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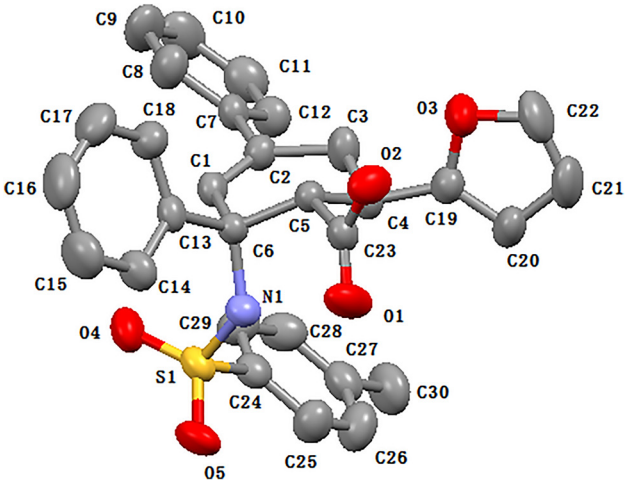
The crystal structure of 5′-(furan-2-yl)-3′-((4-methylphenyl)sulfonamido)-3′,4′,5′,6′-tetrahydro-[1,1′:3′,1″-terphenyl]-4′-carboxylic acid, $C_{30}H_{27}NO_5S$



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Abstract

$C_{30}H_{27}NO_5S$, monoclinic, $P2_1$ (no. 4), a = 10.4329(16) Å, b = 11.6208(18) Å, c = 11.8268(18) Å, β = 114.257(5)°, V = 1307.3(4) Å^3, Z = 2, $R_{gt}(F)$ = 0.0685, wR_{ref} (F^2) = 0.1547, T = 273(2) 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.

Table 1: Data collection and handling.

Crystal:	Yellow needle
Size:	0.24 × 0.19 × 0.13 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.17 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II
θ_{max}, completeness:	32.1°, 99 %
N (hkl)$_{measured}$, N(hkl)$_{unique}$, R_{int}:	16,448, 8845, 0.059
Criterion for I_{obs}, N(hkl)$_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 4334
N(param)$_{refined}$:	336
Programs:	Bruker [1], SHELX [2, 3]

1 Source of materials

The title compound was obtained via the following synthetic procedure: *N*-((*E*)-1,3-diphenylbut-2-en-1-ylidene)-4-methylbenzenesulfonamide (0.2 mmol, 1 eq) and (*E*)-3-(furan-2-yl)acrylaldehyde (2 eq) were added into a dry Schlenk tube, which was filled with (*R*)-diphenylprolinol trimethyl silyl ether (0.2 eq), dipotassium phosphate (1 eq), monopotassium phosphate (1 eq) and sodium chloride (1 eq). The tube was heated at 60 °C to initiate the reaction under nitrogen atmosphere with 2 mL of MeOH/H_2O (99:1) as solvent. After 12 h, the reaction mixture was separated over silica gel chromatography eluted with petroleum ether/ethyl acetate (7:1) to obtain the intermediate in 55 % yield, which was further transformed to the title compound in 64 % yield using $KMnO_4$ (2 eq) as the oxidant. The structure of the title compound was determined by NMR analyses before XRD analyses. The yellow needles were obtained via recrystallization in dichloromethane/ethyl acetate.

2 Experimental details

The structure was solved and refined using the Bruker SHELXTL Software Package [2, 3].

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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	0.2876 (4)	0.3462 (3)	0.5984 (4)	0.0388 (9)
C2	0.2714 (4)	0.4295 (3)	0.5169 (4)	0.0404 (9)
C3	0.2906 (5)	0.5542 (4)	0.5545 (4)	0.0513 (11)
C4	0.3520 (5)	0.5723 (3)	0.6941 (4)	0.0428 (10)
C5	0.2820 (4)	0.4877 (3)	0.7527 (4)	0.0355 (9)
C6	0.3209 (4)	0.3623 (3)	0.7354 (3)	0.0350 (8)
C7	0.2313 (4)	0.4042 (3)	0.3840 (4)	0.0395 (9)
C8	0.1254 (5)	0.3255 (4)	0.3217 (5)	0.0592 (13)
C9	0.0868 (6)	0.3023 (4)	0.1963 (5)	0.0717 (16)
C10	0.1545 (6)	0.3566 (5)	0.1330 (5)	0.0699 (15)
C11	0.2582 (6)	0.4341 (5)	0.1937 (5)	0.0622 (14)
C12	0.2948 (5)	0.4589 (4)	0.3170 (4)	0.0496 (11)
C13	0.2345 (4)	0.2771 (3)	0.7752 (4)	0.0382 (9)
C14	0.2943 (5)	0.2068 (4)	0.8767 (5)	0.0526 (12)
C15	0.2132 (7)	0.1312 (5)	0.9112 (6)	0.0708 (16)
C16	0.0712 (7)	0.1258 (5)	0.8425 (6)	0.0722 (16)
C17	0.0107 (6)	0.1961 (5)	0.7416 (6)	0.0672 (15)
C18	0.0911 (5)	0.2704 (4)	0.7071 (4)	0.0534 (11)
C19	0.3430 (5)	0.6956 (4)	0.7277 (4)	0.0474 (11)
C20	0.4383 (5)	0.7724 (4)	0.7932 (4)	0.0585 (12)
C21	0.3669 (7)	0.8738 (4)	0.7985 (5)	0.0702 (15)
C22	0.2322 (7)	0.8511 (4)	0.7373 (5)	0.0727 (16)
C23	0.3188 (5)	0.5232 (3)	0.8844 (4)	0.0419 (10)
C24	0.6632 (4)	0.3420 (4)	0.7127 (4)	0.0443 (10)
C25	0.7755 (5)	0.4122 (4)	0.7806 (5)	0.0591 (13)
C26	0.8407 (6)	0.4761 (5)	0.7209 (6)	0.0685 (15)
C27	0.7947 (5)	0.4712 (4)	0.5932 (5)	0.0545 (12)
C28	0.6822 (5)	0.3999 (5)	0.5281 (5)	0.0587 (13)
C29	0.6164 (5)	0.3352 (4)	0.5867 (4)	0.0528 (12)
C30	0.8677 (6)	0.5359 (5)	0.5261 (5)	0.0756 (16)
H1A	0.507903	0.391914	0.876635	0.049*
H2	0.917621	0.600226	0.575578	0.113*
H2A	0.247937	0.613197	0.965108	0.084*
H3	0.200114	0.592562	0.517648	0.062*
H3A	0.932782	0.485673	0.512137	0.113*
H4	0.916345	0.523085	0.767013	0.082*
H5	0.807135	0.416459	0.866332	0.071*
H5A	0.179995	0.495575	0.707244	0.043*
H6	0.390914	0.209818	0.923429	0.063*
H7	0.255383	0.084507	0.980587	0.085*
H8	0.048270	0.316468	0.637284	0.064*
H9	−0.086078	0.193629	0.695622	0.081*
H10	0.016289	0.074863	0.864225	0.087*
H10A	0.129781	0.340611	0.049552	0.084*
H11	0.015171	0.249965	0.155434	0.086*
H12	0.079843	0.287904	0.364157	0.071*
H13	0.304789	0.470599	0.151492	0.075*
H14	0.363841	0.513776	0.355923	0.060*
H15	0.352032	0.589987	0.521622	0.062*
H16	0.160196	0.902788	0.727204	0.087*
H17	0.406781	0.942531	0.837317	0.084*
H18	0.535174	0.761398	0.829318	0.070*
H19	0.451920	0.552272	0.726321	0.051*
H21	0.277685	0.270986	0.569149	0.047*
H22	0.541235	0.287538	0.541057	0.063*
H23	0.649795	0.395437	0.442316	0.070*
H24	0.799205	0.563059	0.448008	0.113*
N1	0.4729 (3)	0.3484 (3)	0.8123 (3)	0.0410 (8)
O1	0.4316 (3)	0.5073 (3)	0.9683 (3)	0.0660 (10)
O2	0.2166 (3)	0.5799 (3)	0.8981 (3)	0.0560 (8)
O3	0.2119 (3)	0.7415 (3)	0.6905 (3)	0.0607 (9)
O4	0.5042 (3)	0.1702 (2)	0.7112 (3)	0.0586 (9)
O5	0.6877 (3)	0.2360 (3)	0.9114 (3)	0.0580 (9)
S1	0.58051 (11)	0.26194 (8)	0.79045 (11)	0.0438 (3)

3 Comment

The title compound belongs to cyclic β-amino acids characterized by well-defined and controllable conformational preferences, demonstrating its potential as building blocks in the field of foldamer research [4, 5]. Additionally, cyclic β-amino acids also exhibited various bioactivities such as anti-tumor and anti-influenza [6–8]. Thus, it has attracted an increased interest in the past two decades, and several effective methods have been developed to access cyclic β-amino acids [9, 10].

There is one crystallographically independent molecule in the asymmetric unit of the title compound with all geometric parameters in normal ranges [11]. The cyclohexene skeleton was shown in its half-chair conformation with C1–C2–C3–C6 almost in the same plane. The dihedral angle between phenyl plane at C2 with C1–C2–C3 plane was determined as 44.5°. Only one intramolecular hydrogen bond was observed between N1–H1A and O1 with donor–acceptor distance of 2.078 Å, and the molecules were packed through the intermolecular hydrogen bond between O2–H2A from the carboxyl group with O5 from the sulfonyl group with donor-acceptor distance of 1.953 Å.

Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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

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