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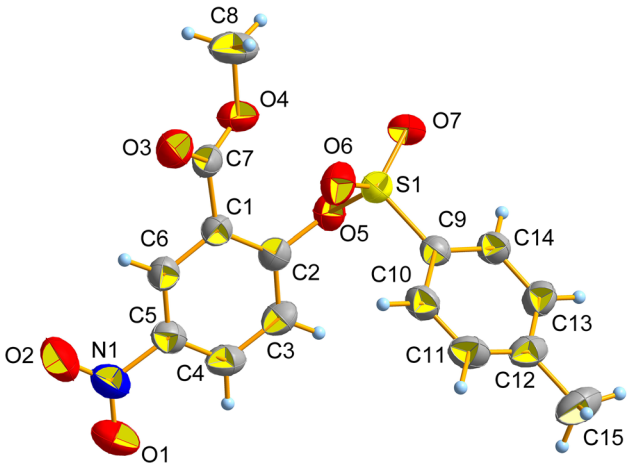
The crystal structure of methyl 5-nitro-2-(tosyloxy) benzoate, $C_{15}H_{13}NO_7S$



https://doi.org/10.1515/ncrs-2023-0360
Received August 3, 2023; accepted September 1, 2023;
published online September 14, 2023

Abstract

$C_{15}H_{13}NO_7S$, triclinic, $P\bar{1}$ (no. 2), a = 6.5990(11) Å, b = 7.7259(13) Å, c = 16.428(3) Å, α = 79.1416(18)°, β = 79.4782(16)°, γ = 81.1835(18)°, V = 802.6(2) Å^3, Z = 2, $R_{gt}(F)$ = 0.0340, $wR_{ref}(F^2)$ = 0.0960, T = 296(2) K.

CCDC no.: 2292371

The molecular structure is shown in the figure. 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 (1.049 g, 5.5 mmol), dissolved in THF (10 mL) was added dropwise to a solution of methyl-2-hydroxy-5-nitrobenzoate (0.986 g, 5 mmol) and trimethylamine (0.505 g, 5 mmol) in THF (10 mL) with constant stirring at room temperature for 12 h. The solvent was removed and the residue was separated using flash column chromatography to obtain the target product (yield: 1.370 g, 78 %). Crystals were obtained by recrystallization from dichloromethane at room temperature.

Table 1: Data collection and handling.

Crystal:	Block
Size:	0.47 x 0.41 x 0.36 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.24 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}:	7654, 2813, 0.013
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 2548
$N(param)_{refined}$:	220
Programs:	Bruker [1], SHELX [2, 3], Olex2 [4]

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 sulfonates can coordinate with metals as catalysts, which can be used as intermediates for the synthesis of drugs for the treatment of Parkinson's disease, and can also be used as self-assembled supramoleculars with potential applications in optoelectronics and pharmaceutical products [5–7]. Their crystal structures have always been in the focus of scientists [8–10].

Herein, a novel *p*-toluene sulfonate compound was synthesized and characterized by single-crystal X-ray diffraction [1–4]. In the crystal structure, a nitro group, a methyl formate group and a para-toluene sulfonate group are connected to the same benzene ring with C(5)–N(1), C(1)–C(7), C(2)–O(5) bonds, respectively. The bond lengths of N(1)–C(5), N(1)–O(1), C(7)–O(3), C(7)–O(4) and C(8)–O(4) are 1.476(2), 1.208(2), 1.200(2), 1.331(2) and 1.444(2) Å, respectively [11]. The bond

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.1349 (2)	0.1088 (2)	0.12603 (10)	0.0437 (4)
C2	−0.0220 (2)	0.1560 (2)	0.19044 (10)	0.0428 (4)
C3	−0.2028 (3)	0.2616 (2)	0.17379 (12)	0.0539 (4)
H3	−0.3040	0.2927	0.2177	0.065*
C4	−0.2333 (3)	0.3210 (2)	0.09195 (12)	0.0579 (5)
H4	−0.3559	0.3902	0.0799	0.069*
C5	−0.0788 (3)	0.2758 (2)	0.02866 (11)	0.0508 (4)
C6	0.1045 (3)	0.1729 (2)	0.04390 (10)	0.0488 (4)
H6	0.2070	0.1466	−0.0005	0.059*
C7	0.3338 (3)	−0.0076 (2)	0.13878 (11)	0.0508 (4)
C8	0.4928 (4)	−0.2518 (4)	0.22346 (18)	0.0916 (8)
H8A	0.5468	−0.3177	0.1785	0.137*
H8B	0.4599	−0.3328	0.2746	0.137*
H8C	0.5950	−0.1815	0.2299	0.137*
C9	0.0056 (2)	0.3421 (2)	0.36042 (9)	0.0430 (4)
C10	0.0187 (3)	0.5068 (2)	0.31029 (11)	0.0541 (4)
H10	0.1010	0.5175	0.2576	0.065*
C11	−0.0920 (3)	0.6540 (2)	0.33980 (13)	0.0610 (5)
H11	−0.0838	0.7650	0.3064	0.073*
C12	−0.2154 (3)	0.6418 (2)	0.41794 (13)	0.0556 (4)
C13	−0.2253 (3)	0.4749 (2)	0.46665 (12)	0.0551 (4)
H13	−0.3074	0.4643	0.5193	0.066*
C14	−0.1165 (3)	0.3241 (2)	0.43890 (10)	0.0489 (4)
H14	−0.1250	0.2129	0.4721	0.059*
C15	−0.3336 (3)	0.8056 (3)	0.44917 (17)	0.0802 (7)
H15A	−0.4455	0.8505	0.4180	0.120*
H15B	−0.2419	0.8945	0.4418	0.120*
H15C	−0.3887	0.7762	0.5076	0.120*
N1	−0.1097 (3)	0.3362 (2)	−0.05944 (11)	0.0654 (4)
O1	−0.2682 (3)	0.4299 (3)	−0.07302 (12)	0.1049 (6)
O2	0.0242 (3)	0.2892 (3)	−0.11453 (10)	0.0956 (6)
O3	0.4951 (2)	0.0139 (2)	0.09306 (9)	0.0733 (4)
O4	0.3075 (2)	−0.13692 (17)	0.20448 (9)	0.0631 (4)
O5	−0.00912 (17)	0.08901 (15)	0.27484 (7)	0.0488 (3)
O6	0.32249 (18)	0.20630 (19)	0.26435 (8)	0.0625 (4)
O7	0.1708 (2)	0.01357 (17)	0.39140 (8)	0.0658 (4)
S1	0.14808 (6)	0.15465 (5)	0.32361 (2)	0.04730 (16)

lengths of S(1)–O(5), S(1)–O(6), S(1)–O(7) and S(1)–C(9) are 1.6104(12), 1.4202(13), 1.4157(13) and 1.7469(16) *Å*, respectively. The bond angles of O(5)–S(1)–O(6), O(5)–S(1)–C(9), O(6)–S(1)–C(9) and O(7)–S(1)–C(9) are 108.36(7), 103.44(7), 109.32(8) and 110.12(8)°, indicating the 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(9)–C(14) is 49.513°.

Acknowledgements: We gratefully acknowledge support by Natural Science Foundation of Hunan Province of China (2021JJ30291), the Scientific Research Fund of Hunan Provincial Education Department (21A0518, 21C0691), Yongzhou Guiding Science and Technology Plan Project (2021).

Author contributions: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.

Competing interests: The authors declare no conflicts of interest regarding this article.

Research funding: Natural Science Foundation of Hunan Province of China (2021JJ30291), the Scientific Research Fund of Hunan Provincial Education Department (21A0518, 21C0691), Yongzhou Guiding Science and Technology Plan Project (2021).

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