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Benzylbutyldimethylammonium bromide

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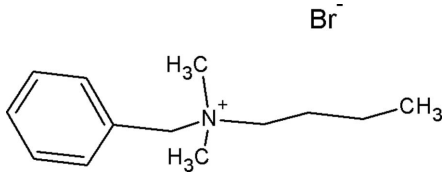
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.038; wR factor = 0.077; data-to-parameter ratio = 34.5.

The crystal structure of the title compound, $C_{13}H_{22}N^+\cdot Br^-$, has been determined as part of a study of the relationship between the sorption properties of montmorillonite and the architecture of the hydrophobic layers formed by modifications of the clay mineral by amphiphilic compounds. In the crystal structure, benzylbutyldimethylammonium and bromide ions are linked *via* weak C—H···Br hydrogen-bonding interactions, with C—H···Br1 = 3.745 (2)–4.016 (2) Å. C—H···π interactions are also observed in the structure. The ammonium cations are packed in a pseudo-tetragonal 'parquet'-style pattern, with encapsulated Br^- ions.

Related literature

For related literature, see: Hodorowicz *et al.* (2003, 2005); Kwolek *et al.* (2003); Kruger *et al.* (2003); Allen *et al.* (1987).



Experimental

Crystal data

$C_{13}H_{22}N^+\cdot Br^-$
M_r = 272.23
Monoclinic, $P2_1/n$
a = 8.924 (2) Å
b = 9.046 (2) Å
c = 17.183 (4) Å
β = 96.787 (1)°
V = 1377.4 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 2.96 mm^{-1}
T = 293 (2) K
0.25 × 0.22 × 0.20 mm

Data collection

Nonius KappaCCD area-detector diffractometer
Absorption correction: multi-scan (*DENZO* and *SCALEPACK*; Otwinowski & Minor, 1997)
T_{min} = 0.493, T_{max} = 0.554
7959 measured reflections
4725 independent reflections
3058 reflections with $I > 2\sigma(I)$
R_{int} = 0.021

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.077
S = 1.08
4725 reflections
137 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.45 e Å^{-3}

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

*Cg*1 is the centroid of the C31–C36 ring.

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3*A*···Br1	0.97	2.82	3.745 (2)	160
C3—H3*B*···Br1^{i}	0.97	2.89	3.825 (2)	162
C33—H33···Br1^{ii}	0.93	3.02	3.814 (2)	145
C36—H36···Br1	0.93	3.13	3.929 (2)	146
C2—H2*C*···Br1^{iii}	0.96	3.12	3.966 (2)	148
C4—H4*B*···Br1^{iv}	0.96	3.04	3.811 (2)	138
C1—H1*A*···Br1^{i}	0.97	3.13	4.016 (2)	153
C12—H12*B*···*Cg*1^{v}	0.97	3.10	3.980	151

Symmetry codes: (i) $-x-\frac{1}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$; (ii) $x-\frac{1}{2}, -y+\frac{1}{2}, z-\frac{1}{2}$; (iii) $x, y+1, z$; (iv) $-x+\frac{1}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$; (v) $-x+\frac{3}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$.

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* and *DENZO* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1999) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *SHELXL97*.

The author thanks the Joint X-ray Laboratory, Faculty of Chemistry, and SLAFiBS, Jagiellonian University, for making the Nonius KappaCCD diffractometer available.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ER2039).

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supplementary materials

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Benzylbutyldimethylammonium bromide

M. Hodorowicz

Comment

Ammonium halides are widely studied cationic surfactants used in many fields such as micelar catalysis, medicine, detergency. Additionally they are able to change the nature of the surface of clay minerals, such as montmorillonite or bentonite, from hydrophilic to hydrophobic one (Kwolek *et al.*, 2003). The title compound was investigated in the project on relationship between sorption properties of montmorillonite and the architecture of hydrophobic layers which are due to modifications of the clay mineral by, in this case, quaternary alkylammonium salts (Hodorowicz *et al.*, 2003, 2005). The crystal structure analysis of benzyldimethylbutylammonium bromide was performed to find out the influence of molecular geometry on the packing properties of the ammonium cations. The molecular structure of the title compound is shown in Fig. 1. The symmetrically independent part of the unit cell is composed of the ammonium cation, showing pseudosymmetry m, and bromide counterion ($N^{+}\cdots Br^{-}$ = 4.287 (2) Å). The bond lengths and angles indicate the typical tetrahedral arragement of substituents at the N atom. The benzene rings are essentially planar, with a mean deviation of the C atoms from the best plane of 0.006 Å. The molecular dimensions are comparable with the values reported in the literature (Allen *et al.*, 1987). Methyl and methylene groups as well as C—H of C31–C36 benzene ring of the quaternary ammonium cation are involved in weak intermolecular hydrogen interactions of C—H$\cdots Br^{-}$ type (Table 1). This kind of interactions are responsible for self-assembly of ammonium cations in hydrophobic layers (Hodorowicz *et al.*, 2003, 2005). The ammonium cations are packed in a pseudo-tetragonal `parquet'-style pattern, with Br^{-} ions in between (Fig. 2).

Experimental

The title compoud was prepared by dissolving a (1:1) mixture of benzyl bromide [$CH_3(CH_2)_3Br$] and *N*,*N*-dimethylbenzylamine [$C_6H_5CH_2N(CH_3)_2$] in acetone at 273 K. The solution was slowly heated to room temperature to give colourless single crystals of the title compound. Recrystallization from acetone afforded crystals suitable for X-ray measurements.

Refinement

All hydrogen atom positions were observed in difference Fourier map. Nevertheless, in the refinement procedure the hydrogen atoms were positioned geometrically and refined using a riding model (including free rotation about the C—C bond), with C—H = 0.95–0.99 Å (C—H = 0.97 Å for CH_2 groups, 0.96 Å for CH_3 groups, and 0.93 Å for aromatic CH) and with U_{iso}(H) = 1.5U_{eq}(C) for methyl groups and U_{iso}(H) = 1.2U_{eq}(C) for all other H atoms.

Figures

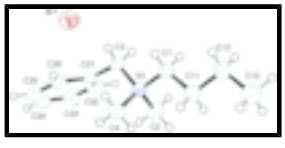

Fig. 1. *ORTEP-3* (Farrugia, 1999) drawing of the title compound with labels. Displacement ellipsoids of non-H atoms drawn at 30% probabilty level.

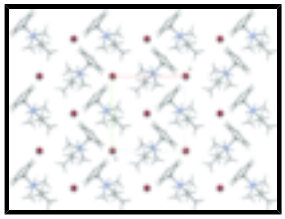

Fig. 2. Mercury (Macrae *et al.*, 2006) drawing of the ammonium cations packed in a pseudo-tetragonal `parquet'-style pattern, with Br^- ions in between; viewed along [001].

Benzylbutyldimethylammonium bromide

Crystal data

$C_{13}H_{22}N^+ \cdot Br^-$	F_{000} = 568
M_r = 272.23	D_x = 1.313 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 4362 reflections
a = 8.924 (2) Å	θ = 1.0–32.0º
b = 9.046 (2) Å	μ = 2.96 mm^{-1}
c = 17.183 (4) Å	T = 293 (2) K
β = 96.7870 (10)º	Prism, colourless
V = 1377.4 (5) Å^3	0.25 × 0.22 × 0.20 mm
Z = 4	

Data collection

Nonius KappaCCD area-detector diffractometer	4725 independent reflections
Radiation source: fine-focus sealed tube	3058 reflections with $I > 2\sigma(I)$
Monochromator: horizontally mounted graphite crystal	R_{int} = 0.021
Detector resolution: 9 pixels mm^{-1}	θ_{max} = 32.0º
T = 293(2) K	θ_{min} = 3.5º
φ and ω scans to fill the asymmetric unit	h = 0→13
Absorption correction: multi-scan (DENZO and SCALEPACK; Otwinowski & Minor, 1997)	k = −13→12
T_{min} = 0.493, T_{max} = 0.554	l = −25→25
7959 measured reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.038	$w = 1/[\sigma^2(F_o^2) + (0.0084P)^2 + 0.6796P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.077	$(\Delta/\sigma)_{max} < 0.001$

S = 1.08	$\Delta\rho_{max}$ = 0.36 e Å^{-3}
4725 reflections	$\Delta\rho_{min}$ = −0.45 e Å^{-3}
137 parameters	Extinction correction: SHELXL97 (Sheldrick, 1997), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.0556 (12)
Secondary atom site location: difference Fourier map	

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}
Br1	0.01487 (2)	0.00013 (2)	0.241455 (12)	0.04883 (9)
N1	−0.06917 (17)	0.45897 (17)	0.19138 (10)	0.0387 (3)
C1	−0.0634 (2)	0.4635 (2)	0.28006 (12)	0.0433 (4)
H1A	−0.1634	0.4870	0.2932	0.052*
H1B	−0.0377	0.3656	0.3005	0.052*
C11	0.0478 (2)	0.5733 (2)	0.32121 (13)	0.0505 (5)
H11A	0.1494	0.5470	0.3116	0.061*
H11B	0.0261	0.6714	0.3000	0.061*
C12	0.0385 (2)	0.5749 (2)	0.40864 (13)	0.0510 (5)
H12A	0.0583	0.4763	0.4296	0.061*
H12B	−0.0628	0.6027	0.4181	0.061*
C13	0.1511 (3)	0.6825 (3)	0.45093 (16)	0.0700 (7)
H13A	0.1421	0.6807	0.5060	0.084*
H13B	0.1306	0.7804	0.4310	0.084*
H13C	0.2515	0.6542	0.4425	0.084*
C2	−0.1167 (3)	0.6065 (2)	0.15765 (13)	0.0539 (5)
H2A	−0.2139	0.6318	0.1724	0.065*
H2B	−0.1223	0.6025	0.1016	0.065*
H2C	−0.0444	0.6800	0.1773	0.065*
C3	−0.1856 (2)	0.3414 (2)	0.16165 (11)	0.0418 (4)
H3A	−0.1579	0.2488	0.1880	0.050*
H3B	−0.2831	0.3705	0.1762	0.050*
C31	−0.2008 (2)	0.3157 (2)	0.07474 (12)	0.0412 (4)
C32	−0.3044 (2)	0.3941 (2)	0.02355 (13)	0.0513 (5)
H32	−0.3641	0.4661	0.0432	0.062*

C33	−0.3195 (3)	0.3662 (3)	−0.05597 (14)	0.0580 (6)
H33	−0.3889	0.4196	−0.0895	0.070*
C34	−0.2324 (3)	0.2596 (3)	−0.08580 (14)	0.0581 (6)
H34	−0.2424	0.2415	−0.1394	0.070*
C35	−0.1303 (3)	0.1797 (2)	−0.03612 (14)	0.0568 (5)
H35	−0.0712	0.1078	−0.0563	0.068*
C36	−0.1154 (2)	0.2061 (2)	0.04369 (13)	0.0488 (5)
H36	−0.0477	0.1503	0.0770	0.059*
C4	0.0826 (2)	0.4170 (3)	0.16919 (13)	0.0513 (5)
H4A	0.1114	0.3222	0.1913	0.062*
H4B	0.1557	0.4897	0.1889	0.062*
H4C	0.0780	0.4121	0.1132	0.062*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.04368 (12)	0.04684 (12)	0.05522 (14)	−0.00025 (9)	0.00274 (8)	0.00673 (10)
N1	0.0350 (7)	0.0334 (7)	0.0490 (9)	−0.0011 (6)	0.0100 (7)	0.0040 (6)
C1	0.0405 (9)	0.0443 (11)	0.0461 (10)	0.0012 (8)	0.0094 (8)	0.0048 (8)
C11	0.0519 (12)	0.0452 (11)	0.0550 (12)	−0.0024 (9)	0.0091 (10)	0.0024 (10)
C12	0.0480 (11)	0.0482 (12)	0.0582 (13)	0.0042 (9)	0.0113 (10)	−0.0054 (10)
C13	0.0710 (16)	0.0664 (16)	0.0727 (17)	−0.0093 (13)	0.0084 (13)	−0.0159 (14)
C2	0.0680 (14)	0.0359 (10)	0.0570 (13)	−0.0008 (10)	0.0050 (11)	0.0069 (9)
C3	0.0368 (9)	0.0366 (9)	0.0537 (11)	−0.0034 (7)	0.0123 (8)	0.0048 (8)
C31	0.0357 (9)	0.0374 (9)	0.0513 (11)	−0.0043 (7)	0.0086 (8)	0.0027 (8)
C32	0.0413 (10)	0.0487 (12)	0.0637 (14)	0.0025 (9)	0.0055 (10)	0.0053 (10)
C33	0.0528 (12)	0.0591 (14)	0.0598 (14)	−0.0068 (10)	−0.0037 (10)	0.0109 (11)
C34	0.0647 (14)	0.0581 (14)	0.0523 (13)	−0.0205 (11)	0.0101 (11)	−0.0015 (11)
C35	0.0617 (13)	0.0462 (12)	0.0647 (14)	−0.0051 (10)	0.0165 (11)	−0.0089 (11)
C36	0.0493 (11)	0.0384 (10)	0.0590 (13)	0.0021 (8)	0.0079 (9)	0.0001 (9)
C4	0.0375 (10)	0.0581 (13)	0.0610 (13)	−0.0043 (9)	0.0174 (9)	−0.0032 (11)

Geometric parameters (Å, °)

N1—C2	1.496 (2)	C2—H2C	0.9600
N1—C4	1.499 (2)	C3—C31	1.501 (3)
N1—C1	1.519 (2)	C3—H3A	0.9700
N1—C3	1.532 (2)	C3—H3B	0.9700
N1—Br1	4.287 (2)	C31—C32	1.392 (3)
C1—C11	1.518 (3)	C31—C36	1.395 (3)
C1—H1A	0.9700	C32—C33	1.380 (3)
C1—H1B	0.9700	C32—H32	0.9300
C11—C12	1.515 (3)	C33—C34	1.375 (3)
C11—H11A	0.9700	C33—H33	0.9300
C11—H11B	0.9700	C34—C35	1.378 (3)
C12—C13	1.520 (3)	C34—H34	0.9300
C12—H12A	0.9700	C35—C36	1.383 (3)
C12—H12B	0.9700	C35—H35	0.9300
C13—H13A	0.9600	C36—H36	0.9300

C13—H13B	0.9600	C4—H4A	0.9600
C13—H13C	0.9600	C4—H4B	0.9600
C2—H2A	0.9600	C4—H4C	0.9600
C2—H2B	0.9600		
C2—N1—C4	110.54 (16)	N1—C2—H2B	109.5
C2—N1—C1	109.82 (15)	H2A—C2—H2B	109.5
C4—N1—C1	109.70 (15)	N1—C2—H2C	109.5
C2—N1—C3	109.90 (15)	H2A—C2—H2C	109.5
C4—N1—C3	109.68 (15)	H2B—C2—H2C	109.5
C1—N1—C3	107.14 (14)	C31—C3—N1	114.67 (14)
C2—N1—Br1	167.48 (12)	C31—C3—H3A	108.6
C4—N1—Br1	70.35 (10)	N1—C3—H3A	108.6
C1—N1—Br1	80.81 (9)	C31—C3—H3B	108.6
C3—N1—Br1	59.34 (9)	N1—C3—H3B	108.6
C11—C1—N1	115.32 (16)	H3A—C3—H3B	107.6
C11—C1—H1A	108.4	C32—C31—C36	118.2 (2)
N1—C1—H1A	108.4	C32—C31—C3	121.68 (18)
C11—C1—H1B	108.4	C36—C31—C3	120.06 (18)
N1—C1—H1B	108.4	C33—C32—C31	120.8 (2)
H1A—C1—H1B	107.5	C33—C32—H32	119.6
C12—C11—C1	111.03 (17)	C31—C32—H32	119.6
C12—C11—H11A	109.4	C34—C33—C32	120.2 (2)
C1—C11—H11A	109.4	C34—C33—H33	119.9
C12—C11—H11B	109.4	C32—C33—H33	119.9
C1—C11—H11B	109.4	C33—C34—C35	119.9 (2)
H11A—C11—H11B	108.0	C33—C34—H34	120.0
C11—C12—C13	111.7 (2)	C35—C34—H34	120.0
C11—C12—H12A	109.3	C34—C35—C36	120.2 (2)
C13—C12—H12A	109.3	C34—C35—H35	119.9
C11—C12—H12B	109.3	C36—C35—H35	119.9
C13—C12—H12B	109.3	C35—C36—C31	120.6 (2)
H12A—C12—H12B	107.9	C35—C36—H36	119.7
C12—C13—H13A	109.5	C31—C36—H36	119.7
C12—C13—H13B	109.5	N1—C4—H4A	109.5
H13A—C13—H13B	109.5	N1—C4—H4B	109.5
C12—C13—H13C	109.5	H4A—C4—H4B	109.5
H13A—C13—H13C	109.5	N1—C4—H4C	109.5
H13B—C13—H13C	109.5	H4A—C4—H4C	109.5
N1—C2—H2A	109.5	H4B—C4—H4C	109.5
C2—N1—C1—C11	−61.3 (2)	N1—C3—C31—C32	−90.8 (2)
C4—N1—C1—C11	60.4 (2)	N1—C3—C31—C36	92.4 (2)
C3—N1—C1—C11	179.40 (16)	C36—C31—C32—C33	−1.4 (3)
Br1—N1—C1—C11	125.59 (16)	C3—C31—C32—C33	−178.28 (19)
N1—C1—C11—C12	176.89 (17)	C31—C32—C33—C34	0.2 (3)
C1—C11—C12—C13	179.01 (19)	C32—C33—C34—C35	0.4 (3)
C2—N1—C3—C31	63.8 (2)	C33—C34—C35—C36	0.1 (3)
C4—N1—C3—C31	−58.0 (2)	C34—C35—C36—C31	−1.4 (3)
C1—N1—C3—C31	−176.96 (15)	C32—C31—C36—C35	2.0 (3)

Br1—N1—C3—C31	−109.11 (16)	C3—C31—C36—C35	178.89 (18)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3A···Br1	0.97	2.82	3.745 (2)	160
C3—H3B···Br1^{i}	0.97	2.89	3.825 (2)	162
C33—H33···Br1^{ii}	0.93	3.02	3.814 (2)	145
C36—H36···Br1	0.93	3.13	3.929 (2)	146
C2—H2C···Br1^{iii}	0.96	3.12	3.966 (2)	148
C4—H4B···Br1^{iv}	0.96	3.04	3.811 (2)	138
C4—H4A···Br1	0.96	3.19	4.038 (2)	149
C11—H11B···Br1^{iii}	0.97	3.14	4.096 (2)	170
C1—H1A···Br1^{i}	0.97	3.13	4.016 (2)	153
C11—H11A···Br1^{iv}	0.97	3.26	4.222 (2)	171
C35—H35···Br1^{v}	0.93	3.42	4.125 (2)	134
C2—H2A···Br1^{i}	0.96	3.43	4.244 (2)	144
C12—H12A···Cg1^{vi}	0.97	3.23	4.111	152
C12—H12B···Cg1^{vii}	0.97	3.10	3.980	151

Symmetry codes: (i) −*x*−1/2, *y*+1/2, −*z*+1/2; (ii) *x*−1/2, −*y*+1/2, *z*−1/2; (iii) *x*, *y*+1, *z*; (iv) −*x*+1/2, *y*+1/2, −*z*+1/2; (v) −*x*, −*y*, −*z*; (vi) *x*−1/2, −*y*−1/2, *z*−1/2; (vii) −*x*+3/2, *y*+1/2, −*z*+1/2.

Fig. 1

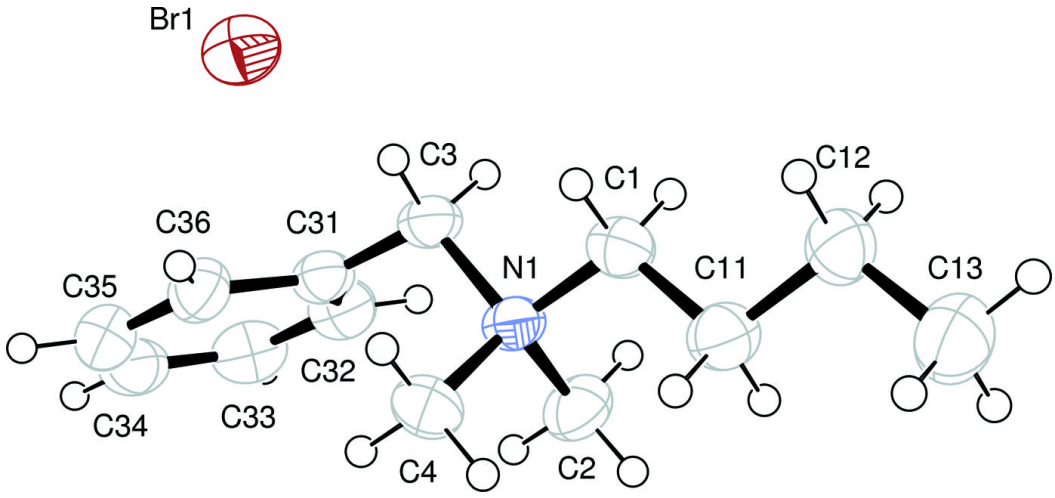

Fig. 2