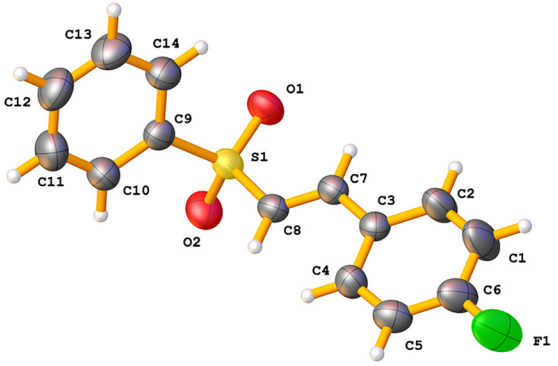
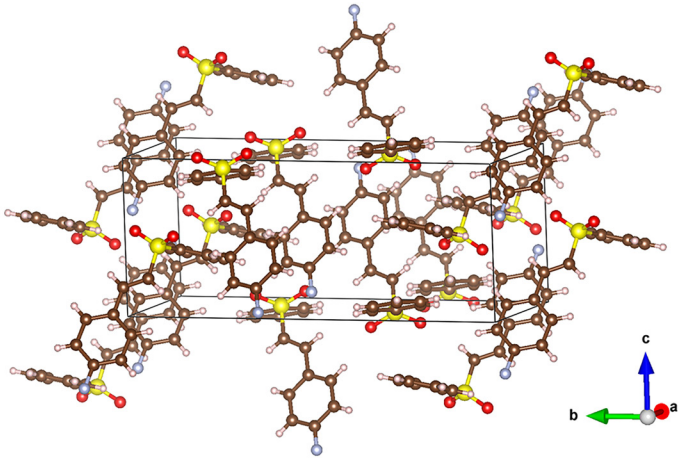
Bin Liu* and Ling-Yun Mao

Crystal structure of (*E*)-1-fluoro-4-(2-(phenylsulfonyl)vinyl)benzene, $C_{14}H_{11}FO_2S$





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Abstract

$C_{14}H_{11}FO_2S$, monoclinic, $P2_1/c$, a = 8.2908(8) Å, b = 19.3233(18) Å, c = 7.9496(7) Å, β = 91.188(2)°, V = 1,273.3(2) Å^3, Z = 4, $R_{gt}(F)$ = 0.0473, $wR_{ref}(F^2)$ = 0.1266, T = 296 K.

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Table 1 contains the crystallographic data and the list of the atoms including atomic coordinates and displacement parameters can be found in the cif-file attached to this article.

Table 1: Data collection and handling.

Crystal:	Clear light colourless block
Size:	0.26 × 0.25 × 0.22 mm
Wavelength:	Mo*K*α radiation (0.71073 Å)
μ:	0.26 mm^{-1}
Diffractometer, scan mode:	Bruker SMART APEX2, φ and ω scans
θ_{max}, completeness:	25.3°, 100 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	6582, 2323, 0.026
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1,919
$N(param)_{refined}$:	151
Programs:	Bruker[1], Olex2[2], SHELX[3,4]

1 Source of materials

The synthesis route of (*E*)-1-fluoro-4-(2-(phenylsulfonyl)vinyl)benzene (FPSVB) mainly refers to the papers published by Guo Ruqing *et al.* and Battace Ahmed *et al.* with minor modifications.[5,6] The synthetic route of FPSVB is as follows: under a nitrogen atmosphere, in a 25 mL three-necked flask, 0.05 mmol of palladium(II) acetate, 1.0 mmol of silver carbonate, 0.6 mmol of sodium benzenesulfonate, and 1.0 mmol of (*E*)-3-(4-fluorophenyl)acrylic acid were added. The sealed three-necked flask was then stirred on a magnetic stirrer at 80 °C for 24 h under a nitrogen atmosphere. After the reaction was completed, the reaction mixture was cooled to room temperature, and ethyl acetate was added to the system. The mixture was filtered using celatom, followed by washing with ethyl acetate. The obtained solution was collected and concentrated using a rotary evaporator to obtain the crude solid product. The solid product was then purified by column chromatography, with a mixture of *n*-hexane and ethyl acetate (v/v = 4:1) as the eluent. The eluate was collected and concentrated again using a rotary evaporator to obtain the target FPSVB compound. The crystallization procedure of FPSVB is as follows: 0.015 g of FPSVB powder was placed in a 10 mL tube, and 6 mL of dichloromethane was added. The

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tube was sealed with parafilm, and small holes were made in the parafilm with a needle to facilitate the evaporation of the solvent. The tube was placed in a cool and dry place for about 3 days, and colourless crystals could be observed at the interface between the solvent and air inside the tube. The yield of the crystals was 43 % (based on FPSVB powder). All the reagents and solvents used in the entire experimental process were purchased from Anhui Zesheng Technology Co., Ltd., and no further purification was performed before use.

2 Experimental details

A high-quality single crystal was carefully selected for structural characterization using X-ray single-crystal diffraction. The initial structural solution and refinement were carried out using the Olex2 software. Specifically, the initial structural determination was achieved using the SHELXT program. During the refinement stage, the SHELXL program was employed. Anisotropic refinement was applied to the C, F, O, and S atoms to account for their anisotropic thermal vibrations within the crystal structure, thereby enhancing the precision of the structural parameters. In contrast, H atoms were positioned theoretically and assigned isotropic thermal parameters.

3 Comment

Vinyl sulfones, as a class of olefin derivatives with unique structures and properties, have gradually become a research hotspot in the fields of bio-medicine and organic synthesis. They are not only capable of effectively inhibiting a variety of key biological targets, including cysteine proteases, Sortase A (SrtA), and caspase-3, but also demonstrate potential in trypanocidal activity, neuroprotective effects in the treatment of Parkinson's disease, and as fluorescent probes for selective labeling of live cells in the field of bioimaging.[7–13] Given the significant advantages and great potential of vinyl sulfones in multiple key areas, the synthesis of new vinyl sulfone compounds holds great significance. To date, the crystal structures of a few vinyl sulfone compounds have been reported. However, the crystal structure of the relatively simple compound (*E*)-1-fluoro-4-(2-(phenylsulfonyl)vinyl)benzene (FPSVB) has not yet been reported.

X-ray single-crystal diffractometer testing (Mo-target) indicate that the crystal system of the title FPSVB crystal is monoclinic, with a space group of $P2_1/c$. The asymmetric unit of the crystal contains one complete FPSVB molecule. All atoms in the molecule are fully occupied, and the thermal ellipsoids of the atoms are within the normal vibration range, with no need for additional disorder splitting treatment (see left part of the figure). The bond lengths of C–C, C–F, C–S, and S–O are 1.325(3)–1.468(2) Å, 1.324(2) Å, 1.737(2)–1.764(2) Å, and 1.4383(17)–1.4446(17) Å, respectively. These bond length ranges are similar to those reported in previous studies with similar structures to FPSVB,[14–19] and are all within the normal range. The FPSVB molecule can be divided into two parts based on the double bond (C7–C8) in the structure. One part can be regarded as a sulfonyl phenyl group (C9–C10–C11–C12–C13–C14 and O1–S1–O2), while the other part can be regarded as a chlorobenzene group (C1–C2–C3–C4–C5–C6 and F1). Clearly, these two parts are located on opposite sides of the double bond, indicating that the configuration of the FPSVB molecule is a trans-structure. The bond angles S1–C8–C7 and C8–C7–C3 are 122.6° and 125.2°, respectively. From another perspective, centered on the sulfonyl group, a specific angle is formed within the FPSVB molecule, making it a non-planar structure and the angle (C9–S1–C8) is 103.82(11)°. Further analysis of the weak interactions in the FPSVB crystal using Mercury and PLATON software revealed the presence of two types of C–H–O hydrogen bonds.[20,21] These include intermolecular C–H–O hydrogen bonds: C2–H2···O1[1] and C8–H8···O2[2], with donor-acceptor distances of 3.370(2) Å and 3.330(3) Å, respectively; and intramolecular C–H–O hydrogen bonds: C7–H7···O1 and C14–H14···O1, with donor-acceptor distances of 2.938(3) Å and 2.932(3) Å, respectively. Additionally, C–H–F hydrogen bonds were observed: C14–H14···F1[3], with a donor-acceptor distance of 3.184(3) Å. Moreover, π–π stacking interactions (see right part of the figure) exist between the aromatic rings in the FPSVB crystal (ring 1: C1–C2–C3–C4–C5–C6; ring 2: C9–C10–C11–C12–C13–C14), with the perpendicular distance between the aromatic rings ranging from 4.2126(17) to 4.5651(11) Å (only those less than 5.0 Å are listed), and the slippage ranging from 1.178 to 2.695 Å. Ultimately, the FPSVB molecules form a three-dimensional supramolecular structure through the aforementioned weak attractive interactions. Symmetry codes: #1: $1-x, 1-y, -z$; #2: $x, 1/2-y, 1/2+z$; #3: $1-x, 1-y, 1-z$.

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