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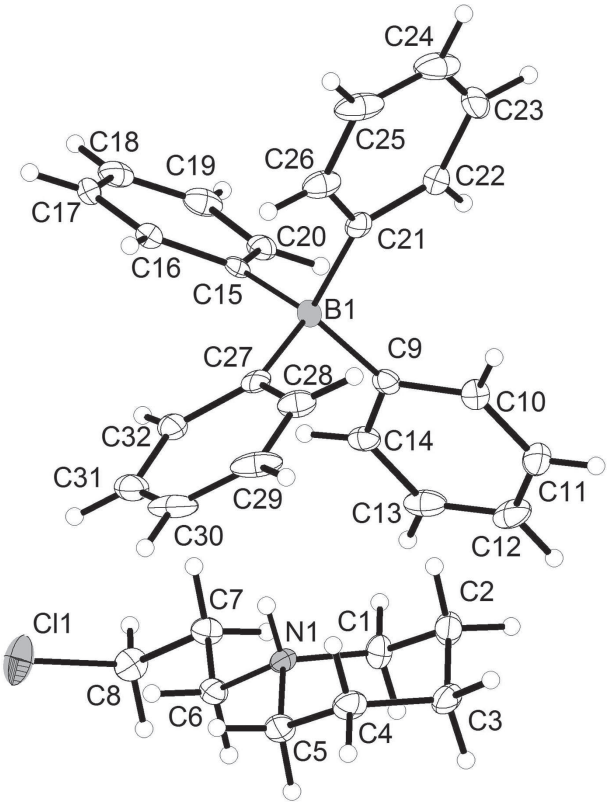
Crystal structure of 1-(3-chloropropyl)piperidin-1-ium tetraphenylborate, $C_{32}H_{37}BClN$



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Abstract

$C_{32}H_{37}BClN$, monoclinic, $P2_1/n$ (no. 14), $a = 15.7303(6)$ Å, $b = 9.1129(3)$ Å, $c = 19.9295(8)$ Å, $\beta = 109.143(2)°$, $V = 2698.9(2)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0483$, $wR_{ref}(F^2) = 0.1265$, $T = 100$ K.

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The asymmetric unit of the title compound is shown in the figure. Tables 1 and 2 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 plates
	Size 0.51 × 0.37 × 0.11 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	1.6 cm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$2\theta_{max}$, completeness:	57.2°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	17136, 4726, 0.065
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 3536
$N(param)_{refined}$:	320
Programs:	Bruker programs [10], SHELXT [11]

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

Atom	x	y	z	U_{iso}*/U_{eq}
B1	0.50294(15)	0.2905(2)	0.73579(12)	0.0148(5)
Cl1	0.14444(4)	−0.10164(6)	0.54614(3)	0.03580(19)
N1	0.24645(11)	0.33519(17)	0.49813(9)	0.0149(4)
C1	0.24139(14)	0.4783(2)	0.53405(11)	0.0213(5)
H1A	0.2583	0.4622	0.5859	0.026*
H1B	0.1787	0.5151	0.5168	0.026*
C2	0.30343(14)	0.5925(2)	0.51959(11)	0.0222(5)
H2A	0.2960	0.6865	0.5419	0.027*
H2B	0.3666	0.5604	0.5414	0.027*
C3	0.28397(14)	0.6161(2)	0.44068(12)	0.0234(5)
H3A	0.3273	0.6877	0.4331	0.028*
H3B	0.2226	0.6565	0.4192	0.028*
C4	0.29174(14)	0.4711(2)	0.40540(11)	0.0224(5)
H4A	0.2758	0.4861	0.3535	0.027*
H4B	0.3547	0.4361	0.4237	0.027*
C5	0.23008(14)	0.3561(2)	0.41963(10)	0.0195(5)
H5A	0.1667	0.3858	0.3961	0.023*
H5B	0.2396	0.2615	0.3987	0.023*
C6	0.18026(13)	0.2232(2)	0.50600(11)	0.0191(5)
H6A	0.1889	0.1313	0.4825	0.023*
H6B	0.1187	0.2591	0.4806	0.023*
C7	0.18703(13)	0.1878(2)	0.58189(11)	0.0193(5)

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
H7A	0.1739	0.2771	0.6050	0.023*
H7B	0.2491	0.1560	0.6086	0.023*
C8	0.12136(14)	0.0673(2)	0.58428(11)	0.0229(5)
H8A	0.0593	0.0997	0.5579	0.027*
H8B	0.1253	0.0496	0.6342	0.027*
C9	0.42754(13)	0.4200(2)	0.72923(10)	0.0162(4)
C10	0.44210(14)	0.5670(2)	0.71484(11)	0.0231(5)
H10A	0.4998	0.5951	0.7139	0.028*
C11	0.37488(15)	0.6727(2)	0.70197(12)	0.0288(5)
H11A	0.3872	0.7710	0.6921	0.035*
C12	0.29040(15)	0.6358(2)	0.70343(12)	0.0294(6)
H12A	0.2445	0.7082	0.6946	0.035*
C13	0.27315(14)	0.4922(2)	0.71786(11)	0.0258(5)
H13A	0.2155	0.4656	0.7196	0.031*
C14	0.34082(13)	0.3873(2)	0.72978(10)	0.0185(5)
H14A	0.3275	0.2889	0.7387	0.022*
C15	0.49854(12)	0.1572(2)	0.79009(10)	0.0141(4)
C16	0.53141(13)	0.0161(2)	0.78440(11)	0.0172(4)
H16A	0.5531	−0.0035	0.7461	0.021*
C17	0.53366(14)	−0.0960(2)	0.83212(11)	0.0235(5)
H17A	0.5553	−0.1904	0.8255	0.028*
C18	0.50426(14)	−0.0704(2)	0.88951(11)	0.0246(5)
H18A	0.5056	−0.1465	0.9224	0.029*
C19	0.47305(13)	0.0677(2)	0.89796(11)	0.0229(5)
H19A	0.4533	0.0872	0.9373	0.027*
C20	0.47038(13)	0.1783(2)	0.84923(10)	0.0176(4)
H20A	0.4485	0.2723	0.8563	0.021*
C21	0.60599(13)	0.3556(2)	0.76787(10)	0.0170(4)
C22	0.63073(15)	0.4626(2)	0.82132(12)	0.0260(5)
H22A	0.5857	0.5025	0.8382	0.031*
C23	0.71879(17)	0.5124(2)	0.85059(13)	0.0391(7)
H23A	0.7326	0.5861	0.8862	0.047*
C24	0.78643(16)	0.4551(3)	0.82802(14)	0.0397(7)
H24A	0.8465	0.4899	0.8475	0.048*
C25	0.76535(15)	0.3473(3)	0.77705(13)	0.0331(6)
H25A	0.8113	0.3053	0.7619	0.040*
C26	0.67727(13)	0.2997(2)	0.74776(11)	0.0228(5)
H26A	0.6644	0.2255	0.7123	0.027*
C27	0.47984(12)	0.2322(2)	0.65359(10)	0.0159(4)
C28	0.50681(13)	0.3129(2)	0.60424(11)	0.0232(5)
H28A	0.5416	0.3992	0.6197	0.028*
C29	0.48459(14)	0.2714(3)	0.53339(12)	0.0303(6)
H29A	0.5048	0.3287	0.5018	0.036*
C30	0.43343(15)	0.1476(3)	0.50871(12)	0.0339(6)
H30A	0.4187	0.1187	0.4604	0.041*
C31	0.40400(15)	0.0668(2)	0.55517(12)	0.0289(5)
H31A	0.3682	−0.0182	0.5388	0.035*
C32	0.42650(13)	0.1089(2)	0.62617(11)	0.0200(5)
H32A	0.4049	0.0519	0.6571	0.024*
H1N1	0.3006(16)	0.300(2)	0.5185(12)	0.024(6)*

Source of material

To a solution of N-(3-chloropropyl) piperidine HCl (0.198 g, 1 mmol) in water (20 mL) was added to solution of sodium tetraphenylborate (0.342 g, 1 mmol) in water (20 mL). A white precipitate was formed and filtered off, washed with cold water until the solution is free of chloride. The precipitate was dried under vacuum. Recrystallization from acetonitrile of the title salt gave 64% yield.

Experimental details

Carbon-bound H atoms were placed in calculated positions and were included in the refinement in the riding model approximation, with U(H) set to 1.2 U_{eq}(C). The N-bound H atom was located on a difference Fourier map.

Discussion

N-(3-chloropropyl) piperidine HCl is an important organic reagent used in many purpose for example as new selective ligands for sigma receptors [1], as dual topoisomerase 1-tyrosyl-DNA phosphodiesterase 1 inhibitors [2], as CCR5 antagonist-based HIV-1 entry inhibitors [3], as potent CCR5 inhibitors [4]. Sodium tetraphenylborate is a popular precipitating reagent [5]. It is also used as association for construct of sensing material used in chemical sensors [6–9].

The title compound crystallizes with one cation *N*-(3-chloropropyl) piperidinum ion and anion tetraphenylborate in the asymmetric unit (*cf*. the figure). The piperidinyl moiety adopts a chair conformation. Typically, the tetraphenylborate anion is in a tetrahedral geometry around the B atom. The molecules in the crystal structure are stacked without any hydrogen bond interactions.

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