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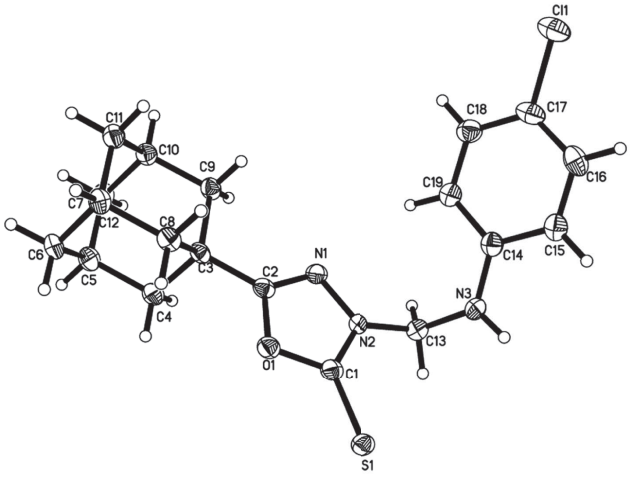
Crystal structure of 5-(adamantan-1-yl)-3-[(4-chloroanilino)methyl]-2,3-dihydro-1,3,4-oxadiazole-2-thione, $C_{19}H_{22}ClN_3OS$



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Abstract

$C_{19}H_{22}ClN_3OS$, orthorhombic, $P2_12_12_1$ (no. 19), $a = 7.0418(2)$ Å, $b = 10.8802(3)$ Å, $c = 23.5506(6)$ Å, $V = 1804.36(8)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0413$, $wR_{ref}(F^2) = 0.1110$, $T = 100$ K.

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The crystal structure is shown in the figure. Tables 1–3 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, prism, size 0.2231×0.5389×0.7274 mm
Wavelength:	CuK_α radiation (1.54184 Å)
μ:	30.49 cm^{-1}
Diffractometer, scan mode:	Xcalibur, Ruby, Gemini, ω scans
$2\theta_{max}$:	140.8°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	15818, 3444
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 3373
$N(param)_{refined}$:	231
Programs:	CrysAlis [17], SHELX [18]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	Site	x	y	z	U_{iso}
H(9A)	4a	0.5080	−0.0521	0.1901	0.031
H(9B)	4a	0.6646	−0.0449	0.1427	0.031
H(15)	4a	−0.3588	−0.1097	0.0216	0.037
H(10)	4a	0.8080	−0.0995	0.2301	0.033
H(8A)	4a	0.4589	0.2715	0.2156	0.033
H(8B)	4a	0.3809	0.1427	0.2350	0.033
H(18)	4a	0.1008	−0.3292	0.1189	0.041
H(4A)	4a	0.7917	0.1621	0.1110	0.036
H(4B)	4a	0.7132	0.2835	0.1386	0.036
H(13A)	4a	0.1514	0.0956	−0.0290	0.036
H(13B)	4a	0.2343	−0.0282	−0.0062	0.036
H(7)	4a	0.6037	0.2154	0.3032	0.035
H(16)	4a	−0.4431	−0.2822	0.0734	0.044
H(19)	4a	0.1838	−0.1522	0.0710	0.038
H(5)	4a	1.0117	0.2359	0.1789	0.043
H(6A)	4a	0.7915	0.3347	0.2400	0.043
H(6B)	4a	0.9234	0.2473	0.2756	0.043
H(12A)	4a	1.0509	0.0515	0.2310	0.042
H(12B)	4a	0.9987	0.0194	0.1678	0.042
H(11A)	4a	0.8000	0.0395	0.3070	0.035
H(11B)	4a	0.5919	−0.0003	0.2915	0.035
H(3)	4a	−0.133(4)	0.032(3)	−0.004(2)	0.05(1)

Table 3: Atomic displacement parameters (Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
S(1)	4*a*	0.08526(8)	0.33243(6)	0.03351(3)	0.0228(3)	0.0306(3)	0.0473(3)	0.0020(2)	−0.0023(2)	0.0118(3)
Cl(1)	4*a*	−0.2398(1)	−0.45144(6)	0.14068(3)	0.0636(5)	0.0355(3)	0.0392(3)	−0.0125(3)	0.0101(3)	0.0039(2)
O(1)	4*a*	0.3371(2)	0.2459(2)	0.10883(7)	0.0238(8)	0.0236(8)	0.0351(9)	0.0009(7)	−0.0003(7)	0.0015(7)
N(2)	4*a*	0.2306(3)	0.1054(2)	0.05303(8)	0.0245(9)	0.0250(9)	0.0279(9)	−0.0030(8)	−0.0031(8)	0.0042(7)
N(1)	4*a*	0.3609(3)	0.0477(2)	0.08851(8)	0.0238(9)	0.0243(9)	0.0292(9)	0.0000(8)	0.0003(8)	0.0044(8)
C(2)	4*a*	0.4201(3)	0.1342(2)	0.12104(9)	0.019(1)	0.023(1)	0.030(1)	−0.0006(9)	0.0042(9)	0.0033(8)
N(3)	4*a*	−0.0360(3)	−0.0064(2)	0.0103(1)	0.026(1)	0.028(1)	0.043(1)	0.0018(9)	−0.0098(9)	0.0059(9)
C(14)	4*a*	−0.0788(4)	−0.1109(2)	0.04094(9)	0.029(1)	0.028(1)	0.028(1)	0.000(1)	−0.000(1)	−0.0050(9)
C(1)	4*a*	0.2174(3)	0.2252(2)	0.0643(1)	0.019(1)	0.029(1)	0.033(1)	−0.0022(9)	0.0034(9)	0.0068(9)
C(9)	4*a*	0.6193(3)	−0.0073(2)	0.1775(1)	0.025(1)	0.021(1)	0.033(1)	0.0008(9)	−0.0020(9)	−0.0027(9)
C(15)	4*a*	−0.2665(4)	−0.1524(2)	0.0418(1)	0.029(1)	0.028(1)	0.035(1)	0.003(1)	−0.001(1)	−0.009(1)
C(10)	4*a*	0.7746(3)	−0.0134(2)	0.2231(1)	0.025(1)	0.024(1)	0.033(1)	0.0010(9)	−0.002(1)	−0.0005(9)
C(3)	4*a*	0.5669(3)	0.1282(2)	0.16640(9)	0.019(1)	0.021(1)	0.031(1)	−0.0024(9)	0.0024(9)	0.0003(8)
C(8)	4*a*	0.4931(3)	0.1863(2)	0.2221(1)	0.024(1)	0.024(1)	0.035(1)	0.0023(9)	0.0016(9)	−0.0016(9)
C(18)	4*a*	0.0083(4)	−0.2835(2)	0.1002(1)	0.042(1)	0.030(1)	0.030(1)	0.000(1)	−0.002(1)	0.000(1)
C(4)	4*a*	0.7451(3)	0.1982(2)	0.1460(1)	0.023(1)	0.033(1)	0.035(1)	−0.008(1)	0.001(1)	0.0067(9)
C(13)	4*a*	0.1510(4)	0.0399(2)	0.0031(1)	0.031(1)	0.031(1)	0.030(1)	−0.002(1)	−0.0042(9)	0.004(1)
C(7)	4*a*	0.6497(4)	0.1787(2)	0.2677(1)	0.031(1)	0.028(1)	0.030(1)	−0.002(1)	−0.0008(9)	−0.005(1)
C(17)	4*a*	−0.1787(4)	−0.3207(2)	0.1017(1)	0.051(2)	0.026(1)	0.029(1)	−0.006(1)	0.009(1)	0.000(1)
C(16)	4*a*	−0.3174(4)	−0.2562(3)	0.0725(1)	0.032(1)	0.036(1)	0.040(1)	−0.007(1)	0.008(1)	−0.011(1)
C(19)	4*a*	0.0581(4)	−0.1785(2)	0.0709(1)	0.029(1)	0.033(1)	0.032(1)	−0.000(1)	−0.002(1)	0.001(1)
C(5)	4*a*	0.8989(4)	0.1911(3)	0.1918(1)	0.024(1)	0.042(2)	0.041(1)	−0.014(1)	−0.002(1)	0.009(1)
C(6)	4*a*	0.8249(4)	0.2494(3)	0.2468(1)	0.036(1)	0.030(1)	0.041(1)	−0.014(1)	−0.011(1)	0.002(1)
C(12)	4*a*	0.9516(3)	0.0563(3)	0.2025(1)	0.019(1)	0.050(2)	0.037(1)	0.001(1)	0.001(1)	−0.001(1)
C(11)	4*a*	0.7023(3)	0.0444(2)	0.2781(1)	0.027(1)	0.030(1)	0.030(1)	−0.003(1)	0.0000(9)	−0.001(1)

Source of material

4-Chloroaniline (1.28 g, 0.01 mol) and 37% formaldehyde solution (1.5 mL) were added to a solution of 5-(1-adamantyl)-1,3,4-oxadiazoline-2-thione (2.36 g, 0.01 mol) in ethanol (15 mL), and the mixture was stirred at room temperature for 2 hours and allowed to stand overnight. The precipitated crude product was filtered, washed with cold ethanol, dried, and crystallized from ethanol to yield 3.46 g (92%) of the title compound. M.p. 450–452 K [9]. Colourless prismatic single crystals were obtained by slow evaporation of chloroform-ethanol solution (1:1) at room temperature. ^{1}H NMR ($CDCl_3$, 500.13 MHz): δ 1.68 (q, 6H, adamantane-CH_2), 1.93 (s, 6H, adamantine-CH_2), 2.07 (s, 3H, adamantane-CH), 5.06 (t, 1H, NH), 5.36 (d, 2H, CH_2), 6.80 (d, 2H, Ar—H, $J = 7.0$ Hz), 7.14 (d, 2H, Ar—H, $J = 7.0$ Hz). ^{13}C NMR ($CDCl_3$, 125.76 MHz): δ 27.30, 34.35, 35.91, 38.94 (adamantane-C), 58.22 (CH_2), 115.30, 124.46, 129.10, 142.75 (Ar—C), 168.20 (C = N), 176.77 (C = S). EI-MS, *m/z* (rel. int.): 377 ($M^+ + 2$, 1), 359 (M^+, 4), 236 (82), 176 (17), 142 (20), 140 (55), 135 (100), 113 (5), 111 (26).

Experimental details

The structure was refined as an inversion twin (BASF 0.43).

Discussion

The adamantyl group was identified as an essential pharmacophore in several bioactive drugs [1, 2]. Several adamantane derivatives are currently used as efficient therapies against influenza [3–5], herpes simplex [6] and HIV [7–9] viruses. In addition, numerous adamantane derivatives were reported to exhibit marked anticancer [10, 11], antibacterial [12–15] and antifungal [16] activities. In continuation to our ongoing interest in the chemical and biological properties of adamantane derivatives, we report herein the crystal structure of the title compound which displayed potent broad spectrum antibacterial activity [9]. The title compound is a functionalized 1,3,4-oxadiazole-2-thione derivative. The benzene ring makes a dihedral angle of 57.43(5)° with the 1,3,4-oxadiazole-2-thione. In the crystal, the N3—H3···S1 hydrogen-bonding interactions link the molecules into chains extending along the c direction. The distance of N and H3 is 2.57(4) Å and the angle is 171.0 (3)°.

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