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Crystal structure of 2-((4-nitrophenyl)thio)ethan-1-ol, $C_8H_9NO_3S$

Table 1: Data collection and handling.

Crystal:	Yellow plate
Size:	0.20 × 0.20 × 0.06 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.33 mm ⁻¹
Diffractometer, scan mode:	SMART, φ and ω
$\theta_{\max}$, completeness:	25.0°, 99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	2228, 1542, 0.028
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1369
$N(\text{param})_{\text{refined}}$:	119
Programs:	SHELX [1], Bruker [2]

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Abstract

$C_8H_9NO_3S$, triclinic, $P\bar{1}$ (no. 2), $a = 6.984(4)$ Å, $b = 7.494(4)$ Å, $c = 9.174(5)$ Å, $\alpha = 97.249(9)^\circ$, $\beta = 95.155(8)^\circ$, $\gamma = 108.515(8)^\circ$, $V = 447.4(4)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.0538$, $wR_{\text{ref}}(F^2) = 0.1617$, $T = 296(2)$ K.

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The asymmetric unit of the title crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

The title compound is synthesized according to the literature known method [3]. In brief, *p*-nitrochlorobenzene (1.57 g, 10 mmol) and 2-mercapto-ethanol (0.86 g, 11 mmol) were dissolved in DMF (10 mL). NaOH (0.44 g, 11 mmol) was added in portions during a period of 3 h. After then, the reaction mixture was stirred at room temperature for another 2 h, and neutralized by concentrated hydrochloric acid. The evaporation of the solvent and recrystallization of the residue in EtOH/H₂O (1:1, v:v) yield yellow plate crystals.

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.2852(3)	0.1541(3)	0.6962(2)	0.0433(6)
H1A	0.390893	0.105597	0.712958	0.052*
C2	0.1665(3)	0.1680(3)	0.8052(2)	0.0433(6)
H2A	0.190757	0.129968	0.895750	0.052*
C3	0.0095(3)	0.2401(3)	0.7776(2)	0.0413(6)
H3A	0.601173	0.144326	0.087239	0.094*
C4	−0.0337(3)	0.2908(3)	0.6430(3)	0.0455(6)
H4A	−0.143052	0.334390	0.625803	0.055*
C5	0.0850(3)	0.2767(3)	0.5343(2)	0.0431(6)
H5A	0.056239	0.310533	0.442775	0.052*
C6	0.2500(3)	0.2115(3)	0.5608(2)	0.0380(5)
C7	0.3472(3)	0.3047(3)	0.2843(2)	0.0455(6)
H7A	0.212532	0.227622	0.234038	0.055*
H7B	0.343364	0.429941	0.322803	0.055*
C8	0.4992(4)	0.3222(3)	0.1778(3)	0.0513(6)
H8A	0.466664	0.387157	0.098975	0.062*
H8B	0.634715	0.395843	0.228498	0.062*
O1	−0.0773(3)	0.2127(3)	1.0123(2)	0.0709(6)
O2	−0.2450(3)	0.3330(3)	0.8710(2)	0.0724(6)
O3	0.4932(3)	0.1385(2)	0.11832(18)	0.0628(6)
N1	−0.1122(3)	0.2632(3)	0.8941(2)	0.0490(5)
S1	0.41818(8)	0.19517(8)	0.43422(6)	0.0474(3)

Experimental details

The structure was solved by direct methods and refined with the SHELX crystallographic software package [1]. The hydrogen atoms were placed at calculated positions and refined as riding atoms with isotropic displacement parameters.

Comment

The *p*-aminophenyl- β -sulfatoethylsulfone is one of the most important intermediates of vinyl-sulfone reactive

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dyes [4]. Recently, a novel route for the production of *p*-aminophenyl- β -sulfatoethylsulfone has been proposed by using *p*-nitrochlorobenzene as the raw material, and the title compound is the product of the first step of this synthetic protocol [3]. Its crystal structure is presented in this paper.

In the title compound, both the S atom and the nitro group are well conjugated with the central six-membered aryl moiety with the r.m.s. deviation of 0.0360 Å. In the crystal, pairs of intermolecular O—H...O hydrogen bonds link two molecules into centrosymmetric dimers. Bond lengths and angles are all in the expected ranges [5].

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