Crystal structure of 1-[(4-chloro-phenyl)-phenyl-methyl]piperazine, $C_{17}H_{19}ClN_2$

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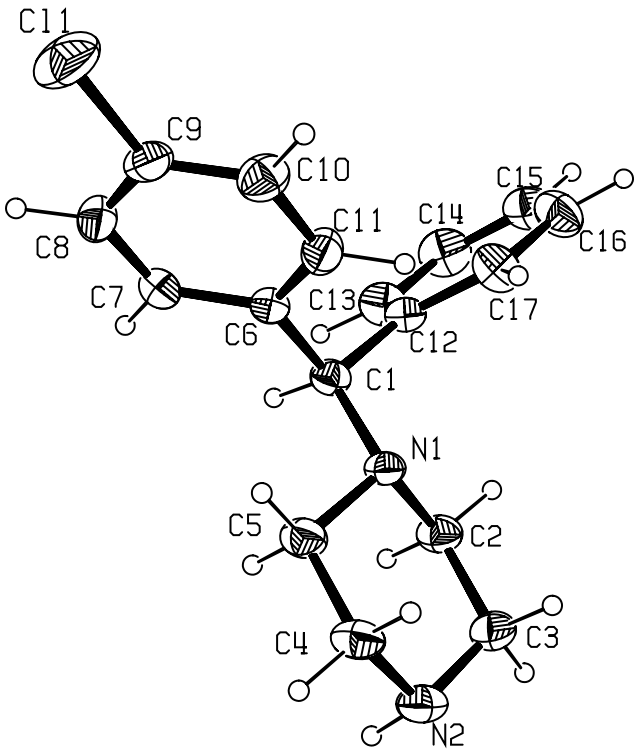

Abstract

$C_{17}H_{19}ClN_2$, orthorhombic, $P2_12_12_1$ (No. 19), a = 18.0920(1) Å, b = 5.8896(1) Å, c = 14.6011(2) Å, V = 1555.8 Å^3, Z = 4, $R_{gt}(F)$ = 0.048, $wR_{ref}(F^2)$ = 0.114, T = 298 K.

Source of material

The mixture of *p*-methylbenzene sulfonyl chloride and dithiolamine was refluxed with stirring for 1 hour, then adding SO_2Cl_2, refluxing for 6 hours. The reaction mixture was treated with 1-[(4-chlorophenyl)(phenyl)]methylamine in diisopropylethylamine and refluxed for 6 hours. The mixture was dissolving in HBr-AcOH 30:70 (v/v) to obtained the title compound crystallized from *n*-BuOH as colorless needles.

Experimental details

The H atoms were localized from differential Fourier maps and included in the final cycles of refinement in riding model (d(C—H) = 0.95 Å – 1.04 Å).

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Discussion

A great deal of attention has been devoted to the medicine with chiral center, since they usually have special biological activity. Consequently, much interest has been focused on obtaining compounds with such a feature.

The title compound is a chiral intermediate used for the synthesis of cetirizine hydrochloride. It consists of three different rings,two phenyl rings and a piperazine ring. The dihedral angle between two phenyl rings is 116.78(6)°. Futhermore, piperazine ring is a chair-like conformation, this can be seen from the distance of N1 and N2 to the plane of C2–C3–C4–C5 (–0.699 Å and 0.597 Å, respectively). In addition, through Van der Waals interation, molecules pack into infinite V-type chains, which are oriented along *a*-axis and *c*-axis.

Table 1. Data collection and handling.

Crystal:	colorless chunk, size 0.10 × 0.11 × 0.29 mm
Wavelength:	Mo K_α radiation (0.7107 Å)
μ:	2.37 cm^{-1}
Diffractometer, scan mode:	Rigaku RAXIS-RAPID, ω
$2\theta_{max}$:	54.96°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	14633, 3565
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2251
$N(param)_{refined}$:	200
Programs:	SIR92 [3], ABSCOR [4], CrystalStructure [5]

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

Atom	Site	x	y	z	U_{iso}
H(1)	4*a*	0.7753(2)	0.3404(5)	0.3422(2)	0.049(1)
H(2)	4*a*	0.8539(2)	0.5663(6)	0.4475(2)	0.058(1)
H(3)	4*a*	0.8799(2)	0.7233(6)	0.3755(2)	0.058(1)
H(4)	4*a*	0.8330(2)	1.0514(6)	0.4414(2)	0.064(1)
H(5)	4*a*	0.8810(2)	0.8824(6)	0.5214(2)	0.064(1)
H(6)	4*a*	0.7820(1)	0.7823(5)	0.5877(2)	0.066(1)
H(7)	4*a*	0.6889(2)	1.0007(7)	0.4671(2)	0.068(1)
H(8)	4*a*	0.6659(2)	0.8567(7)	0.5411(2)	0.069(1)
H(9)	4*a*	0.6615(2)	0.6510(7)	0.3956(2)	0.061(1)
H(10)	4*a*	0.7198(2)	0.5207(7)	0.4713(2)	0.061(1)
H(11)	4*a*	0.6993(2)	0.7822(6)	0.1967(2)	0.064(1)
H(12)	4*a*	0.5877(2)	0.7372(6)	0.1095(2)	0.070(1)
H(13)	4*a*	0.5570(2)	0.0941(7)	0.2168(3)	0.074(2)
H(14)	4*a*	0.6688(2)	0.1498(6)	0.3103(2)	0.065(1)
H(15)	4*a*	0.8167(2)	0.8121(7)	0.1809(2)	0.072(1)
H(16)	4*a*	0.9225(2)	0.7803(8)	0.0733(3)	0.083(2)
H(17)	4*a*	1.0076(2)	0.4734(8)	0.0870(3)	0.081(2)
H(18)	4*a*	0.9831(2)	0.1735(7)	0.1771(3)	0.082(2)
H(19)	4*a*	0.8708(2)	0.1714(6)	0.2747(2)	0.070(1)

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)	4*a*	0.48254(5)	0.3768(2)	0.09389(8)	0.0526(5)	0.135(1)	0.0872(7)	–0.0114(6)	–0.0171(5)	–0.0341(8)
N(1)	4*a*	0.7681(1)	0.6876(4)	0.3646(1)	0.039(1)	0.048(1)	0.032(1)	0.000(1)	–0.004(1)	–0.005(1)
N(2)	4*a*	0.7742(1)	0.9060(5)	0.5460(2)	0.069(2)	0.061(2)	0.035(1)	0.006(2)	–0.007(1)	–0.009(1)
C(1)	4*a*	0.7693(2)	0.4859(5)	0.3029(2)	0.048(2)	0.040(2)	0.035(1)	0.004(1)	–0.004(1)	0.004(1)
C(2)	4*a*	0.8385(2)	0.7088(6)	0.4160(2)	0.039(2)	0.062(2)	0.043(2)	0.004(1)	–0.009(1)	–0.007(2)
C(3)	4*a*	0.8356(2)	0.9112(6)	0.4809(2)	0.061(2)	0.060(2)	0.040(2)	0.002(2)	–0.012(2)	–0.004(2)
C(4)	4*a*	0.7054(2)	0.8644(7)	0.4969(2)	0.054(2)	0.074(2)	0.043(2)	0.012(2)	0.003(1)	–0.019(2)
C(5)	4*a*	0.7076(2)	0.6619(7)	0.4320(2)	0.044(2)	0.070(2)	0.040(2)	0.003(2)	0.003(1)	–0.013(2)
C(6)	4*a*	0.6958(2)	0.4633(5)	0.2521(2)	0.047(2)	0.043(2)	0.035(1)	–0.006(1)	0.005(1)	–0.000(1)
C(7)	4*a*	0.6693(2)	0.6352(6)	0.1956(2)	0.053(2)	0.049(2)	0.058(2)	–0.010(2)	–0.012(2)	–0.002(2)
C(8)	4*a*	0.6035(2)	0.6092(6)	0.1482(2)	0.059(2)	0.059(2)	0.057(2)	0.004(2)	–0.012(2)	–0.002(2)
C(9)	4*a*	0.5641(2)	0.4125(7)	0.1572(2)	0.040(2)	0.080(3)	0.044(2)	–0.008(2)	0.002(1)	–0.014(2)
C(10)	4*a*	0.5886(2)	0.2395(7)	0.2133(3)	0.061(2)	0.070(2)	0.055(2)	–0.027(2)	0.007(2)	–0.012(2)
C(11)	4*a*	0.6556(2)	0.2657(6)	0.2599(2)	0.067(2)	0.055(2)	0.041(2)	–0.012(2)	0.006(2)	0.003(2)
C(12)	4*a*	0.8356(2)	0.4892(5)	0.2386(2)	0.044(2)	0.047(2)	0.036(2)	0.004(1)	–0.005(1)	–0.003(2)
C(13)	4*a*	0.8500(2)	0.6682(7)	0.1800(2)	0.052(2)	0.064(2)	0.065(2)	0.011(2)	0.011(2)	0.008(2)
C(14)	4*a*	0.9107(2)	0.6668(8)	0.1217(3)	0.057(2)	0.091(3)	0.060(2)	–0.007(2)	0.014(2)	0.013(2)
C(15)	4*a*	0.9579(2)	0.4833(8)	0.1218(3)	0.044(2)	0.101(3)	0.058(2)	0.011(2)	0.010(2)	–0.015(2)
C(16)	4*a*	0.9457(2)	0.3046(7)	0.1805(3)	0.056(2)	0.077(3)	0.072(2)	0.024(2)	0.007(2)	–0.014(2)
C(17)	4*a*	0.8845(2)	0.3067(6)	0.2381(2)	0.060(2)	0.056(2)	0.059(2)	0.020(2)	–0.001(2)	–0.000(2)

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