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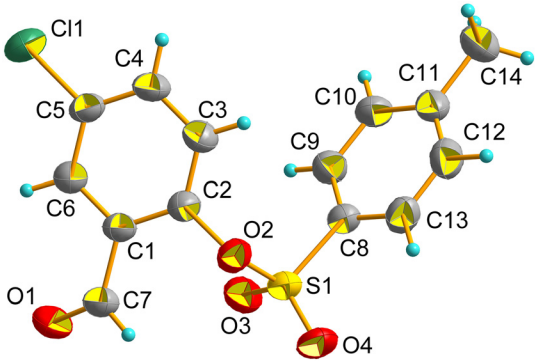
The crystal structure of 4-chloro-2-formylphenyl 4-methylbenzenesulfonate, $C_{14}H_{11}ClO_4S$



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Abstract

$C_{14}H_{11}ClO_4S$, monoclinic, $P2_1/c$ (no. 14), a = 8.2472(13) Å, b = 22.431(4) Å, c = 7.9308(13) Å, β = 106.278(2)°, V = 1,408.3(4) Å^3, Z = 4, $R_{gt}(F)$ = 0.0363, $wR_{ref}(F^2)$ = 0.0996, T = 296(2) K.

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Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

1 Source of material

4–Toluenesulfonyl chloride (0.419 g, 2.2 mmol), dissolved in THF (5 mL) was added dropwise to a solution of 5-chlorosalicylaldehyde (0.313 g, 2 mmol) and trimethylamine (0.202 g, 2 mmol) in THF (5 mL) with constant stirring at room temperature for 12 h. The solvent was removed and the residue was separated using flash column chromatography

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Table 1: Data collection and handling.

Crystal:	Colorless, block
Size:	0.53 × 0.45 × 0.39 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.43 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	25.0°, >99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	13,301, 2,489, 0.015
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2,198
$N(param)_{refined}$:	182
Programs:	Bruker,[1] SHELX,[2,3] Olex2[4]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	x	y	z	U_{iso}*/U_{eq}
C1	0.4593 (2)	0.73572 (8)	0.8239 (2)	0.0419 (4)
C2	0.5113 (2)	0.67884 (8)	0.8826 (2)	0.0420 (4)
C3	0.6788 (2)	0.66605 (9)	0.9668 (3)	0.0517 (5)
H3	0.7107	0.6276	1.0067	0.062*
C4	0.7975 (2)	0.71076 (9)	0.9910 (3)	0.0542 (5)
H4	0.9106	0.7028	1.0461	0.065*
C5	0.7467 (2)	0.76757 (9)	0.9324 (3)	0.0477 (4)
C6	0.5795 (2)	0.78057 (9)	0.8498 (3)	0.0467 (4)
H6	0.5477	0.8192	0.8120	0.056*
C7	0.2797 (2)	0.75035 (10)	0.7363 (3)	0.0523 (5)
H7	0.1970	0.7237	0.7478	0.063*
C8	0.5158 (2)	0.54863 (8)	0.7014 (2)	0.0440 (4)
C9	0.6522 (2)	0.56743 (9)	0.6464 (3)	0.0516 (5)
H9	0.6485	0.6037	0.5886	0.062*
C10	0.7939 (2)	0.53184 (9)	0.6782 (3)	0.0556 (5)
H10	0.8859	0.5445	0.6414	0.067*
C11	0.8018 (2)	0.47775 (9)	0.7636 (3)	0.0522 (5)
C12	0.6619 (3)	0.45924 (10)	0.8137 (3)	0.0591 (5)
H12	0.6644	0.4225	0.8686	0.071*
C13	0.5192 (3)	0.49408 (9)	0.7841 (3)	0.0547 (5)
H13	0.4265	0.4812	0.8190	0.066*
C14	0.9586 (3)	0.43989 (11)	0.8017 (4)	0.0778 (7)
H14A	1.0262	0.4470	0.9199	0.117*
H14B	1.0223	0.4499	0.7213	0.117*
H14C	0.9272	0.3986	0.7882	0.117*
Cl1	0.89659 (7)	0.82400 (3)	0.96406 (9)	0.0720 (2)
O1	0.23565 (19)	0.79481 (7)	0.6513 (2)	0.0712 (5)
O2	0.39155 (16)	0.63279 (6)	0.86087 (17)	0.0491 (3)
O3	0.33457 (18)	0.63775 (7)	0.53985 (19)	0.0595 (4)
O4	0.19887 (17)	0.56313 (7)	0.6853 (2)	0.0727 (5)
S1	0.34313 (6)	0.59613 (2)	0.67744 (7)	0.04947 (17)

to obtain the target product (yield: 0.510 g, 82 %). The single crystal of the product was obtained by recrystallization from dichloromethane at room temperature.

2 Experimental details

All hydrogen atoms were identified in difference Fourier syntheses. The U_{iso} values of the methyl groups and phenolic hydroxyl were set to $1.5U_{eq}$(C), and the U_{iso} values of all other hydrogen atoms were set to $1.2U_{eq}$(C).

3 Comment

p–Toluene sulfonate has a wide range of applications in biology and industry, which can be used as a synthetic intermediate to synthesize drugs for the treatment of Parkinson 's disease, and can also be used as a precursor for the preparation of Schiff base.[5–9]

Herein, a novel *p*-toluene sulfonate compound was synthesized and characterized by single-crystal X-ray diffraction.[1–4] In the crystal structure, the benzene ring C(1)–C(6) connects a formyl group, a chlorine atom and a p-toluene sulfonate group through C(1)–C(7), C(5)–Cl(1) and C(2)–O(2). The bond lengths of Cl(1)–C(5), O(1)–C(7), S(1)–O(2), S(1)–O(3), S(1)–O(4) and S(1)–C(8) are 1.7374(19), 1.201(3), 1.6207(14), 1.4228(16), 1.4174(15) and 1.7455(19) Å, respectively. The bond angles of O(1)–C(7)–C(1), O(2)–S(1)–O(3), O(2)–S(1)–C(8), O(3)–S(1)–C(8) and O(4)–S(1)–C(8) are 123.4(2), 107.59(8), 102.94(8), 110.33(9) and 110.22(10)°, indicating the C(7) atom is in the sp^2 hybridization state and S(1) atom is in the sp^3 unequal hybridization state. The dihedral angle between the plane through C(1)–C(6) and the plane through C(8)–C(13) is 34.8°.

Author contributions: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.
Conflict of interest: The authors declare no conflicts of interest regarding this article.

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