)	-0.019(4)	-0.053(4)
O2	0.031(3)	0.045(3)	0.021(3)	0.009(2)	0.002(2)	0.002(2)
O6	0.051(3)	0.060(4)	0.039(3)	0.023(3)	-0.002(3)	0.017(3)

O21	0.047(3)	0.058(4)	0.030(3)	0.002(3)	0.000(3)	-0.007(3)
O22	0.076(4)	0.057(4)	0.040(3)	0.020(3)	0.006(3)	0.022(3)
O23	0.091(5)	0.105(6)	0.053(4)	0.030(4)	-0.023(3)	0.023(4)
O24	0.079(4)	0.099(5)	0.090(5)	0.060(4)	0.009(4)	0.013(4)
O32	0.060(3)	0.059(4)	0.058(4)	0.019(3)	-0.011(3)	0.010(3)
O36	0.058(3)	0.054(4)	0.037(3)	0.012(3)	0.000(3)	0.002(3)
O51	0.130(6)	0.097(6)	0.082(6)	0.025(5)	0.046(5)	0.014(4)
O52	0.071(5)	0.076(6)	0.207(9)	0.020(4)	-0.058(5)	0.003(5)
O53	0.057(3)	0.061(4)	0.038(4)	0.009(3)	0.001(3)	0.000(3)
O54	0.087(4)	0.068(4)	0.049(4)	-0.007(3)	0.017(3)	0.014(3)
C1	0.033(4)	0.049(6)	0.025(4)	-0.002(4)	0.001(3)	0.003(4)
C2	0.021(4)	0.060(6)	0.027(4)	0.005(4)	-0.001(3)	0.003(4)
C3	0.036(4)	0.062(6)	0.027(5)	-0.001(4)	0.005(4)	0.008(4)
C4	0.027(4)	0.101(8)	0.019(4)	-0.004(4)	0.000(3)	0.007(4)
C5	0.026(4)	0.082(7)	0.039(5)	0.015(4)	0.002(4)	0.014(5)

Table 3. Anisotropic displacement parameters (continued)

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C6	0.038(4)	0.062(6)	0.023(4)	0.004(4)	-0.002(3)	0.010(4)
C7	0.036(4)	0.031(5)	0.022(4)	0.004(3)	0.002(3)	0.005(3)
C8	0.039(4)	0.049(6)	0.037(5)	-0.015(4)	-0.006(4)	-0.006(4)
C9	0.040(4)	0.048(5)	0.030(5)	-0.009(4)	0.010(4)	0.005(4)
C10	0.035(4)	0.038(5)	0.030(5)	0.002(4)	0.001(4)	0.006(4)
C11	0.037(4)	0.047(5)	0.023(5)	-0.001(4)	-0.011(3)	-0.000(4)
C12	0.033(4)	0.036(5)	0.030(4)	0.003(3)	-0.007(3)	0.006(4)
C13	0.039(4)	0.046(5)	0.040(5)	-0.003(4)	-0.002(4)	0.009(4)
C14	0.058(5)	0.044(6)	0.061(6)	0.011(4)	0.020(5)	0.017(4)
C15	0.047(5)	0.064(6)	0.045(5)	0.009(4)	0.017(4)	0.002(4)
C16	0.043(4)	0.053(6)	0.043(5)	-0.007(4)	-0.011(4)	0.007(4)
C17	0.068(5)	0.045(6)	0.043(5)	-0.006(4)	-0.009(4)	0.010(4)
C18	0.056(5)	0.044(5)	0.045(5)	0.002(4)	0.013(4)	0.004(4)
C19	0.064(6)	0.089(8)	0.042(6)	-0.032(5)	0.006(5)	0.009(5)
C20	0.069(6)	0.096(8)	0.055(6)	-0.030(6)	0.007(5)	0.000(6)
C21	0.088(7)	0.129(10)	0.040(6)	-0.024(7)	0.014(5)	0.026(6)
C22	0.041(5)	0.068(7)	0.053(6)	0.011(5)	0.013(5)	-0.011(6)
C23	0.088(8)	0.061(8)	0.086(9)	0.015(6)	0.013(7)	0.019(7)
C31	0.060(5)	0.036(5)	0.035(5)	0.009(4)	-0.008(4)	0.007(4)
C32	0.036(4)	0.048(5)	0.030(5)	0.004(4)	-0.013(3)	0.010(4)
C33	0.034(5)	0.063(6)	0.065(6)	-0.004(4)	-0.012(4)	0.020(5)
C34	0.039(4)	0.052(6)	0.046(5)	-0.007(4)	0.000(4)	0.001(4)

C35	0.059(5)	0.034(5)	0.045(5)	0.003(4)	-0.017(4)	0.019(4)
C36	0.043(4)	0.043(5)	0.014(4)	0.008(4)	0.014(3)	0.005(3)
C37	0.045(5)	0.039(5)	0.055(6)	0.000(4)	-0.015(4)	-0.004(5)
C38	0.057(5)	0.048(6)	0.054(6)	0.006(5)	0.005(5)	0.021(5)
C39	0.059(5)	0.050(6)	0.036(5)	-0.004(4)	-0.012(4)	0.015(4)
C40	0.040(4)	0.050(6)	0.037(5)	0.008(4)	-0.001(4)	0.008(4)
C41	0.064(5)	0.042(6)	0.038(5)	-0.000(4)	0.001(4)	0.008(4)
C42	0.046(5)	0.044(6)	0.042(5)	0.000(4)	-0.012(4)	0.011(4)
C43	0.062(5)	0.040(6)	0.046(5)	-0.013(4)	-0.008(4)	0.006(4)
C44	0.066(6)	0.046(6)	0.101(8)	-0.002(5)	0.030(5)	-0.015(5)
C45	0.068(6)	0.040(7)	0.189(13)	-0.009(5)	0.029(8)	-0.022(7)
C46	0.051(5)	0.030(6)	0.190(12)	-0.002(4)	0.011(7)	0.040(7)
C47	0.050(5)	0.041(6)	0.117(8)	-0.001(4)	0.003(5)	0.028(5)
C48	0.048(5)	0.044(6)	0.080(7)	0.016(4)	0.019(5)	0.024(5)
C49	0.136(5)	0.135(5)	0.137(5)	0.0145(11)	0.0039(11)	0.0123(11)

Table 3. Anisotropic displacement parameters (continued)

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C50	0.138(5)	0.137(5)	0.138(5)	0.0133(11)	0.0050(11)	0.0127(11)
C51	0.210(7)	0.210(7)	0.210(7)	0.0216(9)	0.0066(6)	0.0187(8)
C52	0.067(7)	0.058(7)	0.071(8)	0.020(5)	0.003(6)	-0.003(6)
C53	0.064(7)	0.047(7)	0.093(9)	0.004(5)	0.017(6)	-0.011(6)

The general temperature factor expression: $\exp(-2\pi^2(a^{*2}U_{11}h^2 + b^{*2}U_{22}k^2 + c^{*2}U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$

Table 4. Fragment Analysis

fragment: 1

S(2)	S(6)	F(3)	F(5)	F(21)
F(22)	F(23)	F(24)	F(25)	F(26)
O(2)	O(6)	O(21)	O(22)	O(23)
O(24)	C(1)	C(2)	C(3)	C(4)
C(5)	C(6)	C(7)	C(8)	C(9)
C(10)	C(11)	C(12)	C(13)	C(14)
C(15)	C(16)	C(17)	C(18)	C(19)
C(20)	C(21)	C(22)	C(23)	

fragment: 2

S(32)	S(36)	F(33)	F(35)	F(51)
F(52)	F(53)	F(54)	F(55)	F(56)
O(32)	O(36)	O(51)	O(52)	O(53)
O(54)	C(31)	C(32)	C(33)	C(34)
C(35)	C(36)	C(37)	C(38)	C(39)
C(40)	C(41)	C(42)	C(43)	C(44)
C(45)	C(46)	C(47)	C(48)	C(49)
C(50)	C(51)	C(52)	C(53)	

Table 5. Bond lengths (Å)

atom	atom	distance	atom	atom	distance
S2	O2	1.579(5)	S2	O21	1.427(5)
S2	O22	1.411(6)	S2	C22	1.834(8)
S6	O6	1.570(6)	S6	O23	1.407(7)
S6	O24	1.420(7)	S6	C23	1.825(11)
S32	O32	1.586(6)	S32	O51	1.378(8)
S32	O52	1.403(8)	S32	C52	1.800(9)
S36	O36	1.522(5)	S36	O53	1.406(5)
S36	O54	1.405(6)	S36	C53	1.799(10)
F3	C3	1.393(10)	F5	C5	1.408(9)
F21	C22	1.303(12)	F22	C22	1.327(11)
F23	C22	1.295(10)	F24	C23	1.331(13)
F25	C23	1.322(13)	F26	C23	1.287(14)
F33	C33	1.366(9)	F35	C35	1.364(9)
F51	C52	1.294(11)	F52	C52	1.328(11)
F53	C52	1.329(12)	F54	C53	1.296(12)
F55	C53	1.301(12)	F56	C53	1.294(13)
O2	C2	1.478(8)	O6	C6	1.462(10)
O32	C32	1.467(10)	O36	C36	1.495(8)
C1	C2	1.531(10)	C1	C6	1.515(10)
C1	C7	1.521(9)	C2	C3	1.513(10)
C3	C4	1.495(11)	C4	C5	1.490(13)
C5	C6	1.510(10)	C7	C8	1.394(10)

C7	C12	1.384(9)	C8	C9
		1.363(10)		
C9	C10	1.381(9)	C10	C11
		1.367(10)		
C10	C13	1.522(10)	C11	C12
		1.407(10)		
C13	C14	1.533(11)	C13	C18
		1.533(11)		
C14	C15	1.551(11)	C15	C16
		1.514(12)		
C16	C17	1.500(11)	C16	C19
		1.541(11)		
C17	C18	1.531(11)	C19	C20
		1.537(12)		
C20	C21	1.506(13)	C31	C32
		1.534(10)		
C31	C36	1.521(11)	C31	C37
		1.523(10)		
C32	C33	1.496(10)	C33	C34
		1.468(12)		
C34	C35	1.513(11)	C35	C36
		1.484(10)		
C37	C38	1.395(12)	C37	C42
		1.360(12)		
C38	C39	1.389(11)	C39	C40
		1.374(12)		
C40	C41	1.423(11)	C40	C43
		1.521(10)		
C41	C42	1.359(11)	C43	C44
		1.511(12)		

Table 5. Bond lengths (Å) (continued)

atom	atom	distance	atom	atom	distance
C43	C48	1.542(11)	C44	C45	1.540(12)
C45	C46	1.470(16)	C46	C47	1.502(12)
C46	C49	1.546(14)	C47	C48	1.521(11)
C49	C50	1.548(18)	C50	C51	1.50(2)

Table 6. Bond lengths involving hydrogens (Å)

atom	atom	distance	atom	atom	distance
C1	H1	1.000	C2	H2	1.000
C3	H3	1.000	C4	H4A	0.990
C4	H4B	0.990	C5	H5	1.000
C6	H6	1.000	C8	H8	0.950
C9	H9	0.950	C11	H11	0.950
C12	H12	0.950	C13	H13	1.000
C14	H14A	0.990	C14	H14B	0.990
C15	H15A	0.990	C15	H15B	0.990
C16	H16	1.000	C17	H17A	0.990
C17	H17B	0.990	C18	H18A	0.990
C18	H18B	0.990	C19	H19A	0.990
C19	H19B	0.990	C20	H20A	0.990
C20	H20B	0.990	C21	H21A	0.980
C21	H21B	0.980	C21	H21C	0.980
C31	H31	1.000	C32	H32	1.000
C33	H33	1.000	C34	H34A	0.990
C34	H34B	0.990	C35	H35	1.000
C36	H36	1.000	C38	H38	0.950
C39	H39	0.950	C41	H41	0.950
C42	H42	0.950	C43	H43	1.000
C44	H44A	0.990	C44	H44B	0.990
C45	H45A	0.990	C45	H45B	0.990

C46	H46	1.000	C47	H47A
	0.990			
C47	H47B	0.990	C48	H48A
	0.990			
C48	H48B	0.990	C49	H49A
	0.990			
C49	H49B	0.990	C50	H50A
	0.990			
C50	H50B	0.990	C51	H51A
	0.980			
C51	H51B	0.980	C51	H51C
	0.980			

Table 7. Bond angles (°)

atom	atom	atom	angle	atom	atom	atom	angle
O2	S2	O21	111.7(3)	O2	S2	O22	108.6(3)
O2	S2	C22	98.7(4)	O21	S2	O22	122.1(3)
O21	S2	C22	106.7(4)	O22	S2	C22	106.4(4)
O6	S6	O23	111.3(4)	O6	S6	O24	109.1(4)
O6	S6	C23	96.5(4)	O23	S6	O24	121.6(4)
O23	S6	C23	109.7(5)	O24	S6	C23	105.6(5)
O32	S32	O51	112.0(4)	O32	S32	O52	105.0(4)
O32	S32	C52	100.0(4)	O51	S32	O52	124.7(5)
O51	S32	C52	106.2(5)	O52	S32	C52	106.0(5)
O36	S36	O53	112.8(3)	O36	S36	O54	109.0(3)
O36	S36	C53	98.6(4)	O53	S36	O54	121.5(3)
O53	S36	C53	106.7(5)	O54	S36	C53	105.5(4)
S2	O2	C2	120.8(4)	S6	O6	C6	122.2(4)
S32	O32	C32	120.8(5)	S36	O36	C36	123.2(4)
C2	C1	C6	107.6(5)	C2	C1	C7	110.9(5)
C6	C1	C7	112.2(6)	O2	C2	C1	106.3(5)
O2	C2	C3	107.5(6)	C1	C2	C3	111.9(6)
F3	C3	C2	109.4(6)	F3	C3	C4	110.4(6)
C2	C3	C4	109.5(7)	C3	C4	C5	109.6(6)
F5	C5	C4	109.8(6)	F5	C5	C6	107.3(7)
C4	C5	C6	111.5(6)	O6	C6	C1	106.1(5)
O6	C6	C5	107.8(6)	C1	C6	C5	113.1(7)

C1	C7	C8	121.1(6)	C1	C7	C12
	121.0(6)					
C8	C7	C12	117.9(6)	C7	C8	C9
	121.5(6)					
C8	C9	C10	121.7(7)	C9	C10	C11
	117.0(6)					
C9	C10	C13	121.2(6)	C11	C10	C13
	121.7(6)					
C10	C11	C12	122.7(6)	C7	C12	C11
	119.1(6)					
C10	C13	C14	111.9(6)	C10	C13	C18
	109.2(6)					
C14	C13	C18	110.9(6)	C13	C14	C15
	112.0(6)					
C14	C15	C16	112.3(6)	C15	C16	C17
	110.7(6)					
C15	C16	C19	114.2(6)	C17	C16	C19
	110.9(7)					
C16	C17	C18	111.6(6)	C13	C18	C17
	113.3(6)					
C16	C19	C20	114.5(7)	C19	C20	C21
	111.7(8)					
S2	C22	F21	111.0(6)	S2	C22	F22
	107.0(6)					
S2	C22	F23	110.2(6)	F21	C22	F22
	107.8(7)					
F21	C22	F23	111.3(8)	F22	C22	F23
	109.4(7)					
S6	C23	F24	111.4(7)	S6	C23	F25
	108.7(8)					

Table 7. Bond angles (°) (continued)

atom	atom	atom	angle	atom	atom	atom	angle
S6	C23	F26	111.9(8)	F24	C23	F25	106.3(9)
F24	C23	F26	109.7(9)	F25	C23	F26	108.6(9)
C32	C31	C36	109.1(6)	C32	C31	C37	112.7(6)
C36	C31	C37	113.1(6)	O32	C32	C31	104.3(6)
O32	C32	C33	110.0(6)	C31	C32	C33	112.1(6)
F33	C33	C32	109.4(6)	F33	C33	C34	113.5(7)
C32	C33	C34	111.7(6)	C33	C34	C35	112.5(7)
F35	C35	C34	111.6(6)	F35	C35	C36	110.2(6)
C34	C35	C36	111.8(6)	O36	C36	C31	105.9(5)
O36	C36	C35	109.1(5)	C31	C36	C35	114.3(6)
C31	C37	C38	122.4(8)	C31	C37	C42	119.9(7)
C38	C37	C42	117.7(7)	C37	C38	C39	120.4(8)
C38	C39	C40	122.0(7)	C39	C40	C41	116.4(7)
C39	C40	C43	122.0(7)	C41	C40	C43	121.5(7)
C40	C41	C42	120.7(8)	C37	C42	C41	122.8(7)
C40	C43	C44	110.1(6)	C40	C43	C48	112.3(6)
C44	C43	C48	109.8(7)	C43	C44	C45	111.0(7)
C44	C45	C46	114.2(8)	C45	C46	C47	110.5(9)
C45	C46	C49	117.9(9)	C47	C46	C49	109.7(8)
C46	C47	C48	110.8(7)	C43	C48	C47	111.7(7)
C46	C49	C50	112.1(10)	C49	C50	C51	120.1(11)
S32	C52	F51	113.1(7)	S32	C52	F52	109.2(6)

S32	C52	F53	110.2(6)	F51	C52	F52
	108.8(7)					
F51	C52	F53	107.0(8)	F52	C52	F53
	108.5(8)					
S36	C53	F54	112.7(8)	S36	C53	F55
	110.6(6)					
S36	C53	F56	111.9(8)	F54	C53	F55
	109.0(9)					
F54	C53	F56	107.2(8)	F55	C53	F56
	105.2(9)					

Table 8. Bond angles involving hydrogens (º)

atom	atom	atom	angle	atom	atom	atom	angle
C2	C1	H1	108.7	C6	C1	H1	108.7
C7	C1	H1	108.7	O2	C2	H2	110.4
C1	C2	H2	110.4	C3	C2	H2	110.4
F3	C3	H3	109.2	C2	C3	H3	109.2
C4	C3	H3	109.2	C3	C4	H4A	109.7
C3	C4	H4B	109.7	C5	C4	H4A	109.7
C5	C4	H4B	109.7	H4A	C4	H4B	108.2
F5	C5	H5	109.4	C4	C5	H5	109.4
C6	C5	H5	109.4	O6	C6	H6	109.9
C1	C6	H6	109.9	C5	C6	H6	109.9
C7	C8	H8	119.2	C9	C8	H8	119.2
C8	C9	H9	119.1	C10	C9	H9	119.1
C10	C11	H11	118.6	C12	C11	H11	118.6
C7	C12	H12	120.5	C11	C12	H12	120.5
C10	C13	H13	108.3	C14	C13	H13	108.3
C18	C13	H13	108.3	C13	C14	H14A	109.2
C13	C14	H14B	109.2	C15	C14	H14A	109.2
C15	C14	H14B	109.2	H14A	C14	H14B	107.9
C14	C15	H15A	109.1	C14	C15	H15B	109.1
C16	C15	H15A	109.1	C16	C15	H15B	109.1
H15A	C15	H15B	107.9	C15	C16	H16	106.9
C17	C16	H16	106.9	C19	C16	H16	106.9

C16	C17	H17A	109.3	C16	C17	H17B
109.3						
C18	C17	H17A	109.3	C18	C17	H17B
109.3						
H17A	C17	H17B	108.0	C13	C18	H18A
108.9						
C13	C18	H18B	108.9	C17	C18	H18A
108.9						
C17	C18	H18B	108.9	H18A	C18	H18B
107.7						
C16	C19	H19A	108.6	C16	C19	H19B
108.6						
C20	C19	H19A	108.6	C20	C19	H19B
108.6						
H19A	C19	H19B	107.6	C19	C20	H20A
109.3						
C19	C20	H20B	109.3	C21	C20	H20A
109.3						
C21	C20	H20B	109.3	H20A	C20	H20B
107.9						
C20	C21	H21A	109.5	C20	C21	H21B
109.5						
C20	C21	H21C	109.5	H21A	C21	H21B
109.5						
H21A	C21	H21C	109.5	H21B	C21	H21C
109.5						
C32	C31	H31	107.2	C36	C31	H31
107.2						
C37	C31	H31	107.2	O32	C32	H32
110.1						

Table 8. Bond angles involving hydrogens (°) (continued)

atom	atom	atom	angle	atom	atom	atom	angle
C31	C32	H32	110.1	C33	C32	H32	110.1
F33	C33	H33	107.3	C32	C33	H33	107.4
C34	C33	H33	107.3	C33	C34	H34A	109.1
C33	C34	H34B	109.1	C35	C34	H34A	109.1
C35	C34	H34B	109.1	H34A	C34	H34B	107.8
F35	C35	H35	107.7	C34	C35	H35	107.7
C36	C35	H35	107.7	O36	C36	H36	109.1
C31	C36	H36	109.1	C35	C36	H36	109.1
C37	C38	H38	119.8	C39	C38	H38	119.8
C38	C39	H39	119.0	C40	C39	H39	119.0
C40	C41	H41	119.7	C42	C41	H41	119.7
C37	C42	H42	118.6	C41	C42	H42	118.6
C40	C43	H43	108.2	C44	C43	H43	108.2
C48	C43	H43	108.2	C43	C44	H44A	109.4
C43	C44	H44B	109.4	C45	C44	H44A	109.4
C45	C44	H44B	109.4	H44A	C44	H44B	108.0
C44	C45	H45A	108.7	C44	C45	H45B	108.7
C46	C45	H45A	108.7	C46	C45	H45B	108.7
H45A	C45	H45B	107.6	C45	C46	H46	106.0
C47	C46	H46	106.0	C49	C46	H46	106.0
C46	C47	H47A	109.5	C46	C47	H47B	109.5
C48	C47	H47A	109.5	C48	C47	H47B	109.5

H47A	C47	H47B	108.1	C43	C48	H48A
	109.3					
C43	C48	H48B	109.3	C47	C48	H48A
	109.3					
C47	C48	H48B	109.3	H48A	C48	H48B
	107.9					
C46	C49	H49A	109.2	C46	C49	H49B
	109.2					
C50	C49	H49A	109.2	C50	C49	H49B
	109.2					
H49A	C49	H49B	107.9	C49	C50	H50A
	107.3					
C49	C50	H50B	107.3	C51	C50	H50A
	107.3					
C51	C50	H50B	107.3	H50A	C50	H50B
	106.9					
C50	C51	H51A	109.5	C50	C51	H51B
	109.5					
C50	C51	H51C	109.5	H51A	C51	H51B
	109.5					
H51A	C51	H51C	109.5	H51B	C51	H51C
	109.5					

Table 9. Torsion Angles(º)
(Those having bond angles > 160 or < 20 degrees are excluded.)

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
O21	S2	O2	C2	-10.9(4)	O22	S2	O2	C2	126.6(3)
O2	S2	C22	F21	62.7(6)	O2	S2	C22	F22	-179.9(5)
O2	S2	C22	F23	-61.0(6)	C22	S2	O2	C2	-122.8(4)
O21	S2	C22	F21	-53.2(7)	O21	S2	C22	F22	64.2(6)
O21	S2	C22	F23	-176.9(5)	O22	S2	C22	F21	175.1(5)
O22	S2	C22	F22	-67.5(6)	O22	S2	C22	F23	51.4(7)
O23	S6	O6	C6	16.3(5)	O24	S6	O6	C6	-120.6(4)
O6	S6	C23	F24	-80.0(8)	O6	S6	C23	F25	163.2(7)
O6	S6	C23	F26	43.3(8)	C23	S6	O6	C6	130.4(5)
O23	S6	C23	F24	35.5(9)	O23	S6	C23	F25	-81.3(8)
O23	S6	C23	F26	158.7(7)	O24	S6	C23	F24	168.1(7)
O24	S6	C23	F25	51.3(8)	O24	S6	C23	F26	-68.7(8)
O51	S32	O32	C32	-7.0(5)	O52	S32	O32	C32	131.1(5)
O32	S32	C52	F51	63.1(7)	O32	S32	C52	F52	-175.7(6)
O32	S32	C52	F53	-56.7(7)	C52	S32	O32	C32	-119.1(5)
O51	S32	C52	F51	-53.5(8)	O51	S32	C52	F52	67.7(7)
O51	S32	C52	F53	-173.2(6)	O52	S32	C52	F51	172.0(7)
O52	S32	C52	F52	-66.7(7)	O52	S32	C52	F53	52.3(7)
O53	S36	O36	C36	-8.5(5)	O54	S36	O36	C36	129.6(4)
O36	S36	C53	F54	67.3(7)	O36	S36	C53	F55	-170.5(7)
O36	S36	C53	F56	-53.6(7)	C53	S36	O36	C36	-120.7(5)

O53	S36	C53	F54	-49.7(8)	O53	S36	C53	F55	72.5(8)
O53	S36	C53	F56	-170.6(6)	O54	S36	C53	F54	179.8(6)
O54	S36	C53	F55	-58.0(8)	O54	S36	C53	F56	59.0(7)
S2	O2	C2	C1	149.1(3)	S2	O2	C2	C3	-91.0(5)
S6	O6	C6	C1	-135.5(4)	S6	O6	C6	C5	103.1(5)
S32	O32	C32	C31	148.1(4)	S32	O32	C32	C33	-91.5(5)
S36	O36	C36	C31	-115.1(5)	S36	O36	C36	C35	121.5(5)
C2	C1	C6	O6	-171.3(5)	C2	C1	C6	C5	-53.3(7)
C6	C1	C2	O2	173.2(5)	C6	C1	C2	C3	56.1(7)
C2	C1	C7	C8	-62.7(8)	C2	C1	C7	C12	117.1(6)
C7	C1	C2	O2	-63.8(6)	C7	C1	C2	C3	179.2(5)
C6	C1	C7	C8	57.6(8)	C6	C1	C7	C12	-122.5(6)
C7	C1	C6	O6	66.5(6)	C7	C1	C6	C5	-175.6(5)
O2	C2	C3	F3	61.8(6)	O2	C2	C3	C4	-177.1(4)
C1	C2	C3	F3	178.1(5)	C1	C2	C3	C4	-60.8(7)
F3	C3	C4	C5	-179.7(5)	C2	C3	C4	C5	59.8(7)

Table 9. Torsion angles (º) (continued)

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
C3	C4	C5	F5	-176.3(5)	C3	C4	C5	C6	-57.5(7)
F5	C5	C6	O6	-67.0(7)	F5	C5	C6	C1	176.1(5)
C4	C5	C6	O6	172.8(5)	C4	C5	C6	C1	55.8(7)
C1	C7	C8	C9	-177.3(6)	C1	C7	C12	C11	179.1(6)
C8	C7	C12	C11	-1.0(10)	C12	C7	C8	C9	2.9(10)
C7	C8	C9	C10	-2.9(11)	C8	C9	C10	C11	0.9(11)
C8	C9	C10	C13	179.0(6)	C9	C10	C11	C12	1.0(11)
C9	C10	C13	C14	56.6(9)	C9	C10	C13	C18	-66.5(8)
C11	C10	C13	C14	-125.4(7)	C11	C10	C13	C18	111.4(7)
C13	C10	C11	C12	-177.1(6)	C10	C11	C12	C7	-0.9(11)
C10	C13	C14	C15	-172.3(5)	C10	C13	C18	C17	175.0(5)
C14	C13	C18	C17	51.3(7)	C18	C13	C14	C15	-50.1(7)
C13	C14	C15	C16	53.6(8)	C14	C15	C16	C17	-56.2(7)
C14	C15	C16	C19	177.8(5)	C15	C16	C17	C18	56.3(7)
C15	C16	C19	C20	-67.2(8)	C17	C16	C19	C20	166.9(6)
C19	C16	C17	C18	-175.9(5)	C16	C17	C18	C13	-54.9(8)
C16	C19	C20	C21	-174.2(6)	C32	C31	C36	O36	-171.7(5)
C32	C31	C36	C35	-51.5(7)	C36	C31	C32	O32	172.4(5)
C36	C31	C32	C33	53.4(7)	C32	C31	C37	C38	-68.7(9)
C32	C31	C37	C42	107.4(7)	C37	C31	C32	O32	-61.1(7)
C37	C31	C32	C33	179.9(6)	C36	C31	C37	C38	55.6(9)
C36	C31	C37	C42	-128.3(7)	C37	C31	C36	O36	62.1(7)

C37	C31	C36	C35	-177.8(5)	O32	C32	C33	F33	61.2(8)
O32	C32	C33	C34	-172.4(5)	C31	C32	C33	F33	176.7(6)
C31	C32	C33	C34	-56.9(8)	F33	C33	C34	C35	179.5(5)
C32	C33	C34	C35	55.4(8)	C33	C34	C35	F35	-176.3(6)
C33	C34	C35	C36	-52.3(8)	F35	C35	C36	O36	-65.7(7)
F35	C35	C36	C31	176.0(5)	C34	C35	C36	O36	169.5(5)
C34	C35	C36	C31	51.2(8)	C31	C37	C38	C39	178.7(6)
C31	C37	C42	C41	-179.3(6)	C38	C37	C42	C41	-3.1(11)
C42	C37	C38	C39	2.6(11)	C37	C38	C39	C40	-1.5(12)
C38	C39	C40	C41	0.7(11)	C38	C39	C40	C43	-175.8(7)
C39	C40	C41	C42	-1.1(11)	C39	C40	C43	C44	101.4(8)
C39	C40	C43	C48	-135.9(7)	C41	C40	C43	C44	-75.0(8)
C41	C40	C43	C48	47.7(9)	C43	C40	C41	C42	175.4(6)
C40	C41	C42	C37	2.4(12)	C40	C43	C44	C45	176.3(6)
C40	C43	C48	C47	-178.5(6)	C44	C43	C48	C47	-55.6(8)

Table 9. Torsion angles (º) (continued)

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
C48	C43	C44	C45	52.1(8)	C43	C44	C45	C46	-53.9(11)
C44	C45	C46	C47	55.1(11)	C44	C45	C46	C49	-177.6(7)
C45	C46	C47	C48	-56.3(10)	C45	C46	C49	C50	159.4(9)
C47	C46	C49	C50	-73.0(12)	C49	C46	C47	C48	172.0(8)
C46	C47	C48	C43	57.6(10)	C46	C49	C50	C51	-176.8(9)

Table 10. Intramolecular contacts less than 3.60 Å

atom	atom	distance	atom	atom	distance
S2	F3	3.107(5)	S2	C3	3.391(7)
S6	F5	3.316(5)	S6	C5	3.510(10)
S32	F33	3.156(5)	S32	C33	3.425(9)
S36	C31	3.588(8)	S36	C37	3.576(8)
F3	O2	2.796(6)	F3	O21	3.062(6)
F3	O22	3.367(7)	F5	O6	2.816(6)
F5	O23	3.478(8)	F5	O24	3.442(8)
F21	O2	2.936(6)	F21	O21	2.949(7)
F21	C7	3.332(9)	F21	C8	3.230(9)
F21	C9	3.385(9)	F21	C10	3.581(9)
F21	C12	3.504(9)	F22	O21	2.983(6)
F22	O22	2.998(7)	F23	O2	2.902(6)
F23	O22	2.903(7)	F24	O6	3.081(8)
F24	O23	2.894(8)	F24	C7	3.320(9)
F24	C8	3.367(10)	F24	C9	3.372(9)
F24	C10	3.385(9)	F24	C11	3.335(9)
F24	C12	3.308(10)	F25	O23	3.200(8)
F25	O24	2.867(9)	F26	O6	2.704(8)
F26	O24	3.054(9)	F33	O32	2.802(7)
F33	O51	3.039(8)	F33	O52	3.481(9)
F35	F54	2.851(8)	F35	O36	2.850(7)
F51	O32	2.974(7)	F51	O51	2.922(9)

F51	C37	3.308(10)	F51	C38
	3.213(10)			
F51	C39	3.303(10)	F51	C40
	3.464(9)			
F51	C41	3.507(10)	F51	C42
	3.407(10)			
F52	O51	2.989(9)	F52	O52
	2.990(8)			
F53	O32	2.878(8)	F53	O52
	2.885(9)			
F54	O36	2.948(8)	F54	O53
	2.909(8)			
F55	O53	3.064(8)	F55	O54
	2.915(9)			
F56	O36	2.803(9)	F56	O54
	2.942(9)			
O2	C7	2.906(8)	O2	C12
	3.493(8)			
O6	C7	2.933(8)	O6	C12
	3.598(8)			
O21	C2	2.874(9)	O21	C3
	3.563(9)			
O23	C6	2.885(11)	O24	C6
	3.598(11)			
O32	C37	2.871(9)	O32	C42
	3.303(9)			
O36	C37	2.920(10)	O36	C38
	3.565(10)			
O51	C32	2.858(10)	O51	C33
	3.538(11)			

Table 10. Intramolecular contacts less than 3.60 Å (continued)

atom	atom	distance	atom	atom	distance
O53	C36	2.892(8)	O53	C38	3.528(10)
C1	C4	2.952(10)	C2	C5	2.872(10)
C2	C8	3.110(10)	C2	C12	3.568(9)
C3	C6	2.884(12)	C6	C8	3.084(10)
C7	C10	2.819(10)	C8	C11	2.719(9)
C9	C12	2.755(10)	C9	C14	3.077(11)
C9	C18	3.107(10)	C11	C18	3.496(11)
C13	C16	2.985(11)	C14	C17	2.954(12)
C15	C18	2.924(11)	C15	C20	3.194(12)
C31	C34	2.931(11)	C32	C35	2.903(11)
C32	C38	3.221(10)	C32	C42	3.492(10)
C33	C36	2.888(10)	C36	C38	3.114(10)
C37	C40	2.824(10)	C38	C41	2.746(12)
C39	C42	2.729(11)	C39	C44	3.419(12)
C41	C44	3.215(11)	C41	C48	3.050(10)
C43	C46	2.960(12)	C44	C47	2.932(12)
C45	C48	2.879(13)	C47	C50	3.132(16)

Table 11. Intramolecular contacts less than 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
S2	H2	2.728	S6	H6	2.687
S32	H32	2.722	S36	H36	2.677
F3	H2	2.592	F3	H4A	2.584
F3	H4B	2.589	F5	H4A	2.608
F5	H4B	2.555	F5	H6	2.513
F21	H2	3.546	F21	H8	3.581
F24	H18A	3.278	F33	H32	2.569
F33	H34A	2.582	F33	H34B	2.589
F35	H34A	2.567	F35	H34B	2.618
F35	H36	2.510	F51	H32	3.483
F51	H44B	3.391	F53	H42	3.580
O2	H1	2.556	O2	H3	2.584
O2	H12	3.544	O6	H1	2.514
O6	H5	2.537	O21	H2	2.447
O23	H6	2.402	O23	H8	3.595
O32	H31	2.500	O32	H33	2.564
O32	H42	3.245	O36	H31	2.528
O36	H35	2.532	O51	H32	2.456
O53	H36	2.404	O53	H38	3.247
C1	H3	2.708	C1	H4B	3.278
C1	H5	2.770	C1	H8	2.685
C1	H12	2.693	C2	H4A	3.318

C2	H4B	2.656	C2	H5
	3.246			
C2	H6	2.730	C2	H8
	2.957			
C3	H1	2.725	C3	H5
	2.664			
C3	H6	3.300	C4	H1
	3.300			
C4	H2	2.686	C4	H6
	2.748			
C5	H1	2.752	C5	H2
	3.238			
C5	H3	2.632	C6	H2
	2.716			
C6	H3	3.184	C6	H4A
	3.334			
C6	H4B	2.704	C6	H8
	2.932			
C7	H2	2.696	C7	H6
	2.676			
C7	H9	3.255	C7	H11
	3.255			
C8	H1	3.338	C8	H2
	2.808			
C8	H6	2.749	C8	H12
	3.247			
C9	H11	3.203	C9	H13
	3.323			
C9	H14A	3.401	C9	H14B
	2.728			

Table 11. Intramolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
C9	H18A	3.425	C9	H18B	2.767
C10	H8	3.245	C10	H12	3.284
C10	H14A	2.777	C10	H14B	2.665
C10	H18A	2.701	C10	H18B	2.626
C11	H9	3.204	C11	H13	2.566
C11	H18A	3.384	C12	H1	2.566
C12	H8	3.238	C13	H9	2.675
C13	H11	2.664	C13	H15A	3.398
C13	H15B	2.807	C13	H16	3.241
C13	H17A	2.805	C13	H17B	3.400
C14	H9	2.890	C14	H16	2.707
C14	H17A	3.302	C14	H18A	3.370
C14	H18B	2.789	C15	H13	2.804
C15	H17A	2.707	C15	H17B	3.332
C15	H18B	3.304	C15	H19A	2.725
C15	H19B	3.390	C15	H20A	3.450
C15	H20B	2.925	C16	H13	3.360
C16	H14A	3.385	C16	H14B	2.802
C16	H18A	3.349	C16	H18B	2.750
C16	H20A	2.744	C16	H20B	2.825
C17	H13	2.804	C17	H14B	3.347

C17	H15A	3.328	C17	H15B
		2.710		
C17	H19A	2.780	C17	H19B
		2.591		
C18	H9	3.003	C18	H14A
		3.371		
C18	H14B	2.800	C18	H15B
		3.281		
C18	H16	2.667	C19	H15A
		2.741		
C19	H15B	2.772	C19	H17A
		2.716		
C19	H17B	2.654	C19	H21A
		2.784		
C19	H21B	2.639	C19	H21C
		3.355		
C20	H15A	2.853	C20	H15B
		3.540		
C20	H16	2.697	C21	H19A
		2.740		
C21	H19B	2.656	C31	H33
		2.686		
C31	H34A	3.300	C31	H35
		2.759		
C31	H38	2.723	C31	H42
		2.619		
C32	H34A	2.690	C32	H34B
		3.301		
C32	H35	3.265	C32	H36
		2.762		
C32	H38	3.147	C32	H42
		3.597		

Table 11. Intramolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
C33	H31	2.707	C33	H35	2.709
C33	H36	3.298	C34	H31	3.249
C34	H32	2.715	C34	H36	2.765
C35	H31	2.741	C35	H32	3.281
C35	H33	2.666	C36	H32	2.765
C36	H33	3.172	C36	H34A	2.747
C36	H34B	3.322	C36	H38	2.944
C37	H32	2.739	C37	H36	2.707
C37	H39	3.266	C37	H41	3.234
C38	H31	3.335	C38	H32	2.953
C38	H36	2.799	C38	H42	3.216
C39	H41	3.240	C39	H43	2.588
C39	H44B	3.269	C40	H38	3.266
C40	H42	3.269	C40	H44A	2.642
C40	H44B	2.698	C40	H48A	2.725
C40	H48B	2.747	C41	H39	3.244
C41	H43	3.356	C41	H44A	2.909
C41	H44B	3.560	C41	H48A	2.743
C41	H48B	3.257	C42	H31	2.514
C42	H38	3.218	C43	H39	2.682
C43	H41	2.730	C43	H45A	2.759

C43	H45B	3.355	C43	H46
	3.210			
C43	H47A	3.386	C43	H47B
	2.755			
C44	H39	3.552	C44	H41
	3.207			
C44	H46	2.676	C44	H47B
	3.270			
C44	H48A	2.731	C44	H48B
	3.349			
C45	H43	2.735	C45	H47A
	3.293			
C45	H47B	2.678	C45	H48A
	3.221			
C45	H49A	2.685	C45	H49B
	2.945			
C46	H43	3.289	C46	H44A
	2.793			
C46	H44B	3.358	C46	H48A
	2.710			
C46	H48B	3.338	C46	H50A
	2.690			
C46	H50B	2.736	C47	H43
	2.737			
C47	H44A	3.325	C47	H45A
	2.676			
C47	H45B	3.294	C47	H49A
	3.331			
C47	H49B	2.601	C47	H50A
	3.424			
C47	H50B	2.791	C48	H41
	2.808			

Table 11. Intramolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
C48	H44A	2.753	C48	H44B	3.354
C48	H45A	3.253	C48	H46	2.621
C49	H45A	2.794	C49	H45B	2.761
C49	H47A	2.612	C49	H47B	2.734
C49	H51A	3.027	C49	H51B	2.727
C49	H51C	3.420	C50	H46	2.584
C50	H47A	2.737	C50	H47B	3.578
C51	H49A	2.845	C51	H49B	2.885
H1	H2	2.896	H1	H3	2.531
H1	H5	2.624	H1	H6	2.878
H1	H12	2.361	H2	H3	2.887
H2	H4A	3.586	H2	H4B	2.501
H2	H6	2.609	H2	H8	2.402
H3	H4A	2.358	H3	H4B	2.859
H3	H5	2.465	H4A	H5	2.335
H4B	H5	2.857	H4B	H6	2.598
H5	H6	2.880	H6	H8	2.349
H8	H9	2.290	H9	H14A	3.379
H9	H14B	2.290	H9	H18A	3.517
H9	H18B	2.440	H11	H12	2.344
H11	H13	2.334	H11	H18A	3.463

H13	H14A	2.327	H13	H14B
	2.872			
H13	H15B	2.691	H13	H17A
	2.686			
H13	H18A	2.331	H13	H18B
	2.870			
H14A	H15A	2.415	H14A	H15B
	2.357			
H14B	H15A	2.358	H14B	H15B
	2.885			
H14B	H16	2.592	H14B	H18B
	2.704			
H15A	H16	2.338	H15A	H19A
	2.963			
H15A	H20A	3.061	H15A	H20B
	2.350			
H15B	H16	2.848	H15B	H17A
	2.564			
H15B	H19A	2.563	H15B	H20B
	3.202			
H16	H17A	2.839	H16	H17B
	2.327			
H16	H18A	3.571	H16	H18B
	2.532			
H16	H19A	2.861	H16	H19B
	2.412			
H16	H20A	2.477	H16	H20B
	3.020			
H17A	H18A	2.346	H17A	H18B
	2.870			
H17A	H19A	2.627	H17A	H19B
	2.855			

Table 11. Intramolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H17B	H18A	2.387	H17B	H18B	2.342
H17B	H19A	3.052	H17B	H19B	2.359
H19A	H20A	2.872	H19A	H20B	2.355
H19A	H21A	3.150	H19A	H21B	2.497
H19A	H21C	3.576	H19B	H20A	2.421
H19B	H20B	2.872	H19B	H21A	2.565
H19B	H21B	2.805	H19B	H21C	3.585
H20A	H21A	2.304	H20A	H21B	2.845
H20A	H21C	2.402	H20B	H21A	2.844
H20B	H21B	2.414	H20B	H21C	2.293
H31	H32	2.882	H31	H33	2.496
H31	H35	2.600	H31	H36	2.863
H31	H42	2.264	H32	H33	2.855
H32	H34A	2.575	H32	H36	2.666
H32	H38	2.648	H33	H34A	2.817
H33	H34B	2.289	H33	H35	2.512
H34A	H35	2.852	H34A	H36	2.663
H34B	H35	2.313	H35	H36	2.837
H36	H38	2.360	H38	H39	2.322
H39	H43	2.375	H39	H44B	3.279
H41	H42	2.285	H41	H44A	2.711

H41	H48A	2.219	H41	H48B
	3.133			
H43	H44A	2.858	H43	H44B
	2.322			
H43	H45A	2.609	H43	H47B
	2.583			
H43	H48A	2.882	H43	H48B
	2.369			
H44A	H45A	2.875	H44A	H45B
	2.350			
H44A	H46	2.570	H44A	H48A
	2.618			
H44B	H45A	2.343	H44B	H45B
	2.399			
H44B	H46	3.579	H45A	H46
	2.804			
H45A	H47A	3.572	H45A	H47B
	2.533			
H45A	H49A	2.896	H45A	H49B
	2.821			
H45B	H46	2.285	H45B	H47B
	3.581			
H45B	H49A	2.505	H45B	H49B
	3.266			
H46	H47A	2.327	H46	H47B
	2.834			
H46	H48A	2.458	H46	H48B
	3.524			
H46	H49A	2.470	H46	H49B
	2.849			
H46	H50A	2.332	H46	H50B
	2.791			

Table 11. Intramolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H47A	H48A	2.356	H47A	H48B	2.373
H47A	H49A	3.560	H47A	H49B	2.733
H47A	H50A	3.060	H47A	H50B	2.132
H47B	H48A	2.869	H47B	H48B	2.358
H47B	H49A	3.549	H47B	H49B	2.477
H47B	H50B	3.233	H49A	H50A	2.404
H49A	H50B	2.870	H49A	H51A	2.925
H49A	H51B	2.895	H49B	H50A	2.869
H49B	H50B	2.367	H49B	H51A	3.432
H49B	H51B	2.643	H50A	H51A	2.212
H50A	H51B	2.808	H50A	H51C	2.446
H50B	H51A	2.796	H50B	H51B	2.479
H50B	H51C	2.190			

Table 12. Intermolecular contacts less than 3.60 Å

atom	atom	distance	atom	atom	distance
S36	F33[1]	3.489(5)	F3	F56	3.389(8)
F3	O54	3.571(7)	F5	F23[2]	3.193(7)
F21	F22[3]	2.932(7)	F22	F21[3]	2.932(7)
F22	F22[3]	3.122(7)	F22	C14[3]	3.594(10)
F22	C22[3]	3.557(10)	F23	F5[1]	3.193(7)
F23	O36[1]	3.414(8)	F23	C4[1]	3.472(8)
F23	C42[1]	3.250(10)	F24	F25[4]	2.916(9)
F25	F24[4]	2.916(9)	F25	F25[4]	2.913(9)
F25	O23[4]	3.382(9)	F25	C23[4]	3.371(14)
F26	C19[5]	3.491(10)	F26	C47	3.543(12)
F33	S36[2]	3.489(5)	F33	F33[6]	3.068(7)
F33	F55[2]	2.837(7)	F33	O53[2]	3.311(6)
F33	O54[2]	3.214(7)	F35	C20[7]	3.380(10)
F35	C38[8]	3.462(10)	F35	C39[8]	3.358(10)
F52	C47[2]	3.441(11)	F52	C48[2]	3.585(9)
F53	C12[2]	3.563(9)	F55	F33[1]	2.837(7)
F55	C33[1]	3.353(9)	F55	C49[9]	3.584(13)
F55	C51[9]	3.326(18)	F56	F3	3.389(8)
F56	C14[7]	3.578(10)	O21	C2[7]	3.423(8)
O21	C4[7]	3.560(9)	O21	C6[7]	3.526(8)
O21	C8[7]	3.470(8)	O22	C35[1]	3.447(10)
O23	F25[4]	3.382(9)	O24	C48	3.424(11)

O36	F23[2]	3.414(8)	O51	C34[6]
	3.228(11)			
O52	O54[2]	3.279(9)	O52	C48[2]
	3.532(11)			
O53	F33[1]	3.311(6)	O53	C34[8]
	3.528(9)			
O53	C36[8]	3.289(8)	O54	F3
	3.571(7)			
O54	F33[1]	3.214(7)	O54	O52[1]
	3.279(9)			
O54	C3	3.197(9)	O54	C4
	3.132(9)			
O54	C5	3.589(10)	C2	O21[7]
	3.423(8)			
C3	O54	3.197(9)	C4	F23[2]
	3.472(8)			
C4	O21[7]	3.560(9)	C4	O54
	3.132(9)			
C5	O54	3.589(10)	C6	O21[7]
	3.526(8)			
C8	O21[7]	3.470(8)	C12	F53[1]
	3.563(9)			
C14	F22[3]	3.594(10)	C14	F56[7]
	3.578(10)			
C19	F26[5]	3.491(10)	C20	F35[7]
	3.380(10)			
C22	F22[3]	3.557(10)	C23	F25[4]
	3.371(14)			
C33	F55[2]	3.353(9)	C34	O51[6]
	3.228(11)			

Table 12. Intermolecular contacts less than 3.60 Å (continued)

atom	atom	distance	atom	atom
	distance			
C34	O53[8]	3.528(9)	C35	O22[2]
	3.447(10)			
C36	O53[8]	3.289(8)	C38	F35[8]
	3.462(10)			
C39	F35[8]	3.358(10)	C42	F23[2]
	3.250(10)			
C47	F26	3.543(12)	C47	F52[1]
	3.441(11)			
C48	F52[1]	3.585(9)	C48	O24
	3.424(11)			
C48	O52[1]	3.532(11)	C49	F55[10]
	3.584(13)			
C51	F55[10]	3.326(18)		

Symmetry Operators:

(1) X-1,Y,Z
(2) X+1,Y,Z
(3) -X,-Y+2,-Z+1
(4) -X+1,-Y+1,-Z+1
(5) -X,-Y+1,-Z+1
(6) -X+3,-Y+2,-Z
(7) -X+1,-Y+2,-Z+1
(8) -X+2,-Y+2,-Z
(9) X,Y+1,Z
(10) X,Y-1,Z

Table 13. Intermolecular contacts less than 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
S2	H31[1]	3.324	S6	H48A	3.513
S32	H48B[2]	3.552	S36	H4A	3.541
F3	H8[3]	3.087	F3	H9[3]	3.241
F3	H33[1]	3.355	F3	H51A[4]	3.067
F5	H11[2]	3.365	F5	H41	3.123
F5	H42	3.573	F21	H4B[3]	3.048
F22	H4A[1]	3.436	F22	H4B[1]	3.544
F22	H4B[3]	3.279	F22	H14A[5]	2.802
F22	H14B[5]	3.496	F23	H4A[1]	2.743
F23	H4B[1]	3.535	F23	H31[1]	2.828
F23	H42[1]	2.557	F24	H17A[6]	3.411
F25	H50A	3.395	F25	H50B	3.000
F25	H51C	3.589	F26	H19A[6]	2.528
F26	H21B[6]	3.401	F26	H47A	2.577
F26	H50B	2.772	F33	H3[2]	3.517
F33	H32[7]	3.359	F33	H34A[7]	2.869
F35	H20A[3]	3.011	F35	H20B[3]	2.909
F35	H21C[3]	3.484	F35	H38[8]	2.922
F35	H39[8]	2.694	F51	H45A[9]	3.002
F51	H45B[9]	3.584	F52	H21A[10]	3.486
F52	H21B[10]	3.280	F52	H43[2]	3.581
F52	H45A[9]	3.108	F52	H47A[2]	3.592

F52	H47B[2]	2.756	F52	H48B[2]
	2.982			
F52	H49B[9]	3.597	F53	H1[2]
	2.906			
F53	H12[2]	2.708	F53	H21B[10]
	3.547			
F53	H48B[2]	3.356	F54	H16[3]
	3.506			
F54	H20A[3]	2.967	F54	H38[8]
	2.997			
F54	H45B[4]	3.527	F54	H49A[4]
	2.831			
F55	H33[1]	3.088	F55	H34B[1]
	3.034			
F55	H49A[4]	2.776	F55	H51A[4]
	2.719			
F55	H51B[4]	3.212	F56	H4A
	3.448			
F56	H9[3]	3.531	F56	H14B[3]
	2.682			
F56	H15A[3]	3.508	F56	H16[3]
	3.302			
F56	H49A[4]	3.532	F56	H51A[4]
	3.169			
O2	H31[1]	2.852	O2	H33[1]
	3.332			
O2	H42[1]	3.342	O6	H48A
	3.288			
O6	H48B	3.311	O21	H2[3]
	2.547			

Table 13. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
O21	H4B[3]	2.721	O21	H6[3]	2.711
O21	H8[3]	2.539	O22	H15A[5]	3.276
O22	H31[1]	2.871	O22	H33[1]	3.157
O22	H35[1]	2.513	O22	H51A[4]	3.595
O22	H51B[4]	3.541	O22	H51C[4]	3.309
O23	H13[2]	3.171	O23	H14A[2]	3.288
O23	H15B[2]	2.800	O24	H11[2]	3.390
O24	H13[2]	3.207	O24	H17B[10]	2.847
O24	H18A[10]	3.250	O24	H18B[10]	3.495
O24	H46	3.336	O24	H47A	3.214
O24	H48A	2.610	O32	H1[2]	3.093
O32	H3[2]	3.155	O51	H34A[7]	2.841
O51	H34B[7]	2.720	O51	H49B[9]	3.599
O52	H1[2]	3.099	O52	H3[2]	3.104
O52	H5[2]	2.738	O52	H48B[2]	2.586
O53	H32[8]	2.850	O53	H34A[8]	2.740
O53	H36[8]	2.407	O53	H38[8]	2.714
O54	H3	2.623	O54	H4A	2.487
O54	H5	3.068	C3	H33[1]	3.423
C11	H17A[6]	3.405	C11	H41[1]	3.565
C12	H41[1]	3.581	C12	H42[1]	3.064

C15	H51C^{6}	3.561	C16	H46^{10}
	3.506			
C17	H11^{6}	3.399	C17	H18A^{6}
	3.360			
C17	H46^{10}	3.484	C18	H17A^{6}
	3.445			
C18	H18A^{6}	3.421	C18	H50A^{10}
	3.375			
C19	H44A^{10}	3.287	C20	H45B^{10}
	3.302			
C21	H39^{11}	3.497	C21	H43^{11}
	3.320			
C21	H47B^{6}	3.581	C21	H49B^{6}
	3.458			
C22	H4A^{1}	3.592	C22	H4B^{3}
	3.505			
C22	H42^{1}	3.563	C23	H19A^{6}
	3.441			
C23	H47A	3.471	C23	H50B
	3.393			
C33	H3^{2}	3.474	C34	H51B^{12}
	3.243			
C35	H20B^{3}	3.595	C35	H51B^{12}
	3.372			
C41	H5	3.544	C41	H12^{2}
	3.343			
C42	H12^{2}	3.093	C44	H19B^{10}
	3.232			
C44	H21A^{10}	3.239	C44	H44B^{9}
	3.494			
C45	H19B^{10}	3.551	C45	H20A^{10}
	3.488			

Table 13. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
C45	H44B[9]	3.598	C47	H21B[6]	3.238
C50	H18B[10]	3.421	C51	H20B[6]	3.260
C51	H34B[13]	3.365	C51	H35[13]	3.594
C52	H48B[2]	3.452	C53	H49A[4]	3.247
C53	H51A[4]	3.520	H1	F53[1]	2.906
H1	O32[1]	3.093	H1	O52[1]	3.099
H2	O21[3]	2.547	H2	H2[3]	2.970
H3	F33[1]	3.517	H3	O32[1]	3.155
H3	O52[1]	3.104	H3	O54	2.623
H3	C33[1]	3.474	H3	H33[1]	2.737
H4A	S36	3.541	H4A	F22[2]	3.436
H4A	F23[2]	2.743	H4A	F56	3.448
H4A	O54	2.487	H4A	C22[2]	3.592
H4B	F21[3]	3.048	H4B	F22[2]	3.544
H4B	F22[3]	3.279	H4B	F23[2]	3.535
H4B	O21[3]	2.721	H4B	C22[3]	3.505
H5	O52[1]	2.738	H5	O54	3.068
H5	C41	3.544	H5	H41	3.392
H5	H48A	3.434	H5	H48B	3.063
H6	O21[3]	2.711	H8	F3[3]	3.087
H8	O21[3]	2.539	H9	F3[3]	3.241

H9	F56[3]	3.531	H9	H50A[10]
	3.295			
H9	H51A[10]	3.019	H11	F5[1]
	3.365			
H11	O24[1]	3.390	H11	C17[6]
	3.399			
H11	H17A[6]	2.982	H11	H17B[6]
	2.882			
H11	H41[1]	2.956	H12	F53[1]
	2.708			
H12	C41[1]	3.343	H12	C42[1]
	3.093			
H12	H41[1]	3.016	H12	H42[1]
	2.491			
H13	O23[1]	3.171	H13	O24[1]
	3.207			
H13	H17A[6]	3.245	H13	H17B[6]
	3.442			
H13	H18A[6]	2.966	H14A	F22[5]
	2.802			
H14A	O23[1]	3.288	H14B	F22[5]
	3.496			
H14B	F56[3]	2.682	H15A	F56[3]
	3.508			
H15A	O22[5]	3.276	H15A	H35[3]
	3.357			
H15A	H51C[6]	3.070	H15B	O23[1]
	2.800			
H15B	H51C[6]	3.106	H16	F54[3]
	3.506			
H16	F56[3]	3.302	H16	H45B[10]
	3.413			

Table 13. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H16	H46[10]	2.904	H16	H49A[10]	3.499
H16	H50A[10]	3.111	H17A	F24[6]	3.411
H17A	C11[6]	3.405	H17A	C18[6]	3.445
H17A	H11[6]	2.982	H17A	H13[6]	3.245
H17A	H18A[6]	2.584	H17B	O24[10]	2.847
H17B	H11[6]	2.882	H17B	H13[6]	3.442
H17B	H44A[10]	3.522	H17B	H46[10]	2.799
H17B	H48A[10]	3.533	H17B	H50A[10]	3.514
H18A	O24[10]	3.250	H18A	C17[6]	3.360
H18A	C18[6]	3.421	H18A	H13[6]	2.966
H18A	H17A[6]	2.584	H18A	H18A[6]	2.944
H18B	O24[10]	3.495	H18B	C50[10]	3.421
H18B	H46[10]	3.496	H18B	H50A[10]	2.442
H19A	F26[6]	2.528	H19A	C23[6]	3.441
H19A	H50B[6]	3.274	H19B	C44[10]	3.232
H19B	C45[10]	3.551	H19B	H44A[10]	2.363
H19B	H45B[10]	3.165	H19B	H46[10]	3.165
H20A	F35[3]	3.011	H20A	F54[3]	2.967
H20A	C45[10]	3.488	H20A	H39[11]	3.176
H20A	H44A[10]	3.566	H20A	H45B[10]	2.575
H20B	F35[3]	2.909	H20B	C35[3]	3.595

H20B	C51[6]	3.260		H20B	H35[3]
	3.238				
H20B	H50B[6]	3.299		H20B	H51B[6]
	2.731				
H20B	H51C[6]	3.077		H21A	F52[10]
	3.486				
H21A	C44[10]	3.239		H21A	H39[11]
	3.534				
H21A	H43[11]	3.080		H21A	H44A[10]
	2.870				
H21A	H44B[11]	3.306		H21A	H44B[10]
	2.870				
H21A	H45A[11]	3.243		H21A	H45B[10]
	3.088				
H21B	F26[6]	3.401		H21B	F52[10]
	3.280				
H21B	F53[10]	3.547		H21B	C47[6]
	3.238				
H21B	H47A[6]	2.787		H21B	H47B[6]
	2.787				
H21B	H49B[6]	3.021		H21B	H50B[6]
	3.023				
H21C	F35[3]	3.484		H21C	H39[11]
	2.866				
H21C	H43[11]	2.676		H21C	H47B[6]
	3.571				
H21C	H49B[6]	3.049		H31	S2[2]
	3.324				
H31	F23[2]	2.828		H31	O2[2]
	2.852				
H31	O22[2]	2.871		H32	F33[7]
	3.359				

Table 13. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H32	O53[8]	2.850	H33	F3[2]	3.355
H33	F55[2]	3.088	H33	O2[2]	3.332
H33	O22[2]	3.157	H33	C3[2]	3.423
H33	H3[2]	2.737	H33	H51A[12]	3.588
H33	H51B[12]	3.433	H34A	F33[7]	2.869
H34A	O51[7]	2.841	H34A	O53[8]	2.740
H34B	F55[2]	3.034	H34B	O51[7]	2.720
H34B	C51[12]	3.365	H34B	H51A[12]	3.477
H34B	H51B[12]	2.481	H35	O22[2]	2.513
H35	C51[12]	3.594	H35	H15A[3]	3.357
H35	H20B[3]	3.238	H35	H51B[12]	2.834
H36	O53[8]	2.407	H36	H36[8]	2.968
H36	H38[8]	3.094	H38	F35[8]	2.922
H38	F54[8]	2.997	H38	O53[8]	2.714
H38	H36[8]	3.094	H39	F35[8]	2.694
H39	C21[14]	3.497	H39	H20A[14]	3.176
H39	H21A[14]	3.534	H39	H21C[14]	2.866
H41	F5	3.123	H41	C11[2]	3.565
H41	C12[2]	3.581	H41	H5	3.392
H41	H11[2]	2.956	H41	H12[2]	3.016
H42	F5	3.573	H42	F23[2]	2.557

H42	O2[2]	3.342	H42	C12[2]
	3.064			
H42	C22[2]	3.563	H42	H12[2]
	2.491			
H43	F52[1]	3.581	H43	C21[14]
	3.320			
H43	H21A[14]	3.080	H43	H21C[14]
	2.676			
H44A	C19[10]	3.287	H44A	H17B[10]
	3.522			
H44A	H19B[10]	2.363	H44A	H20A[10]
	3.566			
H44A	H21A[10]	2.870	H44B	C44[9]
	3.494			
H44B	C45[9]	3.598	H44B	H21A[14]
	3.306			
H44B	H21A[10]	2.870	H44B	H44B[9]
	2.663			
H44B	H45A[9]	3.292	H44B	H45B[9]
	3.396			
H45A	F51[9]	3.002	H45A	F52[9]
	3.108			
H45A	H21A[14]	3.243	H45A	H44B[9]
	3.292			
H45B	F51[9]	3.584	H45B	F54[15]
	3.527			
H45B	C20[10]	3.302	H45B	H16[10]
	3.413			
H45B	H19B[10]	3.165	H45B	H20A[10]
	2.575			
H45B	H21A[10]	3.088	H45B	H44B[9]
	3.396			

Table 13. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H46	O24	3.336	H46	$C16^{10}$	3.506
H46	$C17^{10}$	3.484	H46	$H16^{10}$	2.904
H46	$H17B^{10}$	2.799	H46	$H18B^{10}$	3.496
H46	$H19B^{10}$	3.165	H47A	F26	2.577
H47A	$F52^{1}$	3.592	H47A	O24	3.214
H47A	C23	3.471	H47A	$H21B^{6}$	2.787
H47B	$F52^{1}$	2.756	H47B	$C21^{6}$	3.581
H47B	$H21B^{6}$	2.787	H47B	$H21C^{6}$	3.571
H48A	S6	3.513	H48A	O6	3.288
H48A	O24	2.610	H48A	H5	3.434
H48A	$H17B^{10}$	3.533	H48B	$S32^{1}$	3.552
H48B	$F52^{1}$	2.982	H48B	$F53^{1}$	3.356
H48B	O6	3.311	H48B	$O52^{1}$	2.586
H48B	$C52^{1}$	3.452	H48B	H5	3.063
H49A	$F54^{15}$	2.831	H49A	$F55^{15}$	2.776
H49A	$F56^{15}$	3.532	H49A	$C53^{15}$	3.247
H49A	$H16^{10}$	3.499	H49B	$F52^{9}$	3.597
H49B	$O51^{9}$	3.599	H49B	$C21^{6}$	3.458
H49B	$H21B^{6}$	3.021	H49B	$H21C^{6}$	3.049
H50A	F25	3.395	H50A	$C18^{10}$	3.375
H50A	$H9^{10}$	3.295	H50A	$H16^{10}$	3.111

H50A	H17B[10]	3.514	H50A	H18B[10]
	2.442			
H50B	F25	3.000	H50B	F26
	2.772			
H50B	C23	3.393	H50B	H19A[6]
	3.274			
H50B	H20B[6]	3.299	H50B	H21B[6]
	3.023			
H51A	F3[15]	3.067	H51A	F55[15]
	2.719			
H51A	F56[15]	3.169	H51A	O22[15]
	3.595			
H51A	C53[15]	3.520	H51A	H9[10]
	3.019			
H51A	H33[13]	3.588	H51A	H34B[13]
	3.477			
H51B	F55[15]	3.212	H51B	O22[15]
	3.541			
H51B	C34[13]	3.243	H51B	C35[13]
	3.372			
H51B	H20B[6]	2.731	H51B	H33[13]
	3.433			
H51B	H34B[13]	2.481	H51B	H35[13]
	2.834			
H51C	F25	3.589	H51C	O22[15]
	3.309			
H51C	C15[6]	3.561	H51C	H15A[6]
	3.070			
H51C	H15B[6]	3.106	H51C	H20B[6]
	3.077			

Symmetry Operators:

(1) X-1,Y,Z
(2) X+1,Y,Z
(3) -X+1,-Y+2,-Z+1
(4) X,Y+1,Z
(5) -X,-Y+2,-Z+1
(6) -X,-Y+1,-Z+1
(7) -X+3,-Y+2,-Z
(8) -X+2,-Y+2,-Z
(9) -X+2,-Y+1,-Z
(10) -X+1,-Y+1,-Z+1
(11) X-1,Y,Z+1
(12) X+1,Y+1,Z
(13) X-1,Y-1,Z
(14) X+1,Y,Z-1
(15) X,Y-1,Z

X-Ray Data Collection for **17**

Data Collection

A colorless platelet crystal of $C_{23}H_{28}F_8O_6S_2$ having approximate dimensions of 0.300 x 0.060 x 0.020 mm was mounted in a loop. All measurements were made on a Rigaku Mercury70 diffractometer using multi-layer mirror monochromated Mo-Kα radiation.

Cell constants and an orientation matrix for data collection corresponded to a C-centered monoclinic cell with dimensions:

a = 44.560(17) Å
b = 5.5896(15) Å β = 124.357(7)°
c = 26.277(10) Å
V = 5403(3) Å^3

For Z = 8 and F.W. = 616.58, the calculated density is 1.516 g/cm^3. Based on the reflection conditions of:

hkl: h+k = 2n
h0l: l = 2n

packing considerations, a statistical analysis of intensity distribution, and the successful solution and refinement of the structure, the space group was determined to be:

C2/c (#15)

The data were collected at a temperature of -100 ± 1°C to a maximum 2θ value of 50.7°. Readout was performed in the 0.068 mm pixel mode.

Data Reduction

Of the 16828 reflections were collected, where 4913 were unique (R_{int} = 0.0799); equivalent reflections were merged. Data were collected and processed using CrystalClear (Rigaku). [1]

The linear absorption coefficient, μ, for Mo-Kα radiation is 2.889 cm^{-1}. An empirical absorption correction was applied which resulted in transmission factors ranging from 0.608 to 0.994. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by direct methods[2] and expanded using Fourier techniques. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined using the riding model. The final cycle of full-matrix least-squares refinement[3] on F^2 was based on 4913 observed reflections and 353 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R1 = \Sigma \, ||Fo| - |Fc|| \, / \, \Sigma \, |Fo| = 0.0648$$

$$wR2 = [\Sigma (w (Fo^2 - Fc^2)^2) / \Sigma \, w(Fo^2)^2]^{1/2} = 0.1673$$

The goodness of fit[4] was 1.07. Unit weights were used. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.36 and -0.39 $e^-/Å^3$, respectively.

Neutral atom scattering factors were taken from International Tables for Crystallography (IT), Vol. C, Table 6.1.1.4 [5]. Anomalous dispersion effects were included in Fcalc[6]; the values for Δf' and Δf'' were those of Creagh and McAuley[7]. The values for the mass attenuation coefficients are those of Creagh and Hubbell[8]. All calculations were performed using the CrystalStructure[9] crystallographic software package except for refinement, which was performed using SHELXL2013[10].

References

(1) CrystalClear: Data Collection and Processing Software, Rigaku Corporation (1998-2014). Tokyo 196-8666, Japan.

(2) SIR2004: Burla, M. C., Caliandro, R., Camalli, M., Carrozzini, B., Cascarano, G. L., De Caro, L., Giacovazzo, C., Polidori, G. and Spagna R. (2005). J. Appl. Cryst. 38, 381-388.

(3) Least Squares function minimized: (SHELXL2013)

$\Sigma w(F_o^2 - F_c^2)^2$ where w = Least Squares weights.

(4) Goodness of fit is defined as:

$$[\Sigma w(F_O^2-F_C^2)^2/(N_O-N_V)]^{1/2}$$

where: N_O = number of observations
N_V = number of variables

(5) International Tables for Crystallography, Vol.C (1992). Ed. A.J.C. Wilson, Kluwer Academic Publishers, Dordrecht, Netherlands, Table 6.1.1.4, pp. 572.

(6) Ibers, J. A. & Hamilton, W. C.; Acta Crystallogr., 17, 781 (1964).

(7) Creagh, D. C. & McAuley, W.J .; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.6.8, pages 219-222 (1992).

(8) Creagh, D. C. & Hubbell, J.H..; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.4.3, pages 200-206 (1992).

(9) CrystalStructure 4.1: Crystal Structure Analysis Package, Rigaku Corporation (2000-2014). Tokyo 196-8666, Japan.

(10) SHELXL2013: Sheldrick, G. M. (2008). Acta Cryst. A64, 112-122.

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$C_{23}H_{28}F_8O_6S_2$
Formula Weight	616.58
Crystal Color, Habit	colorless, platelet
Crystal Dimensions	0.300 X 0.060 X 0.020 mm
Crystal System	monoclinic
Lattice Type	C-centered
Lattice Parameters	a = 44.560(17) Å b = 5.5896(15) Å c = 26.277(10) Å β = 124.357(7) ° V = 5403(3) Å^3
Space Group	C2/c (#15)
Z value	8
D_{calc}	1.516 g/cm^3
F_{000}	2544.00
μ(MoKα)	2.889 cm^{-1}

B. Intensity Measurements

Diffractometer	Mercury70
Radiation monochromated	MoKα (λ = 0.71075 Å) multi-layer mirror
Voltage, Current	50kV, 16mA
Temperature	-100.0°C
Detector Aperture	70.0 x 70.0 mm
Pixel Size	0.068 mm
$2\theta_{max}$	50.7°
No. of Reflections Measured	Total: 16828 Unique: 4913 (R_{int} = 0.0799)
Corrections	Lorentz-polarization Absorption (trans. factors: 0.608 - 0.994)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SIR2004)
Refinement	Full-matrix least-squares on F^2
Function Minimized	Σ w $(Fo^2 - Fc^2)^2$
Least Squares Weights	w = 1/ [$\sigma^2(Fo^2)$ + $(0.0469 \cdot P)^2$ + 6.6592 · P] where P = (Max(Fo^2,0) + $2Fc^2$)/3
$2\theta_{max}$ cutoff	50.7º
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (All reflections)	4913
No. Variables	353
Reflection/Parameter Ratio	13.92
Residuals: R1 (I>2.00σ(I))	0.0648
Residuals: R (All reflections)	0.1467
Residuals: wR2 (All reflections)	0.1673
Goodness of Fit Indicator	1.067
Max Shift/Error in Final Cycle	0.000
Maximum peak in Final Diff. Map	0.36 $e^-/Å^3$
Minimum peak in Final Diff. Map	-0.39 $e^-/Å^3$

Table 1. Atomic coordinates and B_{iso}/B_{eq}

atom	x	y	z	B_{eq}
S1	0.16515(3)	1.2015(2)	1.10124(6)	3.65(3)
S2	0.19661(3)	1.0852(2)	0.85371(5)	3.65(3)
F1	0.14676(7)	0.6096(4)	0.96489(11)	4.10(6)
F2	0.24468(6)	0.9942(4)	0.99777(11)	3.68(5)
F3	0.22951(10)	1.3582(9)	1.18092(16)	9.82(13)
F4	0.22240(12)	0.9814(9)	1.18620(16)	11.75(16)
F5	0.20102(10)	1.2348(7)	1.21953(14)	8.31(10)
F6	0.14933(9)	1.4096(6)	0.78193(13)	6.70(8)
F7	0.13171(9)	1.0514(6)	0.75427(13)	7.13(9)
F8	0.17074(9)	1.2013(6)	0.74182(13)	6.48(8)
O1	0.17904(8)	1.1401(5)	1.06003(13)	3.25(6)
O2	0.15449(10)	1.4447(5)	1.09055(16)	5.28(8)
O3	0.14240(9)	1.0199(6)	1.09887(16)	5.28(8)
O4	0.17957(7)	1.1479(5)	0.89027(12)	3.16(6)
O5	0.19915(9)	0.8329(6)	0.84947(14)	4.48(7)
O6	0.22632(8)	1.2384(7)	0.87230(15)	5.18(8)
C1	0.18051(12)	0.8869(7)	1.04272(19)	3.11(9)
C2	0.14633(12)	0.8526(7)	0.9783(2)	3.31(9)
C3	0.14526(11)	1.0067(7)	0.92942(18)	2.74(8)
C4	0.18069(11)	0.9735(7)	0.93341(19)	3.03(9)
C5	0.21339(11)	1.0285(8)	0.99770(19)	3.18(9)
C6	0.21484(11)	0.8642(8)	1.04489(19)	3.26(9)
C7	0.11140(11)	0.9671(7)	0.86565(19)	2.90(9)
C8	0.08374(12)	1.1342(8)	0.8400(2)	3.32(9)
C9	0.05235(12)	1.1020(8)	0.7817(2)	3.41(9)
C10	0.04744(11)	0.9030(8)	0.74637(19)	2.94(8)
C11	0.07530(11)	0.7348(8)	0.7725(2)	3.48(9)
C12	0.10664(12)	0.7646(8)	0.8308(2)	3.43(9)
C13	0.01371(12)	0.8766(8)	0.68115(19)	3.26(9)
C14	-0.00467(12)	0.6354(9)	0.6689(2)	4.15(10)
C15	-0.03800(12)	0.6142(9)	0.6034(2)	4.33(11)
C16	-0.02976(12)	0.6565(9)	0.5564(2)	4.14(11)
C17	-0.01169(14)	0.9018(10)	0.5681(2)	5.31(12)
C18	0.02207(13)	0.9296(10)	0.6339(2)	4.78(11)
C19	-0.06299(13)	0.6238(11)	0.4902(2)	5.46(13)
C20	-0.05600(15)	0.6106(14)	0.4425(2)	7.76(18)
C21	-0.08867(14)	0.5554(12)	0.3786(2)	6.85(16)

Table 1. Atomic coordinates and B_{iso}/B_{eq} (continued)

atom	x	y	z	B_{eq}
C22	0.20744(19)	1.1884(14)	1.1774(3)	6.34(15)
C23	0.15954(14)	1.1925(9)	0.7786(2)	4.23(11)

$B_{eq} = 8/3\ \pi^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$

Table 2. Atomic coordinates and B_{iso} involving hydrogen atoms

atom	x	y	z	B_{iso}
H1	0.18080	0.77280	1.07230	3.732
H2	0.12441	0.88622	0.97871	3.976
H3	0.14470	1.17714	0.94043	3.292
H4	0.18230	0.80687	0.92127	3.634
H5	0.21224	1.19863	1.00825	3.815
H6A	0.21780	0.69659	1.03626	3.912
H6B	0.23602	0.90628	1.08660	3.912
H8	0.08631	1.27365	0.86284	3.984
H9	0.03370	1.21932	0.76560	4.093
H11	0.07273	0.59558	0.74956	4.174
H12	0.12511	0.64589	0.84738	4.112
H13	-0.00411	1.00004	0.67580	3.912
H14A	-0.01187	0.61126	0.69790	4.978
H14B	0.01283	0.50789	0.67647	4.978
H15A	-0.04852	0.45223	0.59726	5.191
H15B	-0.05644	0.73150	0.59710	5.191
H16	-0.01142	0.53397	0.56349	4.968
H17A	-0.00470	0.92566	0.53871	6.376
H17B	-0.02949	1.02770	0.56007	6.376
H18A	0.04126	0.81919	0.64017	5.740
H18B	0.03143	1.09504	0.63982	5.740
H19A	-0.07562	0.47529	0.48854	6.553
H19B	-0.07986	0.75862	0.48031	6.553
H20A	-0.03737	0.48604	0.45421	9.310
H20B	-0.04565	0.76537	0.44114	9.310
H21A	-0.10839	0.66656	0.36807	8.224
H21B	-0.09667	0.39098	0.37738	8.224
H21C	-0.08217	0.57286	0.34883	8.224

Table 3. Anisotropic displacement parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
S1	0.0496(8)	0.0469(7)	0.0483(8)	-0.0051(6)	0.0312(7)	-0.0030(6)
S2	0.0397(8)	0.0590(8)	0.0399(7)	-0.0014(6)	0.0224(6)	-0.0037(6)
F1	0.0680(19)	0.0367(14)	0.0505(16)	-0.0094(13)	0.0330(15)	-0.0060(12)
F2	0.0355(15)	0.0533(16)	0.0518(16)	-0.0003(12)	0.0250(13)	-0.0023(13)
F3	0.081(3)	0.213(5)	0.078(3)	-0.062(3)	0.044(2)	-0.068(3)
F4	0.154(4)	0.184(5)	0.054(2)	0.102(4)	0.026(2)	0.021(3)
F5	0.126(3)	0.151(3)	0.051(2)	-0.023(3)	0.057(2)	-0.026(2)
F6	0.108(3)	0.071(2)	0.067(2)	0.031(2)	0.044(2)	0.0144(17)
F7	0.062(2)	0.108(3)	0.056(2)	-0.025(2)	0.0065(17)	0.0088(19)
F8	0.084(2)	0.121(3)	0.0522(19)	0.017(2)	0.0451(19)	0.0146(18)
O1	0.050(2)	0.0376(17)	0.0401(18)	-0.0022(15)	0.0282(16)	-0.0007(14)
O2	0.095(3)	0.0414(19)	0.090(3)	0.0115(19)	0.068(2)	0.0026(18)
O3	0.080(3)	0.059(2)	0.090(3)	-0.0274(19)	0.065(2)	-0.0142(19)
O4	0.0428(18)	0.0443(18)	0.0419(18)	-0.0005(14)	0.0292(16)	0.0012(14)
O5	0.066(2)	0.058(2)	0.047(2)	0.0161(18)	0.0322(19)	-0.0008(16)
O6	0.048(2)	0.103(3)	0.054(2)	-0.030(2)	0.0330(19)	-0.012(2)
C1	0.045(3)	0.035(2)	0.036(3)	-0.000(2)	0.021(2)	-0.000(2)
C2	0.041(3)	0.040(3)	0.047(3)	-0.005(2)	0.026(3)	-0.008(2)
C3	0.037(3)	0.032(2)	0.036(3)	-0.001(2)	0.021(2)	-0.001(2)
C4	0.041(3)	0.037(3)	0.038(3)	-0.000(2)	0.023(2)	0.004(2)
C5	0.033(3)	0.046(3)	0.043(3)	-0.002(2)	0.022(2)	-0.002(2)
C6	0.040(3)	0.044(3)	0.036(3)	0.000(2)	0.019(2)	0.002(2)

C7	0.039(3)	0.037(3)	0.034(3)	-0.001(2)	0.020(2)	-0.002(2)
C8	0.047(3)	0.037(3)	0.044(3)	-0.001(2)	0.028(3)	-0.004(2)
C9	0.038(3)	0.044(3)	0.041(3)	0.002(2)	0.018(3)	-0.001(2)
C10	0.033(3)	0.046(3)	0.036(3)	-0.001(2)	0.022(2)	0.000(2)
C11	0.035(3)	0.044(3)	0.045(3)	-0.001(2)	0.018(3)	-0.010(2)
C12	0.037(3)	0.044(3)	0.043(3)	0.007(2)	0.019(3)	0.001(2)
C13	0.035(3)	0.049(3)	0.035(3)	0.003(2)	0.017(2)	0.002(2)
C14	0.045(3)	0.071(3)	0.036(3)	-0.014(3)	0.020(3)	-0.007(2)
C15	0.042(3)	0.072(3)	0.039(3)	-0.010(3)	0.015(3)	0.000(3)
C16	0.033(3)	0.075(4)	0.038(3)	0.008(3)	0.013(3)	-0.006(3)
C17	0.064(4)	0.096(4)	0.036(3)	-0.002(3)	0.025(3)	0.011(3)
C18	0.058(4)	0.079(4)	0.042(3)	-0.015(3)	0.027(3)	0.002(3)
C19	0.047(3)	0.104(5)	0.044(3)	0.009(3)	0.018(3)	-0.006(3)
C20	0.066(4)	0.183(7)	0.040(3)	0.005(4)	0.026(3)	-0.009(4)
C21	0.058(4)	0.151(6)	0.040(3)	0.001(4)	0.020(3)	-0.016(4)

Table 3. Anisotropic displacement parameters (continued)

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C22	0.081(5)	0.104(5)	0.048(4)	-0.009(4)	0.032(4)	-0.017(4)
C23	0.057(4)	0.057(3)	0.042(3)	-0.005(3)	0.026(3)	-0.003(3)

The general temperature factor expression: $\exp(-2\pi^2(a^{*2}U_{11}h^2 + b^{*2}U_{22}k^2 + c^{*2}U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$

Table 4. Bond lengths (Å)

atom	atom	distance	atom	atom	distance
S1	O1	1.559(5)	S1	O2	1.415(3)
S1	O3	1.411(4)	S1	C22	1.818(5)
S2	O4	1.564(4)	S2	O5	1.424(3)
S2	O6	1.412(4)	S2	C23	1.818(4)
F1	C2	1.406(5)	F2	C5	1.406(6)
F3	C22	1.332(10)	F4	C22	1.289(9)
F5	C22	1.315(10)	F6	C23	1.316(7)
F7	C23	1.294(6)	F8	C23	1.315(9)
O1	C1	1.499(5)	O4	C4	1.474(6)
C1	C2	1.520(5)	C1	C6	1.504(8)
C2	C3	1.525(7)	C3	C4	1.533(8)
C3	C7	1.510(5)	C4	C5	1.514(5)
C5	C6	1.515(7)	C7	C8	1.381(6)
C7	C12	1.394(7)	C8	C9	1.384(5)
C9	C10	1.386(7)	C10	C11	1.391(6)
C10	C13	1.520(5)	C11	C12	1.383(5)
C13	C14	1.515(7)	C13	C18	1.514(9)
C14	C15	1.516(5)	C15	C16	1.491(9)
C16	C17	1.531(8)	C16	C19	1.531(5)
C17	C18	1.531(5)	C19	C20	1.456(10)
C20	C21	1.508(6)			

Table 5. Bond lengths involving hydrogens (Å)

atom	atom	distance	atom	atom	distance
C1	H1	1.000	C2	H2	1.000
C3	H3	1.000	C4	H4	1.000
C5	H5	1.000	C6	H6A	0.990
C6	H6B	0.990	C8	H8	0.950
C9	H9	0.950	C11	H11	0.950
C12	H12	0.950	C13	H13	1.000
C14	H14A	0.990	C14	H14B	0.990
C15	H15A	0.990	C15	H15B	0.990
C16	H16	1.000	C17	H17A	0.990
C17	H17B	0.990	C18	H18A	0.990
C18	H18B	0.990	C19	H19A	0.990
C19	H19B	0.990	C20	H20A	0.990
C20	H20B	0.990	C21	H21A	0.980
C21	H21B	0.980	C21	H21C	0.980

Table 6. Bond angles (°)

atom	atom	atom	angle	atom	atom	atom	angle
O1	S1	O2	107.1(3)	O1	S1	O3	111.6(2)
O1	S1	C22	100.7(3)	O2	S1	O3	122.0(3)
O2	S1	C22	106.3(3)	O3	S1	C22	107.0(3)
O4	S2	O5	110.9(2)	O4	S2	O6	109.7(2)
O4	S2	C23	97.7(3)	O5	S2	O6	121.8(2)
O5	S2	C23	107.8(2)	O6	S2	C23	106.0(2)
S1	O1	C1	121.4(3)	S2	O4	C4	120.4(3)
O1	C1	C2	105.6(3)	O1	C1	C6	106.6(4)
C2	C1	C6	113.0(5)	F1	C2	C1	104.7(3)
F1	C2	C3	109.4(4)	C1	C2	C3	113.7(4)
C2	C3	C4	109.4(3)	C2	C3	C7	112.9(4)
C4	C3	C7	113.8(4)	O4	C4	C3	106.3(3)
O4	C4	C5	108.5(3)	C3	C4	C5	110.9(4)
F2	C5	C4	107.6(4)	F2	C5	C6	109.4(3)
C4	C5	C6	111.1(4)	C1	C6	C5	110.2(3)
C3	C7	C8	120.0(4)	C3	C7	C12	122.3(4)
C8	C7	C12	117.8(3)	C7	C8	C9	121.2(4)
C8	C9	C10	121.7(4)	C9	C10	C11	116.9(3)
C9	C10	C13	121.0(4)	C11	C10	C13	122.0(4)
C10	C11	C12	121.8(4)	C7	C12	C11	120.7(4)
C10	C13	C14	113.5(4)	C10	C13	C18	111.2(4)
C14	C13	C18	110.7(4)	C13	C14	C15	112.2(4)

C14	C15	C16	112.7(5)	C15	C16	C17
	108.9(5)					
C15	C16	C19	112.9(5)	C17	C16	C19
	113.3(4)					
C16	C17	C18	112.8(4)	C13	C18	C17
	111.6(5)					
C16	C19	C20	116.4(5)	C19	C20	C21
	115.3(6)					
S1	C22	F3	108.2(5)	S1	C22	F4
	110.0(4)					
S1	C22	F5	109.6(5)	F3	C22	F4
	109.9(7)					
F3	C22	F5	107.7(6)	F4	C22	F5
	111.3(6)					
S2	C23	F6	111.6(3)	S2	C23	F7
	111.5(4)					
S2	C23	F8	108.7(4)	F6	C23	F7
	108.9(5)					
F6	C23	F8	107.6(5)	F7	C23	F8
	108.4(4)					

Table 7. Bond angles involving hydrogens (°)

atom	atom	atom	angle	atom	atom	atom	angle
O1	C1	H1	110.5	C2	C1	H1	110.5
C6	C1	H1	110.5	F1	C2	H2	109.6
C1	C2	H2	109.6	C3	C2	H2	109.6
C2	C3	H3	106.8	C4	C3	H3	106.8
C7	C3	H3	106.8	O4	C4	H4	110.4
C3	C4	H4	110.3	C5	C4	H4	110.4
F2	C5	H5	109.6	C4	C5	H5	109.6
C6	C5	H5	109.6	C1	C6	H6A	109.6
C1	C6	H6B	109.6	C5	C6	H6A	109.6
C5	C6	H6B	109.6	H6A	C6	H6B	108.2
C7	C8	H8	119.4	C9	C8	H8	119.4
C8	C9	H9	119.2	C10	C9	H9	119.2
C10	C11	H11	119.1	C12	C11	H11	119.1
C7	C12	H12	119.7	C11	C12	H12	119.7
C10	C13	H13	107.0	C14	C13	H13	107.0
C18	C13	H13	107.1	C13	C14	H14A	109.2
C13	C14	H14B	109.2	C15	C14	H14A	109.2
C15	C14	H14B	109.2	H14A	C14	H14B	107.9
C14	C15	H15A	109.1	C14	C15	H15B	109.1
C16	C15	H15A	109.1	C16	C15	H15B	109.0
H15A	C15	H15B	107.8	C15	C16	H16	107.1
C17	C16	H16	107.1	C19	C16	H16	107.1

C16	C17	H17A	109.0	C16	C17	H17B
	109.0					
C18	C17	H17A	109.0	C18	C17	H17B
	109.0					
H17A	C17	H17B	107.8	C13	C18	H18A
	109.3					
C13	C18	H18B	109.3	C17	C18	H18A
	109.3					
C17	C18	H18B	109.3	H18A	C18	H18B
	108.0					
C16	C19	H19A	108.2	C16	C19	H19B
	108.2					
C20	C19	H19A	108.2	C20	C19	H19B
	108.2					
H19A	C19	H19B	107.4	C19	C20	H20A
	108.4					
C19	C20	H20B	108.4	C21	C20	H20A
	108.4					
C21	C20	H20B	108.4	H20A	C20	H20B
	107.5					
C20	C21	H21A	109.5	C20	C21	H21B
	109.5					
C20	C21	H21C	109.5	H21A	C21	H21B
	109.5					
H21A	C21	H21C	109.5	H21B	C21	H21C
	109.4					

Table 8. Torsion Angles(º)
(Those having bond angles > 160 or < 20 degrees are excluded.)

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
O2	S1	O1	C1	-158.83(19)	O3	S1	O1	C1	-22.9(2)
O1	S1	C22	F3	63.2(5)	O1	S1	C22	F4	-56.8(6)
O1	S1	C22	F5	-179.5(4)	C22	S1	O1	C1	90.3(3)
O2	S1	C22	F3	-48.3(6)	O2	S1	C22	F4	-168.4(5)
O2	S1	C22	F5	69.0(5)	O3	S1	C22	F3	179.9(5)
O3	S1	C22	F4	59.8(7)	O3	S1	C22	F5	-62.8(5)
O5	S2	O4	C4	22.2(2)	O6	S2	O4	C4	-115.2(2)
O4	S2	C23	F6	49.1(5)	O4	S2	C23	F7	-72.9(4)
O4	S2	C23	F8	167.7(3)	C23	S2	O4	C4	134.7(2)
O5	S2	C23	F6	164.1(4)	O5	S2	C23	F7	42.1(5)
O5	S2	C23	F8	-77.3(4)	O6	S2	C23	F6	-64.0(5)
O6	S2	C23	F7	174.0(4)	O6	S2	C23	F8	54.6(4)
S1	O1	C1	C2	98.4(3)	S1	O1	C1	C6	-141.2(2)
S2	O4	C4	C3	-145.4(2)	S2	O4	C4	C5	95.3(3)
O1	C1	C2	F1	-175.5(4)	O1	C1	C2	C3	65.1(6)
O1	C1	C6	C5	-62.8(4)	C2	C1	C6	C5	52.8(4)
C6	C1	C2	F1	68.3(5)	C6	C1	C2	C3	-51.1(5)
F1	C2	C3	C4	-65.4(4)	F1	C2	C3	C7	62.4(5)
C1	C2	C3	C4	51.2(5)	C1	C2	C3	C7	179.1(4)
C2	C3	C4	O4	-173.4(3)	C2	C3	C4	C5	-55.7(4)
C2	C3	C7	C8	104.0(5)	C2	C3	C7	C12	-75.7(6)

C4	C3	C7	C8	-130.5(4)	C4	C3	C7	C12	49.8(6)
C7	C3	C4	O4	59.3(4)	C7	C3	C4	C5	177.0(3)
O4	C4	C5	F2	-63.6(4)	O4	C4	C5	C6	176.6(3)
C3	C4	C5	F2	180.0(3)	C3	C4	C5	C6	60.3(4)
F2	C5	C6	C1	-176.4(3)	C4	C5	C6	C1	-57.8(5)
C3	C7	C8	C9	-179.8(5)	C3	C7	C12	C11	-179.8(5)
C8	C7	C12	C11	0.5(8)	C12	C7	C8	C9	-0.1(8)
C7	C8	C9	C10	-0.7(9)	C8	C9	C10	C11	0.9(9)
C8	C9	C10	C13	-176.7(5)	C9	C10	C11	C12	-0.5(8)
C9	C10	C13	C14	-130.8(5)	C9	C10	C13	C18	103.6(6)
C11	C10	C13	C14	51.6(7)	C11	C10	C13	C18	-73.9(6)
C13	C10	C11	C12	177.2(5)	C10	C11	C12	C7	-0.3(9)
C10	C13	C14	C15	-179.6(4)	C10	C13	C18	C17	179.6(3)
C14	C13	C18	C17	52.5(5)	C18	C13	C14	C15	-53.7(5)
C13	C14	C15	C16	56.9(6)	C14	C15	C16	C17	-55.8(5)
C14	C15	C16	C19	177.3(4)	C15	C16	C17	C18	55.0(6)

Table 8. Torsion angles (º) (continued)

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
C15	C16	C19	C20	-167.2(4)	C17	C16	C19	C20	68.3(6)
C19	C16	C17	C18	-178.5(5)	C16	C17	C18	C13	-54.4(6)
C16	C19	C20	C21	174.5(5)					

Table 9. Intramolecular contacts less than 3.60 Å

atom	atom	distance	atom	atom	distance
S1	C2	3.447(5)	S2	F2	3.166(3)
S2	C5	3.432(6)	F1	C4	2.919(6)
F1	C5	3.486(6)	F1	C6	2.907(5)
F1	C7	2.939(5)	F1	C12	3.041(5)
F2	O4	2.808(3)	F2	O5	3.345(4)
F2	O6	3.218(5)	F3	O1	2.928(4)
F3	O2	2.844(5)	F4	O1	2.878(5)
F4	O3	2.964(5)	F4	C1	3.168(6)
F4	C6	3.594(8)	F5	O2	3.038(5)
F5	O3	2.995(4)	F6	O4	2.780(4)
F6	O6	3.022(4)	F7	O4	3.001(4)
F7	O5	2.880(4)	F7	C7	3.549(8)
F7	C11	3.322(7)	F7	C12	3.226(7)
F8	O5	3.131(5)	F8	O6	2.889(4)
O1	C3	2.961(5)	O1	C4	3.496(7)
O1	C5	2.871(8)	O3	C1	2.905(8)
O3	C2	3.401(8)	O4	C7	2.907(6)
O4	C12	3.444(5)	O5	C4	2.865(8)
O6	C4	3.548(7)	C1	C4	2.917(8)
C1	C22	3.467(9)	C2	C5	2.905(8)
C2	C8	3.467(5)	C2	C12	3.264(7)
C3	C6	2.973(5)	C4	C12	3.079(5)

C7	C10	2.827(5)	C8	C11
		2.743(7)		
C9	C12	2.748(6)	C9	C18
		3.454(7)		
C11	C14	3.077(5)	C11	C18
		3.213(6)		
C13	C16	2.974(6)	C14	C17
		2.898(8)		
C15	C18	2.914(8)	C17	C20
		3.176(8)		

Table 10. Intramolecular contacts less than 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
S1	H1	2.721	S1	H2	3.189
S2	H4	2.694	F1	H1	2.503
F1	H3	3.228	F1	H4	2.662
F1	H6A	2.660	F1	H12	2.673
F2	H4	2.561	F2	H6A	2.568
F2	H6B	2.618	F4	H1	2.737
F4	H6B	3.031	F7	H12	3.467
O1	H2	2.576	O1	H3	2.614
O1	H5	2.534	O1	H6A	3.277
O1	H6B	2.577	O3	H1	2.583
O3	H2	2.883	O4	H3	2.548
O4	H5	2.592	O4	H12	3.456
O5	H4	2.397	O5	H12	3.432
C1	H3	2.750	C1	H4	3.268
C1	H5	2.702	C2	H4	2.756
C2	H5	3.222	C2	H6A	2.784
C2	H6B	3.365	C2	H8	3.569
C2	H12	3.224	C3	H1	3.406
C3	H5	2.715	C3	H6A	3.330
C3	H8	2.646	C3	H12	2.709
C4	H2	3.364	C4	H6A	2.718
C4	H6B	3.350	C4	H12	2.877

C5	H1	3.348	C5	H3
	2.669			
C6	H2	3.372	C6	H3
	3.268			
C6	H4	2.728	C7	H2
	2.724			
C7	H4	2.776	C7	H9
	3.258			
C7	H11	3.264	C8	H2
	3.321			
C8	H3	2.511	C8	H12
	3.238			
C9	H11	3.228	C9	H13
	2.549			
C9	H18B	3.295	C10	H8
	3.267			
C10	H12	3.274	C10	H14A
	2.733			
C10	H14B	2.726	C10	H18A
	2.686			
C10	H18B	2.692	C11	H9
	3.229			
C11	H13	3.321	C11	H14A
	3.288			
C11	H14B	2.792	C11	H18A
	2.942			
C11	H18B	3.512	C12	H2
	3.566			
C12	H3	3.313	C12	H4
	2.833			

Table 10. Intramolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
C12	H8	3.238	C13	H9	2.675
C13	H11	2.698	C13	H15A	3.358
C13	H15B	2.741	C13	H16	3.252
C13	H17A	3.360	C13	H17B	2.762
C14	H11	2.861	C14	H16	2.672
C14	H17B	3.261	C14	H18A	2.748
C14	H18B	3.340	C15	H13	2.704
C15	H17A	3.311	C15	H17B	2.696
C15	H18A	3.296	C15	H19A	2.609
C15	H19B	2.789	C16	H13	3.284
C16	H14A	3.343	C16	H14B	2.736
C16	H18A	2.797	C16	H18B	3.391
C16	H20A	2.685	C16	H20B	2.762
C17	H13	2.712	C17	H14B	3.263
C17	H15A	3.320	C17	H15B	2.680
C17	H19A	3.383	C17	H19B	2.698
C17	H20A	3.434	C17	H20B	2.880
C18	H9	3.584	C18	H11	3.174
C18	H14A	3.340	C18	H14B	2.738
C18	H15B	3.253	C18	H16	2.725
C19	H15A	2.683	C19	H15B	2.722

C19	H17A	2.735	C19	H17B
	2.757			
C19	H21A	2.669	C19	H21B
	2.778			
C19	H21C	3.322	C20	H16
	2.667			
C20	H17A	2.863	C20	H17B
	3.502			
C21	H19A	2.644	C21	H19B
	2.721			
C22	H1	3.277	H1	H2
	2.406			
H1	H6A	2.358	H1	H6B
	2.389			
H2	H3	2.345	H2	H8
	3.318			
H3	H4	2.880	H3	H5
	2.489			
H3	H8	2.287	H4	H5
	2.891			
H4	H6A	2.572	H4	H12
	2.343			
H5	H6A	2.876	H5	H6B
	2.360			
H8	H9	2.314	H9	H13
	2.328			
H9	H18B	3.323	H11	H12
	2.316			
H11	H14A	3.207	H11	H14B
	2.295			
H11	H18A	2.691	H13	H14A
	2.325			

Table 10. Intramolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H13	H14B	2.850	H13	H15A	3.597
H13	H15B	2.554	H13	H17B	2.582
H13	H18A	2.850	H13	H18B	2.318
H14A	H15A	2.361	H14A	H15B	2.340
H14A	H16	3.569	H14B	H15A	2.341
H14B	H15B	2.859	H14B	H16	2.526
H14B	H18A	2.628	H15A	H16	2.321
H15A	H17B	3.599	H15A	H19A	2.396
H15A	H19B	3.079	H15B	H16	2.833
H15B	H17A	3.583	H15B	H17B	2.539
H15B	H19A	2.858	H15B	H19B	2.630
H16	H17A	2.351	H16	H17B	2.862
H16	H18A	2.603	H16	H19A	2.411
H16	H19B	2.852	H16	H20A	2.430
H16	H20B	2.967	H17A	H18A	2.344
H17A	H18B	2.392	H17A	H19B	2.940
H17A	H20A	3.072	H17A	H20B	2.329
H17B	H18A	2.870	H17B	H18B	2.341
H17B	H19A	3.596	H17B	H19B	2.526
H17B	H20B	3.146	H19A	H20A	2.336
H19A	H20B	2.800	H19A	H21A	2.842

H19A	H21B	2.558	H19A	H21C
	3.557			
H19B	H20A	2.800	H19B	H20B
	2.271			
H19B	H21A	2.519	H19B	H21B
	3.132			
H19B	H21C	3.553	H20A	H21A
	2.841			
H20A	H21B	2.306	H20A	H21C
	2.377			
H20B	H21A	2.398	H20B	H21B
	2.838			
H20B	H21C	2.288		

Table 11. Intermolecular contacts less than 3.60 Å

atom	atom	distance	atom	atom	distance
F1	O1[1]	3.338(4)	F1	O2[1]	3.254(6)
F1	C3[1]	3.487(5)	F2	F2[2]	2.762(3)
F2	F2[3]	2.890(3)	F2	O4[3]	3.584(3)
F2	O6[3]	3.253(5)	F2	C5[2]	3.434(5)
F2	C5[3]	3.221(5)	F2	C6[2]	3.287(6)
F3	F4[4]	3.508(7)	F3	F4[5]	2.969(5)
F3	F5[5]	3.423(5)	F3	O6[3]	3.038(7)
F4	F3[1]	3.508(7)	F4	F3[6]	2.969(5)
F4	F5[6]	3.184(5)	F4	F8[7]	3.504(8)
F4	C22[6]	3.557(7)	F5	F3[6]	3.423(5)
F5	F4[5]	3.184(5)	F5	F8[7]	2.999(6)
F5	O5[7]	3.483(6)	F6	O5[4]	3.038(4)
F6	C12[4]	3.459(7)	F6	C21[8]	3.503(5)
F8	F4[9]	3.504(8)	F8	F5[9]	2.999(6)
F8	O3[9]	3.447(5)	F8	C21[8]	3.489(5)
O1	F1[4]	3.338(4)	O1	O6[3]	3.598(5)
O2	F1[4]	3.254(6)	O2	O3[4]	3.288(5)
O2	C1[4]	3.268(7)	O2	C2[4]	3.580(7)
O2	C19[10]	3.516(6)	O2	C21[10]	3.511(9)
O3	F8[7]	3.447(5)	O3	O2[1]	3.288(5)
O4	F2[3]	3.584(3)	O5	F5[9]	3.483(6)
O5	F6[1]	3.038(4)	O5	O6[1]	3.471(5)

O5	C6[2]	3.406(5)	O6	F2[3]
	3.253(5)			
O6	F3[3]	3.038(7)	O6	O1[3]
	3.598(5)			
O6	O5[4]	3.471(5)	O6	C5[3]
	3.198(5)			
O6	C6[3]	3.179(5)	C1	O2[1]
	3.268(7)			
C2	O2[1]	3.580(7)	C3	F1[4]
	3.487(5)			
C5	F2[2]	3.434(5)	C5	F2[3]
	3.221(5)			
C5	O6[3]	3.198(5)	C6	F2[2]
	3.287(6)			
C6	O5[2]	3.406(5)	C6	O6[3]
	3.179(5)			
C12	F6[1]	3.459(7)	C19	O2[11]
	3.516(6)			
C21	F6[8]	3.503(5)	C21	F8[8]
	3.489(5)			
C21	O2[11]	3.511(9)	C22	F4[5]
	3.557(7)			

Symmetry Operators:

(1) X,Y-1,Z
(2) -X+1,-Y+2,-Z+2
(3) -X+1,-Y+3,-Z+2
(4) X,Y+1,Z
(5) -X+1/2,Y+1/2,-Z+1/2+2
(6) -X+1/2,Y+1/2-1,-Z+1/2+2
(7) X,-Y+2,Z+1
(8) -X,-Y+2,-Z+1
(9) X,-Y+2,Z
(10) -X,Y+1,-Z+1/2+1
(11) -X,Y-1,-Z+1/2+1

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
S1	H1[1]	3.444	S1	H21B[2]	3.561
S2	H6A[3]	3.597	F1	H3[4]	2.489
F1	H5[4]	3.367	F1	H8[4]	3.134
F1	H15B[5]	3.452	F2	H4[3]	3.183
F2	H5[6]	2.646	F2	H6A[3]	2.534
F2	H6B[3]	3.580	F3	H1[1]	3.352
F6	H11[1]	3.190	F6	H12[1]	2.822
F6	H21A[7]	3.306	F6	H21C[7]	3.027
F7	H21A[7]	3.182	F7	H21C[7]	3.142
F8	H21A[7]	2.747	F8	H21C[7]	3.498
O1	H1[1]	3.548	O2	H1[1]	2.367
O2	H2[1]	3.475	O2	H19A[2]	2.908
O2	H19B[2]	3.258	O2	H21A[2]	3.076
O2	H21B[2]	3.145	O3	H19A[2]	3.591
O3	H19B[5]	2.772	O3	H21A[5]	2.905
O3	H21B[2]	3.206	O4	H12[1]	3.438
O5	H6A[3]	3.193	O5	H6B[3]	2.734
O6	H5[6]	2.788	O6	H6A[3]	3.336
O6	H6B[6]	2.425	C2	H15B[5]	3.387
C7	H15B[5]	3.376	C8	H11[1]	3.351
C8	H12[1]	3.349	C8	H13[5]	3.414
C8	H15A[2]	3.355	C8	H15B[5]	3.397

C9	H9[5]	3.361	C9	H11[1]
	3.165			
C9	H13[5]	2.983	C9	H14A[5]
	3.486			
C9	H14A[2]	3.568	C9	H14B[1]
	3.225			
C10	H13[5]	3.558	C10	H14A[5]
	3.152			
C11	H8[4]	3.348	C11	H9[4]
	3.375			
C11	H14A[5]	3.399	C12	H8[4]
	3.149			
C13	H14A[5]	3.550	C13	H14B[1]
	3.530			
C14	H9[4]	3.145	C14	H9[8]
	3.544			
C14	H13[4]	3.556	C14	H14A[5]
	3.147			
C15	H2[5]	3.524	C15	H8[8]
	3.347			
C15	H17B[4]	3.560	C16	H17B[4]
	3.516			
C16	H20A[9]	3.256	C17	H16[1]
	3.536			
C17	H17A[7]	3.413	C17	H20A[9]
	3.356			
C17	H20B[7]	3.278	C18	H14B[1]
	3.520			
C18	H20B[7]	3.199	C19	H2[5]
	3.582			

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
C20	H16^{9}	3.199	C20	H17A^{7}	3.573
C20	H18A^{9}	3.539	C20	H18B^{7}	3.354
C20	H20A^{9}	3.496	C21	H18A^{9}	3.204
C21	H18B^{7}	3.457	C23	H21A^{7}	3.279
C23	H21C^{7}	3.433	H1	S1^{4}	3.444
H1	F3^{4}	3.352	H1	O1^{4}	3.548
H1	O2^{4}	2.367	H2	O2^{4}	3.475
H2	C15^{5}	3.524	H2	C19^{5}	3.582
H2	H15B^{5}	2.661	H2	H19A^{5}	3.585
H2	H19B^{5}	2.832	H3	F1^{1}	2.489
H3	H12^{1}	3.346	H4	F2^{3}	3.183
H5	F1^{1}	3.367	H5	F2^{6}	2.646
H5	O6^{6}	2.788	H5	H6A^{1}	2.854
H6A	S2^{3}	3.597	H6A	F2^{3}	2.534
H6A	O5^{3}	3.193	H6A	O6^{3}	3.336
H6A	H5^{4}	2.854	H6B	F2^{3}	3.580
H6B	O5^{3}	2.734	H6B	O6^{6}	2.425
H8	F1^{1}	3.134	H8	C11^{1}	3.348
H8	C12^{1}	3.149	H8	C15^{2}	3.347
H8	H11^{1}	3.230	H8	H12^{1}	2.877
H8	H13^{5}	3.548	H8	H14A^{2}	3.337

H8	H15A[2]	2.631	H8	H15B[2]
	3.318			
H9	C9[5]	3.361	H9	C11[1]
	3.375			
H9	C14[1]	3.145	H9	C14[2]
	3.544			
H9	H9[5]	2.629	H9	H11[1]
	2.907			
H9	H13[5]	2.811	H9	H14A[1]
	2.830			
H9	H14A[2]	2.778	H9	H14B[1]
	2.548			
H9	H14B[2]	3.575	H9	H15A[2]
	3.526			
H11	F6[4]	3.190	H11	C8[4]
	3.351			
H11	C9[4]	3.165	H11	H8[4]
	3.230			
H11	H9[4]	2.907	H11	H21C[9]
	2.996			
H12	F6[4]	2.822	H12	O4[4]
	3.438			
H12	C8[4]	3.349	H12	H3[4]
	3.346			
H12	H8[4]	2.877	H13	C8[5]
	3.414			
H13	C9[5]	2.983	H13	C10[5]
	3.558			
H13	C14[1]	3.556	H13	H8[5]
	3.548			
H13	H9[5]	2.811	H13	H14A[1]
	3.515			

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H13	H14B[1]	2.935	H13	H15A[1]	3.155
H14A	C9[8]	3.568	H14A	C9[5]	3.486
H14A	C10[5]	3.152	H14A	C11[5]	3.399
H14A	C13[5]	3.550	H14A	C14[5]	3.147
H14A	H8[8]	3.337	H14A	H9[4]	2.830
H14A	H9[8]	2.778	H14A	H13[4]	3.515
H14A	H14A[5]	2.312	H14A	H14B[5]	3.375
H14B	C9[4]	3.225	H14B	C13[4]	3.530
H14B	C18[4]	3.520	H14B	H9[4]	2.548
H14B	H9[8]	3.575	H14B	H13[4]	2.935
H14B	H14A[5]	3.375	H14B	H18B[4]	2.800
H14B	H21C[9]	3.537	H15A	C8[8]	3.355
H15A	H8[8]	2.631	H15A	H9[8]	3.526
H15A	H13[4]	3.155	H15A	H17B[4]	2.871
H15B	F1[5]	3.452	H15B	C2[5]	3.387
H15B	C7[5]	3.376	H15B	C8[5]	3.397
H15B	H2[5]	2.661	H15B	H8[8]	3.318
H16	C17[4]	3.536	H16	C20[9]	3.199
H16	H17A[4]	3.506	H16	H17B[4]	2.930
H16	H18B[4]	3.060	H16	H20A[9]	2.469
H16	H20B[9]	3.103	H16	H21C[9]	3.495

H17A	C17[7]	3.413	H17A	C20[7]
	3.573			
H17A	H16[1]	3.506	H17A	H17A[7]
	2.443			
H17A	H20A[9]	2.907	H17A	H20B[7]
	2.637			
H17B	C15[1]	3.560	H17B	C16[1]
	3.516			
H17B	H15A[1]	2.871	H17B	H16[1]
	2.930			
H17B	H19A[1]	3.108	H17B	H20B[7]
	3.559			
H18A	C20[9]	3.539	H18A	C21[9]
	3.204			
H18A	H20A[9]	2.934	H18A	H20B[7]
	3.235			
H18A	H21B[9]	2.996	H18A	H21C[9]
	2.759			
H18B	C20[7]	3.354	H18B	C21[7]
	3.457			
H18B	H14B[1]	2.800	H18B	H16[1]
	3.060			
H18B	H20A[7]	3.521	H18B	H20B[7]
	2.657			
H18B	H21C[7]	2.808	H19A	O2[8]
	2.908			
H19A	O3[8]	3.591	H19A	H2[5]
	3.585			
H19A	H17B[4]	3.108	H19B	O2[8]
	3.258			
H19B	O3[5]	2.772	H19B	H2[5]
	2.832			

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H20A	C16[9]	3.256	H20A	C17[9]	3.356
H20A	C20[9]	3.496	H20A	H16[9]	2.469
H20A	H17A[9]	2.907	H20A	H18A[9]	2.934
H20A	H18B[7]	3.521	H20A	H20A[9]	2.804
H20A	H20B[9]	3.426	H20B	C17[7]	3.278
H20B	C18[7]	3.199	H20B	H16[9]	3.103
H20B	H17A[7]	2.637	H20B	H17B[7]	3.559
H20B	H18A[7]	3.235	H20B	H18B[7]	2.657
H20B	H20A[9]	3.426	H21A	F6[7]	3.306
H21A	F7[7]	3.182	H21A	F8[7]	2.747
H21A	O2[8]	3.076	H21A	O3[5]	2.905
H21A	C23[7]	3.279	H21B	S1[8]	3.561
H21B	O2[8]	3.145	H21B	O3[8]	3.206
H21B	H18A[9]	2.996	H21C	F6[7]	3.027
H21C	F7[7]	3.142	H21C	F8[7]	3.498
H21C	C23[7]	3.433	H21C	H11[9]	2.996
H21C	H14B[9]	3.537	H21C	H16[9]	3.495
H21C	H18A[9]	2.759	H21C	H18B[7]	2.808

Symmetry Operators:

(1) X,Y+1,Z
(2) -X,Y+1,-Z+1/2+1
(3) -X+1,-Y+2,-Z+2
(4) X,Y-1,Z
(5) -X,Y,-Z+1/2+1
(6) -X+1,-Y+3,-Z+2

(7) -X,-Y+2,-Z+1
(8) -X,Y-1,-Z+1/2+1
(9) -X,-Y+1,-Z+1

X-Ray data Collection for **19**

Data Collection

A colorless prism crystal of $C_{22}H_{27}F_5O_3S$ having approximate dimensions of 0.240 x 0.120 x 0.020 mm was mounted in a loop. All measurements were made on a Rigaku XtaLAB P200 diffractometer using multi-layer mirror monochromated Mo-Kα radiation.

Cell constants and an orientation matrix for data collection corresponded to a primitive triclinic cell with dimensions:

a = 5.4268(8) Å α = 78.369(6)°
b = 10.7827(15) Å β = 82.270(7)°
c = 19.506(3) Å γ = 79.893(6)°
V = 1094.8(3) $Å^3$

For Z = 2 and F.W. = 466.51, the calculated density is 1.415 g/cm^3. Based on a statistical analysis of intensity distribution, and the successful solution and refinement of the structure, the space group was determined to be:

P-1 (#2)

The data were collected at a temperature of -180 ± 1°C to a maximum 2θ value of 50.7°. Readout was performed in the 0.172 mm pixel mode.

Data Reduction

Of the 21593 reflections were collected, where 3957 were unique (R_{int} = 0.0699); equivalent reflections were merged. Data were collected and processed using CrystalClear (Rigaku). [1]

The linear absorption coefficient, μ, for Mo-Kα radiation is 2.111 cm^{-1}. An empirical absorption correction was applied which resulted in transmission factors ranging from 0.422 to 0.996. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by direct methods[2] and expanded using Fourier techniques. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined using the riding model. The final cycle of full-matrix least-squares refinement[3] on F^2 was based on 3957 observed reflections and 281

variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R1 = \Sigma \, ||Fo| - |Fc|| \, / \, \Sigma \, |Fo| = 0.0434$$

$$wR2 = [\Sigma (w (Fo^2 - Fc^2)^2) / \Sigma \, w(Fo^2)^2]^{1/2} = 0.1317$$

The goodness of fit[4] was 1.06. Unit weights were used. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.68 and -0.41 $e^-/Å^3$, respectively.

Neutral atom scattering factors were taken from International Tables for Crystallography (IT), Vol. C, Table 6.1.1.4 [5]. Anomalous dispersion effects were included in Fcalc[6]; the values for Δf' and Δf'' were those of Creagh and McAuley[7]. The values for the mass attenuation coefficients are those of Creagh and Hubbell[8]. All calculations were performed using the CrystalStructure[9] crystallographic software package except for refinement, which was performed using SHELXL2013[10].

References

(1) CrystalClear: Data Collection and Processing Software, Rigaku Corporation (1998-2014). Tokyo 196-8666, Japan.

(2) SHELXS2013: Sheldrick, G. M. (2008). Acta Cryst. A64, 112-122.

(3) Least Squares function minimized: (SHELXL2013)

$\Sigma w(F_o^2 - F_c^2)^2$ where w = Least Squares weights.

(4) Goodness of fit is defined as:

$[\Sigma w(F_o^2 - F_c^2)^2 / (N_o - N_v)]^{1/2}$

where: N_o = number of observations
N_v = number of variables

(5) International Tables for Crystallography, Vol.C (1992). Ed. A.J.C. Wilson, Kluwer Academic Publishers, Dordrecht, Netherlands, Table 6.1.1.4, pp. 572.

(6) Ibers, J. A. & Hamilton, W. C.; Acta Crystallogr., 17, 781 (1964).

(7) Creagh, D. C. & McAuley, W.J .; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.6.8, pages 219-222 (1992).

(8) Creagh, D. C. & Hubbell, J.H..; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.4.3, pages 200-206 (1992).

(9) CrystalStructure 4.1: Crystal Structure Analysis Package, Rigaku Corporation (2000-2014). Tokyo 196-8666, Japan.

(10) SHELXL2013: Sheldrick, G. M. (2008). Acta Cryst. A64, 112-122.

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$C_{22}H_{27}F_5O_3S$
Formula Weight	466.51
Crystal Color, Habit	colorless, prism
Crystal Dimensions	0.240 X 0.120 X 0.020 mm
Crystal System	triclinic
Lattice Type	Primitive
Lattice Parameters	a = 5.4268(8) Å b = 10.7827(15) Å c = 19.506(3) Å α = 78.369(6) ^{o} β = 82.270(7) ^{o} γ = 79.893(6) ^{o} V = 1094.8(3) $Å^3$
Space Group	P-1 (#2)
Z value	2
D_{calc}	1.415 g/cm^3
F_{000}	488.00
μ(MoKα)	2.111 cm^{-1}

B. Intensity Measurements

Diffractometer	XtaLAB P200
Radiation monochromated	MoKα (λ = 0.71075 Å) multi-layer mirror
Voltage, Current	45kV, 66mA
Temperature	-180.0°C
Detector Aperture	83.8 x 70.0 mm
Pixel Size	0.172 mm
$2\theta_{max}$	50.7°
No. of Reflections Measured	Total: 21593 Unique: 3957 (R_{int} = 0.0699)
Corrections	Lorentz-polarization Absorption (trans. factors: 0.422 - 0.996)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SHELXS2013)
Refinement	Full-matrix least-squares on F^2
Function Minimized	Σ w (Fo^2 - Fc^2)^2
Least Squares Weights	w = 1/ [$\sigma^2(Fo^2)$ + (0.0907 · P)^2 + 0.0000 · P] where P = (Max(Fo^2,0) + 2Fc^2)/3
$2\theta_{max}$ cutoff	50.7^o
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (All reflections)	3957
No. Variables	281
Reflection/Parameter Ratio	14.08
Residuals: R1 (I>2.00σ(I))	0.0434
Residuals: R (All reflections)	0.0495
Residuals: wR2 (All reflections)	0.1317
Goodness of Fit Indicator	1.061
Max Shift/Error in Final Cycle	0.000
Maximum peak in Final Diff. Map	0.68 $e^-/Å^3$
Minimum peak in Final Diff. Map	-0.41 $e^-/Å^3$

Table 1. Atomic coordinates and B_{iso}/B_{eq}

atom	x	y	z	B_{eq}
S1	0.72761(8)	0.52670(4)	0.30353(2)	1.749(13)
F1	0.77118(18)	0.51679(9)	0.46836(5)	1.97(2)
F2	0.35914(18)	0.17278(10)	0.49833(5)	2.00(2)
F3	1.0725(2)	0.45405(11)	0.20625(6)	3.22(3)
F4	0.7520(2)	0.58614(12)	0.16812(6)	3.26(3)
F5	0.7203(3)	0.38732(13)	0.20859(6)	3.68(3)
O1	0.8409(2)	0.39452(11)	0.34633(6)	1.65(2)
O2	0.4603(2)	0.54452(12)	0.31381(7)	2.14(3)
O3	0.8649(3)	0.62508(12)	0.30734(7)	2.41(3)
C1	0.7183(3)	0.34504(16)	0.41711(9)	1.55(3)
C2	0.8499(3)	0.38446(16)	0.47079(9)	1.66(3)
C3	0.7873(3)	0.31480(17)	0.54385(9)	1.82(3)
C4	0.6864(3)	0.20803(17)	0.55671(9)	1.86(3)
C5	0.6257(3)	0.14810(17)	0.49955(9)	1.72(3)
C6	0.7484(3)	0.19956(16)	0.42576(9)	1.55(3)
C7	0.6423(3)	0.15118(16)	0.36975(9)	1.55(3)
C8	0.7544(3)	0.03473(16)	0.35036(9)	1.65(3)
C9	0.6532(3)	-0.01673(16)	0.30252(9)	1.67(3)
C10	0.4350(3)	0.04633(16)	0.27227(9)	1.56(3)
C11	0.3245(3)	0.16309(17)	0.29195(9)	1.78(3)
C12	0.4258(3)	0.21485(16)	0.33916(9)	1.76(3)
C13	0.3176(3)	-0.00191(16)	0.21861(9)	1.64(3)
C14	0.4125(3)	0.05691(17)	0.14320(9)	1.82(3)
C15	0.2828(3)	0.01419(17)	0.08901(9)	1.87(3)
C16	0.3219(3)	-0.13229(17)	0.09652(9)	1.72(3)
C17	0.2274(3)	-0.19087(17)	0.17186(9)	1.86(3)
C18	0.3538(3)	-0.14855(17)	0.22692(9)	1.87(3)
C19	0.1974(3)	-0.17344(18)	0.04088(9)	1.98(3)
C20	0.2877(4)	-0.31148(19)	0.03039(11)	2.57(4)
C21	0.1683(4)	-0.3456(2)	-0.02824(11)	3.15(4)
C22	0.8252(4)	0.48475(19)	0.21588(10)	2.40(4)

$B_{eq} = 8/3\ \pi^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$

Table 2. Atomic coordinates and B_{iso} involving hydrogen atoms

atom	x	y	z	B_{iso}
H1	0.53579	0.38219	0.42115	1.860
H2	1.03600	0.36814	0.45806	1.996
H3	0.82109	0.34838	0.58246	2.188
H4	0.65129	0.16791	0.60424	2.227
H5	0.68035	0.05342	0.51104	2.069
H6	0.93248	0.16566	0.42408	1.855
H8	0.90253	-0.01016	0.37022	1.977
H9	0.73353	-0.09600	0.29018	1.999
H11	0.17586	0.20800	0.27233	2.137
H12	0.34696	0.29478	0.35093	2.114
H13	0.13240	0.02833	0.22504	1.968
H14A	0.37927	0.15151	0.13729	2.189
H14B	0.59662	0.03041	0.13514	2.189
H15A	0.35031	0.05205	0.04113	2.240
H15B	0.10034	0.04661	0.09485	2.240
H16	0.50687	-0.16265	0.08923	2.058
H17A	0.04305	-0.16487	0.17963	2.233
H17B	0.26134	-0.28546	0.17769	2.233
H18A	0.53600	-0.18216	0.22230	2.239
H18B	0.28202	-0.18529	0.27454	2.239
H19A	0.22864	-0.11534	-0.00457	2.373
H19B	0.01338	-0.16272	0.05414	2.373
H20A	0.47285	-0.32407	0.01927	3.085
H20B	0.24765	-0.37044	0.07488	3.085
H21A	0.20773	-0.28780	-0.07251	3.784
H21B	-0.01462	-0.33681	-0.01664	3.784
H21C	0.23475	-0.43423	-0.03331	3.784

Table 3. Anisotropic displacement parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
S1	0.0245(3)	0.0181(3)	0.0226(3)	-0.00300(18)	-0.00269(17)	-0.00092(18)
F1	0.0284(5)	0.0163(5)	0.0322(6)	-0.0041(4)	-0.0063(4)	-0.0066(4)
F2	0.0219(5)	0.0275(6)	0.0270(6)	-0.0073(4)	-0.0018(4)	-0.0029(4)
F3	0.0398(7)	0.0386(7)	0.0333(7)	0.0053(5)	0.0085(5)	0.0008(5)
F4	0.0506(7)	0.0397(7)	0.0241(6)	0.0058(6)	-0.0033(5)	0.0045(5)
F5	0.0716(9)	0.0435(8)	0.0306(7)	-0.0210(7)	-0.0004(6)	-0.0134(6)
O1	0.0225(6)	0.0185(6)	0.0207(6)	-0.0036(5)	-0.0007(5)	-0.0021(5)
O2	0.0252(7)	0.0251(7)	0.0283(7)	-0.0007(5)	-0.0032(5)	-0.0011(5)
O3	0.0355(7)	0.0193(7)	0.0371(8)	-0.0091(6)	-0.0054(6)	-0.0004(6)
C1	0.0201(8)	0.0192(9)	0.0192(8)	-0.0051(7)	-0.0006(7)	-0.0014(7)
C2	0.0208(8)	0.0158(8)	0.0279(9)	-0.0031(7)	-0.0030(7)	-0.0063(7)
C3	0.0243(9)	0.0238(9)	0.0221(9)	-0.0019(7)	-0.0045(7)	-0.0064(7)
C4	0.0251(9)	0.0248(9)	0.0202(9)	-0.0045(7)	-0.0023(7)	-0.0025(7)
C5	0.0205(8)	0.0193(9)	0.0253(9)	-0.0043(7)	-0.0041(7)	-0.0008(7)
C6	0.0205(8)	0.0174(9)	0.0216(9)	-0.0035(7)	-0.0039(7)	-0.0037(7)
C7	0.0214(8)	0.0189(8)	0.0193(8)	-0.0076(7)	0.0001(6)	-0.0022(7)
C8	0.0204(8)	0.0207(9)	0.0207(9)	-0.0038(7)	-0.0023(7)	-0.0012(7)
C9	0.0225(8)	0.0185(9)	0.0226(9)	-0.0046(7)	0.0004(7)	-0.0050(7)
C10	0.0221(8)	0.0189(9)	0.0181(8)	-0.0075(7)	0.0004(6)	-0.0008(7)
C11	0.0212(8)	0.0211(9)	0.0248(9)	-0.0026(7)	-0.0049(7)	-0.0018(7)
C12	0.0235(9)	0.0168(8)	0.0271(9)	-0.0034(7)	-0.0025(7)	-0.0050(7)
C13	0.0217(8)	0.0205(9)	0.0205(9)	-0.0050(7)	-0.0021(7)	-0.0033(7)

C14	0.0283(9)	0.0195(9)	0.0223(9)	-0.0073(7)	-0.0039(7)	-0.0016(7)
C15	0.0286(9)	0.0220(9)	0.0212(9)	-0.0069(7)	-0.0053(7)	-0.0017(7)
C16	0.0225(8)	0.0227(9)	0.0209(9)	-0.0050(7)	-0.0025(7)	-0.0045(7)
C17	0.0280(9)	0.0203(9)	0.0236(9)	-0.0072(7)	-0.0035(7)	-0.0034(7)
C18	0.0307(9)	0.0198(9)	0.0212(9)	-0.0069(7)	-0.0044(7)	-0.0016(7)
C19	0.0269(9)	0.0268(10)	0.0226(9)	-0.0067(7)	-0.0032(7)	-0.0046(8)
C20	0.0412(11)	0.0293(10)	0.0299(10)	-0.0057(9)	-0.0067(8)	-0.0095(8)
C21	0.0564(14)	0.0333(12)	0.0365(12)	-0.0133(10)	-0.0084(10)	-0.0133(9)
C22	0.0369(10)	0.0259(10)	0.0245(10)	-0.0012(8)	-0.0001(8)	-0.0010(8)

The general temperature factor expression: $\exp(-2\pi^2(a^{*2}U_{11}h^2 + b^{*2}U_{22}k^2 + c^{*2}U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$

Table 4. Bond lengths (Å)

atom	atom	distance	atom	atom	distance
S1	O1	1.5675(11)	S1	O2	1.4216(13)
S1	O3	1.4177(16)	S1	C22	1.842(2)
F1	C2	1.4089(19)	F2	C5	1.4265(19)
F3	C22	1.322(2)	F4	C22	1.326(2)
F5	C22	1.319(3)	O1	C1	1.4880(19)
C1	C2	1.508(3)	C1	C6	1.525(2)
C2	C3	1.496(2)	C3	C4	1.325(3)
C4	C5	1.495(3)	C5	C6	1.541(2)
C6	C7	1.517(3)	C7	C8	1.397(2)
C7	C12	1.396(2)	C8	C9	1.391(3)
C9	C10	1.399(2)	C10	C11	1.400(2)
C10	C13	1.512(3)	C11	C12	1.383(3)
C13	C14	1.538(2)	C13	C18	1.537(3)
C14	C15	1.528(3)	C15	C16	1.536(3)
C16	C17	1.536(2)	C16	C19	1.526(3)
C17	C18	1.529(3)	C19	C20	1.529(3)
C20	C21	1.522(3)			

Table 5. Bond lengths involving hydrogens (Å)

atom	atom	distance	atom	atom	distance
C1	H1	1.000	C2	H2	1.000
C3	H3	0.950	C4	H4	0.950
C5	H5	1.000	C6	H6	1.000
C8	H8	0.950	C9	H9	0.950
C11	H11	0.950	C12	H12	0.950
C13	H13	1.000	C14	H14A	0.990
C14	H14B	0.990	C15	H15A	0.990
C15	H15B	0.990	C16	H16	1.000
C17	H17A	0.990	C17	H17B	0.990
C18	H18A	0.990	C18	H18B	0.990
C19	H19A	0.990	C19	H19B	0.990
C20	H20A	0.990	C20	H20B	0.990
C21	H21A	0.980	C21	H21B	0.980
C21	H21C	0.980			

Table 6. Bond angles (°)

atom	atom	atom	angle	atom	atom	atom	angle
O1	S1	O2	110.66(7)	O1	S1	O3	111.56(8)
O1	S1	C22	96.07(8)	O2	S1	O3	121.58(8)
O2	S1	C22	107.83(9)	O3	S1	C22	105.81(9)
S1	O1	C1	119.99(9)	O1	C1	C2	107.41(14)
O1	C1	C6	107.68(13)	C2	C1	C6	111.28(14)
F1	C2	C1	107.27(13)	F1	C2	C3	108.97(14)
C1	C2	C3	112.49(15)	C2	C3	C4	122.46(18)
C3	C4	C5	122.72(15)	F2	C5	C4	107.51(13)
F2	C5	C6	108.40(13)	C4	C5	C6	114.24(15)
C1	C6	C5	107.69(14)	C1	C6	C7	114.89(14)
C5	C6	C7	110.66(15)	C6	C7	C8	119.56(15)
C6	C7	C12	122.54(15)	C8	C7	C12	117.79(18)
C7	C8	C9	121.25(15)	C8	C9	C10	121.07(16)
C9	C10	C11	117.22(18)	C9	C10	C13	124.10(16)
C11	C10	C13	118.66(15)	C10	C11	C12	121.80(15)
C7	C12	C11	120.87(16)	C10	C13	C14	111.11(15)
C10	C13	C18	114.44(14)	C14	C13	C18	109.76(14)
C13	C14	C15	111.14(15)	C14	C15	C16	112.03(14)
C15	C16	C17	109.00(15)	C15	C16	C19	111.47(14)
C17	C16	C19	112.73(15)	C16	C17	C18	112.03(15)
C13	C18	C17	111.77(14)	C16	C19	C20	114.87(15)
C19	C20	C21	112.83(16)	S1	C22	F3	110.52(15)

S1	C22	F4	108.00(13)	S1	C22	F5
	110.74(13)					
F3	C22	F4	109.21(15)	F3	C22	F5
	108.93(16)					
F4	C22	F5	109.42(17)			

Table 7. Bond angles involving hydrogens (°)

atom	atom	atom	angle	atom	atom	atom	angle
O1	C1	H1	110.1	C2	C1	H1	110.1
C6	C1	H1	110.1	F1	C2	H2	109.3
C1	C2	H2	109.4	C3	C2	H2	109.3
C2	C3	H3	118.8	C4	C3	H3	118.8
C3	C4	H4	118.6	C5	C4	H4	118.6
F2	C5	H5	108.9	C4	C5	H5	108.9
C6	C5	H5	108.9	C1	C6	H6	107.8
C5	C6	H6	107.8	C7	C6	H6	107.8
C7	C8	H8	119.4	C9	C8	H8	119.4
C8	C9	H9	119.5	C10	C9	H9	119.5
C10	C11	H11	119.1	C12	C11	H11	119.1
C7	C12	H12	119.6	C11	C12	H12	119.6
C10	C13	H13	107.0	C14	C13	H13	107.0
C18	C13	H13	107.0	C13	C14	H14A	109.4
C13	C14	H14B	109.4	C15	C14	H14A	109.4
C15	C14	H14B	109.4	H14A	C14	H14B	108.0
C14	C15	H15A	109.2	C14	C15	H15B	109.2
C16	C15	H15A	109.2	C16	C15	H15B	109.2
H15A	C15	H15B	107.9	C15	C16	H16	107.8
C17	C16	H16	107.8	C19	C16	H16	107.8
C16	C17	H17A	109.2	C16	C17	H17B	109.2
C18	C17	H17A	109.2	C18	C17	H17B	109.2

H17A	C17	H17B	107.9	C13	C18	H18A
	109.3					
C13	C18	H18B	109.3	C17	C18	H18A
	109.3					
C17	C18	H18B	109.3	H18A	C18	H18B
	107.9					
C16	C19	H19A	108.6	C16	C19	H19B
	108.6					
C20	C19	H19A	108.5	C20	C19	H19B
	108.5					
H19A	C19	H19B	107.5	C19	C20	H20A
	109.0					
C19	C20	H20B	109.0	C21	C20	H20A
	109.0					
C21	C20	H20B	109.0	H20A	C20	H20B
	107.8					
C20	C21	H21A	109.5	C20	C21	H21B
	109.5					
C20	C21	H21C	109.5	H21A	C21	H21B
	109.5					
H21A	C21	H21C	109.5	H21B	C21	H21C
	109.5					

Table 8. Torsion Angles(º)
(Those having bond angles > 160 or < 20 degrees are excluded.)

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
O2	S1	O1	C1	-36.91(13)	O3	S1	O1	C1	101.75(10)
O1	S1	C22	F3	-59.52(13)	O1	S1	C22	F4	-178.92(12)
O1	S1	C22	F5	61.30(12)	C22	S1	O1	C1	-148.57(11)
O2	S1	C22	F3	-173.53(11)	O2	S1	C22	F4	67.07(14)
O2	S1	C22	F5	-52.72(13)	O3	S1	C22	F3	54.96(13)
O3	S1	C22	F4	-64.45(14)	O3	S1	C22	F5	175.77(11)
S1	O1	C1	C2	-94.66(12)	S1	O1	C1	C6	145.39(10)
O1	C1	C2	F1	73.95(14)	O1	C1	C2	C3	-166.23(10)
O1	C1	C6	C5	179.01(11)	O1	C1	C6	C7	-57.18(16)
C2	C1	C6	C5	61.54(16)	C2	C1	C6	C7	-174.66(11)
C6	C1	C2	F1	-168.42(11)	C6	C1	C2	C3	-48.60(16)
F1	C2	C3	C4	136.10(14)	C1	C2	C3	C4	17.3(2)
C2	C3	C4	C5	-0.2(2)	C3	C4	C5	F2	-105.70(17)
C3	C4	C5	C6	14.6(2)	F2	C5	C6	C1	75.94(17)
F2	C5	C6	C7	-50.39(16)	C4	C5	C6	C1	-43.90(17)
C4	C5	C6	C7	-170.23(11)	C1	C6	C7	C8	149.06(13)
C1	C6	C7	C12	-34.8(2)	C5	C6	C7	C8	-88.73(16)
C5	C6	C7	C12	87.38(16)	C6	C7	C8	C9	175.89(13)
C6	C7	C12	C11	-175.34(13)	C8	C7	C12	C11	0.8(2)
C12	C7	C8	C9	-0.4(2)	C7	C8	C9	C10	-0.2(2)
C8	C9	C10	C11	0.3(2)	C8	C9	C10	C13	178.42(13)

C9	C10	C11	C12	0.2(2)	C9	C10	C13	C14	-93.48(17)
C9	C10	C13	C18	31.5(2)	C11	C10	C13	C14	84.63(17)
C11	C10	C13	C18	-150.37(13)	C13	C10	C11	C12	-178.09(13)
C10	C11	C12	C7	-0.7(2)	C10	C13	C14	C15	-176.76(11)
C10	C13	C18	C17	179.10(12)	C14	C13	C18	C17	-55.19(17)
C18	C13	C14	C15	55.65(17)	C13	C14	C15	C16	-57.70(16)
C14	C15	C16	C17	56.46(17)	C14	C15	C16	C19	-178.47(12)
C15	C16	C17	C18	-55.62(17)	C15	C16	C19	C20	163.15(12)
C17	C16	C19	C20	-73.88(17)	C19	C16	C17	C18	-179.95(12)
C16	C17	C18	C13	56.43(17)	C16	C19	C20	C21	-176.98(13)

Table 9. Intramolecular contacts less than 3.60 Å

atom	atom	distance	atom	atom	distance
S1	F1	3.2345(12)	S1	C2	3.4125(18)
F1	O1	2.8975(17)	F1	O2	3.5849(18)
F1	O3	3.1285(16)	F1	C4	3.504(2)
F2	C1	3.002(2)	F2	C3	3.299(2)
F2	C7	2.7892(19)	F2	C12	3.027(2)
F3	O1	2.8478(16)	F3	O3	2.9492(19)
F4	O2	3.0511(17)	F4	O3	2.987(2)
F5	O1	2.8685(18)	F5	O2	2.9767(19)
F5	C11	3.522(2)	F5	C12	3.246(2)
O1	C7	2.938(2)	O1	C12	3.252(2)
O2	C1	2.933(2)	O2	C12	3.524(2)
O3	C1	3.480(2)	C1	C4	2.823(2)
C1	C12	3.016(3)	C2	C5	2.936(3)
C3	C6	2.876(3)	C5	C8	3.323(3)
C5	C12	3.349(3)	C7	C10	2.837(3)
C8	C11	2.757(2)	C9	C12	2.769(2)
C9	C14	3.424(3)	C9	C18	3.022(3)
C11	C14	3.276(3)	C13	C16	2.995(3)
C14	C17	2.941(3)	C15	C18	2.933(2)
C17	C20	3.235(3)			

Table 10. Intramolecular contacts less than 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
S1	H1	2.695	S1	H12	3.427
F1	H1	2.468	F1	H3	2.585
F2	H1	2.699	F2	H4	2.755
F2	H6	3.245	F2	H12	2.915
F5	H12	3.300	O1	H2	2.493
O1	H6	2.627	O1	H12	3.043
O2	H1	2.473	O2	H12	2.798
C1	H3	3.355	C1	H5	3.330
C1	H12	2.718	C2	H4	3.327
C2	H6	2.643	C3	H1	2.819
C3	H5	3.177	C3	H6	3.040
C4	H1	3.038	C4	H2	3.018
C4	H6	2.832	C5	H1	2.676
C5	H2	3.441	C5	H3	3.328
C5	H8	3.369	C5	H12	3.418
C6	H2	2.803	C6	H4	3.407
C6	H8	2.658	C6	H12	2.720
C7	H1	2.809	C7	H5	2.767
C7	H9	3.280	C7	H11	3.267
C8	H5	3.149	C8	H6	2.582
C8	H12	3.253	C9	H11	3.250
C9	H13	3.298	C9	H14B	3.249

C9	H18A	2.797	C9	H18B
	3.105			
C10	H8	3.279	C10	H12
	3.282			
C10	H14A	2.683	C10	H14B
	2.730			
C10	H18A	2.770	C10	H18B
	2.757			
C11	H9	3.252	C11	H13
	2.562			
C11	H14A	3.017	C12	H1
	2.823			
C12	H6	3.308	C12	H8
	3.252			
C13	H9	2.747	C13	H11
	2.635			
C13	H15A	3.379	C13	H15B
	2.744			
C13	H16	3.297	C13	H17A
	2.766			
C13	H17B	3.385	C14	H9
	3.522			
C14	H11	3.271	C14	H16
	2.722			
C14	H17A	3.295	C14	H18A
	2.746			
C14	H18B	3.369	C15	H13
	2.696			
C15	H17A	2.726	C15	H17B
	3.357			

Table 10. Intramolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
C15	H18A	3.294	C15	H19A	2.586
C15	H19B	2.830	C16	H13	3.282
C16	H14A	3.388	C16	H14B	2.761
C16	H18A	2.770	C16	H18B	3.387
C16	H20A	2.747	C16	H20B	2.789
C17	H13	2.710	C17	H14B	3.290
C17	H15A	3.357	C17	H15B	2.720
C17	H19A	3.372	C17	H19B	2.651
C17	H20A	3.561	C17	H20B	2.947
C18	H9	2.732	C18	H14A	3.370
C18	H14B	2.744	C18	H15B	3.272
C18	H16	2.729	C19	H15A	2.704
C19	H15B	2.727	C19	H17A	2.739
C19	H17B	2.739	C19	H21A	2.731
C19	H21B	2.746	C19	H21C	3.377
C20	H16	2.662	C20	H17A	3.580
C20	H17B	2.925	C21	H19A	2.697
C21	H19B	2.741	H1	H2	2.872
H1	H6	2.882	H1	H12	2.248
H2	H3	2.532	H2	H6	2.576
H3	H4	2.238	H4	H5	2.369

H5	H6	2.312	H5	H8
	3.008			
H6	H8	2.384	H6	H12
	3.597			
H8	H9	2.324	H9	H13
	3.598			
H9	H14B	3.185	H9	H18A
	2.234			
H9	H18B	2.859	H11	H12
	2.314			
H11	H13	2.366	H11	H14A
	2.856			
H13	H14A	2.362	H13	H14B
	2.870			
H13	H15A	3.596	H13	H15B
	2.534			
H13	H17A	2.564	H13	H18A
	2.868			
H13	H18B	2.357	H14A	H15A
	2.375			
H14A	H15B	2.360	H14B	H15A
	2.362			
H14B	H15B	2.872	H14B	H16
	2.569			
H14B	H18A	2.607	H15A	H16
	2.369			
H15A	H19A	2.390	H15A	H19B
	3.146			
H15B	H16	2.874	H15B	H17A
	2.573			
H15B	H19A	2.805	H15B	H19B
	2.673			

Table 10. Intramolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom
	distance			
H16	H17A	2.873	H16	H17B
	2.363			
H16	H18A	2.586	H16	H19A
	2.446			
H16	H19B	2.852	H16	H20A
	2.463			
H16	H20B	2.923	H17A	H18A
	2.871			
H17A	H18B	2.354	H17A	H19A
	3.557			
H17A	H19B	2.470	H17A	H20B
	3.267			
H17B	H18A	2.354	H17B	H18B
	2.381			
H17B	H19B	2.886	H17B	H20A
	3.231			
H17B	H20B	2.382	H19A	H20A
	2.395			
H19A	H20B	2.864	H19A	H21A
	2.517			
H19A	H21B	2.986	H19A	H21C
	3.588			
H19B	H20A	2.864	H19B	H20B
	2.360			
H19B	H21A	3.030	H19B	H21B
	2.580			
H20A	H21A	2.375	H20A	H21B
	2.859			
H20A	H21C	2.349	H20B	H21A
	2.859			
H20B	H21B	2.363	H20B	H21C
	2.361			

Table 11. Intermolecular contacts less than 3.60 Å

atom	atom	distance	atom	atom	distance
F1	F1[1]	3.0878(14)	F1	F1[2]	2.8551(15)
F1	F2[1]	3.4712(16)	F1	C1[1]	3.5529(19)
F1	C2[1]	3.4753(19)	F1	C2[2]	2.950(2)
F1	C3[1]	3.2600(19)	F1	C3[2]	3.209(2)
F1	C4[1]	3.5165(19)	F2	F1[1]	3.4712(16)
F2	C2[3]	3.2902(18)	F2	C3[3]	3.3020(19)
F2	C5[4]	3.438(2)	F2	C8[4]	3.3807(19)
F3	F5[5]	3.4672(19)	F3	O2[5]	3.538(2)
F3	C11[5]	3.391(2)	F4	C17[6]	3.392(2)
F4	C18[6]	3.557(2)	F5	F3[3]	3.4672(19)
F5	C21[7]	3.594(3)	O1	C11[5]	3.501(2)
O1	C12[5]	3.417(2)	O2	F3[3]	3.538(2)
O2	O3[3]	3.2081(19)	O2	C3[1]	3.434(2)
O2	C18[6]	3.390(2)	O3	O2[5]	3.2081(19)
O3	C17[8]	3.557(2)	C1	F1[1]	3.5529(19)
C2	F1[1]	3.4753(19)	C2	F1[2]	2.950(2)
C2	F2[5]	3.2902(18)	C3	F1[1]	3.2600(19)
C3	F1[2]	3.209(2)	C3	F2[5]	3.3020(19)
C3	O2[1]	3.434(2)	C4	F1[1]	3.5165(19)
C4	C9[4]	3.589(2)	C5	F2[4]	3.438(2)
C8	F2[4]	3.3807(19)	C8	C11[5]	3.564(2)
C9	C4[4]	3.589(2)	C11	F3[3]	3.391(2)

C11	O1[3]	3.501(2)	C11	C8[3]
	3.564(2)			
C12	O1[3]	3.417(2)	C17	F4[9]
	3.392(2)			
C17	O3[10]	3.557(2)	C18	F4[9]
	3.557(2)			
C18	O2[9]	3.390(2)	C21	F5[7]
	3.594(3)			

Symmetry Operators:

(1) -X+1,-Y+1,-Z+1
(2) -X+2,-Y+1,-Z+1
(3) X-1,Y,Z
(4) -X+1,-Y,-Z+1
(5) X+1,Y,Z
(6) X,Y+1,Z
(7) -X+1,-Y,-Z
(8) X+1,Y+1,Z
(9) X,Y-1,Z
(10) X-1,Y-1,Z

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
S1	H18A[1]	3.277	S1	H18B[1]	3.583
F1	H1[2]	2.820	F1	H2[3]	2.504
F1	H3[2]	3.473	F1	H3[3]	2.820
F2	H2[4]	2.557	F2	H3[4]	3.582
F2	H5[5]	2.530	F2	H6[4]	2.911
F2	H8[5]	3.122	F3	H11[6]	2.703
F3	H12[6]	3.378	F3	H17B[7]	3.078
F3	H20B[7]	3.012	F4	H16[1]	3.013
F4	H17A[7]	3.403	F4	H17B[1]	2.778
F4	H17B[7]	3.343	F4	H18A[1]	2.905
F4	H19B[7]	3.516	F4	H20A[1]	3.346
F4	H20B[1]	3.401	F4	H20B[7]	3.089
F4	H21C[8]	3.354	F5	H11[6]	3.095
F5	H21A[8]	3.012	F5	H21C[8]	3.336
O1	H11[6]	2.935	O1	H12[6]	2.774
O2	H3[2]	2.693	O2	H17B[1]	3.113
O2	H18A[1]	3.185	O2	H18B[1]	2.885
O3	H3[3]	3.002	O3	H9[1]	2.928
O3	H17A[7]	3.181	O3	H17B[7]	3.199
O3	H18A[1]	2.892	O3	H18B[1]	3.513
O3	H18B[7]	3.228	C1	H12[6]	3.471
C2	H2[3]	3.425	C2	H3[3]	3.559

C2	H12[6]	3.461	C3	H1[2]
	3.575			
C3	H18B[5]	3.532	C4	H8[9]
	3.085			
C4	H18B[5]	3.277	C5	H5[5]
	3.010			
C6	H11[6]	3.527	C7	H5[5]
	3.396			
C7	H11[6]	3.323	C8	H4[5]
	3.285			
C8	H5[5]	3.437	C8	H11[6]
	3.234			
C8	H13[6]	2.976	C9	H4[5]
	2.763			
C9	H13[6]	2.894	C9	H17A[6]
	3.430			
C10	H4[5]	3.032	C10	H8[4]
	3.322			
C11	H6[4]	3.115	C11	H8[4]
	3.236			
C12	H2[4]	3.404	C12	H6[4]
	3.024			
C13	H4[5]	3.568	C13	H8[4]
	3.464			
C13	H9[4]	3.520	C14	H19A[8]
	3.135			
C14	H21A[8]	3.464	C15	H15A[8]
	3.129			
C15	H19A[10]	3.324	C15	H19A[8]
	3.170			

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
C15	H19B[10]	3.383	C16	H15A[8]	3.073
C17	H9[4]	3.447	C18	H4[5]	3.254
C18	H9[4]	3.416	C19	H14B[8]	3.585
C19	H15A[10]	3.490	C19	H15A[8]	3.086
C19	H15B[10]	3.226	C19	H19A[10]	3.545
C20	H21C[11]	3.423	C21	H15B[10]	3.373
C21	H21B[12]	3.575	C21	H21C[12]	3.439
C22	H11[6]	3.314	H1	F1[2]	2.820
H1	C3[2]	3.575	H1	H2[4]	2.734
H1	H3[2]	3.190	H2	F1[3]	2.504
H2	F2[6]	2.557	H2	C2[3]	3.425
H2	C12[6]	3.404	H2	H1[6]	2.734
H2	H2[3]	3.497	H2	H3[3]	3.204
H2	H12[6]	2.663	H3	F1[2]	3.473
H3	F1[3]	2.820	H3	F2[6]	3.582
H3	O2[2]	2.693	H3	O3[3]	3.002
H3	C2[3]	3.559	H3	H1[2]	3.190
H3	H2[3]	3.204	H3	H18B[5]	3.022
H4	C8[5]	3.285	H4	C9[5]	2.763
H4	C10[5]	3.032	H4	C13[5]	3.568
H4	C18[5]	3.254	H4	H8[9]	2.746

H4	H9[5]	2.823	H4	H18A[5]
	3.426			
H4	H18B[5]	2.488	H5	F2[5]
	2.530			
H5	C5[5]	3.010	H5	C7[5]
	3.396			
H5	C8[5]	3.437	H5	H5[5]
	2.562			
H5	H5[9]	3.456	H5	H6[9]
	3.060			
H5	H8[9]	3.363	H6	F2[6]
	2.911			
H6	C11[6]	3.115	H6	C12[6]
	3.024			
H6	H5[9]	3.060	H6	H11[6]
	3.055			
H6	H12[6]	2.910	H8	F2[5]
	3.122			
H8	C4[9]	3.085	H8	C10[6]
	3.322			
H8	C11[6]	3.236	H8	C13[6]
	3.464			
H8	H4[9]	2.746	H8	H5[9]
	3.363			
H8	H11[6]	3.167	H8	H13[6]
	2.916			
H8	H18B[6]	3.211	H9	O3[13]
	2.928			
H9	C13[6]	3.520	H9	C17[6]
	3.447			
H9	C18[6]	3.416	H9	H4[5]
	2.823			

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H9	H13[6]	2.773	H9	H17A[6]	2.694
H9	H18B[6]	2.958	H11	F3[4]	2.703
H11	F5[4]	3.095	H11	O1[4]	2.935
H11	C6[4]	3.527	H11	C7[4]	3.323
H11	C8[4]	3.234	H11	C22[4]	3.314
H11	H6[4]	3.055	H11	H8[4]	3.167
H12	F3[4]	3.378	H12	O1[4]	2.774
H12	C1[4]	3.471	H12	C2[4]	3.461
H12	H2[4]	2.663	H12	H6[4]	2.910
H13	C8[4]	2.976	H13	C9[4]	2.894
H13	H8[4]	2.916	H13	H9[4]	2.773
H13	H14B[4]	3.586	H14A	H19A[8]	3.165
H14A	H20A[8]	3.318	H14A	H21A[10]	3.549
H14A	H21A[8]	2.903	H14A	H21B[10]	3.379
H14B	C19[8]	3.585	H14B	H13[6]	3.586
H14B	H15B[6]	2.767	H14B	H17A[6]	3.016
H14B	H19A[8]	2.634	H14B	H19B[6]	3.265
H14B	H21A[8]	3.105	H15A	C15[8]	3.129
H15A	C16[8]	3.073	H15A	C19[10]	3.490
H15A	C19[8]	3.086	H15A	H15A[8]	2.421
H15A	H16[8]	2.657	H15A	H19A[10]	3.240

H15A	H19A[8]	2.471	H15A	H19B[10]
	2.835			
H15A	H20A[8]	3.194	H15A	H21B[10]
	3.264			
H15B	C19[10]	3.226	H15B	C21[10]
	3.373			
H15B	H14B[4]	2.767	H15B	H19A[10]
	2.587			
H15B	H19B[10]	3.023	H15B	H21A[10]
	2.822			
H15B	H21B[10]	3.177	H16	F4[13]
	3.013			
H16	H15A[8]	2.657	H16	H17A[6]
	3.594			
H16	H19A[8]	3.557	H16	H19B[6]
	2.742			
H16	H21B[6]	3.598	H17A	F4[14]
	3.403			
H17A	O3[14]	3.181	H17A	C9[4]
	3.430			
H17A	H9[4]	2.694	H17A	H14B[4]
	3.016			
H17A	H16[4]	3.594	H17A	H18A[4]
	2.793			
H17B	F3[14]	3.078	H17B	F4[14]
	3.343			
H17B	F4[13]	2.778	H17B	O2[13]
	3.113			
H17B	O3[14]	3.199	H18A	S1[13]
	3.277			
H18A	F4[13]	2.905	H18A	O2[13]
	3.185			

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom
	distance			
H18A	O3[13]	2.892	H18A	H4[5]
	3.426			
H18A	H17A[6]	2.793	H18B	S1[13]
	3.583			
H18B	O2[13]	2.885	H18B	O3[14]
	3.228			
H18B	O3[13]	3.513	H18B	C3[5]
	3.532			
H18B	C4[5]	3.277	H18B	H3[5]
	3.022			
H18B	H4[5]	2.488	H18B	H8[4]
	3.211			
H18B	H9[4]	2.958	H19A	C14[8]
	3.135			
H19A	C15[10]	3.324	H19A	C15[8]
	3.170			
H19A	C19[10]	3.545	H19A	H14A[8]
	3.165			
H19A	H14B[8]	2.634	H19A	H15A[10]
	3.240			
H19A	H15A[8]	2.471	H19A	H15B[10]
	2.587			
H19A	H16[8]	3.557	H19A	H19A[10]
	3.205			
H19A	H19B[10]	3.072	H19B	F4[14]
	3.516			
H19B	C15[10]	3.383	H19B	H14B[4]
	3.265			
H19B	H15A[10]	2.835	H19B	H15B[10]
	3.023			
H19B	H16[4]	2.742	H19B	H19A[10]
	3.072			
H20A	F4[13]	3.346	H20A	H14A[8]
	3.318			
H20A	H15A[8]	3.194	H20A	H21B[6]
	2.762			
H20A	H21C[11]	2.786	H20B	F3[14]
	3.012			
H20B	F4[14]	3.089	H20B	F4[13]
	3.401			
H20B	H21C[11]	3.314	H21A	F5[8]
	3.012			

H21A	C14[8]	3.464	H21A	H14A[10]
	3.549			
H21A	H14A[8]	2.903	H21A	H14B[8]
	3.105			
H21A	H15B[10]	2.822	H21B	C21[12]
	3.575			
H21B	H14A[10]	3.379	H21B	H15A[10]
	3.264			
H21B	H15B[10]	3.177	H21B	H16[4]
	3.598			
H21B	H20A[4]	2.762	H21B	H21B[12]
	3.427			
H21B	H21C[12]	2.868	H21C	F4[8]
	3.354			
H21C	F5[8]	3.336	H21C	C20[11]
	3.423			
H21C	C21[12]	3.439	H21C	H20A[11]
	2.786			
H21C	H20B[11]	3.314	H21C	H21B[12]
	2.868			
H21C	H21C[12]	3.135	H21C	H21C[11]
	3.289			

Symmetry Operators:

(1) X,Y+1,Z
(2) -X+1,-Y+1,-Z+1
(3) -X+2,-Y+1,-Z+1
(4) X-1,Y,Z
(5) -X+1,-Y,-Z+1
(6) X+1,Y,Z
(7) X+1,Y+1,Z
(8) -X+1,-Y,-Z
(9) -X+2,-Y,-Z+1
(10) -X,-Y,-Z
(11) -X+1,-Y-1,-Z
(12) -X,-Y-1,-Z
(13) X,Y-1,Z
(14) X-1,Y-1,Z