Table 2. *Selected geometric parameters (Å, °) for (II)*

O11—C11	1.350 (6)	O21—C21	1.349 (6)
O12—C128	1.342 (6)	O22—C228	1.359 (6)
N11—C17	1.302 (6)	N21—C27	1.290 (6)
N11—C114	1.480 (6)	N21—C214	1.456 (6)
N12—C116	1.311 (6)	N22—C216	1.292 (6)
N12—C115	1.479 (6)	N22—C215	1.470 (6)
C17—N11—C114	122.8 (5)	O21—C21—C26	121.9 (5)
C116—N12—C115	120.5 (5)	N21—C27—C26	117.6 (5)
O11—C11—C12	117.2 (6)	N21—C27—C28	124.1 (5)
N11—C17—C18	123.6 (5)	N21—C214—C215	110.2 (5)
N11—C17—C16	116.9 (5)	N22—C215—C214	109.1 (5)
N11—C114—C115	110.0 (5)	N22—C216—C223	117.9 (5)
N12—C115—C114	109.0 (5)	N22—C216—C217	122.7 (5)
N12—C116—C123	118.0 (5)	O22—C228—C223	121.2 (6)
N12—C116—C117	122.5 (5)	O22—C228—C227	118.1 (6)
O21—C21—C22	117.9 (6)		
O11—C11—C16—C17	−0.5 (8)		
N11—C114—C115—N12	−63.4 (6)		
C116—C123—C128—O12	−1.4 (9)		
O21—C21—C26—C25	−179.5 (5)		
N21—C214—C215—N22	62.5 (6)		

Data were collected about the ω axis using exposure times of 10 s and frame increments of 0.20°; the crystal-to-detector distance was 4.9 cm. Crystal decay was estimated by comparing the intensities of common reflections at the start and end of data collection and, in both cases, the rate of decay was negligible. The H atoms were added at calculated positions and refined using a riding model. Anisotropic displacement parameters were used for all non-H atoms and H atoms were given isotropic displacement parameters equal to 1.2 (or 1.5 for methyl H atoms) times the equivalent isotropic displacement parameter of the parent atom.

For both compounds, data collection: *MADNES* (Pflugrath & Messerschmidt, 1992); cell refinement: *MADNES*; data reduction: *SHELXTL-Plus* (Sheldrick, 1991); program(s) used to solve structures: *SHELXTL-Plus*; program(s) used to refine structures: *SHELXL93* (Sheldrick, 1993); molecular graphics: *SHELXTL-Plus*; software used to prepare material for publication: *SHELXL93*.

The authors wish to thank Professor M. B. Hursthouse and the EPSRC X-ray crystallographic service (University of Wales, Cardiff) for collecting the diffraction data. We also wish to acknowledge the use of the EPSRC's Chemical Database Service (Allen *et al.*, 1991) at Daresbury and BP for financial support (JPC).

Lists of atomic coordinates, displacement parameters, structure factors and complete geometry have been deposited with the IUCr (Reference: PA1247). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Allen, F. H., Davies, J. E., Galloy, J. J., Johnson, O., Kennard, O., Macrae, C. F., Mitchell, E. M., Mitchell, G. F., Smith, J. M. & Watson, D. G. (1991). *J. Chem. Inf. Comput. Sci.* **31**, 187–204.

Cannadine, J. C., Corden, J. P., Errington, W., Moore, P. & Wallbridge, M. G. H. (1996). *Acta Cryst.* **C52**, 1014–1017.

Corden, J. P., Errington, W., Moore, P. & Wallbridge, M. G. H. (1996). *Acta Cryst.* **C52**, 125–127.

Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.

Pflugrath, J. W. & Messerschmidt, A. (1992). *MADNES. Munich Area Detector Systems.* Enraf–Nonius, Delft, The Netherlands.

Sheldrick, G. M. (1991). *SHELXTL-Plus*. Release 4.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures.* University of Göttingen, Germany.

Acta Cryst. (1997). **C53**, 488–490

Low-Temperature Phase of Tetraethylammonium Bromide

MARTINA RALLE,[a,c] JEFFREY C. BRYAN,[b] ANTON HABENSCHUSS[c] AND BERNHARD WUNDERLICH[a,c]

[a]*Department of Chemistry, University of Tennessee, Knoxville, TN 37996-1600, USA,* [b]*Chemical and Analytical Sciences Division, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6119, USA, and* [c]*Chemical and Analytical Sciences Division, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6197, USA. E-mail: athas@utk.edu*

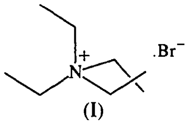
(*Received 30 July 1996; accepted 12 December 1996*)

Abstract

The title compound, $C_8H_{20}N^+.Br^-$, exhibits a trigonal structure. Pairs of alkyl chains form all-*trans* sequences through the N atom.

Comment

Tetra-*n*-alkylammonium bromides and iodides form an interesting series of compounds exhibiting different kinds of disordering transitions depending on alkyl-chain length and temperature. We have been studying the disordering transitions of these materials (up to a chain length of 18 C atoms) using X-ray powder diffraction (Xenopoulos, Ralle, Habenschuss & Wunderlich, 1996; Ralle, Xenopoulos, Habenschuss & Wunderlich, 1996), thermal analysis (Xenopoulos, Cheng, Yasuniva & Wunderlich, 1992; Xenopoulos, Cheng & Wunderlich, 1993) and solid-state NMR (Cheng, Xenopoulos & Wunderlich, 1992*a*,*b*, 1993). The low-temperature crystal structures of tetra-*n*-alkylammonium bromides and iodides containing up to four C atoms per alkyl chain were known for all but the title compound, (I). Solving the crystal structure of the title compound closes an important gap and helps us to complete our study of disorder in these materials.



(I)

Acta Crystallographica Section C
ISSN 0108-2701 © 1997

The four C atoms in the tetraethylammonium ion have distorted tetrahedral symmetry around the N atom, occupying two independent atomic positions (Fig. 1 and Table 2). The N atom occupies one special position and the Br^- ion two.

Along the *c* axis, we find alternating layers of ammonium and bromide ions. These layers are stacked so that triangles of N atoms have Br^- ions above and below the center of each triangle (bromine is located 2.54 Å above and below the plane of each triangle) (Figs. 2 and 3).

This structure is different from the crystal structure of tetraethylammonium iodide (Vincent, Knop, Linden, Cameron & Roberston, 1988). Tetraethylammonium iodide exhibits a tetragonal structure which can be described as a very distorted zincblende structure in which the cation has the shape of a nordic cross.

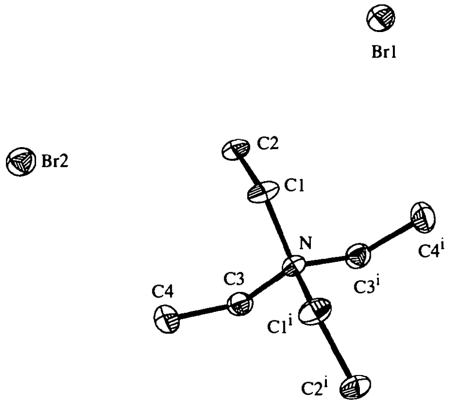


Fig. 1. The structure of tetraethylammonium bromide showing 50% probability displacement ellipsoids. H atoms have been omitted for clarity. [Symmetry code: (i) $\frac{4}{3} - x$, $\frac{2}{3} - x + y$, $\frac{1}{6} - z$.]

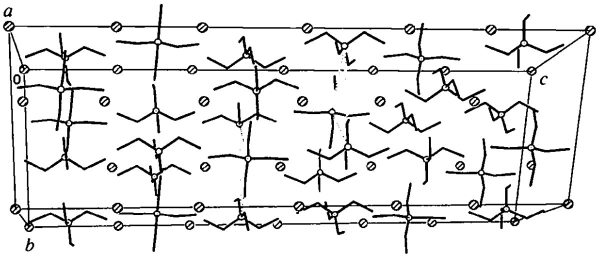


Fig. 2. Packing diagram and unit-cell content of tetraethylammonium bromide. Bromide and ammonium ions are arranged in alternating layers normal to the **c** direction.

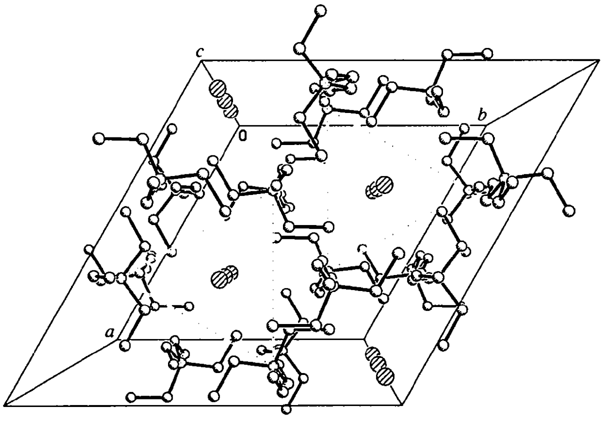


Fig. 3. Packing diagram and unit-cell content of tetraethylammonium bromide viewed down the *c* axis.

Experimental

A sample of tetraethylammonium bromide (Acros Organics) was recrystallized five times from acetone to give transparent plate-shaped single crystals.

Crystal data

$C_8H_{20}N^+.Br^-$
$M_r = 210.16$
Trigonal
$R\bar{3}c$
$a = 12.0128\,(10)$ Å
$c = 35.160\,(3)$ Å
$V = 4394.1\,(6)$ Å^3
$Z = 18$
$D_x = 1.430$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 10.3$–$15.5°$
$\mu = 4.15$ mm^{-1}
$T = 163\,(2)$ K
Block
0.74 × 0.44 × 0.43 mm
Transparent colorless

Data collection

Enraf–Nonius CAD-4 diffractometer
ω scans
Absorption correction: empirical *via* ψ scans (Siemens, 1995)
$T_{min} = 0.113$, $T_{max} = 0.168$
4790 measured reflections
1010 independent reflections

682 reflections with $I > 2\sigma(I)$
$R_{int} = 0.052$
$\theta_{max} = 26.4°$
$h = -15 \rightarrow 0$
$k = -10 \rightarrow 15$
$l = -43 \rightarrow 43$
3 standard reflections
frequency: 120 min
intensity decay: 4%

Refinement

Refinement on F^2
$R(F) = 0.026$
$wR(F^2) = 0.071$
$S = 1.06$
1010 reflections
50 parameters
H atoms not refined
$w = 1/[\sigma^2(F_o^2) + (0.0295P)^2 + 9.6217P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.012$

$\Delta\rho_{max} = 0.35$ e Å^{-3}
$\Delta\rho_{min} = -0.28$ e Å^{-3}
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.00150 (10)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U^{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Br1	0	0	0	0.0240 (2)
Br2	2/3	1/3	0.01103 (1)	0.0269 (2)
N	2/3	0.6543 (2)	1/12	0.0159 (6)

C1	0.6766 (3)	0.7342 (2)	0.04868 (6)	0.0240 (6)
C2	0.6738 (3)	0.6729 (2)	0.01080 (6)	0.0255 (7)
C3	0.5507 (3)	0.5207 (3)	0.08034 (7)	0.0241 (6)
C4	0.4240 (3)	0.5193 (3)	0.07648 (8)	0.0364 (8)

Table 2. *Selected geometric parameters (Å, °)*

N—C1	1.518 (3)	C1—C2	1.514 (3)
N—C3	1.514 (3)	C3—C4	1.519 (4)
C1—N—C1^{i}	107.3 (3)	C3—N—C3^{i}	106.3 (3)
C1—N—C3	110.71 (15)	C2—C1—N	115.0 (2)
C1—N—C3^{i}	110.92 (15)	N—C3—C4	113.9 (2)

Symmetry code: (i) $\frac{4}{3} - x, \frac{2}{3} - x + y, \frac{1}{6} - z$.

All non-H atoms were located from direct methods. H atoms were included in the calculations as riding atoms with common isotropic displacement parameters. Methyl H atoms were allowed to rotate about C—C bonds. Reflections with l odd are systematically weak, giving an apparent pseudo-cell with c = 17.58 Å.

Data collection: *CAD*-4-*PC Software* (Enraf–Nonius, 1993). Cell refinement: *CAD*-4-*PC* Software. Data reduction: *XCAD*4 (Harms, 1995). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1971) and *SHELXTL* (Siemens, 1995).

This work was supported by the Division of Materials Research, National Science Foundation, Polymers Program (Grant No. DMR 90-00520) and Oak Ridge National Laboratory managed by Lockheed Martin Energy Research Corporation for the US Department of Energy under contract number DE-AC05-96OR22464.

Lists of atomic coordinates, displacement parameters, structure factors and complete geometry have been deposited with the IUCr (Reference: BK1293). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Cheng, J., Xenopoulos, A. & Wunderlich, B. (1992*a*). *Mol. Cryst. Liq. Cryst.* **220**, 105–125.

Cheng, J., Xenopoulos, A. & Wunderlich, B. (1992*b*). *Mol. Cryst. Liq. Cryst.* **220**, 127–157.

Cheng, J., Xenopoulos, A. & Wunderlich, B. (1993). *Mol. Cryst. Liq. Cryst.* **225**, 337–353.

Enraf–Nonius (1993). *CAD*-4-*PC Software*. Version 1.2. Enraf–Nonius, Delft, The Netherlands.

Harms, K. (1995). *XCAD*4. *Program for the Lp Correction of Enraf–Nonius Four-Circle Diffractometer Data*. University of Marburg, Germany.

Johnson, C. K. (1971). *ORTEP*II. Report ORNL-3794, revised. Oak Ridge National Laboratory, Tennessee, USA.

Ralle, M., Xenopoulos, A., Habenschuss, A. & Wunderlich, B. (1996). In preparation.

Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.

Sheldrick, G. M. (1993). *SHELXL*93. *Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.

Siemens (1995). *SHELXTL*. Version 5/*IRIX*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Vincent, B. V., Knop, O., Linden, A., Cameron, T. S. & Robertson, K. N. (1988). *Can. J. Chem.* **66**, 3060–3069.

Xenopoulos, A., Cheng, J. & Wunderlich, B. (1993). *Mol. Cryst. Liq. Cryst.* **226**, 87–102.

Xenopoulos, A., Cheng, J., Yasuniva, M. & Wunderlich, B. (1992). *Mol. Cryst. Liq. Cryst.* **214**, 63–79.

Xenopoulos, A., Ralle, M., Habenschuss, A. & Wunderlich, B. (1996). *Powder Diffr.* **11**, 246–249.

Acta Cryst. (1997). C**53**, 490–491

Ethyl *N*-Methyl-2-pyrrolylcarbonylhydrazinocarboxylate

Malva Liu-Gonzalez,[a] Francisco Sanz-Ruiz,[a] Maria Eugenia Gonzalez-Rosende,[b] Oscar Lozano-Lucía,[b] Elena Zaballos-García[b] and Jose Sepulveda-Arqués[b]

[a]*Departamento de Termología, Facultad de Física, Universidad de Valencia, Dr. Moliner 50, 47100 Burjassot (Valencia), Spain, and* [b]*Departamento de Química Orgánica, Facultad de Farmacia, Universidad de Valencia, Avda. Vte. Andrés Estelles s/n, 47100 Burjassot (Valencia), Spain. E-mail: liu@evalrx.scsie.uv.es*

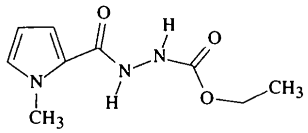
(*Received 23 February 1996; accepted 31 October 1996*)

Abstract

The title compound, $C_9H_{13}N_3O_3$, consists of an *N*-methyl-substituted pyrrolylcarbonyl moiety bonded at position 2 to the N′ atom of a hydrazino chain.

Comment

The C—C and C—N interatomic distances in the title compound, (I), are similar to those found in other pyrrole structures (Cullen, Pepe, Meyer, Falk & Grubmayr, 1979; Fritz, Henlin, Reisen, Tschamber & Zehnderand, 1988; Ruben, Bates, Zalkin & Templeton, 1974).



(I)

The C2—O1 bond [1.232 (2) Å], a urea 'vinylogue', is slightly longer than C3—O2 [1.223(2) Å] in the carbamate group. The C2—N2 interatomic distance is also longer than N3—C3. The torsion angle O1—C2—C2′—N1′ of −8.4 (2)° (Table 1) indicates that the carbonyl group has an *s-cis* disposition with respect to the C—N bond of the pyrrole ring. The relative disposition of the pyrrolylcarbonyl and ethoxycarbonyl substituents on the hydrazine chain afford a torsion