

Manuela Ramos Silva,* Ana
Matos Beja and Jose Antonio
PaixãoCEMDRX, Departamento de Física, Faculdade
de Ciências e Tecnologia, Universidade de
Coimbra, P-3004-516 Coimbra, PortugalCorrespondence e-mail:
manuela@pollux.fis.uc.pt

Key indicators

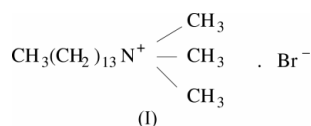
Single-crystal X-ray study
 $T = 293$ K
Mean $\sigma(\text{C}-\text{C}) = 0.004$ Å
 R factor = 0.033
 wR factor = 0.101
Data-to-parameter ratio = 21.3For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.Pseudosymmetry in tetradecyltrimethyl-
ammonium bromide

In the title compound, $\text{C}_{17}\text{H}_{38}\text{N}^+\cdot\text{Br}^-$, the tetradecyl chain is extended in a zigzag form. The molecules are packed in layers with the tetradecyl chains parallel within a layer and antiparallel in alternate layers. There is pseudosymmetry which emulates a c -halved unit cell in $P2_1/m$, but it is not supported by the diffraction pattern, which is consistent with the reported space group and unit cell.

Received 4 July 2003
Accepted 10 July 2003
Online 17 July 2003

Comment

The title compound, (I), is commonly used as a surfactant and germicide. It crystallizes in a centrosymmetric unit cell containing four cations and four anions. The long carbon chain of the cation is not totally planar. Atoms C4 to C14 lie in a plane with a mean deviation of $0.037(3)$ Å from the least-squares plane. This plane makes an angle of $21.87(3)^\circ$ with the plane containing the remaining C atoms (C1, C2 and C3). This differs from what has been observed in a similar compound, *viz.* tetradecyltrimethylammonium salicylate monohydrate, where the tetradecyl chain is fully extended, with a planar chain skeleton (Koh *et al.*, 1993). The average C—C bond length is $1.513(5)$ Å. The average C—C—C angle is $113.8(5)^\circ$. The geometry around the N atom is normal. Due to the lack of donors, no classical hydrogen bonds are found in this structure; only a weak intermolecular interaction exists. There is a short C—H...Br contact between C1N and Br, with a distance of $3.787(2)$ Å and an angle of 161° . The molecules are packed in layers with the tetradecyl chains parallel within a layer and antiparallel in alternate layers.



Experimental

When trying to synthesize a totally different compound, small single crystals were detected on the outside of the flask. One single crystal was analyzed and its origin could be traced to the detergent used to wash the laboratory equipment.

Crystal data

$\text{C}_{17}\text{H}_{38}\text{N}^+\cdot\text{Br}^-$
 $M_r = 336.39$
Monoclinic, $P2_1/c$
 $a = 5.6323(13)$ Å
 $b = 7.240(2)$ Å
 $c = 47.3900(15)$ Å
 $\beta = 91.170(11)^\circ$
 $V = 1932.1(7)$ Å³
 $Z = 4$

$D_x = 1.156$ Mg m⁻³
Cu $K\alpha$ radiation
Cell parameters from 25
reflections
 $\theta = 23.0\text{--}28.3^\circ$
 $\mu = 2.80$ mm⁻¹
 $T = 293(2)$ K
Plate, colourless
 $0.34 \times 0.27 \times 0.12$ mm

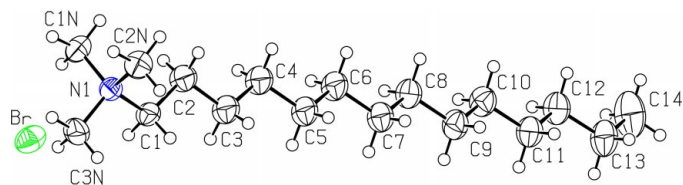


Figure 1
ORTEPII (Johnson, 1976) plot of the title compound. Displacement ellipsoids are drawn at the 50% probability level.

Data collection

Enraf–Nonius CAD-4 diffractometer	$R_{\text{int}} = 0.034$
ω -2 θ scans	$\theta_{\text{max}} = 72.6^\circ$
Absorption correction: ψ scan (North <i>et al.</i> , 1968)	$h = -6 \rightarrow 6$
$T_{\text{min}} = 0.471$, $T_{\text{max}} = 0.711$	$k = 0 \rightarrow 8$
6399 measured reflections	$l = -58 \rightarrow 35$
3750 independent reflections	3 standard reflections
2481 reflections with $I > 2\sigma(I)$	frequency: 180 min
	intensity decay: 10%

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0395P)^2 + 0.767P]$
$R[F^2 > 2\sigma(F^2)] = 0.033$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.101$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.04$	$\Delta\rho_{\text{max}} = 0.97 \text{ e } \text{Å}^{-3}$
3750 reflections	$\Delta\rho_{\text{min}} = -0.51 \text{ e } \text{Å}^{-3}$
176 parameters	
H-atom parameters constrained	

Table 1

Selected geometric parameters (Å , $^\circ$).

N1–C1N	1.493 (3)	N1–C3N	1.499 (2)
N1–C2N	1.495 (3)	N1–C1	1.508 (2)
C1N–N1–C2N	109.41 (17)	C2N–N1–C1	111.53 (17)
C1N–N1–C3N	108.94 (17)	C3N–N1–C1	106.35 (15)
C2N–N1–C3N	108.83 (17)	N1–C1–C2	116.71 (17)
C1N–N1–C1	111.68 (17)		
C1N–N1–C1–C2	54.1 (3)	C3N–N1–C1–C2	172.8 (2)
C2N–N1–C1–C2	–68.7 (2)	N1–C1–C2–C3	166.8 (2)

There is pseudosymmetry which emulates a c -halved unit cell in $P2_1/m$, but it is not supported by the diffraction pattern. Reflections with $l = 2n + 1$ were measured with significant intensities that are reproduced by the structure as described. Refinement attempts in the halved cell, in $P2_1$ and $P2_1/m$, resulted in unrealistic bond lengths and displacement parameters. The methyl H atoms were constrained to an ideal geometry ($\text{C–H} = 0.96 \text{ Å}$) with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$, but were allowed to rotate freely about the C–C bonds. All remaining H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{parent atom})$. Examination of the crystal structure with

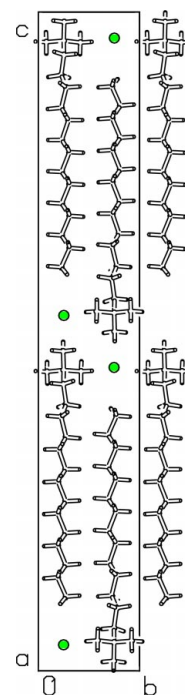


Figure 2
View of the unit-cell packing along a .

PLATON (Spek, 1995) showed that there are no solvent-accessible voids.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *HELENA* (Spek, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

Financial assistance from Fundação para a Ciência e a Tecnologia (Sapiens POCTI/QUI/33495/00) is acknowledged.

References

- Enraf–Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.
- Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
- Koh, L. L., Xu, Y., Gan, L. M., Chew, C. H. & Lee, K. C. (1993). *Acta Cryst.* **C49**, 1032–1035.
- North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* **A24**, 351–359.
- Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
- Spek, A. L. (1995). *PLATON*. University of Utrecht, The Netherlands.
- Spek, A. L. (1997). *HELENA*. University of Utrecht, The Netherlands.