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Redetermination of *tert*-butylhydrazinium chloride

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Abstract

The structure of the title compound, $[(CH_3)_3CNH_2NH_2]Cl$, first determined by Hökelek & Ya~gbasen [Acta Cryst. (1988), C44, 723–725] has been redetermined. In contrast to the previous investigation, no HCl moiety was found. The present structure consists of $[(CH_3)_3C(NH_2)(NH_2)]^+$ cations and Cl^- anions connected by hydrogen bonds.

Comment

The crystal structure of *tert*-butylhydrazine hydrochloride was originally determined by Hökelek & Ya~gbasen (1988). In contrast to the data reported we have found that the title compound exists in the solid state as the hydrazonium salt. The proton is attached to the less hydrogenated N atom (N1) of *tert*-butylhydrazine. This results in a positively charged $[(CH_3)_3C(NH_2)(NH_2)]^+$ cation with Cl^- as counter-ion.

Each Cl^- anion and each H atom connected to N is involved in hydrogen bonding. N—H bond lengths for N1 are 0.864 (16)Å for N1—H1A and 0.907 (16) Å for N1—H1B. The N—H distances for the terminal N (N2) are slightly longer with 0.920 (19)Å for N2—H2A and 0.941 (18)Å for N2—H2B. H···Cl distances are 2.279 (16)Å for N1—H1A···Cl and 2.261 (16)Å for N1—H1B···Cl^{i}, whereas the two N2—H···Cl hydrogen bonds have distances of 2.590 (18)Å for N2—H2A···Cl^{ii} and 2.604 (18)Å for N2—H2B···Cl^{iii}. N1—H···Cl bond angles are 177.3 (14)° for N1—H1A···Cl and 177.4 (14)° for N1—H1B···Cl^{i} and closer to 180° then the corresponding N2—H···Cl angles with 162.8 (15)° for N2—H2A···Cl^{ii} and 166.3 (15)° for N2—H2B···Cl^{iii} [symmetry codes: (i) $x - 1/2, y, 3/2 - z$; (ii) $3/2 - x, y - 1/2, z$; (iii) $1 - x, y - 1/2, 3/2 - z$]. The N2—H···Cl interaction is therefore somewhat weaker then the N1—H···Cl interaction. However, the cations and anions are positionated in such a way, that they build up a two-dimensional hydrogen-bonded network in the a and b direction.

The remaining bond lengths and angles are comparable with the values found by Hökelek & Ya~gbasen (1988).

Experimental

The title compound is a commercial product. Colorless crystals were obtained by recrystallization from water.

Refinement

The structure was solved by direct methods. All non-H atoms were refined anisotropically. All hydrogen atoms connected to nitrogen atoms were located from difference Fourier maps and their displacement parameters were refined isotropically.

The remaining hydrogen atoms were fixed at ideal positions with C—H bond lengths of 0.96 Å and assigned an isotropic displacement parameter of 0.08 $Å^2$. The idealized CH_3 groups were allowed to rotate about their C—C bond.

Computing details

Data collection: *SMART* (Siemens, 1995); cell refinement: *SAINT* (Siemens, 1995); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997a); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997b); software used to prepare material for publication: *SHELXL97*.

t-Butylhydrazine hydrochloride

Crystal data

$C_4H_{13}N_2^{1+}\cdot Cl^{1-}$	V = 1433.00 (3) $Å^3$
M_r = 124.61	Z = 8
Orthorhombic, *Pbca*	Mo $K\alpha$
a = 9.8615 (1) Å	μ = 0.43 mm^{-1}
b = 10.6446 (2) Å	T = 173 (2) K
c = 13.6513 (1) Å	0.62 × 0.58 × 0.35 mm

Data collection

Siemens SMART CCD diffractometer	1627 independent reflections
Absorption correction: empirical (using intensity measurements) (SADABS; Sheldrick, 1996)	1408 reflections with $I > 2\sigma(I)$
T_{min} = 0.717, T_{max} = 0.894	R_{int} = 0.030
10013 measured reflections	

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.029	83 parameters
$wR(F^2)$ = 0.081	H atoms treated by a mixture of independent and constrained refinement
S = 1.07	$\Delta\rho_{max}$ = 0.30 e $Å^{-3}$
1627 reflections	$\Delta\rho_{min}$ = −0.17 e $Å^{-3}$

Table 1
Selected geometric parameters (Å, °)

N1—N2	1.4470 (14)	C2—C4	1.5322 (16)
N1—C4	1.5295 (15)	C3—C4	1.5212 (17)
C1—C4	1.5206 (17)		
N2—N1—C4	119.53 (9)	C1—C4—C2	111.74 (10)
C1—C4—C3	112.33 (10)	C3—C4—C2	110.94 (10)

C1—C4—N1	108.68 (9)	N1—C4—C2	104.79 (9)
C3—C4—N1	107.99 (10)		

Table 2
Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1A···Cl	0.864 (16)	2.279 (16)	3.1419 (11)	177.3 (14)
N1—H1B···Cl^{i}	0.907 (16)	2.261 (16)	3.1670 (10)	177.4 (13)
N2—H2A···Cl^{ii}	0.920 (19)	2.590 (18)	3.4800 (12)	162.8 (15)
N2—H2B···Cl^{iii}	0.941 (18)	2.604 (18)	3.5255 (12)	166.3 (15)

Symmetry codes: (i) $x-1/2, y, -z+3/2$; (ii) $-x+3/2, y-1/2, z$; (iii) $-x+1, y-1/2, -z+3/2$.

Acknowledgements

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References

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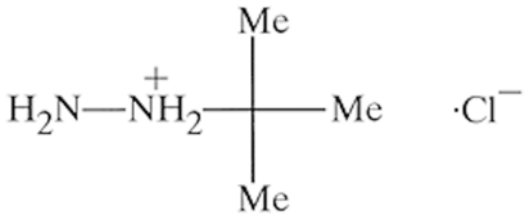
CIF access

Scheme 1



supplementary materials

t-Butylhydrazine hydrochloride

Crystal data

$C_4H_{13}N_2^{1+}\cdot Cl^{1-}$	$D_x = 1.155$ Mg m^{-3}
$M_r = 124.61$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Orthorhombic, *Pbca*	Cell parameters from 7356 reflections
$a = 9.8615$ (1) Å	$\theta = 3.0$–30.6º
$b = 10.6446$ (2) Å	$\mu = 0.43$ mm^{-1}
$c = 13.6513$ (1) Å	$T = 173$ (2) K
$V = 1433.00$ (3) Å^3	Block, colorless
$Z = 8$	0.62 × 0.58 × 0.35 mm
$F_{000} = 544$	

Data collection

Siemens SMART CCD diffractometer	1627 independent reflections
Radiation source: fine-focus sealed tube	1408 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.030$
$T = 173(2)$ K	$\theta_{max} = 27.5$º
ω scans	$\theta_{min} = 3.0$º
Absorption correction: empirical (using intensity measurements) (SADABS; Sheldrick, 1996)	$h = -12\rightarrow12$
$T_{min} = 0.717$, $T_{max} = 0.894$	$k = -13\rightarrow12$
10013 measured reflections	$l = -12\rightarrow17$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.029$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.081$	$w = 1/[\sigma^2(F_o^2) + (0.0397P)^2 + 0.4905P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.07$	$(\Delta/\sigma)_{max} = 0.001$
1627 reflections	$\Delta\rho_{max} = 0.30$ e Å^{-3}
83 parameters	$\Delta\rho_{min} = -0.17$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl	0.75018 (3)	0.40890 (3)	0.78844 (2)	0.02416 (12)
N1	0.51603 (10)	0.24245 (9)	0.70175 (7)	0.0176 (2)
H1A	0.5799 (16)	0.2870 (14)	0.7275 (11)	0.025 (4)*
H1B	0.4408 (15)	0.2911 (14)	0.7065 (10)	0.022 (4)*
N2	0.49815 (12)	0.13638 (11)	0.76677 (8)	0.0236 (2)
H2A	0.575 (2)	0.0871 (15)	0.7625 (14)	0.039 (5)*
H2B	0.4295 (19)	0.0839 (15)	0.7420 (14)	0.038 (5)*
C1	0.67638 (13)	0.14655 (12)	0.58366 (10)	0.0254 (3)
H1D	0.6671	0.0666	0.6154	0.080*
H1E	0.7484	0.1931	0.6140	0.080*
H1F	0.6969	0.1340	0.5156	0.080*
C2	0.55586 (15)	0.35107 (12)	0.54822 (10)	0.0278 (3)
H2D	0.5717	0.3440	0.4791	0.080*
H2E	0.6299	0.3952	0.5782	0.080*
H2F	0.4731	0.3964	0.5593	0.080*
C3	0.42437 (14)	0.14859 (12)	0.55029 (10)	0.0276 (3)
H3A	0.4191	0.0667	0.5795	0.080*
H3B	0.4359	0.1402	0.4808	0.080*
H3C	0.3423	0.1940	0.5636	0.080*
C4	0.54446 (12)	0.21954 (10)	0.59307 (8)	0.0188 (2)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl	0.01935 (18)	0.02054 (18)	0.0326 (2)	0.00123 (10)	−0.00438 (11)	−0.00120 (11)
N1	0.0168 (5)	0.0160 (5)	0.0200 (5)	0.0000 (4)	0.0004 (4)	−0.0004 (4)
N2	0.0257 (6)	0.0223 (5)	0.0228 (5)	0.0001 (4)	0.0024 (5)	0.0057 (4)
C1	0.0231 (6)	0.0282 (6)	0.0248 (6)	0.0048 (5)	0.0037 (5)	0.0002 (5)
C2	0.0332 (7)	0.0232 (6)	0.0269 (6)	0.0002 (5)	0.0016 (5)	0.0060 (5)
C3	0.0285 (7)	0.0276 (6)	0.0267 (6)	−0.0018 (5)	−0.0063 (5)	−0.0028 (5)
C4	0.0206 (6)	0.0184 (5)	0.0174 (5)	0.0003 (4)	0.0005 (4)	0.0002 (4)

Geometric parameters (Å, °)

N1—N2	1.4470 (14)	C2—C4	1.5322 (16)
N1—C4	1.5295 (15)	C3—C4	1.5212 (17)
C1—C4	1.5206 (17)		
N2—N1—C4	119.53 (9)	C1—C4—C2	111.74 (10)
C1—C4—C3	112.33 (10)	C3—C4—C2	110.94 (10)
C1—C4—N1	108.68 (9)	N1—C4—C2	104.79 (9)
C3—C4—N1	107.99 (10)		
N2—N1—C4—C1	−60.93 (13)	N2—N1—C4—C2	179.49 (10)
N2—N1—C4—C3	61.17 (13)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1A···Cl	0.864 (16)	2.279 (16)	3.1419 (11)	177.3 (14)
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Symmetry codes: (i) *x*−1/2, *y*, −*z*+3/2; (ii) −*x*+3/2, *y*−1/2, *z*; (iii) −*x*+1, *y*−1/2, −*z*+3/2.