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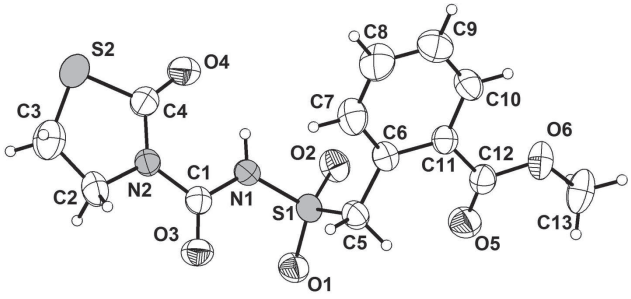
Crystal structure of 2-{[(2-oxo-thiazolidine-3-carbonyl)sulfamoyl]-methy}-benzoic acid methyl ester, $C_{13}H_{14}N_2O_6S_2$



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Abstract

$C_{13}H_{14}N_2O_6S_2$, monoclinic, $C2/c$ (no. 15), $a = 28.1174(10)$Å, $b = 9.8677(4)$Å, $c = 11.2738(4)$Å, $\beta = 98.4457(11)°$, $V = 3094.0(2)$Å^3, $Z = 8$, $R_{gt}(F) = 0.0382$, $wR_{ref}(F^2) = 0.1141$, $T = 296(2)$ K.

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

Table 1: Data collection and handling.

Crystal:	Colourless block
	Size 0.28 × 0.17 × 0.10 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	3.8 cm^{-1}
Diffractometer, scan mode:	Rigaku RAXIS-RAPID, ω-scans
$2\theta_{max}$, completeness:	54.8°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	14936, 3529, 0.050
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2326
$N(param)_{refined}$:	210
Programs:	SHELX [7], CrystalClear [8]

Source of material

2-Isocyanatosulfonylmethyl-benzoic acid methyl ester (2.55 g, 0.01 mol), 2-thiazolidione (1.03 g, 0.01 mol) and anhydrous methylene chloride (50 mL) were added into a three-necked flask. After stirring at ambient temperature for sixteen hours the solvent was removed under reduced pressure. The residue was purified by column chromatography on silica gel using petroleum ether/acetone (1:2 v/v) as the eluent, giving a light yellow solid (yield 85%). m.p.: 132-134 °C. IR(KBr): 3168 (N—H); 1743 (C = O); 1382, 1353, 1196 (O = S=O) cm^{-1}; ^{1}H NMR (TMS, $CDCl_3$): δ 3.35(t, 2H, CH2), 3.93(s, 3H, OCH_3), 4.26(t, 2H, CH_2), 5.35(s, 2H, CH_2), 7.44–7.54(m, 3H, ArH), 7.96–7.98 (t, H, ArH), 10.26(s, H, NH). Elemental Anal. Calcd. (%): C, 43.57; H, 3.94; N, 7.82. Found(%): C, 43.65; H, 4.01; N, 7.71.

Experimental details

The structure was solved by direct methods and successive Fourier difference synthesis [7, 8]. H atoms were positioned geometrically and refined using a riding model [aliphatic C—H = 0.97(2) Å and N—H = 0.86 Å, $U_{iso}(H) = 1.2U_{eq}(C)$].

Discussion

Many scientists have been devoted to study pesticides which have ultra-low application rates and extremely low mammal toxicity. Since chlorsulfuron, a sulfonylureas herbicide, was found in 1982, various novel sulfonylurea herbicides [1] have been found and commercialized, such as Chlorimuron-ethyl, primisulfuron, Monosulfuron and foramsulfuron. Synthesis of a broader spectrum and highly biological sulfonylurea compounds became a hot spot. In view of these facts, and as a part of our work on the synthesis of bioactive lead compounds for drug discover [2], the title compounds were designed by introducing a 2-thiazolidione pharmacophore into sulfonylurea scaffold. A new sulfonylurea derivative containing 2-thiazolidione had been synthesized by the

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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}
S1	0.38056(2)	0.58058(6)	0.71532(5)	0.04198(18)
S2	0.21754(3)	0.62282(8)	0.28584(8)	0.0711(3)
C1	0.34043(8)	0.6800(2)	0.5013(2)	0.0403(5)
O2	0.35616(6)	0.50676(18)	0.79716(14)	0.0520(4)
N2	0.29830(6)	0.68091(19)	0.41667(17)	0.0409(4)
N1	0.33947(7)	0.58930(19)	0.59385(17)	0.0438(5)
H1	0.3159	0.5331	0.5883	0.053*
O3	0.37315(6)	0.75475(18)	0.49105(15)	0.0534(4)
O4	0.25432(7)	0.5098(2)	0.48893(18)	0.0636(5)
C4	0.25991(9)	0.5947(3)	0.4137(2)	0.0473(6)
O5	0.46193(7)	0.39568(19)	0.90780(17)	0.0632(5)
O1	0.39832(7)	0.71296(17)	0.74628(15)	0.0550(5)
O6	0.47368(6)	0.17356(19)	0.93706(16)	0.0604(5)
C6	0.41671(8)	0.3334(2)	0.6607(2)	0.0431(5)
C10	0.41887(9)	0.1029(2)	0.7323(2)	0.0538(6)
H10	0.4281	0.0401	0.7929	0.065*
C12	0.45661(8)	0.2809(3)	0.8724(2)	0.0455(6)
C11	0.43044(8)	0.2389(2)	0.7525(2)	0.0412(5)
C5	0.42798(8)	0.4825(2)	0.6710(2)	0.0474(6)
H5A	0.4348	0.5154	0.5941	0.057*
H5B	0.4567	0.4953	0.7290	0.057*
C2	0.29779(10)	0.7727(3)	0.3141(2)	0.0578(7)
H2A	0.3260	0.7573	0.2756	0.069*
H2B	0.2982	0.8661	0.3410	0.069*
C7	0.39179(10)	0.2869(3)	0.5528(2)	0.0596(7)
H7	0.3825	0.3482	0.4911	0.072*
C8	0.38054(12)	0.1516(3)	0.5354(3)	0.0734(9)
H8	0.3636	0.1229	0.4626	0.088*
C13	0.49567(11)	0.1999(4)	1.0589(3)	0.0777(9)
H13A	0.5182	0.2732	1.0596	0.116*
H13B	0.5122	0.1201	1.0916	0.116*
H13C	0.4713	0.2239	1.1064	0.116*
C9	0.39415(11)	0.0591(3)	0.6246(3)	0.0683(8)
H9	0.3868	−0.0322	0.6126	0.082*
C3	0.25320(13)	0.7463(4)	0.2273(3)	0.0856(10)
H3A	0.2618	0.7148	0.1518	0.103*
H3B	0.2350	0.8296	0.2122	0.103*

reaction of 2-thiazolidiones and 2-isocyanatosulfonylmethylbenzoic acid methyl ester.

In the crystal structure, the bond length of N(1)—S(1) 1.6592(19) Å is shorter than S(1)—C(5) 1.777(2) Å and S(1)—C(5) 1.766(4) Å the bond lengths of N1—C1 and N2—C4 are 1.378(3) and 1.371(3) respectively. The torsion angle of thioether group C5—S1—N1—C1 is -82.4(2)°. In the aromatic rings, the molecular dimensions are as expected with the aromatic C—C bond distances between 1.371(4) and 1.405(3) Å. The aromatic C—C—C bond angles ranging from 118.3(2) to 121.5(3)o are almost within the normal ranges [3].

There exist no classical hydrogen bonds in the molecule. The herbicidal, fungicidal, insecticidal activities of the title compound were measured according to the reference method [4–6]. The results indicated that the title compound showed weak inhibitory activity against Fusarium oxysporum, Rhizoctonia solani, Gibberella zeae, Botryospuaeria berengeriana and Colletotrichum gossypii at 50 ppm, respectively. The title compound exhibited moderate insecticidal activity (48.1%) against aphid. For the herbicidal activity, the compound displayed moderate activity against the root of Brassica napus and Echinochloa crusgalli at 100 ppm, but it showed weak inhibitory at 10 ppm.

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