Crystal structure of *N*-(1-(1-(*p*-chlorophenyl)cyclobutyl)-3-methylbutyl)-*N*,*N*-dimethylamine hydrochloride monohydrate, $(C_{17}H_{27}NCl)Cl \cdot H_2O$

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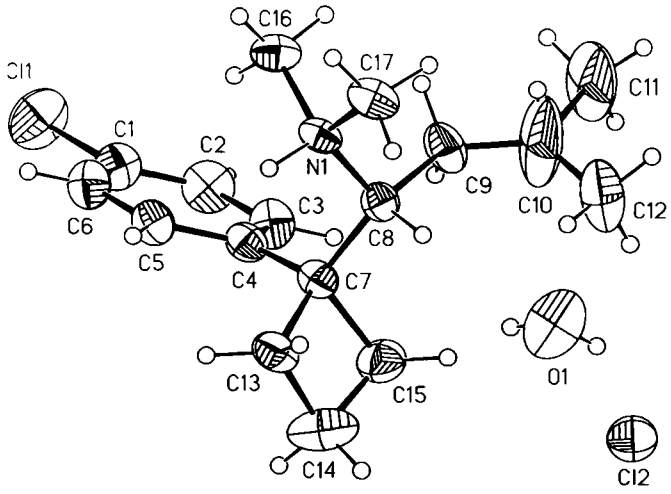

Abstract

$C_{17}H_{29}Cl_2NO$, orthorhombic, *Pbcn* (no. 60), $a = 13.442(3)$ Å, $b = 9.374(2)$ Å, $c = 30.110(7)$ Å, $V = 3794.0$ Å^3, $Z = 8$, $R_{gt}(F) = 0.058$, $wR_{ref}(F^2) = 0.147$, $T = 294$ K.

Source of material

N-(1-(1-(*p*-chlorophenyl)cyclobutyl)-3-methylbutyl)-*N*,*N*-dimethylamine hydrochloride hydrate (0.334 g, 1.000 mmol) was dissolved in 20.0 ml solution of H_2O and methanol (1:1 *v*/*v*). The solution was kept at room temperature for two weeks, and colorless cubic crystals suitable single-crystal X-ray analysis were afforded.

Experimental details

The hydrogen atoms H1A associated with N1 as well as H1WA and H1WB attached to O1 are located from Fourier difference maps and refined. The U_{iso} values of H1WA and H1WB were restrained to 1.5 U_{eq}(O1). All other H atoms were positioned geometrically and treated as riding with $U_{iso}(H) = 1.2U_{eq}(C)$, except for the methyl H atoms with $U_{iso}(H) = 1.5U_{eq}(C)$, and the C–H distances were fixed during refinement.

Discussion

The asymmetric unit consists of one protonated *N*-(1-(1-(*p*-chlorophenyl)cyclobutyl)-3-methylbutyl}-*N*,*N*-dimethylamine, one chlorine anion and one hydrate water molecule. C7, C11 and the phenyl atoms are almost in one plane. C7, C13, C14 and C15 atoms form a four-membered circle which is also coplanar. And these two planes have a dihedral angle of 61.35°. The N1 atom is protonated with an N1—H1A distance of 0.937(2) Å. The NH groups donates the hydrogen atoms to Cl2 anions of adjacent molecules to form intermolecular N–H···Cl hydrogen bonds (d(N···Cl) = 3.09 Å), ∠N–H···Cl = 161.9°). The Cl2 atoms also accept hydrogen atoms from water molecules to generate intermolecular hydrogen bonds (d(O···Cl) = 3.20 Å, ∠O–H···Cl = 172.5°), and accept hydrogen atoms from water groups of neighboring molecules to result intermolecular hydrogen bonds (d(O···Cl) = 3.21 Å, ∠O–H···Cl = 165.3°), which are responsible for supermolecular assembly of 1D chains along [010] direction. Two-dimensional layers are formed by π-π stacking interaction of the adjacent phenyl groups belonging to different chains with the distances of 3.766(2) Å and CH···π interactions between C11–H11B and phenyl groups with the distances of 3.196(3) Å. The C10 atom is slightly disordered and was not split into two positions, since the bond lengths and angles are within normal ranges [1]. The Cl1—C1 bond length (1.735(3) Å) and the other bond distances and angles of the title compound are comparable with corresponding values of a related sibutramine compound, $(C_{17}H_{27}ClN)(C_{18}H_{13}O_8)$ [1].

Table 1. Data collection and handling.

Crystal:	colorless, cubic, size 0.19 × 0.22 × 0.34 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	3.42 cm^{-1}
Diffractometer, scan mode:	Siemens SMART CCD, ω/φ
$2\theta_{max}$:	53.02°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	18855, 3889
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1852
$N(param)_{refined}$:	201
Programs:	SHELXS-97 [2], SHELXL-97 [3]

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

Atom	Site	x	y	z	U_{iso}
H(8A)	8*d*	0.5615	−0.1252	0.3995	0.061
H(17A)	8*d*	0.6006	−0.3264	0.4921	0.098
H(17B)	8*d*	0.5214	−0.2081	0.4821	0.098
H(17C)	8*d*	0.6326	−0.1852	0.4682	0.098
H(5A)	8*d*	0.6272	−0.5265	0.3461	0.076
H(13A)	8*d*	0.7130	−0.2046	0.3642	0.080
H(13B)	8*d*	0.7091	−0.3373	0.3308	0.080
H(6A)	8*d*	0.5541	−0.7361	0.3236	0.089
H(3A)	8*d*	0.3904	−0.3097	0.3038	0.083
H(16A)	8*d*	0.5199	−0.5084	0.4571	0.103
H(16B)	8*d*	0.4849	−0.4996	0.4075	0.103
H(16C)	8*d*	0.4279	−0.4143	0.4443	0.103
H(2A)	8*d*	0.3185	−0.5182	0.2801	0.095
H(9A)	8*d*	0.3878	−0.1916	0.3665	0.088
H(9B)	8*d*	0.3864	−0.2541	0.4143	0.088

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Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(15A)	8d	0.5092	−0.1461	0.2880	0.094
H(15B)	8d	0.5394	−0.0385	0.3270	0.094
H(14A)	8d	0.6996	−0.0595	0.3032	0.136
H(14B)	8d	0.6717	−0.1914	0.2722	0.136
H(10A)	8d	0.3872	−0.0818	0.4430	0.257
H(12A)	8d	0.4983	0.0609	0.4196	0.184
H(12B)	8d	0.4035	0.1221	0.4432	0.184

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(12C)	8d	0.4147	0.1350	0.3915	0.184
H(11A)	8d	0.2386	−0.1406	0.4151	0.265
H(11B)	8d	0.2417	0.0001	0.3870	0.265
H(11C)	8d	0.2458	0.0077	0.4390	0.265
H(1A)	8d	0.622(2)	−0.361(3)	0.4200(9)	0.060(9)
H(1WB)	8d	0.815(3)	−0.142(4)	0.471(1)	0.090
H(1WA)	8d	0.839(3)	−0.269(4)	0.468(1)	0.090

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)	8d	0.37271(9)	−0.8038(1)	0.28272(4)	0.123(1)	0.0941(8)	0.1244(9)	−0.0338(7)	−0.0260(7)	−0.0117(7)
Cl(2)	8d	0.73413(6)	0.0332(1)	0.42645(3)	0.0595(5)	0.0784(6)	0.1022(7)	−0.0152(4)	0.0038(5)	−0.0023(5)
N(1)	8d	0.5595(2)	−0.3216(3)	0.42739(7)	0.037(1)	0.071(2)	0.048(1)	−0.001(1)	0.006(1)	0.000(1)
C(4)	8d	0.5172(2)	−0.3936(3)	0.32847(9)	0.054(2)	0.067(2)	0.044(2)	0.003(2)	−0.000(1)	−0.003(2)
C(7)	8d	0.5627(2)	−0.2572(3)	0.34572(9)	0.051(2)	0.063(2)	0.047(2)	0.003(2)	0.003(1)	−0.001(2)
C(8)	8d	0.5267(2)	−0.2141(3)	0.39237(9)	0.044(2)	0.055(2)	0.054(2)	0.003(1)	−0.002(1)	−0.002(2)
C(17)	8d	0.5804(2)	−0.2543(4)	0.47139(9)	0.053(2)	0.097(3)	0.046(2)	−0.007(2)	−0.003(1)	−0.004(2)
C(5)	8d	0.5643(2)	−0.5238(4)	0.3333(1)	0.055(2)	0.071(2)	0.063(2)	0.005(2)	−0.005(2)	−0.008(2)
C(13)	8d	0.6784(2)	−0.2476(4)	0.3393(1)	0.057(2)	0.078(2)	0.065(2)	−0.009(2)	0.013(2)	−0.011(2)
C(6)	8d	0.5210(3)	−0.6499(4)	0.3197(1)	0.079(3)	0.069(2)	0.075(2)	0.006(2)	−0.004(2)	−0.007(2)
C(3)	8d	0.4240(2)	−0.3954(4)	0.3080(1)	0.070(2)	0.077(2)	0.061(2)	0.009(2)	−0.018(2)	−0.005(2)
C(16)	8d	0.4920(2)	−0.4473(3)	0.4347(1)	0.061(2)	0.079(2)	0.066(2)	−0.017(2)	0.008(2)	0.007(2)
C(2)	8d	0.3804(3)	−0.5201(4)	0.2939(1)	0.070(2)	0.097(3)	0.070(2)	−0.005(2)	−0.024(2)	−0.008(2)
C(1)	8d	0.4286(3)	−0.6468(4)	0.3002(1)	0.080(3)	0.074(3)	0.064(2)	−0.009(2)	−0.009(2)	−0.009(2)
C(9)	8d	0.4155(2)	−0.1799(4)	0.3960(1)	0.045(2)	0.085(3)	0.089(2)	0.010(2)	0.002(2)	−0.020(2)
C(15)	8d	0.5542(3)	−0.1288(4)	0.3126(1)	0.100(3)	0.076(3)	0.059(2)	−0.001(2)	0.003(2)	0.010(2)
C(14)	8d	0.6616(3)	−0.1471(5)	0.3009(1)	0.102(3)	0.143(4)	0.096(3)	−0.033(3)	0.002(3)	0.042(3)
C(10)	8d	0.3804(3)	−0.0547(5)	0.4117(3)	0.057(3)	0.083(4)	0.50(1)	0.018(3)	0.037(5)	−0.041(6)
C(12)	8d	0.4279(3)	0.0759(5)	0.4169(2)	0.103(3)	0.100(4)	0.165(4)	0.025(3)	−0.011(3)	−0.051(3)
C(11)	8d	0.2658(3)	−0.0460(5)	0.4133(2)	0.061(3)	0.123(4)	0.346(9)	0.040(3)	0.023(4)	−0.008(5)
O(1)	8d	0.8458(3)	−0.2037(4)	0.4846(1)	0.116(3)	0.107(3)	0.133(3)	−0.015(2)	−0.067(2)	0.002(2)

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