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Tribenzylammonium chloride

Waly Diallo,[a]* Libasse Diop,[a] Laurent Plasseraud[b] and Hélène Cattey[b]*

[a]Laboratoire de Chimie Minérale et Analytique (LACHIMIA), Département de Chimie, Faculté des Sciences et Techniques, Université Cheikh Anta Diop, Dakar, Senegal, and [b]ICMUB UMR 6302, Université de Bourgogne, Faculté des Sciences, 9 avenue Alain Savary, 21000 Dijon, France
Correspondence e-mail: diallo_waly@yahoo.fr, hcattey@u-bourgogne.fr

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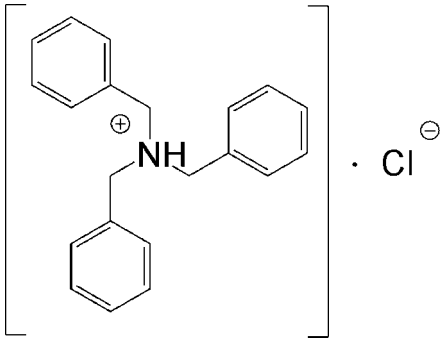
Key indicators: single-crystal X-ray study; T = 115 K; mean σ(C–C) = 0.003 Å; R factor = 0.021; wR factor = 0.052; data-to-parameter ratio = 14.7.

Single crystals of the title salt, $C_{21}H_{21}NH^{+}\cdot Cl^{-}$, were isolated as a side product from the reaction involving $[(C_6H_5CH_2)_3NH]_2[HPO_4]$ and $Sn(CH_3)_3Cl$ in ethanol. Both the cation and the anion are situated on a threefold rotation axis. The central N atom in the cation has a slightly distorted tetrahedral environment, with angles ranging from 107.7 to 111.16 (10)°. In the crystal, the tribenzylammonium cations and chloride anions are linked through N—H···Cl and C—H···Cl hydrogen bonds, leading to the formation of infinite chains along [001]. The crystal studied was a merohedral twin.

Related literature

For related crystal structures containing the tribenzylammonium cation, see: Kozhomuratova *et al.* (2007); Jarvinen *et al.* (1988); Guo *et al.* (2010); Zeng *et al.* (1994); Fazaeli *et al.* (2010); Guan *et al.* (2013); Yousefi *et al.* (2007); Gueye *et al.* (2012); Traore *et al.* (2013). For details of the treatment of intensity data from a twinned crystal, see: Spek (2009).



Experimental

Crystal data

$C_{21}H_{22}N^{+}\cdot Cl^{-}$
M_r = 323.85
Trigonal, $R3$
a = 15.3833 (8) Å
c = 6.7051 (3) Å
V = 1374.15 (18) Å^3
Z = 3
Mo $K\alpha$ radiation
μ = 0.21 mm^{-1}
T = 115 K
0.47 × 0.27 × 0.12 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: multi-scan (Blessing, 1995)
T_{min} = 0.923, T_{max} = 0.963
1884 measured reflections
1047 independent reflections
1045 reflections with $I > 2\sigma(I)$
R_{int} = 0.016

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.021
$wR(F^2)$ = 0.052
S = 1.10
1047 reflections
71 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.12 e Å^{-3}
$\Delta\rho_{min}$ = −0.11 e Å^{-3}
Absolute structure: Flack parameter determined using 348 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2012)
Absolute structure parameter: 0.01 (4)

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N—H···Cl^{i}	1.00	2.00	3.004 (2)	180
C1—H1B···Cl	0.99	2.70	3.5470 (18)	144
C3—H3···Cl^{i}	0.95	3.06	3.683 (2)	125

Symmetry code: (i) $x, y, z-1$.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *DENZO-SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO-SMN*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012) and *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *WinGX* (Farrugia, 2012).

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Supporting information for this paper is available from the IUCr electronic archives (Reference: WM5019).

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supporting information

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Tribenzylammonium chloride

Waly Diallo, Libasse Diop, Laurent Plasseraud and Hélène Cattey

S1. Comment

Tribenzylammonium cations are often used to stabilize metal complex-anions such as $[(C_6H_5CH_2)_3NH\cdot C_6H_5CH_2NH_2][CuCl_4]$ (Zeng *et al.*, 1994), $(Bz_3NH)_3[Mo_6OCl_{13}]$ and $(Bz_3NH)_2[Mo_6Cl_{14}]\cdot 2CH_3CN$ (Bz is benzyl; Kozhomuratova *et al.*, 2007), $[(C_6H_5CH_2)_3NH][AuCl_4]$ (Fazaeli *et al.*, 2010), $2[C_{21}H_{22}N^+]\cdot[MCl_6]^{2-}$ (*M* = Se, Re, Te) (Guo *et al.*, 2010), $2[C_{21}H_{22}N^+]\cdot[CoCl_4]^{2-}$ and $2[C_{21}H_{22}N^+]\cdot[CuCl_4]^{2-}$ (Guan *et al.*, 2013). In the course of our ongoing studies on organotin(IV) chemistry, we serendipitously isolated the title salt, tribenzylammonium chloride $C_{21}H_{21}NH^+\cdot Cl^-$, from the reaction of $[(C_6H_5CH_2)_3NH]_2[HPO_4]$ with $Sn(CH_3)_3Cl$. Together with $C_{21}H_{21}NH^+\cdot Cl^-$, we suggest the formation of the tin(IV) compound, $[(C_6H_5CH_2)_3NH][HPO_4SnMe_3]$. Howver, we were not successful to isolate single crystals of this compound so far.

The asymmetric unit of tribenzylammonium chloride consists of one third of a $(C_6H_5CH_2)_3NH^+$ cation and an Cl^- anion (Fig. 1). The cationic molecule is completed by the symmetry operation associated with a threefold rotation axis. The N—C bond length within the cation [N–C1 1.5145 (17)] is nearly identical to that observed in tris(tribenzylammonium) hexachloridoplatinate(IV) chloride (Yousefi *et al.*, 2007), in tribenzylammonium l,l,l,l,2,2,2,3,3,3-decacarbonyl-2,3-*m*-hydrido-*2,3*-*m*-sulfonyl-*triangulo*-triosmium (Jarvinen *et al.*, 1988), or in dibenzylazanium (oxalato-κ^2O,O')triphenylstannate(IV) (Gueye *et al.*, 2012). The C–N–C angles [C1–N–C1^{ii} 111.16 (10)°] indicate a slight angular distortion in the tetrahedral environment.

In the crystal, the chloride anion is linked to the tribenzylammonium cation *via* N—H···Cl hydrogen bonding (Table 1). In addition and from a supramolecular point of view, the chloride anions are also in intermolecular weak interaction with three methylinic protons of the benzyl groups of neighboring cations (Table 1). The observed distances are in the range of those reported in literature for such interactions, for example in $[(C_6H_5CH_2Ph_3P]^+[SnPh_3Cl_2]^-$ (Traore *et al.*, 2013). The combination of N—H···Cl and C—H···Cl hydrogen bonding interactions leads to the formation of infinite chains along [001] (Fig. 2).

S2. Experimental

All chemicals were purchased from Sigma-Aldrich and were used without further purification. Crystals of the title compound were obtained by reacting $[(C_6H_5CH_2)_3NH]_2[HPO_4]$ (0.300 g, 0.446 mmol), previously synthesized from phosphoric acid (98%$_{wt}$) and tribenzylamine, with $Sn(CH_3)_3Cl$ (0.088 g, 0.446 mmol) in 15 ml of ethanol (98% purity). The mixture was stirred for around two hours at room temperature. Colorless crystals were obtained after one week by slow solvent evaporation.

S3. Refinement

The H atoms,on carbon and nitrogen atoms were placed at calculated positions using a riding model with C—H = 0.95 Å (aromatic), or 0.99 Å (methylene) or N—H = 1.00 Å (amine) with $U_{iso}(H) = 1.2U_{eq}$(C or N). Intensity data revealed

twinning by merohedry. The twin law was found by using TwinRotMat implemented in *PLATON* (Spek, 2009). The use of the twin law (-*h*-*k*, *k*, -*l*) and a refined twin component ratio of 0.93:0.07 reduced the reliability factor *R* (I>2σ(I)) from 0.042 to 0.021. The three reflections (-1 2 0; 1 1 0; -1 1 1) were affected by the beam stop and were omitted from the refinement.

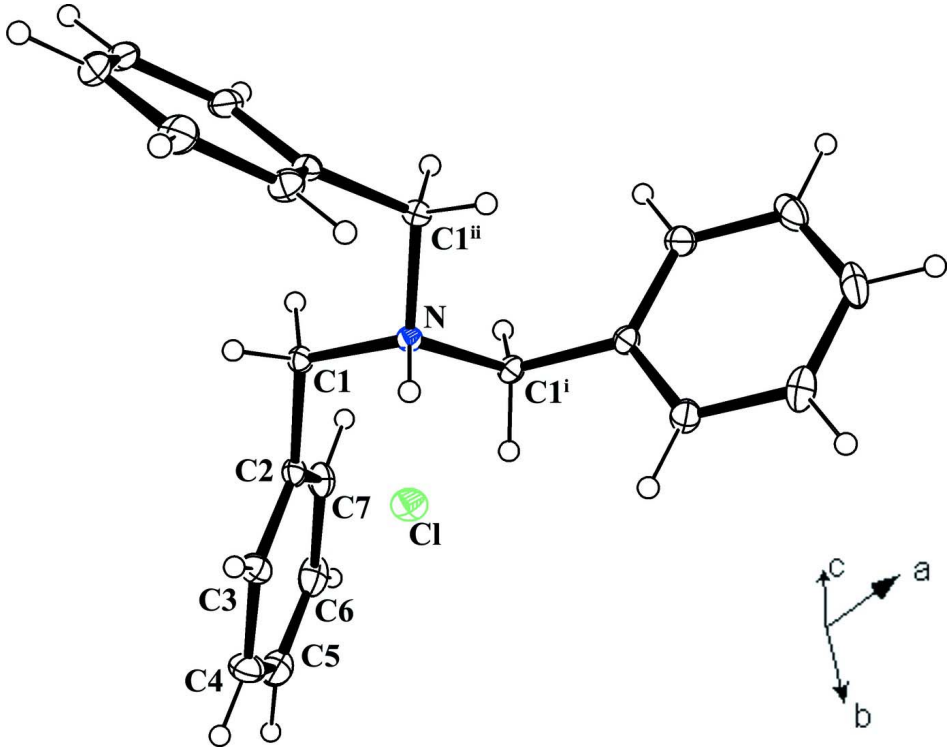

Figure 1

The molecular structure of the title compound with atom labeling. Displacement ellipsoids are draw at the 30% probability level. [Symmetry codes: (i) -*x* + *y*+1, -*x* + 1, *z*; (ii) -*y* + 1, *x*-*y*, *z*.]

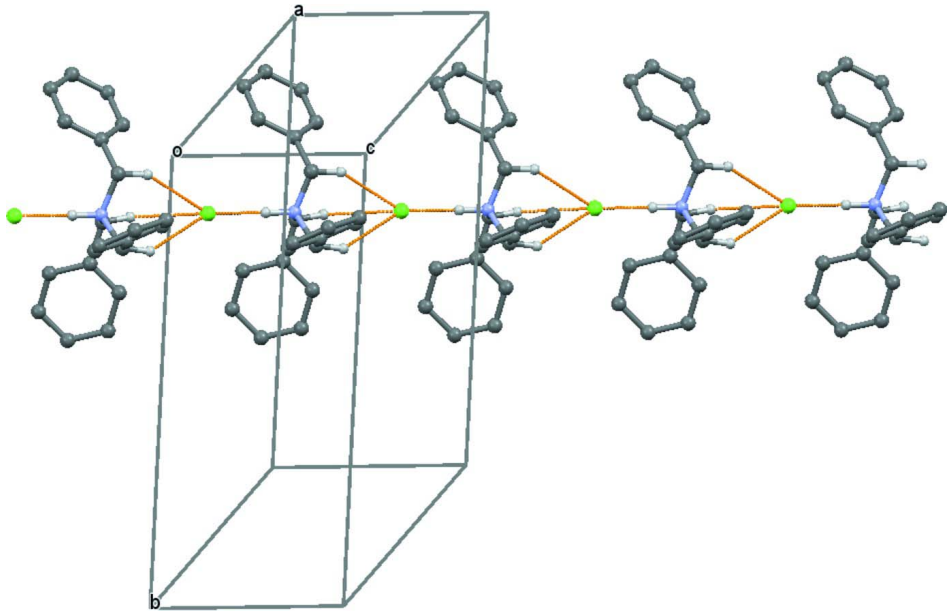

Figure 2

The crystal packing of the title compound showing a chain-like arrangement along [001] through N—H···Cl and C—H···Cl interactions (dashed orange lines; H atoms not involved in hydrogen bonding were omitted for clarity). Colour code: C dark grey, H light grey, N blue, Cl green.

Tribenzylazanium chloride

Crystal data

$C_{21}H_{22}N^+\cdot Cl^-$
M_r = 323.85
Trigonal, $R3$
Hall symbol: R 3
a = 15.3833 (8) Å
c = 6.7051 (3) Å
V = 1374.15 (18) Å^3
Z = 3
$F(000)$ = 516
D_x = 1.174 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2745 reflections
θ = 1.0–27.5°
μ = 0.21 mm^{-1}
T = 115 K
Prism, colourless
0.47 × 0.27 × 0.12 mm

Data collection

Nonius KappaCCD diffractometer
Radiation source: X-ray tube, Siemens KFF Mo 2K-180
Graphite monochromator
Detector resolution: 512 x 512 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (Blessing, 1995)
T_{min} = 0.923, T_{max} = 0.963
1884 measured reflections
1047 independent reflections
1045 reflections with $I > 2\sigma(I)$
R_{int} = 0.016
θ_{max} = 27.5°, θ_{min} = 3.4°
h = −18→17
k = −19→10
l = −8→6

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.021
$wR(F^2)$ = 0.052
S = 1.10
1047 reflections
71 parameters
1 restraint

Primary atom site location: iterative
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + 0.8302P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.12$ e Å^{-3}
$\Delta\rho_{min} = -0.11$ e Å^{-3}
Absolute structure: Flack parameter determined using 348 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2012)
Absolute structure parameter: 0.01 (4)

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. Refined as a 2-component twin.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl	0.6667	0.3333	0.80013 (9)	0.02215 (17)
N	0.6667	0.3333	0.2481 (3)	0.0146 (5)
H	0.6667	0.3333	0.0990	0.018*
C1	0.56631 (12)	0.31837 (13)	0.3169 (3)	0.0168 (3)
H1A	0.5122	0.2574	0.2520	0.020*
H1B	0.5604	0.3064	0.4626	0.020*
C2	0.55016 (13)	0.40504 (12)	0.2725 (3)	0.0176 (4)
C3	0.51543 (14)	0.41462 (15)	0.0861 (3)	0.0231 (4)
H3	0.5043	0.3676	−0.0165	0.028*
C4	0.49715 (17)	0.49308 (17)	0.0510 (3)	0.0313 (5)
H4	0.4731	0.4991	−0.0757	0.038*
C5	0.51373 (16)	0.56246 (15)	0.1988 (4)	0.0332 (5)
H5	0.5011	0.6159	0.1738	0.040*
C6	0.54881 (15)	0.55355 (16)	0.3832 (4)	0.0313 (5)
H6	0.5613	0.6016	0.4844	0.038*
C7	0.56583 (14)	0.47482 (14)	0.4207 (3)	0.0234 (4)
H7	0.5884	0.4683	0.5487	0.028*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl	0.0273 (2)	0.0273 (2)	0.0118 (3)	0.01366 (12)	0.000	0.000
N	0.0156 (6)	0.0156 (6)	0.0128 (13)	0.0078 (3)	0.000	0.000
C1	0.0151 (7)	0.0189 (8)	0.0162 (8)	0.0084 (7)	0.0009 (7)	0.0013 (7)
C2	0.0139 (8)	0.0187 (8)	0.0202 (9)	0.0080 (7)	0.0013 (6)	−0.0002 (7)
C3	0.0240 (9)	0.0268 (9)	0.0220 (10)	0.0155 (8)	−0.0003 (7)	0.0000 (7)
C4	0.0317 (11)	0.0362 (11)	0.0349 (10)	0.0237 (9)	0.0028 (9)	0.0099 (10)
C5	0.0268 (10)	0.0240 (10)	0.0558 (15)	0.0178 (8)	0.0105 (10)	0.0066 (10)
C6	0.0227 (9)	0.0242 (9)	0.0478 (14)	0.0123 (8)	0.0087 (9)	−0.0072 (9)
C7	0.0186 (9)	0.0258 (10)	0.0254 (10)	0.0108 (8)	0.0014 (7)	−0.0053 (8)

Geometric parameters (Å, º)

N—H	1.0000	C3—H3	0.9500
N—C1^{i}	1.5145 (17)	C3—C4	1.389 (3)
N—C1^{ii}	1.5145 (17)	C4—H4	0.9500
N—C1	1.5145 (17)	C4—C5	1.384 (3)
C1—H1A	0.9900	C5—H5	0.9500
C1—H1B	0.9900	C5—C6	1.383 (3)
C1—C2	1.503 (2)	C6—H6	0.9500
C2—C3	1.396 (3)	C6—C7	1.384 (3)
C2—C7	1.392 (2)	C7—H7	0.9500
C1^{ii}—N—H	107.7	C2—C3—H3	120.1
C1^{i}—N—H	107.7	C4—C3—C2	119.86 (19)
C1—N—H	107.7	C4—C3—H3	120.1
C1^{ii}—N—C1^{i}	111.16 (10)	C3—C4—H4	119.7
C1^{i}—N—C1	111.16 (10)	C5—C4—C3	120.6 (2)
C1^{ii}—N—C1	111.16 (10)	C5—C4—H4	119.7
N—C1—H1A	108.7	C4—C5—H5	120.2
N—C1—H1B	108.7	C6—C5—C4	119.63 (19)
H1A—C1—H1B	107.6	C6—C5—H5	120.2
C2—C1—N	114.39 (13)	C5—C6—H6	119.9
C2—C1—H1A	108.7	C5—C6—C7	120.2 (2)
C2—C1—H1B	108.7	C7—C6—H6	119.9
C3—C2—C1	120.83 (16)	C2—C7—H7	119.7
C7—C2—C1	120.07 (16)	C6—C7—C2	120.62 (19)
C7—C2—C3	119.04 (17)	C6—C7—H7	119.7
N—C1—C2—C3	83.7 (2)	C2—C3—C4—C5	0.5 (3)
N—C1—C2—C7	−99.0 (2)	C3—C2—C7—C6	−1.0 (3)
C1^{ii}—N—C1—C2	174.09 (11)	C3—C4—C5—C6	0.0 (3)
C1^{i}—N—C1—C2	49.7 (2)	C4—C5—C6—C7	−1.0 (3)
C1—C2—C3—C4	177.39 (18)	C5—C6—C7—C2	1.5 (3)
C1—C2—C7—C6	−178.38 (17)	C7—C2—C3—C4	0.1 (3)

Symmetry codes: (i) −*x*+*y*+1, −*x*+1, *z*; (ii) −*y*+1, *x*−*y*, *z*.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N—H···Cl^{iii}	1.00	2.00	3.004 (2)	180
C1—H1*B*···Cl	0.99	2.70	3.5470 (18)	144
C3—H3···Cl^{iii}	0.95	3.06	3.683 (2)	125

Symmetry code: (iii) *x*, *y*, *z*−1.