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The Crystal Structure of Tetramethylammonium Mercury Tribromide, $N(CH_3)_4HgBr_3$

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The crystal structure of tetramethylammonium mercury tribromide was determined and refined by two-dimensional Fourier methods. Each mercury atom forms approximately equal bonds of average length 2·52 Å to three bromine atoms but these bonds are not coplanar. In both of the crystallographically independent anions the mercury atoms are distorted about 0·3 Å out of the planes of the bonded bromine atoms. In addition, there is a close approach of 2·9 Å between the mercury atom of each anion and one bromine atom of a neighboring anion in the z direction. Thus the structure is intermediate between one consisting of discrete anions and a structure with infinite anion chains running through the crystal, analogous to the metasilicates.

The ferroelectric reversal which occurs at room temperature in these crystals may be explained by a relative movement of each mercury atom to a corresponding position on the opposite side of its three bonded bromine atoms.

Introduction

A new ferroelectric crystal, tetramethylammonium mercury trichloride, $N(CH_3)_4HgCl_3$, was recently described (Fatuzzo & Nitsche, 1960), and several isomorphous compounds, also ferroelectric, were later prepared by substitution of other halogens for chlorine and phosphorus for nitrogen. The spontaneous polarization of these crystals was in the range 1–3μ coulomb.cm^{-2} at room temperature and the coercive field was very high. No Curie point was observed, as on raising the temperature the crystals decomposed while still in the ferroelectric state (Fatuzzo, Nitsche, Roetschi & Zingg, 1962).

The crystal structure analysis described in the present paper was carried out as an essential first step in understanding the ferroelectric mechanism in these compounds. Since it was believed that the exact disposition of the halogen atoms with respect to the mercury atoms might be of key importance, the compound tetramethylammonium mercury tribromide was selected. Here, the ratio of the average scattering contribution of the mercury atom to that of the rest of the molecule is almost ideal for finding the positions of the bromine atoms by the heavy atom method. While the very high scattering power of the mercury atoms would necessarily make the location of the cation atoms difficult and imprecise, no derivatives were known where a lighter atom was substituted for mercury in the same structure.

Crystal data

Tetramethylammonium mercury tribromide,
$N(CH_3)_4HgBr_3$; formula weight 514·5.
Unit cell dimensions:

$$a = 9{\cdot}05 \pm 0{\cdot}02,\ b = 15{\cdot}90 \pm 0{\cdot}05,$$
$$c = 7{\cdot}94 \pm 0{\cdot}02\ \text{Å};\ \beta = 93{\cdot}6 \pm 0{\cdot}2^\circ;$$

volume of unit cell, 1140 Å^3.
d_c 3·00 g.cm^{-3}; d_{meas} 2·96 g.cm^{-3}.
Monoclinic; absent spectra $0k0$ when k is odd; space group $P2_1(C_2^2)$.
Four molecules per unit cell; two molecules per crystal asymmetric unit.
Absorption coefficient for Cu $K\alpha$ radiation, $\mu = 392$ cm^{-1}.

Experimental

The crystals used in the X-ray investigation were prepared in Laboratories RCA Ltd., Zürich (Fatuzzo, Nitsche, Roetschi & Zingg, 1961). These were in the form of needles elongated along the c axis. They cleaved most easily on (100) and poorly on (001). All the crystals examined showed multiple twinning such that the a and b axes of adjacent twins were parallel but the c axes were at an angle of 2β. In a preliminary examination of the first member of the family prepared, $N(CH_3)_4HgCl_3$, oscillation photographs gave the appearance of orthorhombic symmetry,* but Weissenberg photographs of the $h0l$ zone showed clearly the true nature of the twinning involved.

X-ray intensity data for the three main zones were recorded with Ni filtered Cu $K\alpha$ radiation on Weissenberg photographs using a multiple film technique. Intensities were estimated visually by comparison with a standard scale. Four crystals were used. For the $hk0$ zone a crystal was cut to a cross-

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* Tetramethyl ammonium mercury trichloride is monoclinic with $a = 8{\cdot}68 \pm 0{\cdot}04$ Å, $b = 15{\cdot}75 \pm 0{\cdot}08$ Å, $c = 7{\cdot}69 \pm 0{\cdot}04$ Å, $\beta = 93{\cdot}0 \pm 0{\cdot}2^\circ$, space group $P2_1$.

section of $0{\cdot}08 \times 0{\cdot}12$ mm and accurate absorption corrections were made for all the reflections (Evans, 1952). Two approximately cylindrical crystals, radii 0·055 and 0·096 mm respectively were obtained for the $0kl$ data. Both intensity sets were estimated, cylindrical absorption corrections were applied, and a mean weighted 2:1 in favor of the smaller specimen was taken. The crystal used for the $h0l$ zone was again roughly cylindrical and had a cross-sectional radius of 0·078 mm. The $h0l$ photographs recorded reflections from two sets of twins oriented as described above, and the intensity estimates in the small regions where the two sets of reflections overlapped were necessarily of lower accuracy than elsewhere. While this $h0l$ data was used only in a very limited way as described below it is clear that it would not be worthwhile to collect full three-dimensional data from such twinned crystals. As the analysis proceeded the observed and calculated structure factor comparison could be used to check for the presence of extinction. Only the 110 reflection appeared to be at all seriously affected and this discrepancy was omitted from the later stages of the difference density calculations.

Structure determination

Patterson projections of the $hk0$ and $0kl$ zones, with F^2s sharpened to atoms at rest, showed clearly the positions of the two crystallographically independent mercury atoms in the unit cell. Electron density maps of the two zones, using the phase angles calculated from the mercury atom positions, showed five of the six bromine atoms. From this stage on both ϱ_o and $\varrho_o - \varrho_c$ maps were computed for each refinement. For $\varrho 2$ $hk0$ the positions of the five strongest peaks other than mercury were added into the phasing calculations and the difference electron density map showed decisively that one of these peaks was an unresolved double peak of two nearly overlapping atoms (Br3 and Br6). The analysis of the $0kl$ zone was not quite so straightforward because of the occurrence of false mirrors of certain bromine atoms. However, from $\varrho 3$ $0kl$ the correct positions of all the bromine atoms were established.

The two zones investigated were not by themselves sufficient to give a unique solution since the same coordinates referred to an acute or to an obtuse β angle would give identical projections. Accordingly a Patterson projection of the $h0l$ zone was prepared and this established very clearly which of the two alternatives was correct.

Refinement was carried out up to $\varrho 5$ $hk0$ and $\varrho 6$ $0kl$ which are shown in Figs. 1 and 2. At each stage coordinate changes of $1{\cdot}96 \times$ the change in peak position between ϱ_c and ϱ_o were applied for the $hk0$

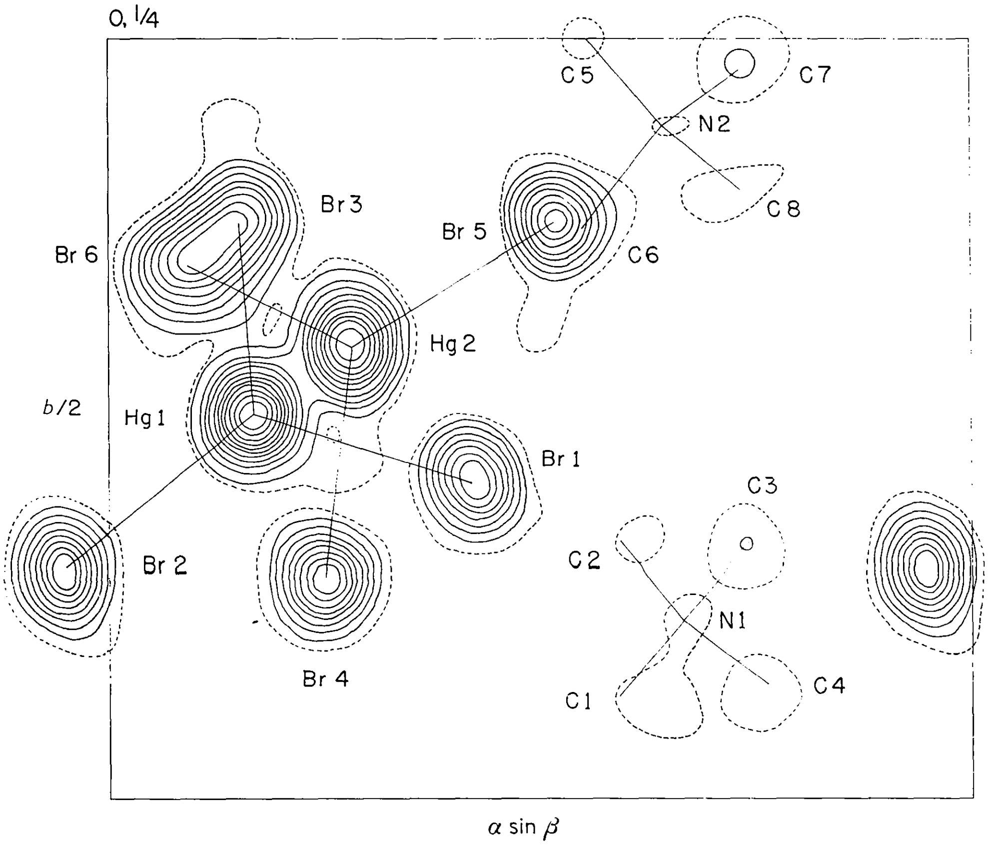


Fig. 1. The electron density $\varrho 5$ projected down the c axis. Contours are drawn at intervals of 5 e.Å^{-2} for the bromine and lighter atoms starting at the 5 el. line which is dotted. For the mercury atoms the contours are drawn at 5, 10, 20, 30, *etc.* e.Å^{-2}. The molecular skeletons indicate the final atomic positions.

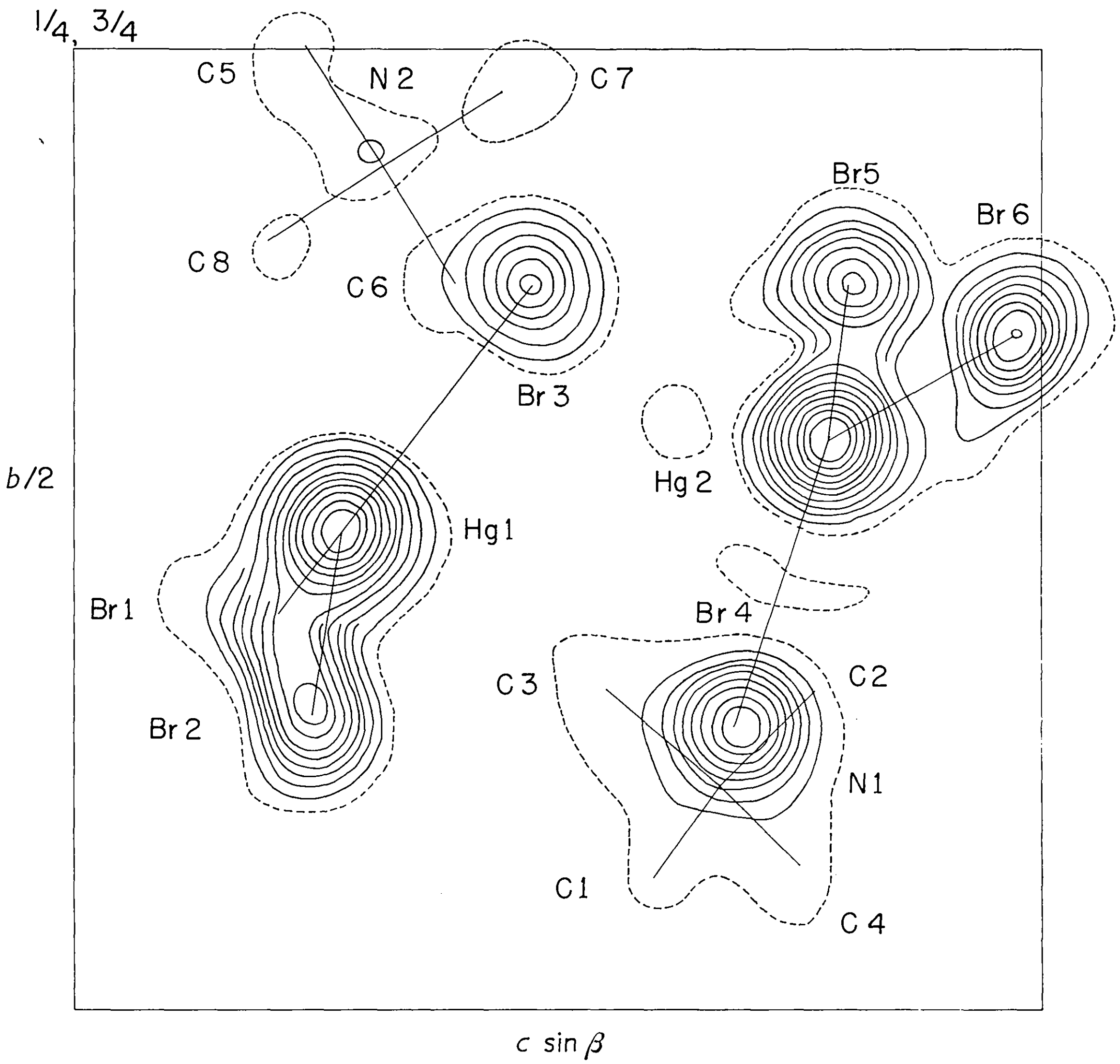


Fig. 2. The electron density $\varrho 6$ projected down the a axis. Contours are as for Fig. 1.

zone and 1·93 shifts were used for the $0kl$ zone. In the difference density maps of the later refinements the regions of largest residual density could be correlated with approximately tetrahedral tetramethyl groups and for $\varrho 5$ $hk0$ and $\varrho 6$ $0kl$ these atomic positions were added into the phasing calculations.

The final coordinates for all atoms are listed in Table 1 together with the estimated standard deviations (Cruickshank, 1949). The final observed and calculated values of the structure factors are collected in Table 2. The scattering factors used are those given in *Internationale Tabellen zur Bestimmung von Kristallstrukturen* (1935), corrected for anomalous dispersion (Dauben & Templeton, 1955). An isotropic temperature factor with $B=4·7$ was found to give the best agreement with the observed data, and this high value is probably indicative of some disorder in the structure. While the final difference density maps could be interpreted in terms of anisotropic vibrations,

Table 1. *Coordinates of the atoms with estimated standard deviations for the positions of the mercury and bromine atoms*

	x	y	z	e.s.d.
Hg_1	0·165	0·000	0·025	0·009 Å
Br_1	0·421	−0·045	−0·023	0·022
Br_2	−0·052	−0·101	−0·007	
Br_3	0·148	0·127	0·223	
Hg_2	0·278	0·046	0·528	0·009
Br_4	0·250	−0·106	0·430	0·022
Br_5	0·514	0·126	0·550	
Br_6	0·089	0·100	0·720	
N_1	0·67	−0·13	0·42	
C_1	0·57	−0·18	0·35	
C_2	0·59	−0·08	0·51	
C_3	0·74	−0·08	0·30	
C_4	0·77	−0·17	0·50	
N_2	0·64	0·19	0·07	
C_5	0·56	0·25	−0·01	
C_6	0·56	0·13	0·14	
C_7	0·73	0·23	0·19	
C_8	0·73	0·15	−0·05	

Table 2. *Calculated and observed values of the structure factors for one asymmetric unit*

hkl	F_c	F_o
001	1	<9
002	180	180
003	26	26
004	135	132
005	32	27
006	25	35
007	22	20
008	5	<12
020	185	181
040	33	31
060	8	12
080	73	62
0,10,0	39	37
0,12,0	29	29
0,14,0	29	29
0,16,0	13	10
100	19	32
200	64	71
300	63	63
400	23	29
500	30	41
600	11	<11
700	38	39
800	2	<13
900	35	44
10,0,0	11	10
011	17	14
021	69	68
031	37	45
041	124	132
051	20	16
061	80	79
071	29	25
081	62	66
091	23	23
0,10,1	55	60
0,11,1	6	<14
0,12,1	49	52
0,13,1	13	18
0,14,1	31	35
0,15 1	11	<12
0,16,1	17	18
012	72	71
022	143	143
032	70	86
042	72	76
052	15	27
062	68	75
072	24	22
082	55	48
092	14	16
0,10,2	4	17
0,11,2	19	20
0,12,2	26	26
0,13,2	4	<13
0,14,2	14	16
0,15,2	14	<12
0,16,2	8	<12
013	36	39
023	71	65
033	36	30
043	66	54
053	16	<14
063	39	25
073	10	15
083	43	32
093	45	45
0,10,3	56	49
0,11,3	48	49
0,12,3	41	39
0,13,3	21	<13
0,14,3	22	19
0,15,3	12	<12
014	29	34
024	87	77
034	58	55
044	21	17
054	53	47
064	4	<14
074	24	30
084	23	26
094	17	<14
0,10,4	15	19
0,11,4	7	<14
0,12,4	6	<13
0,13,4	10	<12
0,14,4	11	<12
015	24	30
025	35	29
035	32	31
045	41	32
055	22	18
065	21	18
075	28	22
085	34	33
095	32	28
0,10,5	21	16
0,11,5	27	21
0,12,5	10	14
016	54	53
026	30	28
036	48	44
046	31	25
056	18	23
066	14	23
076	17	<13
086	8	18
096	17	<13
0,10,6	2	<12
0,11,6	10	<12
0,12,6	6	<11
017	13	16
027	19	18
037	13	16
047	12	16
057	17	19
067	7	19
077	16	19
087	13	18
097	21	17
0,10,7	17	15
0,11,7	22	15
0,12,7	6	11
018	16	<12
028	8	<12
038	20	18
048	8	<11
058	21	17
068	2	<10
078	18	14
110	182	152
120	32	32
130	113	120
140	13	21
150	41	32
160	22	12
170	40	50
180	41	41
190	26	32
1,10,0	59	52
1,11,0	8	<11
1,12,0	34	30
1,13,0	18	20
1,14,0	6	<11
1,15,0	12	11
1,16,0	2	<11
210	74	71
220	67	59
230	50	64
240	84	76
250	27	19
260	91	84
270	78	77
280	43	41
290	67	66
2,10,0	12	<11
2,11,0	41	41
2,12,0	15	16
2,13,0	24	20
2,14,0	9	<11
2,15,0	22	22
2,16,0	13	<11
310	46	51
320	55	65
330	10	11
340	19	29
350	69	66
360	90	84
370	92	88
380	79	68
390	39	40
3,10,0	40	35
3,11,0	21	19
3,12,0	18	15
3,13,0	32	29
3,14,0	24	23
3,15,0	14	<11
3,16,0	22	19
410	46	52
420	8	12
430	72	74
440	33	39
450	53	51
460	61	62
470	38	36
480	48	42
490	36	29
4,10,0	26	19
4,11,0	28	26
4,12,0	33	25
4,13,0	16	20
4,14,0	21	17
4,15,0	15	13
4,16,0	7	<9
510	19	22
520	54	60
530	5	13
540	55	55
550	35	36
560	32	32
570	51	51
580	15	21
590	45	45
5,10,0	18	21
5,11,0	26	26
5,12,0	20	15
5,13,0	23	21
5,14,0	15	10
5,15,0	17	12
5,16,0	10	<9
610	43	46
620	23	29
630	20	20
640	24	24
650	21	24
660	53	55
670	27	29
680	40	36
690	13	18
6,10,0	19	15
6,11,0	8	<12
6,12,0	14	14
6,13,0	14	10
6,14,0	24	24
6,15,0	9	<9
6,16,0	16	14
710	10	16
720	38	45
730	22	24
740	35	44
750	39	37
760	16	20
770	20	20
780	11	<13
790	5	<12
7,11,0	11	11
7,12,0	9	<10
7,13,0	18	20
810	40	50
820	13	16
830	30	34
840	15	18
850	21	22
860	5	<13
870	16	12
880	6	<12
890	3	<11
8,10,0	6	<10
8,11,0	12	<10
8,12,0	8	<9
910	6	<13
920	26	33
930	3	<12
940	12	12
950	5	<12
960	12	11
970	10	10
980	15	13
990	6	<9
9,10,0	7	12
9,11,0	3	<7
9,12,0	6	8
10,1,0	13	10
10,2,0	9	10
10,3,0	16	13
10,4,0	6	<10
10,5,0	16	14
10,6,0	5	<8
10,7,0	8	10

for some of the heavier atoms, particularly in the y direction, these effects may be due to residual absorption errors and the experimental data does not seem to justify anisotropic refinement. For 218 observed reflections the reliability coefficient R is 0·120.

Description of the structure

The structure may be visualized from Figs. 1 and 2 together with Fig. 3 which shows the atomic positions as projected down the crystal b axis. The dimensions of the two HgBr$_3$ groups are illustrated in Fig. 5, and the mercury–bromine bond distances and bond angles are listed in Table 3 with the corresponding estimated standard deviations (Cruickshank, 1949; Jeffrey & Parry, 1952). These groups are considerably closer to a planar configuration than to a pyramidal one in which there are bonds from an atom at the centroid to three atoms at apices of a regular tetrahedron. Referred to orthogonal axes X, Y, Z where $X=x+z\cos\beta$, $Y=y$, $Z=z\sin\beta$ the equations of the planes of the bromine atoms in anions A and B respectively are

$$X-3{\cdot}881Y+6{\cdot}772Z-5{\cdot}355=0$$

and

$$X-1{\cdot}275Y+2{\cdot}519Z-12{\cdot}788=0\,.$$

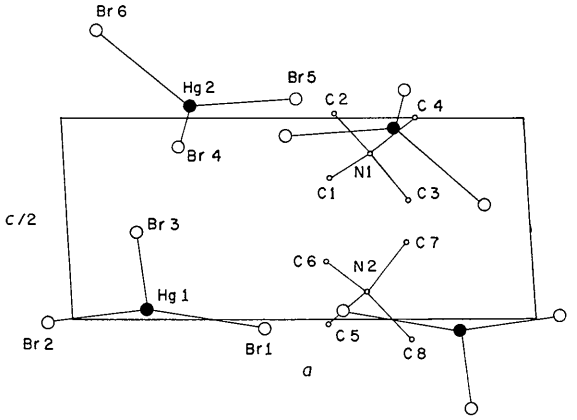


Fig. 3. The final atomic positions as viewed in projection on (010).

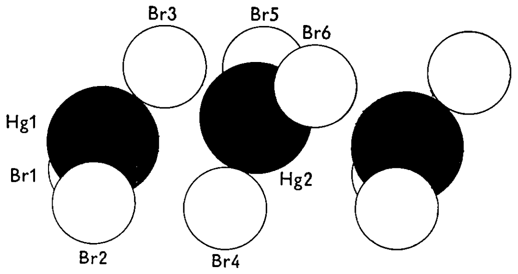


Fig. 4. A view of the anion chain which runs through the crystal in the direction of the c axis.

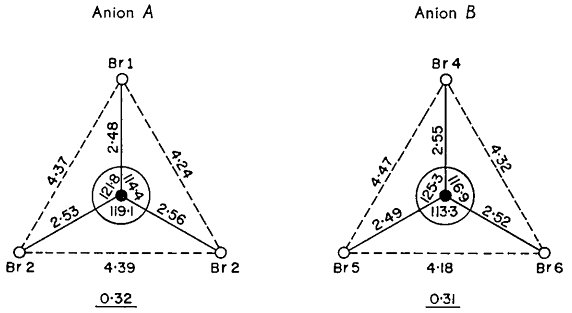


Fig. 5. Dimensions of the two crystallographically independent anions. Distances are in Å and angles in degrees. The underlined figures at the bottom are the distance in Å of the mercury atoms from the planes of the bromine atoms.

Table 3. *Bond distances and bond angles for the anions and estimated standard deviations*

Hg–Br distances

Atoms	Intra-anion distance	e.s.d.
Hg1–Br1	2·48 Å	0·025 Å
Hg1–Br2	2·53	
Hg1–Br3	2·56	
Hg2–Br4	2·55	
Hg2–Br5	2·49	
Hg2–Br6	2·52	

Atoms	Inter-anion distance	e.s.d.
Hg1–Br6	2·94 Å	0·025 Å
Hg2–Br3	2·92	

Br–Hg–Br bond angles

Atoms	Angle	e.s.d.
Br1–Hg1–Br2	121·8°	0·8°
Br2–Hg1–Br3	119·1	
Br1–Hg1–Br3	114·4	
Br4–Hg2–Br5	125·3	
Br5–Hg2–Br6	113·3	
Br4–Hg2–Br6	116·9	

The deviations of the mercury atoms from these planes are 0·32 Å for anion A and 0·31 Å for anion B.

The most interesting feature of the structure is the arrangement of these approximately planar anions. They lie roughly perpendicular to the c axis and in this direction they are not interleaved by cations. Instead there appears to be an interaction between successive anions in the c direction. Atoms Hg2 and Br3 are only 2·92 Å apart and the distance Hg1–Br6 is 2·94 Å. These distances are appreciably less than the expected van der Waals approaches and they must be regarded as the result of polar attractions between the mercury atom of one anion and one bromine atom of the next. Thus this part of the structure may be regarded as being intermediate between a set of discrete anions HgBr$_3^-$, and a structure consisting of infinite chains (HgBr$_3$)$_n^{n-}$ running through the crystal parallel to the c axis and analogous to the metasilicate chain, (SiO$_3$)$_n^{2n-}$. A view of this anion chain, which is reproduced twice in each unit cell by the operation of the screw axis, is shown in Fig. 4. The difficulty of cleaving the crystals across the c axis can readily be understood on this model.

The atomic coordinates for the carbon and nitrogen atoms, derived in a structure containing so many very much heavier atoms, have necessarily quite low accuracy. The tetramethylammonium groups appear to be at least approximately tetrahedral. The eight carbon–nitrogen bond distances computed from the coordinates of Table 1 average 1·35 Å with individual variations of up to $\pm 0{\cdot}1$ Å. This mean value is considerably lower than the expected C–N single bond distance but the discrepancy is probably an artifact of low resolution. The average fractional contribution of the cations to the intensities falls off very rapidly with increasing $\sin\theta/\lambda$ and a systematic error may be produced by a tendency of the separate peaks to cohere. The cations link the separate anion chains together. All the carbon–bromine approaches of less than 4 Å are listed in Table 4. Bromine atoms with coordinates related by the screw axis to those of Table 1 are dashed and atoms which are translated along a, b or c from the standard molecule are so indicated.

Table 4. *Cation-anion approaches less than* 4 Å

Atoms	Distance
C_1–Br_1	3·88 Å
C_1–Br_4	3·28
C_1–$Br_5'(+a-b+c)$	3·25
C_2–Br_4	3·14
C_2–Br_5	3·43
C_3–Br_1	3·80
C_3–$Br_2(+a)$	3·18
C_5–$Br_1'(+a)$	3·27
C_6–Br_1	3·25
C_6–Br_3	3·78
C_6–Br_5	3·28
C_7–Br_5	3·91
C_8–$Br_5(-c)$	3·65

Discussion

(i) *Chemical considerations*

The configuration found for the $HgBr_3$ groups in $N(CH_3)_4HgBr_3$ appears to be unique among mercury compounds. Divalent mercury compounds tend to form either two colinear bonds, presumably *sp*, or four tetrahedral bonds by sp^3 hybridization. Thus in $HgBr_2$ (Verweel & Bijvoet, 1931) each mercury atom forms two colinear bonds to bromine atoms at 2·48 Å, while 4 bromine atoms at 3·23 Å complete a distorted octahedron. In crystals of NH_4HgCl_3 (Harmsen, 1938) two chlorine atoms are again covalently bonded to mercury at 180° while four chlorine atoms are the next neighbors at larger distances. The compound $[N(CH_3)_4]_2HgBr_4$, which exists in solution equilibrium with the tribromide, probably has tetrahedral coordination of the bromine atoms about the mercury atoms as found for the coordination of zinc by chlorine in $[N(CH_3)_4]_2ZnCl_4$ (Morosin & Lingafelter, 1959).

The coordination of mercury found in the present investigation is very close to trigonal. Most probably these bonds are formed by sp^2 hybridization, which would require exactly planar anion groups, and the distortions found are due to the highly polar environment. The reduction in the potential energy achieved by the interaction between adjacent anions must override the tendency of the groups themselves to be planar. It is noteworthy that while the largest deviation of the mercury–bromine bond distances within anions from the mean value of 2·52 Å is only two standard deviations and cannot be considered certainly real, there are deviations in Br–Hg–Br bond angles from the mean value of 118·5° of eight times the standard deviations. In a low symmetry crystal it would be surprising if the mercury atoms were distorted out of the planes of the bromine atoms in a symmetrical fashion and it seems that the extreme variations in the bond angles are real.

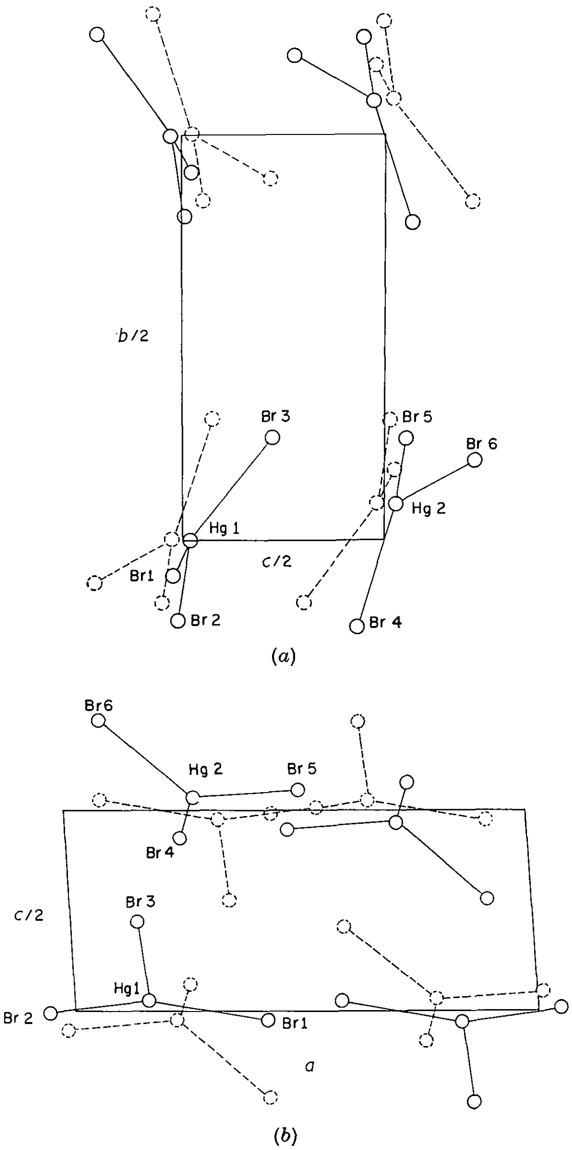
(ii) *Ferroelectricity*

Before the present work was begun it was speculated that the ferroelectric reversal might occur by a movement of each mercury atom through the plane of its three bonded bromine atoms to a corresponding position on the other side of the plane (Nitsche, 1959). This hypothesis would require that the mercury atoms are displaced out of the bromine planes by an amount sufficient for an appreciable resultant dipole to be created, but not so far that their required movement on reversal of the electric field would cause breakdown in the crystal. As a result of the crystal structure analysis it appears that both conditions are fulfilled.

The deviation of the mercury atom from the plane of the three bromine atoms is in the same sense for the two crystallographically independent anions in the unit cell, and hence there is a resultant dipole component which is additive along the b axis for the four anions in the unit cell, but which cancels in all other directions because of the symmetry of the screw axis. From the atomic coordinates given above the dipole component along b may be calculated to be $0{\cdot}136\mu$ for anion A and $0{\cdot}0915\mu$ for anion B (where μ is the dipole moment of the Hg–Br bond and the moments of the six independent bonds are assumed to be equal). Thus the resultant dipole component along b is $0{\cdot}455\mu$ for each unit cell.

It would be surprising if the cations did not suffer some distortion in the asymmetric field of the anion part of the structure. Unfortunately the present analysis gives no concrete information on this point because of the relative lightness of these groups. However, it seems reasonable that induced dipoles in the tetramethylammonium groups could contribute a significant fraction of the total polarization.

A detailed examination of the atomic movements required in the polar reversal of the anion part of structure leads to the conclusion that this is most easily accomplished by maintaining the β angle for a given unit cell constant in both states. Change of the β angle from obtuse to acute would require progressively larger atomic movements through different unit cells, almost certainly incompatible with fast switching. The reversal mechanism which seems to minimize the atomic movements is illustrated in Fig. 6, which shows the positions of the anions in two projections under both polarities of the applied field.



(a)

(b)

Fig. 6. An illustration of the reversal of the anions under an external field viewed in (a) down the crystal *a* axis and in (b) down the crystal *b* axis. The full lines connect bonded atoms in the positions for one polarity of the applied field and the dotted lines the positions for the other polarity.

Anion *A* moves to the configuration of the mirror image in (010) of anion *B* related by the screw axis to the standard ion, and anion *B* takes up the reversed configuration of the corresponding anion *A*. Thus the reversed structure is an exact mirror image of the original structure and the resultant dipole component along the *b* axis is reversed. The origin referred to the coordinates of Table 1 has been translated by $a/2$, $c/2$ and approximately $-0{\cdot}454b$. (Since there is no unique origin in the *y* direction, no translational motions are required to accommodate to symmetry elements. The mercury atoms, however, might move slightly in this direction to minimize movement of the bromine atoms.) The movements of the atoms required for reversal under this mechanism are quite large, about 0·7 Å for the mercury atoms alone, and the high coercive field observed is understandable.

One interesting feature is that the close inter-ion approaches between Hg1 and Br6 and between Hg2 and Br3 are destroyed on reversal, but identical close approaches are set up between the new positions of Hg1 and Br4, and Hg2 and Br1 respectively. It may well be that these interactions are dominant in creating the two energy minima for the two polarities of the structure.

Anomalous dispersion X-ray experiments under an applied field could correlate the absolute polarity of the Hg–Br bonds with the direction of the external field. Such experiments have not yet been carried out because the crystals obtained so far are rather unsuitable for precision measurements close to an absorption edge.

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