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# Supporting Information



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# Fluoro-Bis(phenylsulfonyl)methane: A Fluoromethide Equivalent and Palladium-Catalyzed Enantioselective Allylic Monofluoromethylation

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**Reference 11a**
**Preparation of 1-fluoro-bis(phenylsulfonyl)methane (1)**
**Method A**: To a stirred suspension of NaH (60% oil dispersion, 270 mg, 6.75 mmol) in THF (25 ml) was added commercially available, bis(phenylsulfonyl)methane (2.00 g, 6.75 mmol) at 0 °C under nitrogen atmosphere. After 30 min stirring at room temperature, a solution of Selectfluor (2.39 g, 6.75 mmol) in MeCN (5 ml) was added at 0 °C, and the mixture was stirred for 3 h at room temperature. The reaction was quenched by addition of saturated aqueous ammonium chloride. The mixture was then extracted by dichloromethane, washed with brine, and dried over anhydrous $Na_2SO_4$. The solvent was evaporated and the residue was purified by silica gel column chromatography using 2:8 hexane /dichloromethane as the eluent to give **1** (1.28 g, 60%) as colorless needles.

**Method B**: To a stirred suspension of NaH (60% oil dispersion, 110 mg, 2.75 mmol) in THF (10 ml) was added bis(phenylsulfonyl)methane (500 mg, 1.69 mmol) at 0 °C under nitrogen atmosphere. After 30 min stirring at room temperature, the solvent was removed under reduced pressure. MeCN (17 ml) was added to the residue, and the mixture was cooled to -40°C. Gaseous 10% $F_2$ in $N_2$ was carefully bubbled through a solution for 2 min. The reaction was quenched by addition of saturated sodium bicarbonate. The mixture was then extracted by dichloromethane, washed with brine, and dried over anhydrous $Na_2SO_4$. The solvent was evaporated and the residue was purified by silica gel column chromatography using 2:8 hexane /dichloromethane as the eluent to give **1** (128 mg, 24%) as colorless needles.

Mp: 114-114.5 °C (from hexane); $^1$H NMR (200 MHz; $CDCl_3$, TMS reference): δ 5.70 (1H, d, *J*= 45.8 Hz, CH), 7.55-7.65 (4H, m, Ar), 7.70-7.80 (2H, m, Ar), 7.95-8.05 (4H, d, *J*=7.6 Hz, Ar); $^{13}$C NMR (50 MHz; $CDCl_3$) δ 105.3 (d, *J* = 263.4), 129.2, 129.8, 134.9, 135.4; $^{19}$F NMR (188 MHz; $CDCl_3$, $CFCl_3$ reference): δ -167.2 (d, *J* = 45.8 Hz); IR (KBr): ν=1354, 1174 $cm^{-1}$; MS (ESI-TOF) 314 ($M^+$), 173 ($M^+$-$SO_2Ph$), 141 ($M^+$-$PhSO_2CHF$); *Anal. Calcd* for $C_{26}H_{22}F_2O_8S_4$: C, 49.67; H, 3.53. Found: C, 49.82; H, 3.41.

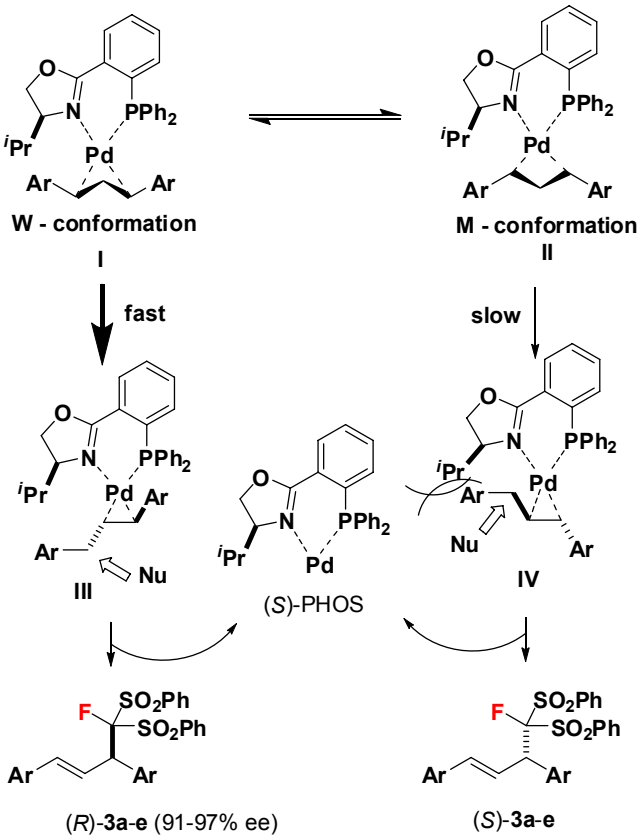
**Reference 11b**
**Typical Experimental Procedure**

A solution of **2** (249 mg, 0.683 mmol), (*S*)-PHOX (12.7 mg, 0.034mmol, 5 mol%), and $[Pd(C_3H_5)Cl]_2$ (6.2 mg, 0.017 mmol, 2.5 mol%) in $CH_2Cl_2$ (0.7 mL) was stirred at room temperature for 5 min. To the solution, **1** (236 mg, 0.751 mmol) and $Cs_2CO_3$ (244 mg, 0.749 mmol) were added at 0 °C. The resulting solution was stirred at 0 °C for 6 h, when **2** was completely consumed as indicated by TLC analysis. The reaction mixture was poured into sat. $NH_4Cl$, and it was extracted with $CH_2Cl_2$. The organic phase was dried over sodium sulfate. After evaporation of the solvent under reduced pressure, the product was purified by silica-gel column chromatography to give **3a** as colourless solids (350 mg, 83%, 94% ee). The

enantiomeric excess was determined by HPLC (Daicel CHIRALPACK AD-H, Hexane/$^{i}$PrOH=80:20, 1.0 ml/min, (*S*)-**3a** (10.2 min) and (*R*)-**3a** (16.2 min). mp. 55.2-56.0 °C; $^{1}$H NMR (200 MHz; $CDCl_3$, 25 °C, TMS) δ 0.87 (*d*, *J* = 5.8 Hz, 6H; $CH_3$), 0.90 (*d*, *J* = 5.8 Hz, 6H; $CH_3$), 1.71-1.92 (*m*, 2H; 2xCH), 2.38 (*d*, *J* = 7.2 Hz, 2H; $CH_2$), 2.45 (*d*, *J* = 7.0 Hz, 2H; $CH_2$), 4.70 (*dd*, *J* = 9.6, 14.5 Hz, 1H; CH=CH-CH), 6.46 (*d*, *J* = 15.8 Hz, 1H; CH=CH-CH), 6.98-7.90 (*m*, 19H; CH=CH-CH, Ar); $^{13}$C NMR (50 MHz; $CDCl_3$, 25 °C) δ 22.4, 22.5, 30.0, 30.1, 44.9, 45.1, 51.2 (d, *J* = 17.9 Hz), 116.2 (d, *J* = 269.0 Hz), 121.5, 121.6, 126.2, 128.0, 128.2, 128.5, 128.9, 129.9, 130.6, 132.3, 133.8, 133.9, 134.4, 135.4, 135.8, 136.5, 140.9, 141.1; $^{19}$F NMR (188 MHz; $CDCl_3$, 25 °C, $CFCl_3$) δ -127.8 (*d, J* = 14.5 Hz); $[\alpha]_D^{20}$ = +40.7 (*c*=1.51 in $CHCl_3$); IR (KBr): ν=1349, 1163, 1079 cm$^{-1.}$

**Reference 13a**

The enantiomer formed in the palladium catalyzed enantioselective allylic substitution reaction of **2a-e** using (*S*)-PHOX ligand can be rationalized by using the model previously described by Helmchen et al.[1] There are two preferred conformations for the (η3-allyl)(PHOX)palladium complexes, so-called W-conformation (**I**) and M-conformation (**II**). Sufficient information from the literature suggests that the nucleophile preferentially attacks at the allylic terminal carbon *trans* to phosphorous, therefore the two dominant transition states of the reaction **III** and **IV** should be considered. Because of the steric interactions between *i*Pr and Ar groups in **IV**, the transition state model **III** is more stable than the transition state model **IV**. Therefore, the nucleophile **1** prefers to approach via the model **III** to furnish the (*R*)-isomer.

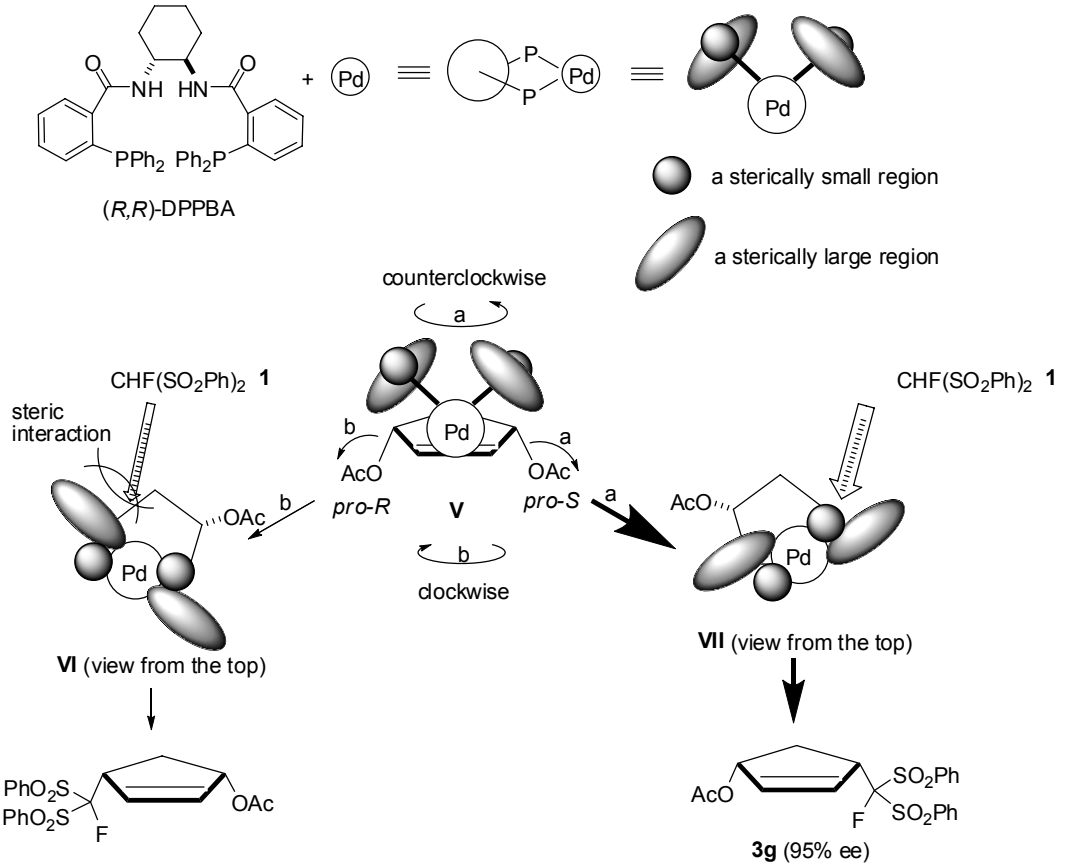



***Scheme 1***. Model for the origin of enantioselectivity of the allylic substitution reaction of **2a-e** using (*S*)-PHOX ligand

[1] M. Kollmar,H. Steinhagen, J. P. Janssen, B. Goldfuss, S. A. Malinovskaya, J. Vazquez, F. Rominger, G. Helmchen, *Chem. Eur. J.* **2002**, *8*, 3103-3114.

**Reference 13c**

Scheme 2 provides a working hypothesis for the origin of the steric interactions which lead to the observed enantioselectivity in the palladium catalyzed desymmetrization-substitution of meso-diester **2f** with **1** using (*R,R*)-DPPBA ligand. According to the report by Trost et al.,[2] two diastereotopic transition states **VI** and **VII** should be considered. A counterclockwise twist of the Pd-ligand on the complex **V** leads to the ionization of the *pro-S* leaving group to afford **VII** (route a). On the other hand, a clockwise twist on the complex **V** gives the model **VI** via the ionization of the *pro-R* leaving group (route b). The nucleophile **1** prefers to approach underneath a sterically small region of **VII** and in an orientation that minimizes steric interactions with a nearby sterically large region.



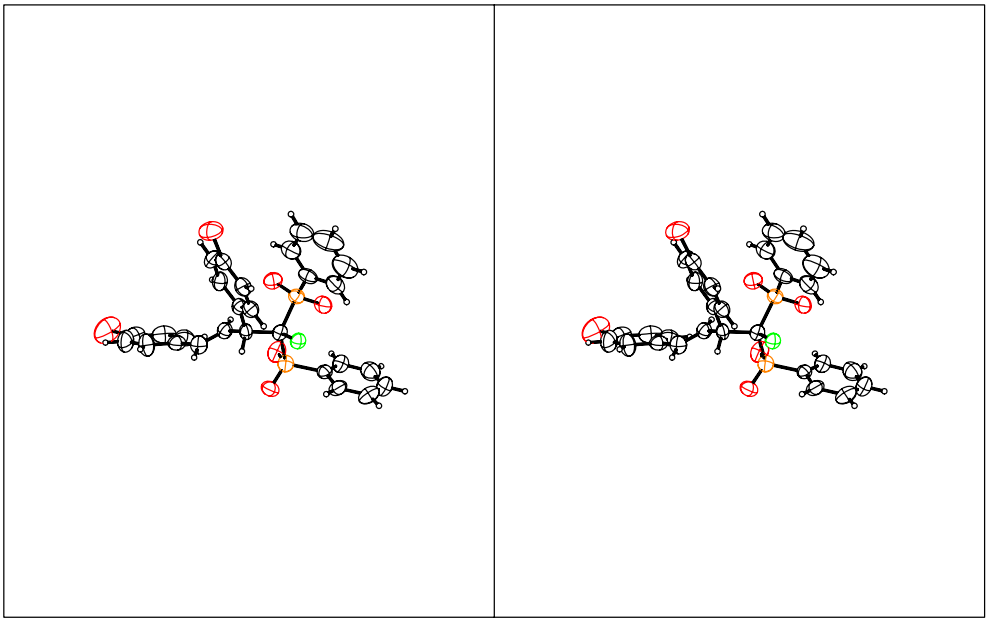

***Scheme 2.*** A working hypothesis for the origin of the steric interactions which lead to the observed enantioselectivity in the palladium catalyzed desymmetrization-substitution of meso-diester **2g** with **1** using (*R,R*)-DPPBA ligand.

**Reference 13b**

(*R*)-**3a** (94% ee): $[\alpha]_D^{20}$ = +40.7 (*c*= 1.51 in $CHCl_3$); (*S*)-**3a** (91% ee): $[\alpha]_D^{30}$ = -39.5 (*c* = 1.45 in $CHCl_3$); Non-fluorinated analogue of (*R*)-**3a**; $[\alpha]_D^{30}$ = +20 (*c* = 1.5 in $CHCl_3$), see, L. Acemoglu, J. M. J. Williams, *J. Mol. Catal. A: Chemical* **2003**, *196,* 3-11.; (*R*)-**3b** (96% ee): $[\alpha]_D^{20}$ = +13.8 (*c*= 1.54 in $CHCl_3$). Non-fluorinated analogue of (*R*)-**3b**; $[\alpha]_D^{25}$ = +5.6 (*c*= 1.0 in $CHCl_3$), see, H. Kubota, K. Koga, *Heterocycles* **1996**, *42*, 543-547.

[2] B. M. Trost, D. L. V. Vranken, C. Binge1, *J. Am. Chem. Soc*. **1992**, *114*, 9327-9343.

# X-ray Crystallography of (*R*)-**3d**



ABSOLUTE CONFIGURATION

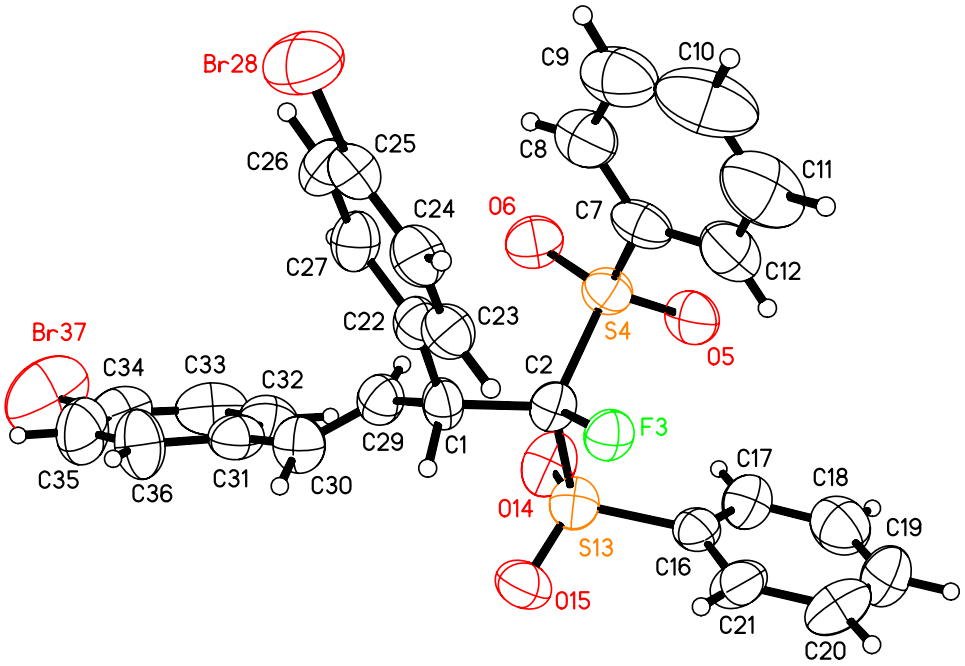

Br28
C25
C26
C24
C27
C22
C23
Br37
C34
C33
C32
C29
C1
C35
C36
C31
C30
C9
C10
C8
C11
O6
C7
C12
S4
O5
C2
F3
C17
C18
O14
S13
C19
C16
O15
C21
C20

Table 1. Crystal data and structure refinement for FT-1891FROM-VIAL.

| | | |
|---|---|---|
| Identification code | N050 | |
| Empirical formula | $C_{28}H_{21}O_4S_2FBr_2$ | |
| Formula weight | 664.39 | |
| Temperature | 298(2) K | |
| Wavelength | 0.71073 Å | |
| Crystal system | Orthorhombic | |
| Space group | P2(1)2(1)2(1) | |
| Unit cell dimensions | a = 7.0016(4) Å | α= 90°. |
| | b = 12.9149(7) Å | β= 90°. |
| | c = 30.8303(17) Å | γ = 90°. |
| Volume | 2787.8(3) $Å^3$ | |
| Z | 4 | |
| Density (calculated) | 1.583 $Mg/m^3$ | |
| Absorption coefficient | 3.096 $mm^{-1}$ | |
| F(000) | 1328 | |
| Crystal size | 0.20 x 0.08 x 0.03 $mm^3$ | |
| Theta range for data collection | 2.06 to 20.82°. | |
| Reflections collected | 14514 | |
| Independent reflections | 2905 [R(int) = 0.0601] | |
| Completeness to theta = 20.82° | 99.9 % | |
| Absorption correction | None | |
| Refinement method | Full-matrix least-squares on $F^2$ | |
| Data / restraints / parameters | 2905 / 0 / 334 | |
| Goodness-of-fit on $F^2$ | 1.021 | |
| Final R indices [I>2sigma(I)] | R1 = 0.0414, wR2 = 0.0963 | |
| R indices (all data) | R1 = 0.0517, wR2 = 0.1023 | |
| Absolute structure parameter | 0.005(14) | |
| Largest diff. peak and hole | 0.506 and -0.359 $e.Å^{-3}$ | |

## Experimental

**Single Crystal X-Ray Analysis.** A representative crystal was surveyed and a 1 Å data set (maximum sin $\Theta/\lambda$=0.5) was collected on a Bruker APEX diffractometer. Friedel pairs were collected in order to facilitate the determination of the absolute configuration. Atomic scattering factors were taken from the International Tables for Crystallography[1]. All crystallographic calculations were facilitated by the SHELXTL[2] system. All diffractometer data were collected at room temperature. Pertinent crystal, data collection, and refinement are summarized in Table I.

A trial structure was obtained by direct methods. This trial structure refined routinely. Hydrogen positions were calculated wherever possible. The hydrogen parameters were added to the structure factor calculations but were not refined. The shifts calculated in the final cycles of least squares refinement were all less than 0.1 of the corresponding standard deviations. The final R-index was 4.14%. A final difference Fourier revealed no missing or misplaced electron density.

The refined structure was plotted using the SHELXTL plotting package (Figure 1). The absolute configuration was determined by the method of Flack[3]. Coordinates, anisotropic temperature factors, distances and angles are available as supplementary material (Tables 1-5).

## References


1. **International Tables for Crystallography**, Vol. C, pp. 219, 500, Kluwer Academic Publishers,1992.

2. **SHELXTL**, Version 5.1, Bruker AXS, 1997.

3. H. D. Flack, Acta Crystallogr., **A39**, 876, 1983.

Table 2. Atomic coordinates ( x $10^4$) and equivalent isotropic displacement parameters ($Å^2$x $10^3$) for FT-1891FROM-VIAL. U(eq) is defined as one third of the trace of the orthogonalized $U_{ij}$ tensor.

| | x | y | z | U(eq) |
|---|---|---|---|---|
| C(1) | 6118(9) | 13786(5) | 8786(2) | 44(2) |
| C(2) | 4379(8) | 14398(5) | 8620(2) | 41(2) |
| F(3) | 4801(5) | 15435(3) | 8620(1) | 45(1) |
| S(4) | 2143(2) | 14276(1) | 8942(1) | 48(1) |
| O(5) | 641(6) | 14619(4) | 8667(2) | 60(1) |
| O(6) | 2105(7) | 13260(4) | 9119(2) | 62(1) |
| C(7) | 2418(9) | 15191(6) | 9362(2) | 51(2) |
| C(8) | 2734(10) | 14836(7) | 9780(3) | 65(2) |
| C(9) | 2830(11) | 15572(10) | 10107(3) | 84(3) |
| C(10) | 2638(17) | 16596(11) | 10020(4) | 109(4) |
| C(11) | 2291(16) | 16926(8) | 9613(4) | 98(3) |
| C(12) | 2167(11) | 16243(7) | 9273(3) | 69(2) |
| S(13) | 3941(3) | 14080(1) | 8037(1) | 51(1) |
| O(14) | 2904(8) | 13138(4) | 8028(2) | 68(2) |
| O(15) | 5797(7) | 14141(4) | 7849(2) | 63(1) |
| C(16) | 2542(10) | 15088(5) | 7824(2) | 46(2) |
| C(17) | 683(12) | 14897(7) | 7720(2) | 65(2) |
| C(18) | -390(13) | 15676(8) | 7537(3) | 77(3) |
| C(19) | 407(17) | 16623(9) | 7466(3) | 89(3) |
| C(20) | 2272(16) | 16821(7) | 7569(3) | 78(3) |
| C(21) | 3380(12) | 16043(6) | 7746(2) | 60(2) |
| C(22) | 6599(9) | 14029(5) | 9259(2) | 45(2) |
| C(23) | 7304(9) | 14999(5) | 9369(2) | 49(2) |
| C(24) | 7847(9) | 15216(6) | 9788(2) | 53(2) |
| C(25) | 7686(10) | 14452(6) | 10102(2) | 55(2) |
| C(26) | 6979(11) | 13503(6) | 10000(3) | 58(2) |
| C(27) | 6463(11) | 13282(5) | 9584(3) | 53(2) |
| Br(28) | 8560(1) | 14751(1) | 10672(1) | 82(1) |
| C(29) | 5983(11) | 12635(5) | 8694(2) | 50(2) |
| C(30) | 7467(12) | 12080(6) | 8587(2) | 57(2) |
| C(31) | 7502(12) | 10946(6) | 8528(2) | 57(2) |
| C(32) | 5948(12) | 10401(7) | 8388(2) | 64(2) |
| C(33) | 5987(16) | 9327(8) | 8360(3) | 80(3) |
| C(34) | 7643(18) | 8814(6) | 8488(3) | 79(3) |
| C(35) | 9207(15) | 9340(7) | 8619(3) | 84(3) |
| C(36) | 9153(12) | 10414(7) | 8638(3) | 76(2) |
| Br(37) | 7713(3) | 7354(1) | 8471(1) | 143(1) |

Table 3. Bond lengths [Å] and angles [°] for FT-1891FROM-VIAL.

| | |
|---|---|
| C(1)-C(29) | 1.517(9) |
| C(1)-C(22) | 1.528(9) |
| C(1)-C(2) | 1.539(9) |
| C(2)-F(3) | 1.372(7) |
| C(2)-S(4) | 1.860(6) |
| C(2)-S(13) | 1.872(7) |
| S(4)-O(6) | 1.419(5) |
| S(4)-O(5) | 1.423(4) |
| S(4)-C(7) | 1.763(7) |
| C(7)-C(12) | 1.397(10) |
| C(7)-C(8) | 1.386(10) |
| C(8)-C(9) | 1.388(11) |
| C(9)-C(10) | 1.357(14) |
| C(10)-C(11) | 1.346(14) |
| C(11)-C(12) | 1.374(12) |
| S(13)-O(14) | 1.417(5) |
| S(13)-O(15) | 1.425(5) |
| S(13)-C(16) | 1.755(7) |
| C(16)-C(17) | 1.363(10) |
| C(16)-C(21) | 1.387(10) |
| C(17)-C(18) | 1.378(11) |
| C(18)-C(19) | 1.362(13) |
| C(19)-C(20) | 1.367(14) |
| C(20)-C(21) | 1.383(12) |
| C(22)-C(23) | 1.388(9) |
| C(22)-C(27) | 1.394(10) |
| C(23)-C(24) | 1.376(9) |
| C(24)-C(25) | 1.385(9) |
| C(25)-C(26) | 1.360(10) |
| C(25)-Br(28) | 1.901(7) |
| C(26)-C(27) | 1.362(10) |
| C(29)-C(30) | 1.305(10) |
| C(30)-C(31) | 1.475(10) |
| C(31)-C(32) | 1.367(10) |
| C(31)-C(36) | 1.387(11) |
| C(32)-C(33) | 1.389(11) |
| C(33)-C(34) | 1.392(13) |
| C(34)-C(35) | 1.350(12) |
| C(34)-Br(37) | 1.887(8) |
| C(35)-C(36) | 1.389(11) |

| | |
|---|---|
| C(29)-C(1)-C(22) | 113.3(5) |
| C(29)-C(1)-C(2) | 113.0(5) |
| C(22)-C(1)-C(2) | 112.7(5) |
| F(3)-C(2)-C(1) | 109.3(5) |
| F(3)-C(2)-S(4) | 105.3(4) |
| C(1)-C(2)-S(4) | 116.4(4) |
| F(3)-C(2)-S(13) | 104.4(4) |
| C(1)-C(2)-S(13) | 109.7(4) |
| S(4)-C(2)-S(13) | 110.9(3) |
| O(6)-S(4)-O(5) | 120.2(3) |
| O(6)-S(4)-C(7) | 109.9(3) |
| O(5)-S(4)-C(7) | 108.0(3) |
| O(6)-S(4)-C(2) | 107.4(3) |
| O(5)-S(4)-C(2) | 106.1(3) |
| C(7)-S(4)-C(2) | 104.0(3) |
| C(12)-C(7)-C(8) | 121.6(7) |
| C(12)-C(7)-S(4) | 119.6(6) |
| C(8)-C(7)-S(4) | 118.5(6) |
| C(9)-C(8)-C(7) | 117.2(8) |
| C(10)-C(9)-C(8) | 121.3(9) |
| C(11)-C(10)-C(9) | 120.7(9) |
| C(10)-C(11)-C(12) | 121.4(10) |
| C(11)-C(12)-C(7) | 117.8(9) |
| O(14)-S(13)-O(15) | 120.5(3) |
| O(14)-S(13)-C(16) | 110.1(3) |
| O(15)-S(13)-C(16) | 108.4(3) |
| O(14)-S(13)-C(2) | 106.8(3) |
| O(15)-S(13)-C(2) | 103.3(3) |
| C(16)-S(13)-C(2) | 106.7(3) |
| C(17)-C(16)-C(21) | 121.6(7) |
| C(17)-C(16)-S(13) | 119.1(6) |
| C(21)-C(16)-S(13) | 119.3(6) |
| C(16)-C(17)-C(18) | 119.0(8) |
| C(19)-C(18)-C(17) | 119.9(9) |
| C(20)-C(19)-C(18) | 121.5(9) |
| C(19)-C(20)-C(21) | 119.4(8) |
| C(16)-C(21)-C(20) | 118.6(8) |
| C(23)-C(22)-C(27) | 118.3(6) |
| C(23)-C(22)-C(1) | 119.7(6) |
| C(27)-C(22)-C(1) | 121.9(6) |
| C(24)-C(23)-C(22) | 120.7(6) |
| C(23)-C(24)-C(25) | 119.2(7) |
| C(26)-C(25)-C(24) | 120.6(7) |
| C(26)-C(25)-Br(28) | 121.0(6) |
| C(24)-C(25)-Br(28) | 118.3(6) |
| C(25)-C(26)-C(27) | 120.3(7) |
| C(26)-C(27)-C(22) | 120.9(7) |
| C(30)-C(29)-C(1) | 122.5(7) |
| C(29)-C(30)-C(31) | 126.1(7) |
| C(32)-C(31)-C(36) | 119.0(7) |
| C(32)-C(31)-C(30) | 122.5(8) |
| C(36)-C(31)-C(30) | 118.4(8) |
| C(31)-C(32)-C(33) | 121.2(9) |
| C(32)-C(33)-C(34) | 118.3(9) |
| C(35)-C(34)-C(33) | 121.4(8) |
| C(35)-C(34)-Br(37) | 119.3(9) |
| C(33)-C(34)-Br(37) | 119.3(8) |
| C(34)-C(35)-C(36) | 119.5(9) |
| C(35)-C(36)-C(31) | 120.5(9) |

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for FT-1891FROM-VIAL. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U_{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U_{12}\ ]$

| | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{23}$ | $U_{13}$ | $U_{12}$ |
|---|---|---|---|---|---|---|
| C(1) | 33(4) | 40(4) | 58(5) | 5(3) | -1(3) | 3(3) |
| C(2) | 39(4) | 33(4) | 50(4) | 2(3) | -5(3) | -9(3) |
| F(3) | 46(2) | 38(2) | 52(2) | 3(2) | 0(2) | -5(2) |
| S(4) | 40(1) | 52(1) | 52(1) | 3(1) | 5(1) | -5(1) |
| O(5) | 34(3) | 90(4) | 55(3) | -1(3) | -10(2) | 1(2) |
| O(6) | 58(3) | 52(3) | 75(3) | 15(3) | 11(3) | -15(3) |
| C(7) | 33(4) | 69(6) | 50(5) | -14(4) | 7(4) | -12(4) |
| C(8) | 49(5) | 90(6) | 57(5) | -12(5) | -6(4) | -1(4) |
| C(9) | 55(5) | 132(10) | 64(6) | -9(7) | 2(4) | -7(6) |
| C(10) | 112(9) | 130(11) | 86(9) | -52(8) | 24(7) | -38(8) |
| C(11) | 113(8) | 64(6) | 117(9) | -37(7) | 30(7) | -20(6) |
| C(12) | 64(6) | 68(6) | 76(6) | -10(5) | 15(5) | 6(4) |
| S(13) | 55(1) | 49(1) | 50(1) | -6(1) | -7(1) | 1(1) |
| O(14) | 84(4) | 52(3) | 69(3) | -9(3) | -25(3) | -6(3) |
| O(15) | 59(3) | 80(4) | 51(3) | -3(3) | 11(3) | 9(3) |
| C(16) | 42(5) | 60(5) | 37(4) | 2(3) | -1(3) | 4(4) |
| C(17) | 64(6) | 67(6) | 63(5) | 9(4) | -4(4) | 8(5) |
| C(18) | 66(6) | 93(8) | 72(6) | -7(6) | -1(5) | 5(6) |
| C(19) | 100(9) | 96(9) | 72(6) | 26(6) | 1(6) | 38(7) |
| C(20) | 97(8) | 68(6) | 68(6) | 24(5) | 2(6) | -13(6) |
| C(21) | 64(5) | 66(6) | 49(5) | 18(4) | 0(4) | -5(5) |
| C(22) | 34(4) | 49(5) | 50(5) | 3(4) | -6(3) | 2(3) |
| C(23) | 38(4) | 55(5) | 55(5) | 1(4) | -1(4) | -3(3) |
| C(24) | 48(4) | 49(4) | 60(5) | -9(4) | -2(4) | 1(4) |
| C(25) | 46(4) | 63(6) | 56(5) | 0(4) | 7(4) | 10(4) |
| C(26) | 59(5) | 56(6) | 57(6) | 19(4) | -6(4) | -2(4) |
| C(27) | 50(5) | 40(5) | 67(5) | 3(4) | -7(4) | -1(4) |
| Br(28) | 87(1) | 108(1) | 51(1) | 0(1) | -7(1) | -10(1) |
| C(29) | 44(5) | 50(5) | 56(5) | 6(4) | -3(4) | -2(4) |
| C(30) | 51(5) | 53(5) | 67(5) | -2(4) | -6(4) | -5(4) |
| C(31) | 74(6) | 50(5) | 49(5) | -1(4) | 12(4) | -1(5) |
| C(32) | 71(6) | 60(7) | 60(5) | -10(4) | 11(4) | -6(5) |
| C(33) | 103(8) | 72(7) | 65(6) | -16(5) | 8(6) | -33(6) |
| C(34) | 122(9) | 52(6) | 62(6) | -4(4) | 9(6) | -1(6) |
| C(35) | 93(8) | 56(7) | 104(7) | 6(5) | -2(6) | 19(5) |
| C(36) | 72(6) | 56(7) | 101(7) | -9(5) | -9(5) | 14(5) |
| Br(37) | 240(2) | 48(1) | 141(1) | -1(1) | -4(1) | -9(1) |

Table 5. Hydrogen coordinates ( x $10^4$) and isotropic displacement parameters (Å$^2$x $10^3$) for FT-1891FROM-VIAL.

| | x | y | z | U(eq) |
|---|---|---|---|---|
| H(1A) | 7208 | 14036 | 8617 | 80 |
| H(8A) | 2877 | 14134 | 9839 | 80 |
| H(9A) | 3031 | 15359 | 10392 | 80 |
| H(10A) | 2747 | 17076 | 10244 | 80 |
| H(11A) | 2133 | 17630 | 9562 | 80 |
| H(12A) | 1924 | 16473 | 8992 | 80 |
| H(17A) | 146 | 14250 | 7773 | 80 |
| H(18A) | -1656 | 15556 | 7461 | 80 |
| H(19A) | -333 | 17147 | 7346 | 80 |
| H(20A) | 2790 | 17474 | 7520 | 80 |
| H(21A) | 4660 | 16157 | 7811 | 80 |
| H(23A) | 7410 | 15508 | 9157 | 80 |
| H(24A) | 8318 | 15867 | 9860 | 80 |
| H(26A) | 6846 | 13002 | 10214 | 80 |
| H(27A) | 6015 | 12624 | 9516 | 80 |
| H(29A) | 4798 | 12313 | 8714 | 80 |
| H(30A) | 8610 | 12432 | 8543 | 80 |
| H(32A) | 4845 | 10755 | 8309 | 80 |
| H(33A) | 4934 | 8961 | 8259 | 80 |
| H(35A) | 10312 | 8985 | 8696 | 80 |
| H(36A) | 10232 | 10779 | 8726 | 80 |