Crystal structure of 4-chloro-*N*-[2-(5-methyl-4,6-dioxo-3-phenyl-hexahydro-pyrrolo-[3,4-*d*]isoxazol-2-yl)-ethyl]-benzenesulfonamide, $C_{20}H_{20}ClN_3O_5S$

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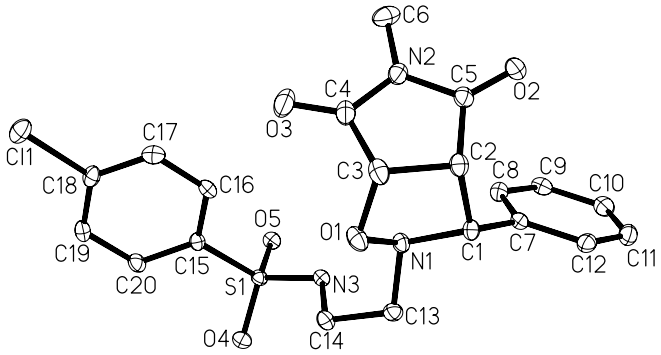

Abstract

$C_{20}H_{20}ClN_3O_5S$, orthorhombic, *Fdd*2 (no. 43), a = 39.856(4) Å, b = 19.058(1) Å, c = 10.5849(8) Å, V = 8039.9 Å^3, Z = 16, $R_{gt}(F)$ = 0.061, $wR_{obs}(F^2)$ = 0.173, T = 160 K.

Source of material

All chemicals used for the preparation of the fused-ring compound were of reagent grade quality. The title compound was prepared by a procedure that reported in the literature [1] which involved first converting aziridine and oxime into a corresponding nitrone and then 1,3-dipolar cycloaddition with *N*-methylmaleimide as dipolarophile. The compound was recrystallized from: petroleum ether/ethyl acetate (b.p. 60-80 °C) afforded the title compound (m.p. 196–198 °C).
Elemental analysis: found – C, 53.05 %; H, 4.5 %; N, 9.6 %; calc. for $C_{20}H_{20}N_3O_5ClS$ – C, 53.35 %; H, 4.5 %; N, 9.35 %.

Discussion

The use of 1,3-dipolar cycloaddition cascades for the synthesis of cyclic rings containing a nitrogen atom, such as pyrrolizidines, indolizidines and quinolizidines is important as they occur in a number of alkaloids [2-4]. Some of the *N*-heterocyclic bearing isoxazolidine rings possess various biological activity [5,6]. We have recently published a range of aza-cyclic *N*-heterocycles including isoxazolidine and pyrrolidine ring compounds [7-9]. The title compound is another example of our newly synthesized isoxazolidine derivatives that contains two fused five-membered rings, i.e. a 1,2-oxazolidine and an *N*-methylpyrrolidine ring. The title cycloadduct, 4-chloro-*N*-[2-(5-methyl-4,6-dioxo-3-phenyl-hexahydro-pyrrolo[3,4-d]isoxazol-2-yl)-ethyl]-benzenesulfonamide, has been synthesized via electrophile induced oxime → nitrone → cycloaddition cascades.

The title compound contains two fused five membered rings; a nearly planar pyrrolidine ring and oxazolidine ring in an envelope conformation with the N1 atom in the flap position. The oxazolidine system, by contrast, is a puckered ring. The relevant torsion angles are ∠N1–C1–C2–C3 = 23.4(5)° and ∠N1–O1–C3–C2 = –35.4(5)°. The pyrrolidine and isoxazolidine rings are not in plane, viz. ∠O1–C3–C4–N2 = –121.0(4)° and ∠C1–C2–C5–N2 = 103.4(4)°. The bond lengths of the C—N and C—C bonds, viz. d(N2—C4) = 1.386(7) Å, d(N2—C5) = 1.379(7) Å, d(C3—C4) = 1.514(8) Å and d(C2—C5) = 1.517(7) Å, in the pyrrolidine ring are all shorter than the average of C—N and C—C single bonds, respectively. These results indicate extensive delocalization of electrons within the pyrrolidine ring. On the contrary, the bond lengths of the isoxazolidine ring, viz. d(C3—O1) = 1.429(6) Å, d(O1—N1) = 1.470(5) Å, d(N1—C1) = 1.483(6) Å, d(C1—C2) = 1.550(7) Å and d(C2—C3) = 1.533(7) Å, are within the average single bonds. These results agree with similar distances obtained in previously reported structure [1,10]. All the other bond lengths and angles fall within the expected range. The absolute structure is confirmed by the Flack x parameter of 0.04(2).

Table 1. Data collection and handling.

Crystal:	colorless, prismatic, size 0.12 × 0.27 × 0.38 mm
Wavelength:	Cu K_α radiation (1.54184 Å)
μ:	29.97 cm^{-1}
Diffractometer, scan mode:	Stoe STADI4, ω-θ
$2\theta_{max}$:	129.0°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	3271, 2969
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2898
$N(param)_{refined}$:	276
Programs:	SHELXS-86 [11], SHELXL-93 [12]

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

Atom	Site	x	y	z	U_{iso}
H(3N)	16*b*	0.248(2)	0.159(3)	0.600(6)	0.04(2)
H(1)	16*b*	0.3085	–0.0249	0.4641	0.030
H(2)	16*b*	0.3569	–0.0276	0.5811	0.031
H(3)	16*b*	0.3297	–0.0330	0.7697	0.037
H(6A)	16*b*	0.3609	0.2172	0.7057	0.061
H(6B)	16*b*	0.3925	0.1771	0.7652	0.061
H(6C)	16*b*	0.3598	0.1912	0.8493	0.061
H(8)	16*b*	0.3116	0.1639	0.4676	0.034

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Table 2. Continued.

Atom	Site	*x*	*y*	*z*	U_{iso}
H(9)	16*b*	0.3240	0.2318	0.2913	0.035
H(10)	16*b*	0.3421	0.1808	0.1038	0.039
H(11)	16*b*	0.3481	0.0585	0.0926	0.040
H(12)	16*b*	0.3359	–0.0111	0.2669	0.033
H(13A)	16*b*	0.2506	–0.0149	0.5091	0.031
H(13B)	16*b*	0.2530	0.0568	0.4305	0.031

Table 2. Continued.

Atom	Site	*x*	*y*	*z*	U_{iso}
H(14A)	16*b*	0.2042	0.0541	0.5618	0.031
H(14B)	16*b*	0.2279	0.0493	0.6838	0.031
H(16)	16*b*	0.2614	0.1627	0.8303	0.030
H(17)	16*b*	0.2705	0.1520	1.0500	0.033
H(19)	16*b*	0.1706	0.1723	1.1125	0.033
H(20)	16*b*	0.1615	0.1815	0.8918	0.031

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}
Cl(1)	16*b*	0.22840(3)	0.15954(7)	0.2606(1)	0.0494(7)	0.0383(7)	0.0251(7)	0.0009(6)	–0.0049(6)	0.0022(6)
S(1)	16*b*	0.20321(2)	0.18989(6)	0.6799(1)	0.0171(5)	0.0253(6)	0.0227(6)	0.0004(4)	–0.0006(4)	0.0011(5)
N(1)	16*b*	0.2868(1)	0.0506(2)	0.5732(4)	0.023(2)	0.026(2)	0.021(2)	0.000(2)	0.003(2)	0.004(2)
N(2)	16*b*	0.3540(1)	0.1135(2)	0.7235(4)	0.026(2)	0.037(2)	0.026(2)	0.009(2)	–0.002(2)	–0.003(2)
N(3)	16*b*	0.2294(1)	0.1446(2)	0.5980(4)	0.020(2)	0.027(2)	0.025(2)	–0.002(2)	0.002(2)	–0.002(2)
O(1)	16*b*	0.28810(8)	0.0044(2)	0.6842(4)	0.026(2)	0.039(2)	0.032(2)	0.001(1)	0.004(2)	0.012(2)
O(2)	16*b*	0.38590(9)	0.1024(2)	0.5451(4)	0.027(2)	0.045(2)	0.033(2)	–0.005(2)	0.004(2)	0.001(2)
O(3)	16*b*	0.3164(1)	0.0968(3)	0.8861(4)	0.039(2)	0.087(3)	0.026(2)	0.019(2)	–0.002(2)	–0.010(2)
O(4)	16*b*	0.17015(8)	0.1645(2)	0.6522(4)	0.021(2)	0.038(2)	0.033(2)	–0.002(1)	–0.001(1)	–0.003(2)
O(5)	16*b*	0.21039(8)	0.2629(2)	0.6560(3)	0.025(2)	0.024(2)	0.027(2)	0.001(1)	–0.001(1)	0.004(2)
C(1)	16*b*	0.3148(1)	0.0228(2)	0.4956(5)	0.023(2)	0.026(2)	0.025(3)	0.003(2)	0.004(2)	–0.005(2)
C(2)	16*b*	0.3428(1)	0.0148(2)	0.5964(5)	0.025(2)	0.025(2)	0.027(3)	0.007(2)	0.003(2)	0.001(2)
C(3)	16*b*	0.3227(1)	0.0089(3)	0.7194(5)	0.027(2)	0.039(3)	0.027(3)	0.007(2)	0.003(2)	0.011(2)
C(4)	16*b*	0.3298(1)	0.0764(3)	0.7897(5)	0.024(2)	0.053(3)	0.022(3)	0.013(2)	–0.002(2)	0.004(3)
C(5)	16*b*	0.3639(1)	0.0804(3)	0.6138(5)	0.022(2)	0.034(3)	0.027(3)	0.006(2)	–0.001(2)	0.000(2)
C(6)	16*b*	0.3679(1)	0.1801(3)	0.7642(6)	0.037(3)	0.038(3)	0.048(4)	0.010(2)	–0.016(3)	–0.015(3)
C(7)	16*b*	0.3224(1)	0.0686(2)	0.3854(5)	0.017(2)	0.027(2)	0.024(2)	–0.001(2)	0.000(2)	–0.001(2)
C(8)	16*b*	0.3190(1)	0.1421(3)	0.3917(5)	0.029(2)	0.030(2)	0.027(3)	–0.001(2)	0.003(2)	–0.004(2)
C(9)	16*b*	0.3264(1)	0.1823(3)	0.2866(5)	0.023(2)	0.030(2)	0.034(3)	–0.004(2)	–0.002(2)	0.005(2)
C(10)	16*b*	0.3371(1)	0.1524(3)	0.1751(6)	0.028(2)	0.046(3)	0.025(3)	–0.002(2)	–0.003(2)	0.008(3)
C(11)	16*b*	0.3406(1)	0.0798(3)	0.1688(6)	0.028(2)	0.047(3)	0.025(3)	0.003(2)	0.000(2)	–0.005(3)
C(12)	16*b*	0.3334(1)	0.0383(3)	0.2725(5)	0.024(2)	0.031(3)	0.027(3)	0.003(2)	–0.003(2)	–0.004(2)
C(13)	16*b*	0.2539(1)	0.0364(2)	0.5165(5)	0.025(2)	0.022(2)	0.031(3)	–0.002(2)	0.001(2)	–0.004(2)
C(14)	16*b*	0.2263(1)	0.0676(3)	0.5965(5)	0.024(2)	0.025(2)	0.028(3)	–0.002(2)	0.005(2)	0.003(2)
C(15)	16*b*	0.2107(1)	0.1745(2)	0.8424(5)	0.023(2)	0.022(2)	0.020(3)	–0.001(2)	0.002(2)	0.003(2)
C(16)	16*b*	0.2431(1)	0.1651(3)	0.8874(5)	0.018(2)	0.028(2)	0.030(3)	–0.001(2)	0.004(2)	0.001(2)
C(17)	16*b*	0.2485(1)	0.1591(2)	1.0178(5)	0.027(2)	0.022(3)	0.034(3)	0.001(2)	–0.008(2)	0.005(2)
C(18)	16*b*	0.2215(1)	0.1637(2)	1.0983(5)	0.035(3)	0.022(3)	0.020(3)	0.001(2)	0.001(2)	0.004(2)
C(19)	16*b*	0.1889(1)	0.1710(3)	1.0551(5)	0.030(3)	0.027(2)	0.024(3)	0.000(2)	0.008(2)	0.003(2)
C(20)	16*b*	0.1836(1)	0.1765(3)	0.9241(5)	0.019(2)	0.029(2)	0.030(3)	0.003(2)	0.004(2)	–0.002(2)

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