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Tetra-\textit{n}-butylammonium bromide: a redetermination at 150 K addressing the merohedral twinning

Mark R. J. Elsegood

\textit{Acta Cryst.} (2011). \textit{E67}, o2599

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The redetermined, low temperature (150 K), structure of tetra-\(n\)-butylammonium bromide, (\(\text{C}_4\text{H}_{9}\))\(_4\)N\(^+\)Br\(^-\), has been found to be merohedrally twinned via twin law \(\overline{1}00\), \(010\), \(10\overline{1}\). The structure was previously determined, with low precision, no inclusion of H atoms and only the bromide ion refined with anisotropic displacement parameters, by Wang \textit{et al.} (1995). \textit{Mol. Cryst. Liq. Cryst. Sci. Tech. A}, \textbf{264}, 115–129. The redetermined structure has considerably improved precision in all geometrical parameters, has all non-H atoms refined anisotropically, H atoms included, and is isomorphous with the iodide analogue. The structure is otherwise routine, with the shortest cation to anion contacts being between the bromide anion and the \(\text{CH}\) atoms close to the ammonium nitrogen centre at a distance of 2.98–3.11 Å. Each anion makes eight such contacts to four different anions. The \(\text{CH}\) chains are fully extended, adopting an all-\textit{anti} conformation with approximate \(S_4\) point symmetry.

**Related literature**

The structure was previously determined by Wang \textit{et al.} (1995). For the uses of \(\text{tetra-}\(n\)-alkylammonium salts and the isomorphous structure of \(\text{tetra-}\(n\)-butyl ammonium iodide, see: Pruakala \textit{et al.} (2007). For a related stucture, see: McMullan \& Jeffrey (1959). For the conformation of \(n\)-butyl chains, see: Alder \textit{et al.} (1990). For details of the Cambridge Structural Database, see: Fletcher \textit{et al.} (1996); Allen (2002).

**Experimental**

**Crystal data**

\[ \text{C}_{16}\text{H}_{36}\text{N}^+\text{Br}^- \]

\[ M_r = 322.37 \]

Monoclinic, \(C2/c\)

\( a = 13.9773 \) (9) Å

\( b = 13.8623 \) (9) Å

\( c = 20.0450 \) (14) Å

\( \beta = 110.383 \) (10)

\[ V = 3640.7 \) (4) Å\(^3\)

\[ Z = 8 \]

Mo \(K\alpha\) radiation

\[ \mu = 2.25 \) mm\(^{-1}\)

\[ T = 150 \) K

\[ 0.41 \times 0.31 \times 0.16 \) mm

**Data collection**

Bruker APEXIID CCD diffractometer

Absorption correction: multi-scan

(SADABS; Sheldrick, 2006a)

\( T_{\text{min}} = 0.459, T_{\text{max}} = 0.715 \)

\[ R[F^2 > 2\sigma(F^2)] = 0.028 \]

\[ wR(F^2) = 0.073 \]

\[ 21135 \) measured reflections

4415 reflections with \( I > 2\sigma(I) \)

\[ R_{\text{int}} = 0.029 \]

168 parameters

H-atom parameters constrained

\[ \Delta \rho_{\text{max}} = 0.62 \) e Å\(^{-3}\)

\[ \Delta \rho_{\text{min}} = -0.24 \) e Å\(^{-3}\)

**Supplementary data**

Data collection: \textit{APEX2} (Bruker, 2008); cell refinement: \textit{SAINT} (Bruker, 2008); data reduction: \textit{SAINT}; program(s) used to solve structure: \textit{SHELXS97} (Sheldrick, 2008b); program(s) used to refine structure: \textit{SHELXL97} (Sheldrick, 2008b) and \textit{PLATON} (Spek, 2009); molecular graphics: \textit{SHELXTL} (Sheldrick, 2008b); software used to prepare material for publication: \textit{SHELXTL} and local programs.

We wish to acknowledge the use of the EPSRC’s Chemical Database Service at Daresbury.

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supplementary materials
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Tetra-n-butylammonium bromide: a redetermination at 150 K addressing the merohedral twinning

M. R. J. Elsegood

Comment

While many common reagents have had their crystal structures well determined, some many times, some deliberately and many by accident, no good quality structure was available for the title compound, tetra-n-butylammonium bromide (I). Compound (I) is used in a number of synthesis applications (see Prukała et al., 2007, and references therein for further details) and as a source of the large tetra-n-butylammonium cation, which is useful in crystallizing large anions. A search of the Cambridge Structural Database (version 5.32 + 3 updates, Fletcher, et al., 1996, Allen, 2002) revealed just one reported structure of this compound with an R1 of 0.098 that had clearly been problematic (Wang et al., 1995). This earlier determination had only the bromide ion refined anisotropically and did not include hydrogen atoms in the model. The authors ruled out dynamic disorder as the cause of the difficulties and concluded that static disorder was the cause of the poor residual.

The crystals of (I) formed readily by vapour diffusion of diethyl ether into an acetonitrile solution. The data collection set-up was trouble free. After data reduction the structure did not solve readily with SHELXS (Sheldrick, 2008a); only the bromide, the nitrogen and two n-butyl chains being evident. When the structure failed to develop, the coordinates from the published structure were used as a starting point (Wang et al., 1995), but the R1 was ca. 35% for an isotropic model with all non-H atoms in the model. Twinning was suspected and confirmed by the TWINROTMAT routine in PLATON (Spek, 2009). Application of the merohedral twin law -1 0 0, 0 -1 0, 1 0 1, led to a reduction in R1 to ca. 5.0% at the same, isotropic, stage of refinement. Anisotropic refinement, and addition of H atoms, led to a good final R1 <3% with no adverse indicators. The ratio of major to minor twin components is 60.69: 39.31 (7)%

The structure is isomorphous with that of the iodide analogue described in detail recently (Prukała et al., 2007). The n-butyl chains are fully extended adopting an all-anti conformation with approximate S4 point symmetry (Alder et al., 1990). The bromide anion resides in a pocket between four cations, making four pairs of weak C—H···Br contacts in the range 2.98–3.11 Å to methylene hydrogens located one or two carbon atoms from the nitrogen cationic centre. The structures of the chloride and fluoride analogues have not been determined to date, although the unit cell of the hydrate of the chloride has been reported (McMullan & Jeffrey, 1959).

Experimental

The title compound (I) was used as received and crystallized from an acetonitrile solution via vapour diffusion with diethyl-ether to give colourless blocks.

Refinement

H atoms were included in a riding model with constrained bond lengths: for CH2 = 0.99 and CH3 = 0.98 Å with Uiso(H) = 1.2 Ueq(CH2) and =1.5Ueq(CH3).
Figures

Fig. 1. The asymmetric unit in the structure of (I) with displacement ellipsoids drawn at the 50% probability level.

Tetra-n-butylammonium bromide

Crystal data

$C_{16}H_{36}^+\text{N}^+\text{Br}^-$

$M_r = 322.37$

Monoclinic, $C2/c$

Hall symbol: -C 2yc

$a = 13.9773$ (9) Å

$b = 13.8623$ (9) Å

$c = 20.0450$ (14) Å

$\beta = 110.383$ (10)$^\circ$

$V = 3640.7$ (4) Å$^3$

$Z = 8$

$F(000) = 1392$

$D_x = 1.176$ Mg m$^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 7468 reflections

$\theta = 2.6-30.1^\circ$

$\mu = 2.25$ mm$^{-1}$

$T = 150$ K

Block, colourless

$0.41 \times 0.31 \times 0.16$ mm

Data collection

Bruker APEXII CCD
diffractometer

Radiation source: fine-focus sealed tube

graphite

$\omega$ rotation with narrow frames scans

Absorption correction: multi-scan

(SADABS; Sheldrick, 2008a)

$T_{\text{min}} = 0.459$, $T_{\text{max}} = 0.715$

21135 measured reflections

5485 independent reflections

4415 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.029$

$\theta_{\text{max}} = 30.5^\circ$, $\theta_{\text{min}} = 1.1^\circ$

$h = -19 \rightarrow 19$

$k = -18 \rightarrow 19$

$l = -28 \rightarrow 28$

Refinement

Refinement on $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.028$

$wR(F^2) = 0.073$

$S = 1.04$

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0379P)^2 + 0.7322P]$

sup-2
supplementary materials

5485 reflections
168 parameters
0 restraints

where \( P = (F_o^2 + 2F_c^2)/3 \)

\((\Delta \sigma)_{\text{max}} = 0.001\)
\(\Delta \rho_{\text{max}} = 0.62 \text{ e Å}^{-3}\)
\(\Delta \rho_{\text{min}} = -0.24 \text{ e Å}^{-3}\)

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 \( F^2 > \sigma(F^2) \) 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 (Å²)

|      | \( x \)     | \( y \)     | \( z \)     | \( U_{iso} \) or \( U_{eq} \) |
|------|-------------|-------------|-------------|-----------------------------|
| Br1  | 0.737682 (14) | 0.00074 (2) | 0.475441 (8) | 0.03037 (6) |
| N1   | 0.49621 (18)  | 0.25167 (8) | 0.49516 (13) | 0.0172 (2)  |
| C1   | 0.44659 (14)  | 0.30519 (13)| 0.54096 (10)| 0.0195 (4)  |
| H1A  | 0.4927        | 0.3579      | 0.5664      | 0.023*       |
| H1B  | 0.3826        | 0.3352      | 0.5093      | 0.023*       |
| C2   | 0.4221 (2)     | 0.24346 (12)| 0.59514 (15)| 0.0252 (6)  |
| H2A  | 0.4848        | 0.2105      | 0.6258      | 0.030*       |
| H2B  | 0.3718        | 0.1934      | 0.5702      | 0.030*       |
| C3   | 0.37887 (16)   | 0.30443 (14)| 0.64107 (11)| 0.0253 (4)  |
| H3A  | 0.3187        | 0.3406      | 0.6101      | 0.030*       |
| H3B  | 0.4309        | 0.3519      | 0.6682      | 0.030*       |
| C4   | 0.3476 (3)     | 0.24244 (16)| 0.69282 (16)| 0.0302 (6)  |
| H4A  | 0.2984        | 0.1936      | 0.6663      | 0.045*       |
| H4B  | 0.3163        | 0.2833      | 0.7194      | 0.045*       |
| H4C  | 0.4081        | 0.2106      | 0.7261      | 0.045*       |
| C5   | 0.42140 (14)   | 0.17734 (13)| 0.45040 (10)| 0.0191 (4)  |
| H5A  | 0.4085        | 0.1292      | 0.4827      | 0.023*       |
| H5B  | 0.3559        | 0.2101      | 0.4249      | 0.023*       |
| C6   | 0.45519 (19)   | 0.12433 (12)| 0.39615 (10)| 0.0230 (4)  |
| H6A  | 0.5208        | 0.0911      | 0.4206      | 0.028*       |
| H6B  | 0.4659        | 0.1712      | 0.3621      | 0.028*       |
| C7   | 0.37484 (16)   | 0.05075 (14)| 0.35601 (11)| 0.0259 (4)  |
| H7A  | 0.3613        | 0.0065      | 0.3905      | 0.031*       |
| H7B  | 0.3104        | 0.0847      | 0.3296      | 0.031*       |
| C8   | 0.4089 (3)     | -0.00752 (19)| 0.30414 (11)| 0.0347 (5)  |
| H8A  | 0.4195        | 0.0358      | 0.2687      | 0.052*       |
| H8B  | 0.3563        | -0.0551     | 0.2801      | 0.052*       |
| H8C  | 0.4729        | -0.0409     | 0.3301      | 0.052*       |
**supplementary materials**

|     | \( U^{11} \)    | \( U^{22} \)    | \( U^{33} \)    | \( U^{12} \)    | \( U^{13} \)    | \( U^{23} \)    |
|-----|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|
| Br1 | 0.02772 (9)     | 0.02330 (8)     | 0.04278 (10)    | 0.00074 (10)    | 0.01565 (8)     | 0.0010 (2)      |
| N1  | 0.0158 (9)      | 0.0157 (5)      | 0.0192 (6)      | -0.0003 (5)     | 0.0048 (12)     | 0.0008 (7)      |
| C1  | 0.0217 (9)      | 0.0173 (8)      | 0.0213 (9)      | 0.0011 (7)      | 0.0099 (8)      | -0.0019 (7)     |
| C2  | 0.0345 (15)     | 0.0197 (10)     | 0.0255 (12)     | -0.0008 (7)     | 0.0157 (11)     | 0.0002 (7)      |
| C3  | 0.0274 (10)     | 0.0261 (9)      | 0.0271 (10)     | 0.0023 (8)      | 0.0154 (8)      | 0.0008 (8)      |
| C4  | 0.0316 (15)     | 0.0361 (12)     | 0.0308 (12)     | 0.0011 (9)      | 0.0208 (14)     | 0.0059 (10)     |
| C5  | 0.0177 (9)      | 0.0181 (8)      | 0.0221 (10)     | -0.0031 (6)     | 0.0076 (7)      | -0.0022 (7)     |
| C6  | 0.0215 (10)     | 0.0229 (8)      | 0.0251 (9)      | -0.0023 (8)     | 0.0088 (9)      | -0.0047 (6)     |
| C7  | 0.0289 (10)     | 0.0219 (9)      | 0.0284 (10)     | -0.0029 (8)     | 0.0117 (8)      | -0.0061 (8)     |
| C8  | 0.0435 (15)     | 0.0270 (10)     | 0.0368 (9)      | -0.0008 (11)    | 0.0182 (12)     | -0.0112 (11)    |
| C9  | 0.0189 (9)      | 0.0189 (8)      | 0.0223 (9)      | 0.0029 (7)      | 0.0067 (7)      | 0.0004 (7)      |
| C10 | 0.0218 (12)     | 0.0236 (9)      | 0.0254 (12)     | -0.0009 (8)     | 0.0017 (10)     | 0.0011 (8)      |
| C11 | 0.0193 (9)      | 0.0299 (10)     | 0.0341 (11)     | 0.0015 (8)      | 0.0042 (8)      | -0.0038 (9)     |
| C12 | 0.0218 (13)     | 0.0513 (16)     | 0.0310 (12)     | -0.0006 (13)    | 0.0026 (11)     | -0.0076 (12)    |
| C13 | 0.0227 (10)     | 0.0169 (8)      | 0.0227 (10)     | -0.0026 (7)     | 0.0108 (8)      | 0.0013 (7)      |
| C14 | 0.0247 (11)     | 0.0243 (8)      | 0.0252 (9)      | 0.0029 (8)      | 0.0109 (9)      | 0.0057 (7)      |
| C15 | 0.0291 (10)     | 0.0222 (9)      | 0.0276 (11)     | -0.0012 (8)     | 0.0100 (8)      | 0.0037 (8)      |
| C16 | 0.0371 (15)     | 0.0302 (8)      | 0.0319 (8)      | 0.0019 (11)     | 0.0073 (9)      | 0.0101 (14)     |
### Geometric parameters (Å, °)

| Bond/Interaction | Length (Å) | Angle (°)  |
|------------------|------------|------------|
| N1—C5            | 1.519 (3)  | C8—H8B     | 0.9800 |
| N1—C1            | 1.522 (3)  | C8—H8C     | 0.9800 |
| N1—C13           | 1.524 (3)  | C9—C10     | 1.522 (3) |
| N1—C9            | 1.526 (3)  | C9—H9A     | 0.9900 |
| C1—C2            | 1.513 (3)  | C9—H9B     | 0.9900 |
| C1—H1A           | 0.9900     | C10—C11    | 1.520 (3) |
| C1—H1B           | 0.9900     | C10—H10A   | 0.9900 |
| C2—C3            | 1.521 (3)  | C10—H10B   | 0.9900 |
| C2—H2A           | 0.9900     | C11—C12    | 1.527 (4) |
| C2—H2B           | 0.9900     | C11—H11A   | 0.9900 |
| C3—C4            | 1.523 (4)  | C11—H11B   | 0.9900 |
| C3—H3A           | 0.9900     | C12—H12A   | 0.9800 |
| C3—H3B           | 0.9900     | C12—H12B   | 0.9800 |
| C4—H4A           | 0.9800     | C12—H12C   | 0.9800 |
| C4—H4B           | 0.9800     | C13—C14    | 1.521 (3) |
| C4—H4C           | 0.9800     | C13—H13A   | 0.9900 |
| C5—C6            | 1.518 (3)  | C13—H13B   | 0.9900 |
| C5—H5A           | 0.9900     | C14—C15    | 1.526 (3) |
| C5—H5B           | 0.9900     | C14—H14A   | 0.9900 |
| C6—C7            | 1.523 (3)  | C14—H14B   | 0.9900 |
| C6—H6A           | 0.9900     | C15—C16    | 1.526 (3) |
| C6—H6B           | 0.9900     | C15—H15A   | 0.9900 |
| C7—C8            | 1.518 (3)  | C15—H15B   | 0.9900 |
| C7—H7A           | 0.9900     | C16—H16A   | 0.9800 |
| C7—H7B           | 0.9900     | C16—H16B   | 0.9800 |
| C8—H8A           | 0.9800     | C16—H16C   | 0.9800 |
| C5—N1—C1         | 108.81 (17)| C7—C8—H8C  | 109.5 |
| C5—N1—C13        | 111.35 (18)| H8A—C8—H8C | 109.5 |
| C1—N1—C13        | 108.81 (12)| H8B—C8—H8C | 109.5 |
| C5—N1—C9         | 108.62 (12)| C10—C9—N1  | 114.89 (16) |
| C1—N1—C9         | 110.88 (17)| C10—C9—H9A | 108.5 |
| C13—N1—C9        | 108.39 (18)| N1—C9—H9A  | 108.5 |
| C2—C1—N1         | 114.96 (15)| C10—C9—H9B | 108.5 |
| C2—C1—H1A        | 108.5      | N1—C9—H9B  | 108.5 |
| N1—C1—H1A        | 108.5      | H9A—C9—H9B | 107.5 |
| C2—C1—H1B        | 108.5      | C11—C10—C9 | 110.05 (17) |
| N1—C1—H1B        | 108.5      | C11—C10—H10A | 109.7 |
| H1A—C1—H1B       | 107.5      | C9—C10—H10A | 109.7 |
| C1—C2—C3         | 110.93 (15)| C11—C10—H10B | 109.7 |
| C1—C2—H2A        | 109.5      | C9—C10—H10B | 109.7 |
| C3—C2—H2A        | 109.5      | H10A—C10—H10B | 108.2 |
| C1—C2—H2B        | 109.5      | C10—C11—C12 | 110.9 (2) |
| C3—C2—H2B        | 109.5      | C10—C11—H11A | 109.5 |
| H2A—C2—H2B       | 108.0      | C12—C11—H11A | 109.5 |
| C2—C3—C4         | 111.53 (18)| C10—C11—H11B | 109.5 |
| C2—C3—H3A        | 109.3      | C12—C11—H11B | 109.5 |
supplementary materials

| Bond          | Angle (°) | Bond          | Angle (°) |
|---------------|-----------|---------------|-----------|
| C4—C3—H3A    | 109.3     | H11A—C11—H11B| 108.1     |
| C2—C3—H3B    | 109.3     | C11—C12—H12A | 108.5     |
| C4—C3—H3B    | 109.3     | C11—C12—H12B | 109.5     |
| H3A—C3—H3B   | 108.0     | H12A—C12—H12B| 109.5     |
| C3—C4—H4A    | 109.5     | C11—C12—H12C | 109.5     |
| C3—C4—H4B    | 109.5     | H12A—C12—H12C| 109.5     |
| H4A—C4—H4B   | 109.5     | H12B—C12—H12C| 109.5     |
| C3—C4—H4C    | 109.5     | C14—C13—N1   | 115.22 (17)|
| H4A—C4—H4C   | 109.5     | C14—C13—H13A | 108.5     |
| H4B—C4—H4C   | 109.5     | N1—C13—H13A  | 108.5     |
| C6—C5—N1     | 115.45 (17)| C14—C13—H13B | 108.5     |
| C6—C5—H5A    | 108.4     | N1—C13—H13B  | 108.5     |
| N1—C5—H5A    | 108.4     | H13A—C13—H13B| 107.5     |
| C6—C5—H5B    | 108.4     | C13—C14—C15  | 109.86 (18)|
| N1—C5—H5B    | 108.4     | C13—C14—H14A | 109.7     |
| H5A—C5—H5B   | 107.5     | C15—C14—H14A | 109.7     |
| C5—C6—C7     | 110.28 (19)| C13—C14—H14B | 109.7     |
| C5—C6—H6A    | 109.6     | C15—C14—H14B | 109.7     |
| C7—C6—H6A    | 109.6     | H14A—C14—H14B| 108.2     |
| C5—C6—H6B    | 109.6     | C14—C15—C16  | 111.1 (2)  |
| C7—C6—H6B    | 109.6     | C14—C15—H15A | 109.4     |
| H6A—C6—H6B   | 108.1     | C16—C15—H15A | 109.4     |
| C8—C7—C6     | 111.6 (2) | C14—C15—H15B | 109.4     |
| C8—C7—H7A    | 109.3     | C16—C15—H15B | 109.4     |
| C6—C7—H7A    | 109.3     | H15A—C15—H15B| 108.0     |
| C8—C7—H7B    | 109.3     | C15—C16—H16A | 109.5     |
| C6—C7—H7B    | 109.3     | C15—C16—H16B | 109.5     |
| H7A—C7—H7B   | 108.0     | H16A—C16—H16B| 109.5     |
| C7—C8—H8A    | 109.5     | C15—C16—H16C | 109.5     |
| C7—C8—H8B    | 109.5     | H16A—C16—H16C| 109.5     |
| H8A—C8—H8B   | 109.5     | H16B—C16—H16C| 109.5     |
| C5—N1—C1—C2  | 63.9 (2)  | C5—N1—C9—C10 | 172.7 (2) |
| C13—N1—C1—C2 | -174.6 (2)| C1—N1—C9—C10  | 53.2 (2)  |
| C9—N1—C1—C2  | -55.5 (2) | C13—N1—C9—C10 | 66.2 (2)  |
| N1—C1—C2—C3  | 176.5 (2) | N1—C9—C10—C11 | -179.95 (19)|
| C1—C2—C3—C4  | 176.5 (2) | C9—C10—C11—C12| 176.4 (2) |
| C1—N1—C5—C6  | 174.16 (17)| C5—N1—C13—C14 | 54.1 (2)  |
| C13—N1—C5—C6 | 54.2 (2)  | C1—N1—C13—C14 | -65.8 (2) |
| C9—N1—C5—C6  | -65.0 (2) | C9—N1—C13—C14 | 173.53 (16)|
| N1—C5—C6—C7  | 178.84 (17)| N1—C13—C14—C15| -179.49 (17)|
| C5—C6—C7—C8  | -176.82 (18)| C13—C14—C15—C16| -177.50 (18)|
