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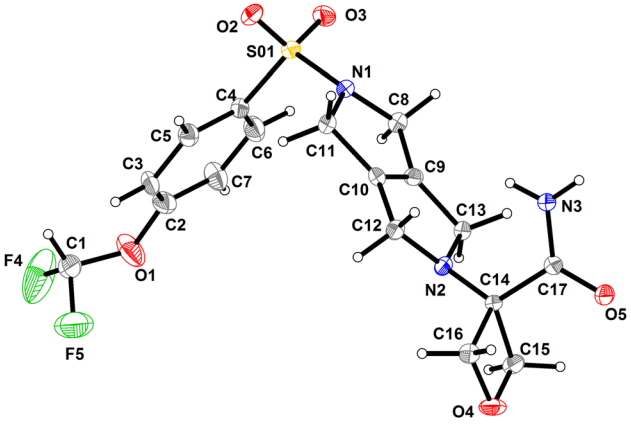
Crystal structure of 3-(5-((4-(difluoromethoxy) phenyl) sulfonyl)-3,4,5,6-tetrahydropyrrolo[3,4-*c*] pyrrol-2(1*H*)-yl) oxetane-3-carboxamide, $C_{17}H_{19}F_2N_3O_5S$



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Abstract

$C_{17}H_{19}F_2N_3O_5S$, monoclinic, $P2_1/c$ (no. 14), a = 14.157(7) Å, b = 11.133(6) Å, c = 11.400(6) Å, β = 91.965(13)°, V = 1795.6(16) Å^3, Z = 4, $R_{gt}(F)$ = 0.0611 $wR_{ref}(F^2)$ = 0.1809, T = 205 K.

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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.

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Table 1: Data collection and handling.

Crystal:	Colourless plate
Size:	0.19 × 0.12 × 0.06 mm
Wavelength:	Mo *K*α 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}:	12776, 3160, 0.116
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 1955
$N(param)_{refined}$:	281
Programs:	Bruker [1], SHELX [2, 3], Olex2 [4]

1 Source of materials

To a stirred mixture of 3-(5-((4-(difluoromethoxy)phenyl) sulfonyl)-3,4,5,6-tetrahydropyrrolo[3,4-*c*]pyrrol-2(1*H*)-yl) oxetane-3-carbonitrile (397 mg, 1.0 mmol) and potassium carbonate (83 mg, 0.6 mmol) in dimethyl sulfoxide (10 mL) was added hydrogen peroxide (5 mL, 30 %) and then the reaction mixture was stirred at room temperature for 6 h. The progress of the reaction was monitored by TLC. After completion of reaction, the reaction was quenched by water (1 mL). The mixture was extracted with ethyl acetate (3 × 15 mL) and the organic layers combined and dried over anhydrous sodium sulfate and concentrated under vacuum. The title compound was separated by silica-gel column chromatography with ethyl acetate-petroleum ether (10 %) gradient solvent system. The target product was obtained as a white solid. Yield: 83.6 %.

2 Experimental details

Hydrogen atoms were positioned in their ideal locations and refined as riding atoms, with U_{iso} values set to 1.2 times the equivalent isotropic displacement parameter (U_{eq}) of the parent atoms.

3 Comment

Oxetane, referred to as oxygen-containing cyclobutane compounds, are cyclic compounds containing a single

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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	0.0607 (4)	0.2849 (4)	−0.0327 (4)	0.0517 (13)
H1A[a]	0.006979	0.338876	−0.017260	0.062*
H1B[b]	0.002683	0.324868	−0.006383	0.062*
C2	0.1505 (3)	0.4243 (4)	0.0849 (4)	0.0430 (11)
C3	0.0816 (3)	0.4527 (4)	0.1623 (4)	0.0415 (11)
H3	0.025055	0.408597	0.163030	0.050*
C4	0.1808 (3)	0.6121 (3)	0.2389 (3)	0.0291 (9)
C5	0.0972 (3)	0.5485 (4)	0.2397 (3)	0.0357 (10)
H5	0.050544	0.569618	0.292691	0.043*
C6	0.2502 (3)	0.5815 (4)	0.1613 (4)	0.0439 (11)
H6	0.307511	0.624220	0.161527	0.053*
C7	0.2345 (3)	0.4868 (4)	0.0828 (4)	0.0477 (12)
H7	0.280666	0.465909	0.029059	0.057*
C8	0.3454 (3)	0.6221 (3)	0.4605 (3)	0.0319 (10)
H8A	0.361144	0.590642	0.383312	0.038*
H8B	0.391641	0.683534	0.484840	0.038*
C9	0.3393 (3)	0.5240 (3)	0.5498 (3)	0.0278 (9)
C10	0.2547 (3)	0.5125 (3)	0.5911 (3)	0.0268 (9)
C11	0.1839 (3)	0.5967 (3)	0.5365 (3)	0.0314 (9)
H11A	0.153778	0.646423	0.595587	0.038*
H11B	0.135175	0.553847	0.489914	0.038*
C12	0.2514 (3)	0.4171 (3)	0.6843 (3)	0.0316 (9)
H12A	0.241398	0.451753	0.761892	0.038*
H12B	0.202071	0.357516	0.666211	0.038*
C13	0.4096 (3)	0.4382 (3)	0.6034 (3)	0.0280 (9)
H13A	0.439758	0.389571	0.543580	0.034*
H13B	0.458250	0.479953	0.651061	0.034*
C14	0.3917 (3)	0.3081 (3)	0.7776 (3)	0.0252 (9)
C15	0.4571 (3)	0.2030 (3)	0.7442 (3)	0.0369 (10)
H15A	0.458671	0.189241	0.659379	0.044*
H15B	0.521402	0.210431	0.778191	0.044*
C16	0.3322 (3)	0.2064 (3)	0.8340 (4)	0.0378 (10)
H16A	0.325736	0.216096	0.918840	0.045*
H16B	0.270181	0.194623	0.794836	0.045*
C17	0.4380 (3)	0.3896 (3)	0.8720 (3)	0.0285 (9)
F4[a]	0.0589 (12)	0.2498 (13)	−0.1406 (13)	0.090 (4)
F4A[b]	0.0862 (17)	0.312 (5)	−0.148 (2)	0.115 (11)
F5[a]	0.0563 (6)	0.1802 (10)	0.0333 (16)	0.075 (4)
F5A[b]	0.0558 (14)	0.1727 (18)	−0.041 (5)	0.116 (10)
N1	0.2463 (2)	0.6694 (3)	0.4609 (3)	0.0288 (8)
N2	0.3471 (2)	0.3646 (3)	0.6769 (3)	0.0282 (8)
N3	0.4092 (2)	0.5027 (3)	0.8776 (3)	0.0319 (8)
H3A	0.432490	0.550017	0.932025	0.038*
H3B	0.366974	0.529574	0.826949	0.038*
O1[a]	0.1435 (10)	0.3387 (13)	−0.0075 (13)	0.055 (3)
O1A[b]	0.1428 (17)	0.313 (2)	0.032 (2)	0.048 (5)
O2	0.1141 (2)	0.7815 (2)	0.3684 (3)	0.0463 (8)
O3	0.2772 (2)	0.8021 (2)	0.2955 (2)	0.0439 (8)
O4	0.4001 (2)	0.1164 (2)	0.8043 (3)	0.0480 (8)
O5	0.4978 (2)	0.3475 (2)	0.9421 (2)	0.0422 (8)
S01	0.20337 (8)	0.72923 (8)	0.34131 (9)	0.0348 (3)

[a]Occupancy: 0.65 (4), [b]Occupancy: 0.35 (4).

oxygen atom within their molecular structure. Oxetanes are high-energy non-aromatic heterocycles with oxygen content, exhibiting notable anti-tumor, antiviral, and antifungal properties. Furthermore, they display activity as angiogenic stimulants, respiratory stimulants, and anti-allergic agents [5]. Oxetanes are currently a subject of great interest as they represent potential pharmacophores with significant and promising biological activity. In this article, we present the precise single-crystal structure of a novel oxetane compound containing oxygen.

In the crystal structure depicted in the figure of this study, the (difluoromethoxy)phenyl group and 3,4,5,6-(tetrahydropyrrolo[3,4-c] pyrrol-2(1H)-yl)oxetane-3-carboxamide group replace two oxygen atoms on the sulfonic acid moiety. The substitution position is characterized by a bond angle of 107.82(19)° for N1–S01–C4. Notably, the four atoms within the oxetane group are nearly coplanar, exhibiting a dihedral angle of only 3.1(4)° for C14–C15–O4–C16. This arrangement aligns with the structural features observed in previous literature reports on oxetane derivatives [6–13].

Additionally, the crystal structure reveals the presence of two hydrogen bonds between the carboxamide groups in the molecule. The N3–H3A–O5 bond angle measures 173.3(3)°, while the bond length (H3A–O5) is determined to be 2.057(4) Å, indicating the prominent role of hydrogen bonding in maintaining intermolecular stability. The molecules undergo stacking in the b-axis direction, resulting in the formation of the crystal lattice structure.

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