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Crystal structure of 1,4-bis(tosyl)-1,4-diazacycloheptane-6-amine, $C_{19}H_{25}N_3O_4S_2$

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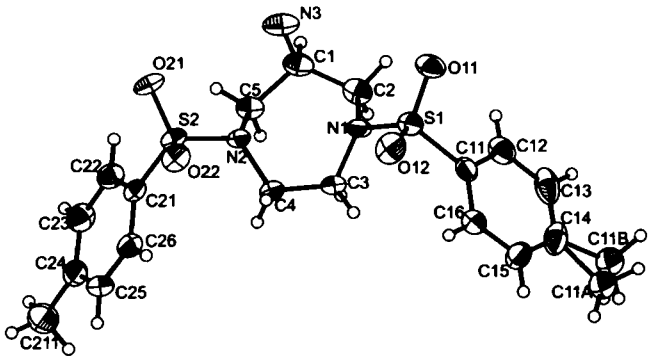

Abstract

$C_{19}H_{25}N_3O_4S_2$, orthorhombic, $P2_12_12_1$ (No. 19), $a = 6.396(1)$ Å, $b = 10.661(2)$ Å, $c = 30.430(6)$ Å, $V = 2075.0$ Å^3, $Z = 4$, $R_{gt}(F) = 0.056$, $wR_{ref}(F^2) = 0.136$, $T = 293$ K.

Source of material

The title compound was obtained from the corresponding azide [1] by hydrogenation (in DMF, Pd (10 %)/C, 4 bar, 298 K). The solid amine was precipitated by adding H_2O and single crystals were grown by slow cooling of a hot DMSO solution of the title compound to room temperature.

Experimental details

Positions of the H(-C) hydrogen atoms were calculated (riding model) with U_{iso} fixed at $1.2U_{eq}$ of the corresponding carbon atom. The hydrogen atoms of the amino group (N3) could not be located and were not considered. One of the methyl groups (C11) proved to be disordered and was refined with two positions (C11a and C11b) having site occupancies of each 50 %.

Discussion

1,4-Diazepane-6-amine could serve as an interesting, facially coordinating complexing agent for divalent transition metal cations [2], and the title compound was therefore prepared in our laboratory as an intermediate within the synthesis of this ligand. The seven-membered 1,4-diazepane ring adopts an approximate twist-chair (TC) conformation which is very similar to the conformation observed for the azide precursor [1], with an axial orientation of the amino group, a comparatively short C3—C4 bond of 145.9(7) pm, and a significantly flattened pyramidal geometry around the two ring nitrogen atoms (sum of bond angels around N1 and N2: 351.6° and 352.0°, respectively). The two S—N bonds with bond distances of 161.8(4) pm and 162.8(4) pm are again slightly shorter than a regular N—S single bond.

Table 1. Data collection and handling.

Crystal:	colorless prism, size 0.1 × 0.2 × 0.4 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	2.87 cm^{-1}
Diffractometer, scan mode:	Stoe IPDS, φ
$2\theta_{max}$:	48.14°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	11630, 3088
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2538
$N(param)_{refined}$:	252
Programs:	SHELXS-97 [3], SHELXL-97 [4]

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

Atom	Site	Occ.	x	y	z	U_{iso}
H(1)	4a		0.2877	0.6147	0.1983	0.072
H(2A)	4a		0.4062	0.4381	0.1629	0.075
H(2B)	4a		0.4382	0.5538	0.1318	0.075
H(3A)	4a		0.6586	0.3311	0.1913	0.083
H(3B)	4a		0.8871	0.3399	0.1734	0.083
H(4A)	4a		0.9944	0.4507	0.2278	0.074
H(4B)	4a		0.8285	0.3650	0.2508	0.074
H(5A)	4a		0.4508	0.6039	0.2650	0.065
H(5B)	4a		0.4484	0.4675	0.2456	0.065
H(12)	4a		0.5001	0.3737	0.0619	0.088
H(13)	4a		0.4679	0.1840	0.0240	0.111
H(15)	4a		1.0411	0.0872	0.0603	0.104
H(16)	4a		1.0824	0.2739	0.0971	0.079
H(22)	4a		0.5256	0.5725	0.3508	0.072
H(23)	4a		0.4906	0.4371	0.4095	0.084
H(25)	4a		1.0509	0.2920	0.3858	0.080
H(26)	4a		1.0934	0.4259	0.3273	0.071
C(11A)	4a	0.50	0.786(2)	–0.012(1)	0.0143(3)	0.069(3)
H(11A)	4a	0.50	0.9218	–0.0448	0.0217	0.103
H(11B)	4a	0.50	0.7774	–0.0004	–0.0169	0.103
H(11C)	4a	0.50	0.6805	–0.0699	0.0237	0.103
C(11B)	4a	0.50	0.672(2)	0.009(1)	0.0088(3)	0.073(3)
H(11D)	4a	0.50	0.7771	–0.0555	0.0067	0.110
H(11E)	4a	0.50	0.6410	0.0401	–0.0201	0.110
H(11F)	4a	0.50	0.5477	–0.0260	0.0216	0.110
H(21A)	4a		0.6066	0.2760	0.4564	0.146
H(21B)	4a		0.8491	0.2760	0.4639	0.146
H(21C)	4a		0.7472	0.1762	0.4327	0.146

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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)	4*a*	0.8289(2)	0.4914(1)	0.10851(4)	0.0463(7)	0.0462(6)	0.0641(6)	–0.0077(6)	–0.0108(5)	0.0045(5)
S(2)	4*a*	0.8454(2)	0.6136(1)	0.28748(4)	0.0405(7)	0.0317(5)	0.0651(6)	–0.0001(5)	0.0069(5)	–0.0036(5)
O(22)	4*a*	1.0650(5)	0.6224(3)	0.2783(1)	0.037(2)	0.067(2)	0.081(2)	–0.007(2)	0.003(2)	–0.007(2)
O(12)	4*a*	1.0466(5)	0.5022(4)	0.1174(1)	0.046(2)	0.083(3)	0.105(3)	–0.014(2)	–0.014(2)	–0.019(2)
O(21)	4*a*	0.7316(5)	0.7264(3)	0.2965(1)	0.063(2)	0.029(2)	0.094(2)	0.006(1)	0.015(2)	–0.005(2)
N(2)	4*a*	0.7348(5)	0.5450(3)	0.2457(1)	0.036(2)	0.032(2)	0.058(2)	0.005(1)	–0.003(2)	–0.002(2)
O(11)	4*a*	0.7258(6)	0.5847(3)	0.0826(1)	0.082(3)	0.053(2)	0.076(2)	–0.005(2)	–0.009(2)	0.023(2)
N(1)	4*a*	0.7089(6)	0.4848(3)	0.1553(1)	0.050(3)	0.040(2)	0.059(2)	0.012(2)	–0.014(2)	0.004(2)
N(3)	4*a*	0.5325(9)	0.7284(4)	0.1926(2)	0.101(4)	0.048(3)	0.118(4)	0.014(3)	0.002(3)	0.021(3)
C(1)	4*a*	0.4396(8)	0.6032(5)	0.1972(2)	0.034(3)	0.067(3)	0.080(3)	0.010(2)	0.000(2)	0.013(3)
C(2)	4*a*	0.4849(8)	0.5150(5)	0.1589(2)	0.046(3)	0.067(3)	0.075(3)	–0.006(2)	–0.006(2)	0.010(3)
C(3)	4*a*	0.7750(9)	0.3880(5)	0.1867(2)	0.099(5)	0.044(3)	0.065(3)	0.028(3)	–0.016(3)	–0.003(2)
C(4)	4*a*	0.846(1)	0.4320(4)	0.2296(2)	0.080(4)	0.048(3)	0.057(3)	0.026(3)	–0.004(3)	–0.005(2)
C(5)	4*a*	0.5063(7)	0.5509(5)	0.2419(2)	0.040(3)	0.050(3)	0.072(3)	0.000(2)	0.006(2)	0.009(2)
C(11)	4*a*	0.7970(8)	0.3437(4)	0.0818(1)	0.062(4)	0.054(3)	0.043(2)	–0.006(2)	–0.013(2)	0.006(2)
C(12)	4*a*	0.611(1)	0.3171(5)	0.0611(2)	0.084(5)	0.065(4)	0.070(3)	–0.015(3)	–0.019(3)	0.011(3)
C(13)	4*a*	0.592(1)	0.2025(7)	0.0387(2)	0.132(7)	0.088(5)	0.058(3)	–0.055(5)	–0.019(4)	0.004(3)
C(14)	4*a*	0.754(1)	0.1165(6)	0.0379(2)	0.160(7)	0.058(4)	0.052(3)	–0.031(4)	0.006(4)	–0.004(3)
C(15)	4*a*	0.933(1)	0.1452(5)	0.0601(2)	0.142(7)	0.058(4)	0.061(3)	0.014(4)	0.012(4)	–0.005(3)
C(16)	4*a*	0.9587(9)	0.2570(5)	0.0822(2)	0.076(4)	0.066(3)	0.054(3)	0.013(3)	–0.005(3)	0.002(2)
C(21)	4*a*	0.8163(8)	0.5135(4)	0.3332(1)	0.050(3)	0.038(2)	0.053(2)	–0.001(2)	0.000(2)	–0.009(2)
C(22)	4*a*	0.6324(8)	0.5167(5)	0.3578(2)	0.056(3)	0.059(3)	0.064(3)	0.007(3)	0.011(2)	–0.004(2)
C(23)	4*a*	0.613(1)	0.4349(5)	0.3929(2)	0.077(4)	0.069(3)	0.064(3)	–0.001(3)	0.019(3)	0.000(3)
C(24)	4*a*	0.767(1)	0.3500(5)	0.4041(2)	0.102(5)	0.052(3)	0.053(3)	0.000(3)	0.002(3)	–0.001(2)
C(25)	4*a*	0.945(1)	0.3486(5)	0.3790(2)	0.085(4)	0.047(3)	0.068(3)	0.018(3)	–0.007(3)	0.002(2)
C(26)	4*a*	0.9712(9)	0.4289(5)	0.3438(2)	0.059(4)	0.055(3)	0.062(3)	0.011(2)	0.004(2)	–0.008(2)
C(211)	4*a*	0.740(1)	0.2614(6)	0.4428(2)	0.138(7)	0.080(4)	0.075(4)	0.001(4)	0.010(4)	0.015(3)

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