surrounded by nine O atoms [three O(1), three O(2) and three O(3)] at a mean distance of 2·741 Å, whereas the Pb(2) atom [6(h) position] is surrounded by six O atoms [O(1), O(2) and four O(3)] at a mean distance of 2·570 Å.

The shape of the PO_4 tetrahedron is almost regular. The mean P—O distance of 1·588 Å is rather long compared with those of other apatites, for example 1·527 Å in $Cd_5OH(PO_4)_3$ (Hata et al., 1978) and 1·540 Å in $Ba_5Cl(PO_4)_3$ (Hata, Aoki, Marumo & Iwai, 1979). The O(1)—P—O(2) and O(1)—P—O(3) bond angles are larger than the O(2)—P—O(3) and O(3)—P—O(3)^{iii} angles, as observed in chlorapatites and hydroxyapatites.

The edge length of the triangle formed by Pb(2) is 4·303 Å, whereas those in $Ba_5Cl(PO_4)_3$ (Hata et al., 1979) and $Sr_5OH(PO_4)_3$ (Sudarsanan & Young, 1972) are 4·508 and 4·284 Å respectively. The size of the triangle seems normal for an apatite structure, though anions at the $(0,0,z)$ sites are completely missing in the present crystal.

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Barium Diiodate

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Abstract. $Ba(IO_3)_2$, monoclinic, $C2/c$, $a = 13·638$ (9), $b = 7·979$ (2), $c = 9·036$ (6) Å, $\beta = 133·62$ (4)°, $V = 711·8$ (6) Å³, $Z = 4$, $D_x = 4·71$, $D_m = 4·73$ Mg m⁻³ (pycnometrically under xylene at 296 K), μ(Mo $K\alpha$) = 14·5 mm⁻¹. $R = 0·055$ for 1036 reflections. The structure consists of pyramidal IO_3^- anions and Ba^{2+} cations. The environment of each I atom is completed by two O atoms from two other IO_3^- ions, so the resulting polyhedron can be described as a distorted trigonal bipyramid. Ba^{2+} cations have ten O neighbours.

Introduction. Single crystals of $Ba(IO_3)_2$ were prepared by hydrothermal synthesis from an oversaturated aqueous solution of $Ba(IO_3)_2.2H_2O$ at 473 K. Systematic absences of the reflections hkl for $h + k = 2n + 1$ and $h0l$ for $l = 2n + 1$ correspond to space group $C2/c$ or Cc. The former was chosen on the basis of the successful refinement of the structure. The cell dimensions were refined from 28 reflections centred on the diffractometer. A crystal $0·07 \times 0·08 \times 0·5$ mm was chosen for intensity measurements on a Hilger & Watts four-circle diffractometer. Mo $K\alpha$ radiation was used. 1036 independent reflections were measured. Only 999 of them were considered as observed with $I \geq$

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Table 1. *The final positional parameters* ($\times 10^3$)

	x	y	z
Ba	0	358·6 (1)	250
I	188·09 (8)	33·9 (1)	178·8 (1)
O(1)	186 (1)	89 (1)	371 (2)
O(2)	357 (1)	−49 (1)	346 (2)
O(3)	87 (1)	−157 (1)	78 (2)

$1.96\sigma(I)$, where $\sigma(I)$ was derived from counter statistics. Data were corrected for Lorentz and polarization factors and for absorption.

The structure was solved by Patterson and Fourier techniques. The parameters were refined by full-matrix least squares with a local version of *ORFLS* (Busing, Martin & Levy, 1962). $\sum w(|F_o| - |F_c|)^2$ was minimized, where $w = 1/\sigma^2(F_o)$. In the final cycle no parameter shift was >0·02 times its e.s.d. Unobserved reflections were not used in the refinement. The final R = 0·055 (R_w = 0·075) for all reflections and 0·054 (R_w = 0·075) for the observed reflections.* The maximum peak in the final difference map was 1·9 e Å^{-3} located 1·11 Å from the Ba atom. The tabulated values (*International Tables for X-ray Crystallography*, 1974) of the scattering factors for Ba, I and O and real and imaginary dispersion corrections for Ba and I were used in the calculations. The final positional parameters are given in Table 1.

Discussion. The polyhedron of O atoms around the I atom in divalent metal iodates can be characterized in some detail from a number of known crystal structures of these compounds. The structures show that the trigonal pyramidal ion IO$_3^-$ (with I—O distances from 1·786 to 1·874 Å) is completed by two, three or four further O atoms (I—O from 2·577 to 3·209 Å) belonging to adjacent iodate anions with formation of the following geometrical arrangements of the iodate polyhedron: most frequently, a distorted octahedral arrangement is formed with α-Cu(IO$_3$)$_2$ (Liminga, Abrahams & Bernstein, 1975), 3Cu(IO$_3$)$_2$.2H$_2$O (Ghose & Wan, 1974), CuIO$_3$(OH) (Ghose, 1962), Ca(IO$_3$)$_2$ (Ghose, Wan & Wittke, 1978), Ca(IO$_3$)$_2$.6H$_2$O (Braibanti, Manotti Lanfredi, Pellinghelli & Tiripicchio, 1971), Cd(IO$_3$)$_2$ (Bach & Küppers, 1978), Ni(IO$_3$)$_2$.2H$_2$O (Abrahams, Bernstein, Elemans & Verschoor, 1973), Ni(IO$_3$)$_2$.-4H$_2$O (Elemans & Verschoor, 1973) and α-Co-(IO$_3$)$_2$.2H$_2$O (Elemans & Verschoor, 1973); a distorted trigonal bipyramid is formed with γ-Cu(IO$_3$)$_2$ (Liminga, Booles, Ghose & Wan, 1978) and 3Cu(IO$_3$)$_2$.2H$_2$O; a distorted pentagonal bipyramidal
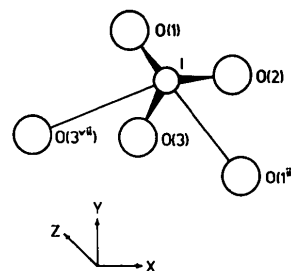
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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35305 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
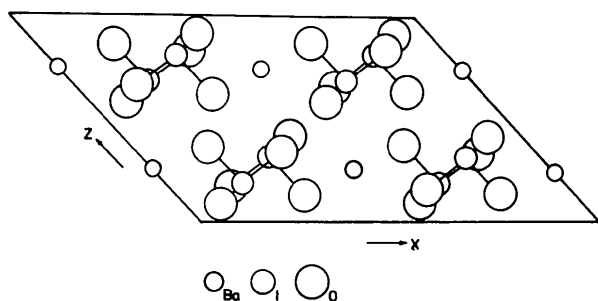
arrangement with Sr(IO$_3$)$_2$.H$_2$O (Manotti Lanfredi, Pellinghelli, Tiripicchio & Tiripicchio Camellini, 1972), γ-Cu(IO$_3$)$_2$, Ca(IO$_3$)$_2$ or irregular polyhedron with 3Cu(IO$_3$)$_2$.2H$_2$O.

The interatomic distances and angles in the structure of Ba(IO$_3$)$_2$ are summarized in Table 2. Every I atom is surrounded by five O atoms in a distorted trigonal bipyramidal arrangement (Fig. 1). The next nearest O atoms are at 3·36 (1) [I—O(2^{vi})] and 3·32 (1) Å [I—O(3^{vi})]. Compared with data for other divalent metal iodates, these distances are certainly too long for O atoms to be assumed as completing the iodine polyhedron to form some of the configurations mentioned above. A comparison of the I—O distances in Ba(IO$_3$)$_2$ with other structures where a trigonal bipyramid is formed, γ-Cu(IO$_3$)$_2$ (I—O 1·786, 1·833,

Table 2. *Interatomic distances* (Å) *and angles* (°)

Symmetry code

(i) $\bar{x}, y, \frac{1}{2} - z$	(v) $x - \frac{1}{2}, y + \frac{1}{2}, z$
(ii) $x, \bar{y}, z - \frac{1}{2}$	(vi) $\frac{1}{2} - x, y + \frac{1}{2}, \frac{1}{2} - z$
(iii) $\frac{1}{2} - x, \frac{1}{2} - y, 1 - z$	(vii) $\bar{x}, \bar{y}, \bar{z}$
(iv) $x - \frac{1}{2}, \frac{1}{2} - y, z - \frac{1}{2}$	(viii) $x, \bar{y}, z + \frac{1}{2}$

Pyramidal anion IO$_3^-$

I—O(1)	1·81 (2)	O(1)—O(2)	2·73 (2)
I—O(2)	1·80 (2)	O(1)—O(3)	2·78 (2)
I—O(3)	1·82 (1)	O(2)—O(3)	2·80 (2)
O(1)—I—O(2)	98·2 (7)	O(2)—O(1)—O(3)	61·2 (6)
O(1)—I—O(3)	100·1 (7)	O(1)—O(2)—O(3)	60·4 (6)
O(2)—I—O(3)	101·6 (6)	O(1)—O(3)—O(2)	58·4 (7)

Distorted trigonal bipyramid

I—O(1^{ii})	2·93 (2)	O(1)—O(1^{ii})	4·74 (2)
I—O(3^{vii})	2·90 (2)	O(1)—O(3^{vii})	3·13 (2)
		O(2)—O(1^{ii})	3·19 (2)
		O(2)—O(3^{vii})	4·69 (2)
		O(3)—O(1^{ii})	3·02 (2)
		O(3)—O(3^{vii})	3·05 (2)

Barium environment

Ba—O(1) Ba—O(1^i)	2·90 (1)	Ba—O(2^{iii}) Ba—O(2^{iv})	3·10 (1)
Ba—O(1^{iii}) Ba—O(1^{iv})	3·16 (2)	Ba—O(3^{vii}) Ba—O(3^{viii})	2·82 (1)
Ba—O(2^v) Ba—O(2^{vi})	2·73 (2)		

Fig. 1. View of the iodate polyhedron.

Fig. 2. Projection of the structure of $Ba(IO_3)_2$ on to the xz plane.

1·833, 2·600, 2·600 Å) and $3Cu(IO_3)_2.2H_2O$ (I—O 1·795, 1·801, 1·823, 2·771, 2·873 Å), shows there are no significant differences except a small expansion of the bonds in $Ba(IO_3)_2$.

Among the known structures of divalent metal iodates, $Ba(IO_3)_2$ contains the largest cation $(r_{Ba^{2+}} = 1.66$ Å, Shannon, 1976). As a consequence, the coordination number of Ba^{2+} is ten, with an irregular arrangement of the O neighbours. The projection of the structure on to the xz plane is in Fig. 2.

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Mercuric Chloride, a Redetermination

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Abstract. $HgCl_2$, orthorhombic, Pnma, at 298 K a = 12·765 (6), b = 5·972 (3), c = 4·330 (2) Å, D_o = 5·44 (1), D_c = 5·464 Mg m^{-3}, Z = 4; at 303 K a = 12·776 (4), b = 5·986 (3), c = 4·333 (2) Å. Using all data, the Cl—Hg—Cl angle is 178·6 (4)° and the two Hg—Cl distances [2·292 (10), 2·274 (10) Å] average to 2·283 (9) Å. A refinement using only those data for which $(\sin\theta)/\lambda > 0.5$ Å^{-1} led to Cl—Hg—Cl = 178·9 (5)° and Hg—Cl (mean) = 2·291 (9) Å. The shortest non-bonded interactions are Hg—Cl = 3·37 and Cl—Cl = 3·33 Å. (The final R_1 value is 0·064.)

Introduction. Initially, crystals were grown by slowly evaporating a saturated solution of $HgCl_2$ in 95% ethanol. A single crystal of extreme dimensions 0·44 × 0·29 × 0·21 mm was selected and studied as described below. Full-matrix anisotropic least-squares refinement (Busing, Martin & Levy, 1962) converged at R_1 = $\sum (|F_o - |F_c||)/\sum F_o$ = 0·23 and R_2 = $[\sum w(F_o - |F_c|)^2/\sum wF_o^2]^{1/2}$ = 0·25. The Cl—Hg—Cl bond angle

and lengths were found to be 178·7 (1·1)°, 2·28 (3) and 2·31 (3) Å. However, the transmission coefficients in the absorption correction were very small and unreliable, ranging from 0·008 to 0·113, approximately. Furthermore, the slightly hygroscopic crystal decayed steadily by about 20% and lost its clarity during the data-collection process. To minimize these problems, the work was repeated as follows.

Very small single crystals of $HgCl_2$ were prepared by cooling a warm saturated toluene solution of $HgCl_2$. Crystals formed as needles elongated along a with lateral (001) and (00$\bar{1}$) faces, as before. A single crystal of extreme dimensions 0·20 × 0·07 × 0·05 mm was selected under paraffin oil and sealed in a fine glass capillary. X-ray diffraction data collected with this crystal were corrected for absorption and refined to give Cl—Hg—Cl = 179·5 (4)° and Hg—Cl = 2·272 (13) and 2·262 (13) Å, but the error indices remained relatively high: R_1 = 0·11 and R_2 = 0·14. It is probable that available moisture attacked the fine hygroscopic