anisotropic temperature factors (*SHELX76*; Sheldrick, 1976). Empirical secondary-extinction correction applied according to the formula $F_{cor} = F(1 - xF^2/\sin\theta)$ where x converged to 0·00121 (6). The function minimized was $\sum w(|F_o| - |F_c|)^2$ with $w = 3{\cdot}6713/\sigma^2(F_o)$. Final $R = 0{\cdot}031$, $wR = 0{\cdot}037$ for 59 refined parameters, $(\Delta/\sigma)_{max} = 0{\cdot}001$. Minimum and maximum heights in the final $\Delta\rho$ map were $-1{\cdot}41$ and 2·85 e Å^{-3}, near the barium positions. Scattering factors including corrections for anomalous dispersion were taken from *International Tables for X-ray Crystallography* (1974). Figures were drawn with *PLUTO*78 (Motherwell & Clegg, 1978) and *ORTEP* (Johnson, 1965). All calculations were performed with an IBM PC/AT.

Discussion. Positional and equivalent isotropic thermal parameters are given in Table 1,* while details of interatomic distances and bond angles are given in Table 2. The structure of $BaCu_2Si_2O_7$ is shown in Fig. 1.

The characteristic feature of the structure is the existence of isolated anions of $[Si_2O_7]^{6-}$. The interatomic Si—O distances ranging from 1·610 (4) to 1·662 (2) Å are typical of this group and comparable with distances for other pyrosilicates (Batalieva & Patenko, 1967; Betechtin, 1950), and, especially $Ba_2CuSi_2O_7$ (Malinovskij, 1984). However, the angle Si—O—Si [133·4 (1)°] is smaller than in $Ba_2CuSi_2O_7$. This difference is a consequence of the smaller distance between barium and the bridging oxygen. The coordination polyhedra of barium and copper are also different from those in $Ba_2CuSi_2O_7$. The cations Ba^{2+} and Cu^{2+} coordinated by O atoms from $[Si_2O_7]^{6-}$ groups form irregular polyhedra (Fig. 2). The Ba^{2+} is coordinated by seven O atoms with distances ranging from 2·713 (6) to 2·932 (4) Å. The Cu^{2+} is coordinated by 4 + 1 O atoms. The nearest four O atoms and copper cation [with distances ranging from 1·926 (3) to 1·973 (3) Å] do not lie on the same plane and with the fifth oxygen [with Cu—O distance 2·789 (4) Å] the coordinated O atoms form an irregular pyramid.

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52778 (9 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of Pentakis(methylammonium) Undecabromodibismuthate

By Janusz Matuszewski

Department of Inorganic Chemistry, Faculty of Engineering, School of Economics, 50-345 Wroclaw, Poland

and Ryszard Jakubas, Lucjan Sobczyk and Tadeusz Głowiak

Institute of Chemistry, University of Wroclaw, 50-383 Wroclaw, Poland

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Abstract. $[NH_3(CH_3)]_5[Bi_2Br_{11}]$, $M_r = 1457{\cdot}3$, orthorhombic, $Pca2_1$, $a = 13{\cdot}405\ (3)$, $b = 14{\cdot}462\ (3)$, $c = 16{\cdot}006\ (3)$ Å, $V = 3102{\cdot}9\ (7)$ Å^3, $Z = 4$, $D_x = 3{\cdot}119$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 257{\cdot}7$ cm^{-1}, $F(000) = 2584$, $T = 297$ K, refinement based on 1976 diffractometric data with $I > 3\sigma(I)$

corrected for absorption and extinction gave $R = 0{\cdot}063$, $wR = 0{\cdot}052$. The crystals consist of $Bi_2Br_{11}^{5-}$ complex anions and methylammonium cations. Some of the cations exhibit partial disorder. The terminal Bi—Br bond lengths vary from 2·694 (7) to 2·938 (8) Å and the bridging Bi—Br distances are 3·060 (8) and 3·085 (8) Å. The structure of the bioctahedral anions is pseudocentrosymmetric.

Introduction. Solid alkylammonium halogenodiantimonates and -bismuthates are of interest because in some cases they show transitions to polar phases (Jakubas, 1986; Jakubas, Czapla, Galewski & Sobczyk, 1986; Jakubas, Sobczyk & Matuszewski, 1987; Jakubas, Krzewska, Bator & Sobczyk, 1988).

The large number of known compounds of this type results from the variety of possible cations and of anion stoichiometries. Various modes of bridging the Sb and Bi atoms *via* the halogen atoms have been found. Recently, Jakubas (1989) reported the preparation of $[NH_3(CH_3)]_5[Bi_2Br_{11}]$ which displays a ferroelectric phase transition at 312 K. The stoichiometry of the compound suggested the presence of bioctahedral anions with five negative charges. In the present paper we describe an X-ray diffraction study of $[NH_3(CH_3)]_5[Bi_2Br_{11}]$ at room temperature, *i.e.* for the ferroelectric phase.

Experimental. The single crystals were grown by slow evaporation of an aqueous solution of $[NH_3(CH_3)]$-Br and $(BiO)_2CO_3$ with a small excess of HBr. The compound crystallized in the form of pseudohexagonal prisms. Preliminary oscillation and Weissenberg photographs did not reveal the true space group initially (reflexions $hk0$ with $k = 2n + 1$ remained pseudoabsent even for long-duration Weissenberg exposures). Therefore the space group *Pcab* was assumed at first. The absence of a centre of symmetry, as imposed by the result of the ferroelectric hysteresis-loop examination, was successfully confirmed by completing the refinement of the structure in space group $Pca2_1$.

Small parallelepiped with dimensions not exceeding 0·15 mm along its edges was used for data acquisition. Enraf–Nonius CAD-4 diffractometer with Mo $K\alpha$ graphite-monochromatized radiation, ω–2θ scans, $\theta_{\min} = 1°$, $\theta_{\max} = 30°$, cell parameters by least squares from setting angles of 25 reflexions in range $15 < 2\theta < 33°$, ω-scan width $(0{\cdot}65 + 0{\cdot}35\tan\theta)°$, max. scan time 60 s, h −1–18, k −1–20, l −1–22. Intensities of three control reflexions ($64\bar{1}$, $1\bar{5}6$, $5\bar{1}\bar{2}$) varied ±5%. Data corrected for Lorentz and polarization effects. For subsequent calculations 1976 reflexions with $I > 3\sigma(I)$ were retained of the 4952 unique reflexions measured. An absorption correction was applied using *DIFABS* (Walker & Stuart, 1983). Min. and max. absorption corrections 0·705 and 1·433. Isotropic extinction correction included in the last cycles of refinement (9×10^{-4}).

As indicated above, the structure was first solved in space group *Pcab* using *SHELXS*86 (Sheldrick, 1986). The correct orientation of the structure in the $Pca2_1$ space group was found using *MULTAN*80 (Main *et al.*, 1980). Most of the light atoms were found in successive difference syntheses, except for two methylammonium cations [C(11)—N(11) and C(12)—N(12)], which were placed in geometrically calculated positions as related to their pseudosymmetrically related counterparts in *Pcab*. (The corresponding peaks were identified in a subsequent difference synthesis but with heights representing 'lowered' electron density.) The refinement of F with anisotropic temperature factors for Bi and Br atoms, and constrained C—N bond lengths (1·49 Å) converged to $R = 0{\cdot}063$ and $wR = 0{\cdot}052$ [$w = 1/\sigma^2(F)$]. Isotropic temperature factors for C(11), N(11), C(12), N(12), C(13), N(13) remained high during refinement indicating at least partial disorder of those cations. Difference syntheses also support this conclusion. Efforts to refine the structure assuming shared occupancy between possible positions of those cations failed. $\Delta\rho$ in final difference synthesis was $\pm 2{\cdot}0$ e Å^{-3}. $(\Delta/\rho)_{\max} = 0{\cdot}07$. The refinement of the parameters for the inverted structure led to $R = 0{\cdot}068$ and $wR = 0{\cdot}058$. The refinement was executed with *SHELX*76 (Sheldrick, 1976), the interatomic distances and angles were calculated with *XTL*/*XTLE* (Syntex, 1976) programs. Atomic scattering factors for neutral atoms were taken from Cromer & Mann (1968) and values of f' and f'' for the anomalous-dispersion correction from Cromer & Liberman (1970). No allowance was made for the scattering of H atoms.

Discussion. Atomic coordinates and isotropic thermal parameters are listed in Table 1.* Bond lengths and angles within the anion are given in Table 2. Selected N···Br distances involving possible hydrogen bonds are given in Table 3.

The crystals are built of discrete undecabromodibismuthate anions and methylammonium cations which make contacts with one another of between 3·37 (3) and 3·66 (3) Å. The content of the unit cell is depicted in Fig. 1, whereas one complete anion is shown in Fig. 2.

The title compound appears to be the first halogenobismuthate in which discrete binuclear $Bi_2Br_{11}^{5-}$ anions are formed by a single bromine bridge. The

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52755 (16 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Fractional atomic coordinates and isotropic temperature parameters* (Å^2) *with e.s.d.'s in parentheses*

$B_{eq} = \frac{1}{3}\sum_i B_{ii}$.

	x	y	z	B_{eq}/B_{iso}
Bi(1)	0·8443 (1)	0·3322 (1)	0·31	2·11 (8)
Bi(2)	0·1676 (1)	0·1660 (1)	0·0833 (1)	1·66 (7)
Br(1)	0·0095 (4)	0·2459 (7)	0·2008 (6)	4·3 (2)
Br(11)	0·7343 (4)	0·1594 (4)	0·2962 (5)	3·8 (3)
Br(12)	0·9636 (4)	0·4909 (5)	0·3220 (5)	5·3 (3)
Br(13)	0·7102 (5)	0·4012 (6)	0·4196 (5)	5·3 (4)
Br(14)	0·9541 (5)	0·2568 (6)	0·4535 (5)	5·2 (4)
Br(15)	0·7485 (6)	0·4021 (5)	0·1724 (5)	5·7 (4)
Br(21)	0·2692 (4)	0·3363 (4)	0·0773 (5)	4·3 (3)
Br(22)	0·0610 (4)	0·9933 (4)	0·0913 (4)	3·5 (2)
Br(23)	0·2985 (4)	0·0867 (5)	0·9776 (5)	3·5 (3)
Br(24)	0·0516 (5)	0·2227 (6)	0·9488 (4)	5·3 (4)
Br(25)	0·2849 (5)	0·1094 (5)	0·2268 (5)	4·5 (3)
N(11)	−0·013 (6)	0·517 (5)	0·071 (5)	11·4 (21)
C(11)	0·043 (7)	0·430 (6)	0·053 (6)	15·2 (34)
C(12)	0·224 (5)	0·365 (4)	0·313 (4)	7·6 (16)
N(12)	0·189 (3)	0·283 (3)	0·361 (3)	5·3 (10)
N(13)	1·007 (3)	0·772 (4)	0·171 (3)	5·4 (12)
C(13)	0·998 (5)	0·724 (5)	0·253 (4)	7·2 (19)
N(21)	1·087 (2)	0·981 (3)	0·882 (2)	3·7 (8)
C(21)	0·990 (3)	0·980 (3)	0·836 (3)	3·1 (10)
C(22)	0·283 (3)	0·823 (3)	0·055 (3)	4·0 (11)
N(22)	0·328 (2)	0·900 (2)	0·103 (2)	3·3 (7)

Table 2. *Bond lengths* (Å) *and angles* (°) *in the complex anion with e.s.d.'s in parentheses*

Bi(1)—Br(1)	3·085 (8)	Bi(2)—Br(1)	3·060 (8)
Bi(1)—Br(11)	2·910 (6)	Bi(2)—Br(21)	2·816 (6)
Bi(1)—Br(12)	2·804 (7)	Bi(2)—Br(22)	2·880 (6)
Bi(1)—Br(13)	2·703 (8)	Bi(2)—Br(23)	2·694 (7)
Bi(1)—Br(14)	2·938 (8)	Bi(2)—Br(24)	2·779 (7)
Bi(1)—Br(15)	2·743 (8)	Bi(2)—Br(25)	2·901 (8)

Br(1)—Bi(1)—Br(11)	88·5 (2)	Br(1)—Bi(2)—Br(21)	91·5 (2)
Br(1)—Bi(1)—Br(12)	87·7 (2)	Br(1)—Bi(2)—Br(22)	87·5 (2)
Br(1)—Bi(1)—Br(13)	174·0 (2)	Br(1)—Bi(2)—Br(23)	176·3 (2)
Br(1)—Bi(1)—Br(14)	86·2 (2)	Br(1)—Bi(2)—Br(24)	88·7 (2)
Br(1)—Bi(1)—Br(15)	91·7 (2)	Br(1)—Bi(2)—Br(25)	89·7 (2)
Br(11)—Bi(1)—Br(12)	175·7 (2)	Br(21)—Bi(2)—Br(22)	179·0 (2)
Br(11)—Bi(1)—Br(13)	91·7 (2)	Br(21)—Bi(2)—Br(23)	92·1 (2)
Br(11)—Bi(1)—Br(14)	89·7 (2)	Br(21)—Bi(2)—Br(24)	89·2 (2)
Br(11)—Bi(1)—Br(15)	91·1 (2)	Br(21)—Bi(2)—Br(25)	90·7 (2)
Br(12)—Bi(1)—Br(13)	91·9 (2)	Br(22)—Bi(2)—Br(23)	89·0 (2)
Br(12)—Bi(1)—Br(14)	88·0 (2)	Br(22)—Bi(2)—Br(24)	90·7 (2)
Br(12)—Bi(1)—Br(15)	91·2 (2)	Br(22)—Bi(2)—Br(25)	89·4 (2)
Br(13)—Bi(1)—Br(14)	88·9 (2)	Br(23)—Bi(2)—Br(24)	90·2 (2)
Br(13)—Bi(1)—Br(15)	94·2 (2)	Br(23)—Bi(2)—Br(25)	91·4 (2)
Br(14)—Bi(1)—Br(15)	177·8 (2)	Br(24)—Bi(2)—Br(25)	178·4 (2)
Bi(1)—Br(1)—Bi(2)	176·5 (3)		

Table 3. *Selected* N···Br *distances* (Å) *with e.s.d.'s in parentheses*

N(11)···Br(21^{i})	3·61 (8)	N(21)···Br(11^{vi})	3·43 (4)
N(12)···Br(14^{ii})	3·50 (4)	N(21)···Br(22^{vii})	3·37 (3)
N(12)···Br(21^{iii})	3·59 (4)	N(21)···Br(23^{viii})	3·57 (3)
N(12)···Br(23^{iv})	3·40 (5)	N(22)···Br(22^{ix})	3·48 (3)
N(13)···Br(14^{v})	3·55 (5)	N(22)···Br(23^{x})	3·39 (3)
N(13)···Br(22)	3·52 (6)	N(22)···Br(25^{viii})	3·66 (3)

Symmetry code: (i) $-\frac{1}{2}+x, 1-y, z$; (ii) $-1+x, y, z$; (iii) $\frac{1}{2}-x, y, \frac{1}{2}+z$; (iv) $\frac{1}{2}-x, y, -\frac{1}{2}+z$; (v) $1-x, 1-y, -\frac{1}{2}+z$; (vi) $1-x, 1-y, \frac{1}{2}+z$; (vii) $x, y, 1+z$; (viii) $x, 1+y, z$; (ix) $\frac{1}{2}+x, 2-y, z$; (x) $x, 1+y, -1+z$.

bridging bonds [3·060 (8) and 3·085 (8) Å] are elongated and the *trans* Bi—Br(terminal) bonds [2·694 (7) and 2·703 (8) Å] shortened compared with the remaining Bi—Br bond lengths of 2·743 (8) to 2·938 (8) Å. It seems probable that the deformation of the octahedra is in part caused by the hydrogen-bonding interactions with the methylammonium cations, as was suggested for diethylammonium hexabromobismuthate(III) by Lazarini (1985). However, the N···Br distances in the present study do not indicate clearly the nature of such interactions. As can be seen from Table 3, Br(23) could form contacts with three N atoms *via* hydrogen bonding. On the other hand, Br(13) makes no contacts with methylammonium cations. Nevertheless, the bridging effects seem to dominate and the Bi(2)—Br(23) bond remains as short as the Bi(1)—Br(13) bond.

Two of the five methylammonium cations [N(21)—C(21) and N(22)—C(22)] are not disordered and are positioned so that the nearest Br atoms are at distances of 3·37 (3) to 3·57 (3) Å and 3·39 (3) to 3·66 (3) Å from N(21) and N(22), respectively. In the case of the C(13)—N(13) methylammonium cation three Br atoms closest to C(13) are at distances of 3·30 (6) and 3·66 (7) Å while three Br atoms closest to N(13) are at distances of 3·52 (6) to 3·55 (5) Å. It

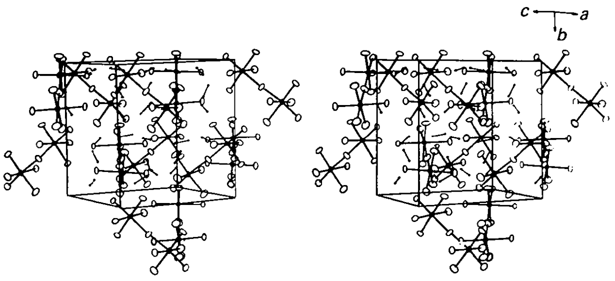

Fig. 1. Stereopacking diagram of the structure of $[NH_3(CH_3)]_5[Bi_2Br_{11}]$. Filled ellipsoids correspond to Bi atoms; solid-line-contoured ellipsoids represent Br atoms; discrete vectors show C—N bond axes in methylammonium cations.

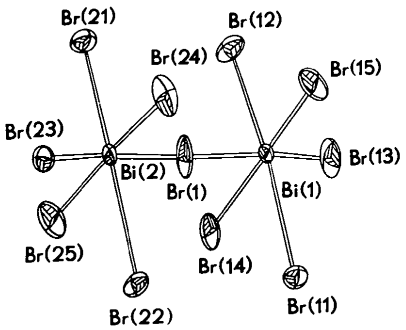

Fig. 2. An *ORTEP* (Johnson, 1976) view of the $Bi_2Br_{11}^{5-}$ anion with atom-numbering scheme.

should be pointed out that in the false *Pcab* space group N(13) and C(13) are symmetry related and cannot be distinguished. It is therefore very probable that these atoms can statistically interchange their positions in the crystal (the refinement of the structure in *Pcab* stopped at $R = 0{\cdot}112$ and $wR = 0{\cdot}113$ and led to unacceptable temperature factors and bond lengths for C—N groups). The disordering of the N(11)—C(11) and N(12)—C(12) cations precludes any interpretation of their interactions with neighbouring anions.

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Structure of 2-(1-Hydroxyethyl)-1-ferrocenecarboxylic Acid Methyl Ester

By Yaoguang Luo, Richard J. Barton and Beverly E. Robertson

Department of Chemistry, University of Regina, Regina, Saskatchewan, Canada S4S 0A2

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Abstract. Methyl 2-(1-hydroxyethyl)-1-ferrocenecarboxylate, [Fe($C_{14}H_{16}O_3$)], $M_r = 288{\cdot}13$, monoclinic, $P2_1/c$, $a = 8{\cdot}378\ (3)$, $b = 9{\cdot}2845\ (8)$, $c = 16{\cdot}669\ (5)$ Å, $\beta = 103{\cdot}13°$, $V = 1262{\cdot}7$ Å^3, $Z = 4$, $D_x = 1{\cdot}52$ g cm^{-3}, $\lambda(\text{Mo } K\bar{\alpha}) = 0{\cdot}71069$ Å, $\mu = 11{\cdot}79$ cm^{-1}, $F(000) = 600$, $T = 293\ (2)$ K. $R = 0{\cdot}036$ for 1913 reflections with $I/\sigma(I) \geq 2{\cdot}0$. The configurations of the molecules are (*R*,*S*) and (*S*,*R*). The hydroxyl groups are hydrogen bonded to carbonyl groups on adjacent molecules.

Introduction. Transition-metal catalysts containing chiral ferrocenyl complexes as a ligand have been found to be useful in asymmetric syntheses (Sun, 1986; Cullen & Woollins, 1981; Hayashi & Kumada, 1982). α-Substituted derivatives of *N*,*N*-dimethylaminoethylferrocene possessing either a center, a plane or both a center and a plane of chirality are effective chiral ligands in asymmetric syntheses catalyzed by rhodium, platinum, palladium and nickel complexes (Hayashi, Yamamoto & Kumada, 1974; Hayashi, Mise, Mitachi, Yamamoto & Kumada, 1976; Cullen & Woollins, 1982; Appleton, Cullen, Evans, Kim & Trotter, 1985; Hayashi, Katsumura, Konishi & Kumada, 1979; Hayashi, Tamao, Katsuro, Nakae & Kumada, 1980; Hayashi, Konishi, Fukushima, Mise, Kagotani, Tajika & Kumada, 1982; Hayashi, Yamamoto & Ito, 1988). In attempts to obtain diketone derivatives of *N*,*N*-dimethylaminoferrocene, the title compound was obtained and its crystallographic study is reported here.

Experimental. A crystal sample of the title compound was provided by W. R. Cullen of the University of British Columbia. It was obtained from the reaction of 2-(*N*,*N*-dimethylaminoethylferrocene)carboxylic acid with sodium bicarbonate and methyl iodide in dimethylacetamide followed by hydrolysis by addition of 10% aqueous sodium chloride.

An orange crystal having an irregular shape and approximate dimensions $0{\cdot}1 \times 0{\cdot}1 \times 0{\cdot}15$ mm was sealed in a quartz capillary. Cell dimensions were determined from the refinement of 34 reflections with $31 \leq 2\theta \leq 39°$. Intensity data collected with modified Picker four-circle diffractometer with graphite-monochromated Mo $K\alpha$ radiation, $\theta/2\theta$ scan tech-

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