Stereochemistry of Some Organic Derivatives of Group Vb Elements. Part VI.[1] Crystal and Molecular Structure of μ-Oxo-bis[perchloratotriphenylbismuth(V)]

By **Frank C. March** and **George Ferguson**,* Chemistry Department, The University, Guelph, Ontario, Canada, N1G 2W1

The structure of the title compound has been determined by X-ray methods. Crystals are monoclinic, space group $P2_1/c$, with $Z = 4$ in a unit cell of dimensions $a = 18{\cdot}750(3)$, $b = 10{\cdot}108(2)$, $c = 18{\cdot}927(3)$ Å, $\beta = 92{\cdot}19(1)°$. The structure was solved by the heavy-atom method and refined by block-diagonal least-squares methods to a residual R of 0·070 for 3145 data considered observed. The structure contains well separated discrete molecules. The two bismuth atoms are linked by an oxygen bridge [mean Bi–O(br) 2·065(10) Å, Bi–O–Bi 142·4(7)°]. The configuration about the bismuth atoms is distorted trigonal bipyramidal with the oxygen atoms apical and phenyl groups equatorial. The perchlorate groups are weakly co-ordinated to the metal [mean Bi–O(perchlorate) 2·65(1) Å], one being disordered. Other mean dimensions: Bi–C(Ph) 2·19(2) Å, O(br)–Bi–C(Ph) 97·1(1)°, Bi–O–Cl 132·3(8)°.

THE class of compounds $X{\cdot}MR_3{-}O{-}MR_3{\cdot}X$ (1; R = Ph or Me, M = Sb or Bi, X = halide, NO_3, or pseudohalide) are well known,[2-8] and their i.r. and conductance data have been interpreted in favour of non-ionic structures containing five-co-ordinate antimony and bismuth. A recent crystal structure analysis[2,3] has shown that for the compound $[\{Ph_3Sb(N_3)\}_2O]$ co-ordination about the metal is close to regular trigonal bipyramidal. On the other hand, tetrahedral co-ordination about the metal, with ionic perchlorate groups, had been suggested, from i.r. studies, for $[\{R_3Sb(ClO_4)\}_2O]$ [R = Ph (ref. 8) or Me (ref. 9)]. A reinvestigation of the i.r. spectra of these compounds and of $[\{Ph_3Bi(ClO_4)\}_2O]$ and their hydrates[2] has led to the postulation that the anhydrous perchlorate compounds have structures similar to those of the series of compounds (1), and an X-ray analysis[10] has established that $[\{Me_3Sb(ClO_4)\}_2O]$ also has a trigonal bipyramidal configuration at the Sb atoms. Since crystals of the anhydrous antimony compound could not be obtained in a satisfactory form, we have investigated the structure of the title bismuth compound in order to elucidate the stereochemistry at the metal atom and the nature of the perchlorate group environment in the solid.

EXPERIMENTAL

Crystal Data.—$C_{18}H_{15}Bi_2Cl_2O_9$, $M = 1095{\cdot}5$, Monoclinic, $a = 18{\cdot}750(3)$, $b = 10{\cdot}108(2)$, $c = 18{\cdot}927(3)$ Å, $\beta = 92{\cdot}19(1)°$, $U = 3585$ Å^3, $D_m = 2{\cdot}01$, $Z = 4$, $D_c = 2{\cdot}03$, $F(000) = 2072$. Space group $P2_1/c$ (C^5_{2h}, No. 14) from systematic absences: $0k0$ for k odd, $h0l$ for l odd. Cu-K_α radiation, $\lambda = 1{\cdot}5418$ Å; μ(Cu-K_α) = 206·6 cm^{-1}.

The crystals used were obtained by recrystallisation from dry acetone–light petroleum. Two crystals were used for the data collection, because the compound decomposed in the X-ray beam.

Cell dimensions were obtained from a least-squares refinement of the setting angles of 12 reflexions measured on a Hilger and Watts Y 290 diffractometer. Intensity data were collected to $\theta_{max.}$ 57° by the θ—2θ scan technique with stationary-counter–stationary-crystal background counts being measured at each end of the integrated scan. The intensities of three standard reflexions, monitored throughout data collection, were used to scale the reflexion intensities within the data sets collected from each crystal. The data sets were overlapped sufficiently so that the intensities from each crystal could be put on a common scale after absorption corrections had been applied. Of 4476 independent intensities obtained, 3145 having $I > 3(\sigma)I$ were considered observed and used in the analysis.

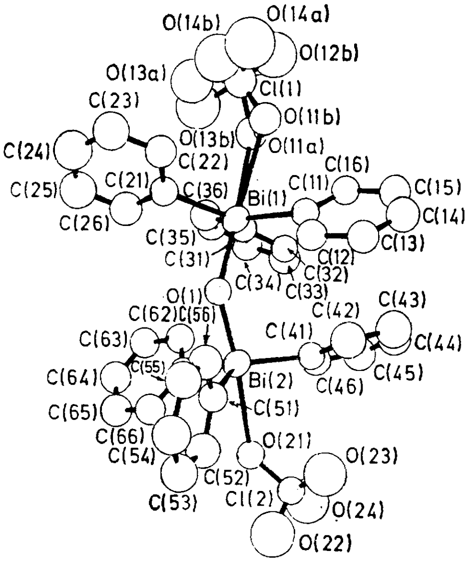

FIGURE 1 The molecule, showing the disordered perchlorate group and the atom numbering scheme used

Structure Determination and Refinement.—The co-ordinates of the two bismuth atoms were obtained from an

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[1] Part V, G. Ferguson, R. G. Goel, and D. R. Ridley, *J.C.S. Dalton*, preceding paper.

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analysis of the three-dimensional Patterson function. Difference-Fourier maps revealed sensible positions for all the non-hydrogen atoms. Eight distinguishable peaks of electron density about Cl(1) (see Figure 1) could be divided into two groups of four atoms, each forming an approximate tetrahedron about the chlorine and conforming closely to a two-fold disorder pattern. Accordingly, a simple two-fold disorder was assumed, and half-weight oxygen atoms were placed on each of the eight positions. The structure was then refined by least-squares methods using the block-diagonal approximation. Absolute weights [based on $\sigma(F) = 0{\cdot}5\sigma(F)^2/F$] were used throughout the refinement. Atomic scattering factors for carbon, oxygen, chlorine, and bismuth taken from ref. 11 were used for all structure

TABLE 1

Fractional co-ordinates ($\times 10^5$ for Bi, $\times 10^4$ for others) and thermal parameters (Å^2 $\times 10^4$ for Bi, $\times 10^3$ for others)

Atom	x/a	y/b	z/c	U_{iso}
Bi(1)	29288(5)	26459(9)	23731(4)	†
Bi(2)	18011(5)	52112(9)	33948(4)	†
Cl(1)	4116(4)	164(7)	1547(4)	83(2)
Cl(2)	1473(3)	8311(6)	4484(3)	60(2)
C(11)	3458(13)	4145(22)	1800(11)	58(6)
C(12)	3060(13)	5051(24)	1405(11)	66(7)
C(13)	3400(14)	6177(24)	1082(12)	71(7)
C(14)	4125(15)	6332(27)	1169(13)	85(8)
C(15)	4541(15)	5420(26)	1576(13)	78(8)
C(16)	4189(13)	4285(22)	1880(11)	60(6)
C(21)	2226(13)	1102(23)	1928(11)	63(6)
C(22)	2269(14)	790(25)	1203(12)	73(7)
C(23)	1806(17)	−235(30)	914(15)	98(10)
C(24)	1412(18)	−913(32)	1394(16)	108(11)
C(25)	1373(17)	−620(31)	2135(15)	103(10)
C(26)	1828(16)	417(28)	2406(14)	86(8)
C(31)	3428(13)	2175(23)	3406(11)	611(6)
C(32)	3828(13)	3170(23)	3738(11)	62(6)
C(33)	4082(16)	2879(28)	4463(14)	88(8)
C(34)	3949(16)	1583(28)	4772(13)	88(8)
C(35)	3587(16)	663(27)	4403(13)	86(8)
C(36)	3285(15)	889(27)	3697(13)	83(8)
C(41)	2815(12)	6225(22)	3627(11)	58(6)
C(42)	3133(17)	6892(31)	3057(15)	99(10)
C(43)	3873(19)	7385(32)	3247(16)	110(11)
C(44)	4114(17)	7236(30)	3872(15)	101(10)
C(45)	3795(17)	6600(30)	4450(14)	95(9)
C(46)	3128(14)	6025(25)	4283(13)	75(7)
C(51)	898(13)	5979(23)	2684(11)	61(6)
C(52)	373(15)	6736(28)	2991(13)	84(8)
C(53)	−255(16)	7064(29)	2540(15)	93(9)
C(54)	−231(17)	6631(30)	1800(15)	97(9)
C(55)	323(15)	5876(27)	1565(13)	86(8)
C(56)	901(15)	5562(27)	2019(13)	80(8)
C(61)	1431(13)	3891(22)	4194(11)	62(6)
C(62)	1853(14)	2907(25)	4419(12)	72(7)
C(63)	1583(15)	1959(27)	4930(13)	80(8)
C(64)	924(15)	2087(28)	5149(13)	87(8)
C(65)	486(16)	3063(28)	4909(14)	88(8)
C(66)	710(14)	4039(26)	4380(12)	77(8)
O(1)	2074(8)	3756(14)	2691(7)	56(4)
O(11a)	3945(19)	1109(34)	2098(16)	80(10)
O(11b)	3963(18)	1480(33)	1582(16)	76(9)
O(12a)	4765(28)	−417(50)	1771(25)	138(18)
O(12b)	4803(31)	131(54)	1541(27)	159(20)
O(13a)	3628(29)	−792(52)	1644(24)	143(18)
O(13b)	3629(30)	−476(54)	2056(27)	160(20)
O(14a)	4198(33)	181(57)	775(31)	191(22)
O(14b)	3784(32)	−200(53)	957(28)	163(20)
O(21)	1367(8)	6874(15)	4375(7)	63(4)
O(22)	804(14)	8891(26)	4463(12)	149(9)
O(23)	1806(14)	8832(25)	3933(13)	144(9)
O(24)	1802(15)	8521(26)	5151(13)	161(10)

† Anisotropic thermal parameters from the expression: $\exp[-2\pi^2(h^2a^{*2}U_{11} + k^2b^{*2}U_{22} + l^2c^{*2}U_{33} + 2hka^*b^*U_{12} + 2hla^*c^*U_{13} + 2klb^*c^*U_{23})]$.

	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Bi(1)	710(5)	491(5)	595(5)	−7(5)	16(5)	−32(5)
Bi(2)	699(5)	511(5)	578(5)	−4(5)	40(5)	−1(5)

TABLE 2

Selected interatomic distances (Å) and angles (°)

(*a*) Bonded distances

Bi(1)–O(1)	2·065(15)	Cl(1)–O(11b)	1·36(3)
Bi(2)–O(1)	2·062(14)	Cl(1)–O(12a)	1·40(5)
Bi(1)–C(11)	2·13(2)	Cl(1)–O(12b)	1·29(6)
Bi(1)–C(21)	2·19(2)	Cl(1)–O(13a)	1·35(5)
Bi(1)–C(31)	2·19(2)	Cl(1)–O(13b)	1·50(5)
Bi(2)–C(41)	2·19(2)	Cl(1)–O(14a)	1·47(6)
Bi(2)–C(51)	2·26(2)	Cl(1)–O(14b)	1·31(5)
Bi(2)–C(61)	2·15(2)	Cl(2)–O(21)	1·480(17)
Bi(1)–O(11a)	2·53(3)	Cl(2)–O(22)	1·38(3)
Bi(1)–O(11b)	2·76(3)	Cl(2)–O(23)	1·34(3)
Bi(2)–O(21)	2·655(15)	Cl(2)–O(24)	1·40(3)
Cl(1)–O(11a)	1·46(3)	C–C (mean)	1·407(8)

(*b*) Angles

Bi(1)–O(1)–Bi(2)	142·2(7)	C(51)–Bi(2)–C(61)	112·2(9)
O(1)–Bi(1)–C(11)	98·2(7)	O(1)–Bi(2)–O(21)	173·5(5)
O(1)–Bi(1)–C(21)	92·1(7)	O(11a)–Cl(1)–O(12a)	106(2)
O(1)–Bi(1)–C(31)	99·7(7)	O(11a)–Cl(1)–O(13a)	102(3)
O(11a)–Bi(1)–C(11)	87·9(10)	O(11a)–Cl(1)–O(14a)	137(3)
O(11b)–Bi(1)–C(11)	71·2(9)	O(12a)–Cl(1)–O(13a)	104(3)
O(11a)–Bi(1)–C(21)	86·0(10)	O(12a)–Cl(1)–O(14a)	101(3)
O(11b)–Bi(1)–C(21)	84·9(9)	O(13a)–Cl(1)–O(14a)	104(3)
O(11a)–Bi(1)–C(31)	75·5(9)	O(11b)–Cl(1)–O(12b)	104(3)
O(11b)–Bi(1)–C(31)	96·1(9)	O(11b)–Cl(1)–O(13b)	105(3)
C(11)–Bi(1)–C(21)	126·6(8)	O(11b)–Cl(1)–O(14b)	103(3)
C(11)–Bi(1)–C(31)	114·7(8)	O(12b)–Cl(1)–O(13b)	129(3)
C(21)–Bi(1)–C(31)	114·8(8)	O(12b)–Cl(1)–O(14b)	115(4)
O(1)–Bi(1)–O(11a)	173·5(8)	O(13b)–Cl(1)–O(14b)	98(3)
O(1)–Bi(1)–O(11b)	163·7(7)	O(21)–Cl(2)–O(22)	107·2(13)
O(1)–Bi(2)–C(41)	103·2(7)	O(21)–Cl(2)–O(23)	109·9(13)
O(1)–Bi(2)–C(51)	93·4(7)	O(21)–Cl(2)–O(24)	109·1(13)
O(1)–Bi(2)–C(61)	96·1(7)	O(22)–Cl(2)–O(23)	105·1(15)
O(21)–Bi(2)–C(41)	81·4(7)	O(22)–Cl(2)–O(24)	109·3(15)
O(21)–Bi(2)–C(51)	87·5(6)	O(23)–Cl(2)–O(24)	116·0(16)
O(21)–Bi(2)–C(61)	77·6(7)	Bi(1)–O(11a)–Cl(1)	137·5(19)
C(41)–Bi(2)–C(51)	125·7(8)	Bi(1)–O(11b)–Cl(1)	126·7(19)
C(41)–Bi(2)–C(61)	116·7(8)	Bi(2)–O(21)–Cl(2)	132·5(8)

(*c*) Selected intramolecular non-bonded contacts

(i) Ph ··· ClO_4

O(11a) ··· C(11)	3·24(4)	O(21) ··· C(41)	3·18(3)
O(11a) ··· C(21)	3·24(4)	O(21) ··· C(51)	3·41(3)
O(11a) ··· C(31)	2·90(4)	O(21) ··· C(52)	3·16(3)
O(11b) ··· C(11)	2·89(4)	O(21) ··· C(61)	3·04(3)
O(11b) ··· C(22)	3·30(4)	O(21) ··· C(60)	3·12(3)

(ii) Ph ··· Ph

C(11) ··· C(32)	3·83(2)	C(32) ··· C(41)	3·36(4)
C(12) ··· C(42)	3·64(4)	C(32) ··· C(45)	3·72(4)
C(13) ··· C(42)	3·86(4)	C(32) ··· C(46)	3·35(3)
C(16) ··· C(32)	3·78(3)	C(32) ··· C(62)	3·98(4)
C(21) ··· C(36)	3·83(3)	C(36) ··· C(62)	3·68(4)
C(26) ··· C(36)	3·63(3)	C(52) ··· C(66)	3·82(4)

(*d*) Shortest non-bonded intermolecular contacts

C(13) ··· C(35^{I})	3·71(4)	O(12a) ··· C(32^{V})	3·18(6)
C(22) ··· C(63^{I})	3·52(4)	O(12b) ··· C(44^{V})	3·06(6)
C(23) ··· C(61^{I})	3·57(4)	O(13a) ··· C(14^{II})	3·19(6)
C(25) ··· C(52^{II})	3·68(4)	O(13b) ··· C(43^{II})	3·14(6)
C(25) ··· C(53^{III})	3·50(4)	O(14a) ··· C(45^{I})	3·16(6)
C(26) ··· C(53^{III})	3·39(4)	O(14a) ··· C(33^{I})	3·16(6)
C(26) ··· C(54^{III})	3·62(4)	O(14b) ··· C(45^{I})	3·18(6)
C(54) ··· C(65^{IV})	3·56(4)	O(22) ··· C(65^{VI})	3·37(4)
C(54) ··· C(66^{IV})	3·40(4)	O(23) ··· C(26^{VII})	3·31(4)
O(12a) ··· C(16^{V})	3·17(5)	O(24) ··· C(13^{VIII})	3·43(4)

Roman numeral superscripts denote the following equivalent positions:

I	$x, \frac{1}{2} - y, z - \frac{1}{2}$	V	$1 - x, y - \frac{1}{2}, \frac{1}{2} - z$
II	$x, y - 1, z$	VI	$-x, 1 - y, 1 - z$
III	$-x, y - \frac{1}{2}, \frac{1}{2} - z$	VII	$x, 1 + y, z$
IV	$-x, \frac{1}{2} + y, \frac{1}{2} - z$	VIII	$x, 1\frac{1}{2} - y, z - \frac{1}{2}$

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[11] 'International Tables for *X*-Ray Crystallography', vol. III, 1965, Kynoch Press, Birmingham, Tables 3.3.1A, 3.3.1B, 3.3.2.

calculations, and corrections for anomalous dispersion for the bismuth atoms were included at later stages of the refinement. With all atoms restricted to isotropic motion, the refinement converged with R 0·085. Finally the bismuth atoms were allowed anisotropic thermal vibration and the refinement converged with R 0·070. In the last refinement cycle the maximum shift in any parameter was 0·5σ [for the temperature factor of O(14A)]; all other parameter shifts were $<0{\cdot}2\sigma$. The final temperature factors of the disordered perchlorate group are reasonable when compared to those of the non-disordered group and confirm the validity of the assumptions made.

A final difference-Fourier synthesis revealed a number of small peaks (highest 1·34 eÅ^{-3}). Most of these were close to the heavy atoms or around the two chlorine atoms, indicating that considerable anisotropic motion of both the chlorine and oxygen atoms had not been taken into account. The final R for all data, including unobserved reflexions, is 0·095, R' being 0·093 $\{R' = [\Sigma w(F_o - F_c)^2/\Sigma wF_o^2]^{1/2}\}$. The error in an observation of unit weight calculated for all data is 1·82. Final atomic positional and thermal parameters are listed in Table 1, and bond distances and angles and selected non-bonded contacts in Table 2. Observed and final calculated structure factors and details of several mean-plane calculations are listed in Supplementary Publication No. SUP 21260 (8 pp., 1 microfiche).*

DESCRIPTION AND DISCUSSION

The structure consists of discrete [Ph$_3$Bi(ClO$_4$)]$_2$O molecules, as shown in Figure 1, which also gives the atom numbering scheme used. The configuration at the bismuth atoms linked by the oxygen bridge is distorted trigonal bipyramidal with oxygens apical and phenyl groups equatorial. The mean O(br)–Bi–C(Ph) angles are 97·1°, and the bismuth atoms are displaced from the plane formed by the three phenyl carbon atoms by 0·24 [Bi(1)] and 0·29 Å [Bi(2)]. The result is that the trigonal bipyramid about each bismuth atom is displaced towards the tetrahedral configuration which would be expected if the perchlorates were not co-ordinated. The contents of the unit cell are illustrated in Figure 2.

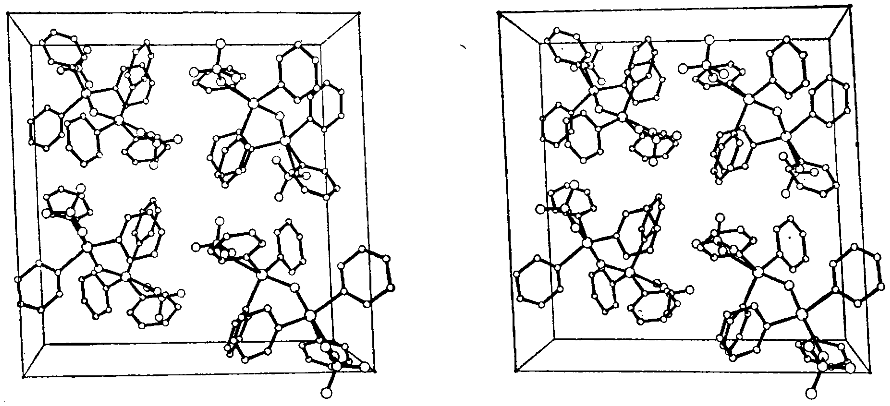

FIGURE 2 Stereoview of the contents of a unit cell. For clarity, only one set of disordered perchlorate oxygens is shown. The origin of the unit cell is at the lower left-hand corner with *a* horizontal, *c* vertical, and *b* into the page

From Bi–C distances in this and other bismuth-containing molecules [12] a covalent bond radius of *ca.* 1·50 Å can be predicted for bismuth. The Bi–O(br) distance [2·065(10) Å] is thus 0·1 Å shorter than that expected for a single-bond distance. A similar apparent bond-shortening was also found for Sb–O in (Ph$_3$SbN$_3$)$_2$O.[3] The metal–oxygen–metal angles in the two compounds [Ph$_3$Bi(ClO$_4$)]$_2$O [142·4(7)°] and (Ph$_3$SbN$_3$)$_2$O [139·8(4)°] also agree well. If the oxygen atom is regarded as being basically tetrahedral, the observed angle could be considered as having been opened from the tetrahedral by intramolecular Ph ··· Ph contacts; however only one contact [C(46) ··· C(32) 3·35 Å] is particularly short, and the majority are considerably greater than the combined van der Waals distances. It is also therefore possible that some π-bonding mechanism is at least partly responsible for the M–O–M angle being much larger than tetrahedral. A similar large angle has been observed in bis(diphenylarsenic)oxide [13] where the angle As–O–As is 137(2)°, and mean As–O 1·67 Å, much shorter than the sum of the single-bond covalent radii.

The mean Bi–C(Ph) distance [2·19(2) Å] is not significantly different from that determined for dichlorotriphenylbismuth [12] [2·12(8) Å] and the mean C(Ph)–Bi–C(Ph) angle is 118·5°. The geometry of the phenyl groups is as expected. The angle of rotation about their respective Bi–C bonds, as determined by the dihedral angle between the phenyl ring planes and the O(1), Bi, C(Ph) plane for each ring, varies from 18·3 to 78·4°. The phenyl groups about Bi(1) are in the propeller configuration, but those about Bi(2) are rotated in different senses. The widely varying rotations result in minimisation of contacts between the phenyl groups and appear to have no other significance.

One of the two perchlorate groups is disordered between two sites, with two interleaved tetrahedral groups of oxygen atoms. The chlorine atom [Cl(1)] of the disordered group has a higher temperature factor than that of the ordered group [Cl(2)] and may also be

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* See Notice to Authors No. 7 in *J.C.S. Dalton*, 1974, Index issue.

[12] D. M. Hawley and G. Ferguson, *J. Chem. Soc.* (*A*), 1968, 2539.

[13] W. R. Cullen and J. Trotter, *Canad. J. Chem.*, 1963, **41**, 2983.

slightly disordered between two positions which have not been resolved. The bond angles and distances associated with the disordered perchlorate group vary widely and inconsistently and are not discussed further; the mean dimensions are in reasonable agreement with those of the ordered perchlorate (Table 2).

The bismuth–oxygen (ordered perchlorate) distance [2·65(1) Å] is extremely long for a covalent bond and suggests that the bonding is intermediate in character between a formal covalent bond and an ionic interaction. A number of examples of this type of semi-co-ordination have been reported with the perchlorate and tetrafluoroborate complexes of some first-row transition metals, *e.g.*, with severely tetragonally distorted octahedral complexes of copper(II) [14–17] metal–ligand distances vary between 2·5 and 3·0 Å. An interesting example is found in the structure of the six-co-ordinate complex bis[*o*-phenylenebis(dimethylarsine)]-cobalt(II) perchlorate,[18] where the co-ordinated perchlorate appears to be disordered in a manner similar to that found for the disordered perchlorate here. The Cl–O(co-ordinated) distance [1·48(2) Å] is significantly longer than the other Cl–O bonds [mean 1·38(3) Å]. This may reflect the effect of a partial covalency between the metal and the oxygen weakening the Cl–O bond. A similar lengthening has been observed in the case of a strongly co-ordinated perchlorate.[19]

The O(br)–Bi–O(perchlorate) angles (163·7—173·5°) are non-linear presumably because of $ClO_4 \cdots Ph$ intramolecular overcrowding; in this respect it is probably significant that the Bi–O–Cl angles are larger than tetrahedral (mean 132·3°) as this would have the effect of minimising such interactions (see Figure 1).

There are [Table 2(*c*)] a number of relatively short intermolecular contacts (3·14—3·18 Å) between the disordered-perchlorate oxygen atoms and phenyl carbon atoms of neighbouring molecules but no $C-H \cdots O$ type hydrogen bonding is inferred from this, since the phenyl C–H groups are not suitably oriented.

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