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Crystal structure of (2*S*, 4*R*)-2-benzyl 1-*tert*-butyl 4-(tosyloxy)pyrrolidine-1,2-dicarboxylate, $C_{24}H_{29}NO_7S$

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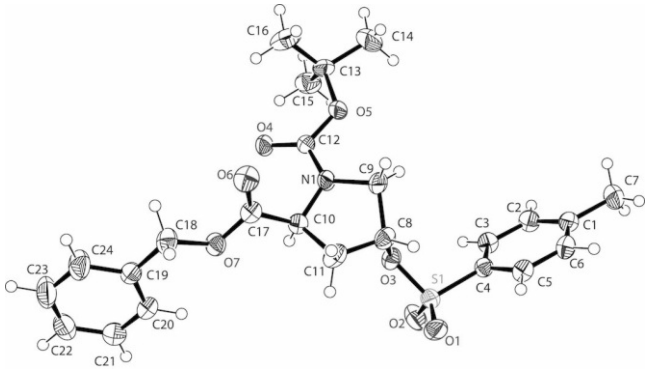

Abstract

$C_{24}H_{29}NO_7S$, triclinic, $P\bar{1}$ (no. 1), a = 6.5903(3) Å, b = 9.7193(5) Å, c = 9.9874(4) Å, α = 82.462(4)°, β = 77.197(3)°, γ = 85.6180(4)°, V = 617.7 Å^3, Z = 1, $R_{gt}(F)$ = 0.0351, $wR_{ref}(F^2)$ = 0.0967, T = 123 K.

Table 1. Data collection and handling.

Crystal:	colourless prisms, size 0.1551×0.2145×0.3113 mm
Wavelength:	Cu K_α radiation (1.54184 Å)
μ:	15.30 cm^{-1}
Diffractometer, scan mode:	SuperNova System, ω
$2\theta_{max}$:	147.46°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	22769, 4737
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 4714
$N(param)_{refined}$:	298
Programs:	SIR-97 [4], SHELXL-97 [5], PLATON [6]

Source of material

All chemicals were purchased from commercial suppliers and used without further purification. The title compound was synthesized following the general procedure reported in the literature [1]. *p*-Toluenesulfonyl chloride (1.31 g, 6.84 mmol) was added portionwise to a solution of 2-benzyl 1-*tert*-butyl (2*S*,4*R*)-4-hydroxypyrrolidine-1,2-dicarboxylate [2] (2 g, 6.22 mmol) in pyridine (10 mL) at 0 °C. The mixture was stirred for 12 h at room temperature, the solvent evaporated in vacuo and the residue poured into an ice bath. The mixture was extracted with ethyl acetate (3 × 50 mL) and washed subsequently with 1M HCl, H_2O, Na_2CO_3 (10%) and H_2O. The combined organic phases were dried over anhydrous Na_2SO_4, filtered and the solvent evaporated to give a yellow oil, which was purified by column chromatography (eluent = hexane:ethyl acetate 8:2). The title compound was obtained as a colourless oil (yield 75%) and displayed spectroscopic data identical to those reported in the literature [3]. The compound spontaneously crystallized over time to white crystals suitable for *X*-ray analysis (m.p. 76-77 ºC). An Ortep diagram with ellipsoids drawn at the 50% probability level is shown.

Experimental details

The data were collected at 123K using an Oxford Instruments Cryojet Cooler. The structure was solved by direct methods (SIR-97 [4]) and refined by full-matrix anisotropic least squares (SHELXL-97 [5]).

All hydrogen atoms on carbons were generated geometrically with d(C–H) = 0.95-1.00 Å and U_{iso}(H) = 1.28 U_{eq}(C).

Discussion

Substituted prolines have attracted a great deal of attention in the development of molecular probes, ligands, and asymmetric organocatalysts [3]. In the title compound, the characteristic C=O, C–O and C–N bond distances are in the ranges 1.217(2)-1.197(2) Å, 1.332(2)-1.482(2) Å and 1.354(2)-1.464(3) Å, respectively. The C–S bond distance is 1.7560(19) Å. The characteristic ∠O–C–C, ∠O–S–C and ∠C–N–C bond angles are in the ranges 102.22-125.48°, 105.17-108.76° and 113.48-125.84°, respectively. Other bond distances and angles are all in normal ranges. The molecular structure is further extended through intermolecular CH–O interactions (H–O bond distances are in the range 2.352(1)-2.715(1) Å) and CH–π interactions (centroid–H distances are 3.150 and 3.082 Å).

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(2)	1*a*	0.4169	–0.0555	0.7089	0.045
H(3)	1*a*	0.2365	0.1057	0.5796	0.04
H(5)	1*a*	0.7837	0.2138	0.3212	0.039
H(6)	1*a*	0.9613	0.0508	0.4516	0.044
H(7A)	1*a*	0.7744	–0.2185	0.6763	0.063
H(7B)	1*a*	0.7987	–0.1032	0.7716	0.063
H(7C)	1*a*	0.9732	–0.1247	0.6354	0.063
H(8)	1*a*	0.4911	0.1747	0.1141	0.042
H(9A)	1*a*	0.3662	–0.0281	0.0678	0.05
H(9B)	1*a*	0.2568	–0.0288	0.2293	0.05
H(10)	1*a*	–0.0799	0.2401	0.1016	0.035
H(11A)	1*a*	0.3297	0.2442	–0.0716	0.046
H(11B)	1*a*	0.223	0.3528	0.0353	0.046
H(14A)	1*a*	–0.1169	–0.4765	0.325	0.082
H(14B)	1*a*	0.0042	–0.3603	0.3711	0.082
H(14C)	1*a*	0.0886	–0.4158	0.2239	0.082
H(15A)	1*a*	–0.3234	–0.2104	0.4102	0.061
H(15B)	1*a*	–0.4447	–0.3262	0.3637	0.061
H(15C)	1*a*	–0.4462	–0.1706	0.288	0.061

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Table 2. continued.

Atom	Site	*x*	*y*	*z*	U_{iso}
H(16A)	1*a*	–0.2955	–0.4074	0.1216	0.07
H(16B)	1*a*	–0.0886	–0.3384	0.031	0.07
H(16C)	1*a*	–0.3036	–0.2477	0.058	0.07
H(18A)	1*a*	–0.2665	0.2438	–0.2762	0.049
H(18B)	1*a*	–0.099	0.3617	–0.3263	0.049

Table 2. continued.

Atom	Site	*x*	*y*	*z*	U_{iso}
H(20)	1*a*	–0.3461	0.5183	–0.0725	0.052
H(21)	1*a*	–0.6241	0.6823	–0.0573	0.06
H(22)	1*a*	–0.8358	0.7059	–0.2182	0.061
H(23)	1*a*	–0.7739	0.5589	–0.3914	0.074
H(24)	1*a*	–0.496	0.3942	–0.4069	0.065

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
S(1)	1*a*	0.35568(5)	0.29985(4)	0.34512(4)	0.0409(2)	0.0305(2)	0.0327(2)	–0.0020(2)	–0.0141(2)	0.0011(2)
O(1)	1*a*	0.5015(3)	0.3823(2)	0.2459(2)	0.063(1)	0.0363(8)	0.0454(8)	–0.0202(7)	–0.0259(7)	0.0126(6)
O(2)	1*a*	0.1956(3)	0.3629(2)	0.4421(2)	0.0607(9)	0.0487(9)	0.0453(8)	0.0200(7)	–0.0200(7)	–0.0115(7)
O(3)	1*a*	0.2320(2)	0.2161(1)	0.2668(1)	0.0280(6)	0.0407(8)	0.0329(6)	–0.0059(5)	–0.0075(5)	–0.0012(5)
O(4)	1*a*	–0.2583(2)	–0.0116(1)	0.0989(1)	0.0251(6)	0.0338(7)	0.0443(7)	–0.0041(5)	–0.0131(5)	0.0026(5)
O(5)	1*a*	–0.0284(2)	–0.1629(1)	0.1902(2)	0.0380(7)	0.0254(7)	0.0650(9)	–0.0039(5)	–0.0260(6)	0.0023(6)
O(6)	1*a*	0.0331(2)	0.1131(2)	–0.1843(2)	0.0408(7)	0.0527(9)	0.0342(7)	0.0021(6)	–0.0017(5)	–0.0145(6)
O(7)	1*a*	–0.1579(2)	0.3039(2)	–0.1205(1)	0.0434(7)	0.0400(8)	0.0263(6)	0.0019(6)	–0.0117(5)	–0.0056(6)
N(1)	1*a*	0.0643(2)	0.0492(2)	0.0999(2)	0.0203(7)	0.0309(8)	0.0454(8)	–0.0028(5)	–0.0108(6)	0.0008(6)
C(1)	1*a*	0.7078(3)	–0.0201(2)	0.5930(2)	0.039(1)	0.033(1)	0.0367(9)	–0.0018(7)	–0.0137(8)	–0.0040(7)
C(2)	1*a*	0.4915(3)	–0.0014(2)	0.6296(2)	0.0390(9)	0.039(1)	0.0311(9)	–0.0074(7)	–0.0061(7)	0.0061(7)
C(3)	1*a*	0.3838(3)	0.0939(2)	0.5532(2)	0.0299(8)	0.038(1)	0.0311(9)	–0.0034(7)	–0.0017(7)	–0.0011(7)
C(4)	1*a*	0.4934(3)	0.1729(2)	0.4367(2)	0.0315(8)	0.031(1)	0.0259(8)	–0.0025(7)	–0.0078(6)	–0.0025(7)
C(5)	1*a*	0.7091(3)	0.1583(2)	0.3993(2)	0.0312(8)	0.041(1)	0.0255(8)	–0.0070(7)	–0.0027(6)	–0.0022(7)
C(6)	1*a*	0.8139(3)	0.0617(2)	0.4774(2)	0.0280(8)	0.046(1)	0.0364(9)	–0.0019(7)	–0.0080(7)	–0.0055(8)
C(7)	1*a*	0.8237(4)	–0.1259(3)	0.6764(3)	0.052(1)	0.047(1)	0.061(1)	0.001(1)	–0.028(1)	0.006(1)
C(8)	1*a*	0.3362(3)	0.1697(2)	0.1328(2)	0.0236(8)	0.041(1)	0.0399(9)	–0.0057(7)	–0.0039(7)	–0.0083(8)
C(9)	1*a*	0.2683(3)	0.0236(2)	0.1362(3)	0.0234(8)	0.039(1)	0.069(1)	0.0018(7)	–0.0193(8)	–0.013(1)
C(10)	1*a*	0.0301(3)	0.1894(2)	0.0375(2)	0.0269(8)	0.0318(9)	0.0262(8)	–0.0048(6)	–0.0024(6)	–0.0001(7)
C(11)	1*a*	0.2414(3)	0.2533(2)	0.0212(2)	0.0356(9)	0.048(1)	0.0308(9)	–0.0179(8)	–0.0026(7)	–0.0009(8)
C(12)	1*a*	–0.0894(3)	–0.0418(2)	0.1280(2)	0.0261(8)	0.0268(9)	0.0371(9)	–0.0029(6)	–0.0100(6)	–0.0026(7)
C(13)	1*a*	–0.1689(3)	–0.2803(2)	0.2275(2)	0.050(1)	0.0243(9)	0.049(1)	–0.0099(8)	–0.0182(9)	–0.0004(8)
C(14)	1*a*	–0.0365(5)	–0.3933(3)	0.2926(4)	0.078(2)	0.032(1)	0.100(2)	–0.003(1)	–0.038(2)	0.011(1)
C(15)	1*a*	–0.3628(4)	–0.2437(3)	0.3314(3)	0.065(1)	0.038(1)	0.046(1)	–0.012(1)	–0.008(1)	0.0024(9)
C(16)	1*a*	–0.2185(5)	–0.3221(3)	0.0982(3)	0.083(2)	0.048(1)	0.047(1)	–0.030(1)	–0.012(1)	–0.008(1)
C(17)	1*a*	–0.0296(3)	0.1938(2)	–0.1019(2)	0.0278(8)	0.037(1)	0.0264(8)	–0.0059(7)	–0.0005(6)	–0.0036(7)
C(18)	1*a*	–0.2190(3)	0.3301(2)	–0.2527(2)	0.046(1)	0.053(1)	0.0263(8)	0.0007(9)	–0.0124(8)	–0.0071(8)
C(19)	1*a*	–0.3921(3)	0.4400(2)	–0.2417(2)	0.039(1)	0.037(1)	0.0261(8)	–0.0117(8)	–0.0076(7)	0.0045(7)
C(20)	1*a*	–0.4324(4)	0.5263(2)	–0.1380(2)	0.048(1)	0.041(1)	0.048(1)	–0.0044(9)	–0.0228(9)	–0.0091(9)
C(21)	1*a*	–0.5976(4)	0.6240(2)	–0.1292(3)	0.056(1)	0.040(1)	0.059(1)	0.002(1)	–0.022(1)	–0.010(1)
C(22)	1*a*	–0.7240(4)	0.6377(3)	–0.2238(3)	0.049(1)	0.049(1)	0.053(1)	0.002(1)	–0.017(1)	0.008(1)
C(23)	1*a*	–0.6864(5)	0.5512(3)	–0.3266(3)	0.067(2)	0.073(2)	0.053(1)	0.010(1)	–0.035(1)	–0.003(1)
C(24)	1*a*	–0.5211(4)	0.4531(3)	–0.3356(2)	0.065(1)	0.068(2)	0.037(1)	0.006(1)	–0.025(1)	–0.009(1)

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