Table 3. *Intermolecular* S···S *distances* (Å) *less than* 3·9 Å

S1K—S8N	3·766 (6)	S1K—S3L	3·589 (6)
S3K—S8N	3·830 (5)	S5K—S7L	3·817 (6)
S2K—S7N	3·870 (5)	S6K—S2M	3·527 (4)
S4K—S7N	3·777 (5)	S2K—S2M	3·339 (3)
S3K—S4N	3·868 (5)	S6K—S4M	3·432 (4)

K, L, M and *N* refer to the BEDT-TTF molecules in Fig. 4.

of at least one of the ethylene groups has been found among other BEDT-TTF salts, including β-(BEDT-TTF)₂I₃ (Leung *et al.*, 1985) and (BEDT-TTF)₂[Cu(NCS)₂] (Urayama *et al.*, 1988). Short H···X (X = Br,I,NCS) contacts less than the van der Waals radii sums are believed to be the dominating factor for the disorder of the ethylene group. For the present structure, accurate hydrogen positions are not available. From the calculated positions of H atoms, the shortest H···I contacts are estimated to be of the order of 3·1 Å.

A number of BEDT-TTF salts containing Hg have been reported including a low-temperature superconducting phase (BEDT-TTF)₄[Hg₂.₈Br₈], but the present report is the first structural study of a Cd-containing salt.

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Structure of Tetraaquacobalt(II) Bromide Dihydrate (Cobalt Bromide Hexahydrate)

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Abstract. Tetraaquacobalt(II) bromide dihydrate, [Co(H₂O)₄]Br₂.2H₂O, $M_r = 326·83$, monoclinic, C2/m, $a = 11·0158$ (12), $b = 7·1694$ (10), $c = 6·9111$ (10) Å, $\beta = 124·761$ (7)°, $V = 448·4$ (1) Å³, $Z = 2$, $F(000) = 314$, $D_x = 2·420$ g cm⁻³, $\lambda(\text{Mo } K\bar{\alpha}) = 0·71069$ Å, $T = 296$ K, $\mu = 107·13$ cm⁻¹, $R = 0·022$ for 532 unique reflections having $I > 0$. The single type of Co ion is octahedrally coordinated by four equivalent water-molecule O atoms at the observed distance 2·081 (2) Å and two equivalent Br ions at the observed distance 2·6048 (5) Å in an array which, with respect to its angles, departs only slightly from regular octahedral geometry. The cobalt–oxygen– bromine complex was found to manifest rigid-body behavior. The cobalt–oxygen distance corrected for rigid-body motion was found to be 2·088 Å; the corrected cobalt–bromine distance, 2·6081 Å. Associated with each complex are two water molecules of hydration. Assignment and refinement of the coordinates of the four non-equivalent H atoms permitted detailed analysis of the hydrogen bonding, which involves each of the four non-equivalent H atoms significantly. The structure as determined in this study differs markedly from that presented in the sole published single-crystal study [Stroganov, Andreev, Kožina & Solov'ev (1961). *Vestn. Leningr. Univ. Ser. Mat. Fiz. Khim.* **16**, 114–119]. In particular, the space-group assignment differs and the pat-

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tern of bromine–oxygen distances is quite different. Contrary to Stroganov *et al.*, this structure is isomorphic with that of cobalt chloride hexahydrate.

Introduction. During a recent study of the structure of hexaaquacobalt(II) bromate, we became aware that quite limited structural data were available for hydrated cobalt(II) bromide. The only previous single-crystal X-ray study is that of Stroganov, Andreev, Kožina & Solov'ev (1961) and it provided only approximate coordinates for the O atoms. The present study was undertaken to determine definitively the heavy-atom positions and, if possible, the H-atom positions in order to permit detailed analysis of the hydrogen bonding. It became apparent as our study progressed that the study by Stroganov *et al.* (1961) was in error. They assigned the space group as $P2_1$, whereas it is $C2/m$, and obtained sets of interatomic distances irreconcilable with the present data.

Experimental. Crystalline tetraaquacobalt(II) bromide dihydrate was obtained at room temperature by evaporation of an aqueous solution prepared from G. F. Smith reagent grade anhydrous cobalt(II) bromide. The experimental sample was a purple, compact prism with two prominent hexagonal faces and twelve circumferential faces. The principal dimensions were approximately $0.19 \times 0.27 \times 0.35$ mm. The sample was coated with a thin layer of Apiezon L grease to retard any potential gain or loss of water and was analyzed at 296 K with a Rigaku AFC5S diffractometer using graphite-monochromated Mo $K\bar{\alpha}$ radiation.

Unit-cell parameters were obtained from a symmetry-constrained least-squares fit of the setting angles for 25 centered reflections with $23 < 2\theta < 30°$. Intensity data were measured for 1321 reflections (exclusive of standards) with $+h$, $+k$, $\pm l$ and $-h$, $-k$, $\pm l$ indices ($h_{max} = 14$; $k_{max} = 9$; $l_{max} = 8$) and with 2θ values in the range $4 \le 2\theta \le 55°$. The ω–2θ scan technique was employed with scan widths $(1.15 + 0.35 \tan\theta)°$ in ω, and a background/scan time ratio of 0.5. The variance assigned to each reflection was $\sigma_I^2 = \sigma_{cs}^2(I) + (0.03I)^2$, where σ_{cs} is based on counting statistics and I is the integrated intensity. Six standard reflections ($40\bar{1}$, 111, 220, $33\bar{4}$, 402 and $62\bar{4}$) were measured after every 150 reflections and exhibited, on average, a maximum non-systematic relative intensity variation of $\pm 1.6\%$. Accordingly, no decay correction was applied. The data were corrected for Lorentz and polarization effects. A ψ-scan absorption correction was applied based on ψ-scan data obtained at the midpoint of intensity-data collection; the range of transmission factors was 0.595–1.000, with an average value 0.834.

The crystal was found to belong to Laue group $2/m$ so that, consistent with the systematic absences

(hkl: $h + k$ odd), three C-centered monoclinic space groups were allowed. The centrosymmetric alternative, $C2/m$ (No. 12), was given initial preference and, since refinement proceeded well, it was adopted. Initial coordinates for Co in a special position with site symmetry $2/m$, for Br in a special position with site symmetry m, and for O(1) in a general position were obtained using a Patterson map. Fourier difference methods were then used to assign O(2) to a special position with site symmetry m, H(1) and H(2) in general positions, and H(3) and H(4) in special positions with site symmetry m. Full-matrix least-squares refinement was performed using the TEXSAN (Molecular Structure Corporation, 1988) structure analysis package to minimize the function $\Sigma\sigma_F^{-2}(|F_o| - |F_c|)^2$ in which $\sigma_F = \sigma_I/2F$Lp. Neutral-atom scattering factors and anomalous-dispersion factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 71, 148) for Co, Br and O; the scattering factor for hydrogen was taken from Stewart, Davidson & Simpson (1965). Following refinement to the isotropic stage for all atoms except hydrogen using the data having $I > 3\sigma_I$, the ψ-scan absorption correction was applied and the data were averaged ($R_{int} = 0.017$ for 539 pairs and 28 quadruples of reflections). Then, using all the unique reflections with $I > 0$ and treating the secondary extinction as an adjustable parameter, least-squares refinement proceeded to the anisotropic stage for all atoms but hydrogen and to the isotropic stage for hydrogen.

The results for the final refinement cycle were: 532 independent observations having $I > 0$; 41 variables; $R = 0.022$; $wR = 0.024$; $w = \sigma_F^{-2}$; $S = 1.13$; $(\Delta/\sigma)_{max} < 0.01$. The optimized secondary-extinction coefficient was $5.31 (28) \times 10^{-6}$. Maximum and minimum peaks in the final electron density difference map were respectively, $+0.53$ e Å^{-3} at $x = 0.22$, $y = 0.00$, $z = 0.03$, approximately 0.9 Å from Br, and -0.59 e Å^{-3} at $x = 0.30$, $y = 0.00$, $z = 0.05$, also approximately 0.9 Å from Br.*

The final atomic coordinates, isotropic and equivalent isotropic displacement parameters and their uncertainties are given in Table 1.† Selected interatomic distances and angles in the coordination

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* For the final refinement cycle for the 478 independent observations having $I > 3\sigma_I$: $R = 0.019$; $wR = 0.024$; $S = 1.18$; $(\Delta/\sigma)_{max} < 0.01$. Similarly, for the 508 independent observations having $I > \sigma_I$: $R = 0.020$; $wR = 0.024$; $S = 1.15$; $(\Delta/\sigma)_{max} < 0.01$. These latter two refinements were performed for the purposes of comparison only.

† Lists of structure factors, anisotropic displacement parameters and supplementary material relating to the rigid-body analysis have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53390 (7 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Final positional parameters and equivalent isotropic and isotropic displacement parameters,* B_{eq}/B (Å²), *for tetraaquacobalt(II) bromide dihydrate at 296 K, with e.s.d.'s in parentheses*

$$B_{eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij}a_i{}^*a_j{}^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}/B
Co	0	0	0	1·62 (2)
Br	0·28174 (4)	0	0·17896 (6)	2·09 (2)
O(1)	0·03639 (21)	0·20060 (28)	0·24543 (34)	2·46 (7)
O(2)	0·28784 (38)	0	0·69990 (60)	2·73 (11)
H(1)	0·095 (4)	0·279 (5)	0·262 (7)	5·3 (11)*
H(2)	−0·031 (3)	0·251 (4)	0·236 (6)	3·4 (8)*
H(3)	0·275 (8)	0	0·823 (12)	6·9 (16)*
H(4)	0·218 (9)	0	0·608 (14)	7·8 (25)*

* Refined isotropically.

polyhedron about cobalt and in the water molecules are given in Table 2 together with their uncertainties. Hydrogen bonds involving water-molecule oxygens, their protons and bromine are delineated in Table 3.

Rigid-body analysis of the cobalt–oxygen–bromine complex was performed using the program *THMA*11 (Trueblood, 1986), based on the work of Schomaker & Trueblood (1968). The maximum magnitude of the differences in the mean square displacement amplitudes (MSDA) along the interatomic vectors for the eight unique atom pairs of the cobalt–oxygen–bromine complex was found to be 29 (10) × 10⁻⁴ Å², while the average was 8 (11) × 10⁻⁴ Å². On the basis of these values, we concluded that Hirshfeld's rigid bond test (Hirshfeld, 1976) is satisfied for the cobalt–oxygen–bromine complex and have applied corrections for the rigid-body motion. The corrected bond lengths are given in Table 2; the corrected angles differed from the observed angles by less than their e.s.d.'s and were therefore not tabulated.

Discussion. The cobalt coordination octahedron has one independent Co—O(1) distance and one Co—Br distance. The O(1)—Co—O(1)^{ii,iii} angles of the octahedron differ by ∼2·6° from the 90° value characterizing a regular octahedron; similarly, the Br—Co—O(1)$^{-,i}$ angles differ by ∼0·6° from the 90° value. The Co—O(1) distance may be compared with the corresponding distance recently reported for hexaaquacobalt(II) bromate (also at 296 K): 2·095 Å observed; 2·099 Å rigid-body-motion corrected (Blackburn, Gallucci & Gerkin, 1990).

A stereoview of this structure is given in Fig. 1. The H-atom positions illustrated are the neutron-adjusted positions as described below. While all hydrogen bonds involving O(2), H(3) and H(4) have been indicated, most of those involving O(1), H(1) and H(2) have been omitted for clarity.

The hydrogen bonds involving the water not coordinated to Co [O(2), H(3) and H(4)], *i.e.* the 'hydrate'

Table 2. *Bond lengths (Å) and bond angles (°) for tetraaquacobalt(II) bromide dihydrate, with e.s.d.'s in parentheses*

Coordination polyhedron: rigid body 1

	Observed distance	Rigid-body distance
Co—O(1)	2·081 (2)	2·088
O(1)—O(1)^i	4·162 (4)	4·175
O(1)—O(1)^{ii}	2·876 (4)	2·885
O(1)—O(1)^{iii}	3·008 (4)	3·018
Co—Br	2·6048 (5)	2·6081
Br—O(1)	3·317 (2)	3·324
Br—O(1)^i	3·351 (2)	3·357
Br—O(1)^{ii}	3·317 (2)	3·324
Br—O(1)^{iii}	3·351 (2)	3·357

	Observed angle
O(1)—Co—O(1)^i	180
O(1)—Co—O(1)^{ii}	87·43 (12)
O(1)—Co—O(1)^{iii}	92·57 (12)
Br—Co—Br^i	180
Br—Co—O(1)	89·39 (5)
Br—Co—O(1)^i	90·61 (5)

Water molecules

O(1)—H(1)	0·81 (4)	H(1)—O(1)—H(2)	109 (3)
O(1)—H(2)	0·79 (3)		
O(2)—H(3)	0·94 (7)	H(3)—O(2)—H(4)	100 (7)
O(2)—H(4)	0·66 (8)		
O(2)—Br^{iv}	3·350 (3)		
O(2)—Br	3·562 (3)		

Code for symmetry-related atoms: none x, y, z; (i) $-x, -y, -z$; (ii) $x, -y, z$; (iii) $-x, y, -z$; (iv) $x, y, 1 + z$.

water, form by themselves a three-dimensional network (Fig. 1). This network is further elaborated by the remaining hydrogen bonds involving the water molecules which are coordinated to Co.

Concerning the water molecules [H(1)—O(1)—H(2)] which participate in the coordination octahedron, we note that the observed H—O—H angle (Table 2) is 109° with an e.s.d. of 3°. This value compares favorably with the value for water-molecule H—O—H angles determined by neutron diffraction as tabulated by Chiari & Ferraris (1982): for water molecules of class *H* (Chiari & Ferraris, 1982; Ferraris & Franchini-Angela, 1972), to which the cobalt-coordinating water molecules belong, the range is 104·0–109·7° with an average value 106·6°. Similarly, for the 'hydrate' water molecules [H(3)—O(2)—H(4)] the observed H—O—H angle is 100° with an e.s.d. of 7°. This value is in reasonable agreement with the values from neutron diffraction for the H—O—H angle for water molecules of class *E*, to which these hydrate water molecules belong: the range is 103·5–109·5° with an average value 106·1° (Chiari & Ferraris, 1982; Ferraris & Franchini-Angela, 1972).

The presence of a single type of cobalt coordination octahedron and a single type of water molecule not coordinated to cobalt leads to a rather simple

Table 3. *Hydrogen-bond parameters for tetraaquacobalt*(II) *bromide dihydrate, with e.s.d.'s in parentheses*

See text for details.

Water oxygen—acceptor atom	Distance, Å	Proton—acceptor atom	Observed distance, Å	Neutron-adjusted distance, Å	Observed O—H—acceptor angle, °	
$O(1)$—$O(2)^v$	2·769 (3)	$H(1)$—$O(2)^v$	1·97 (4)	1·81	$O(1)$—$H(1)$—$O(2)^v$	170 (4)
$O(1)^{vii}$—$O(2)^v$	2·769 (3)	$H(1)^{vii}$—$O(2)^v$	1·97 (4)	1·81	$O(1)^{vii}$—$H(1)^{vii}$—$O(2)^v$	170 (4)
$O(1)$—$O(1)^{iii}$	3·008 (4)	$H(1)$—$O(1)^{iii}$	2·99 (4)	3·01	$O(1)$—$H(1)$—$O(1)^{iii}$	83 (3)
$O(2)$—$O(1)$	3·112 (4)	$H(4)$—$O(1)$	2·58 (7)	2·35	$O(2)$—$H(4)$—$O(1)$	140 (4)
$O(2)$—$O(1)^{ii}$	3·112 (4)	$H(4)$—$O(1)^{ii}$	2·58 (7)	2·35	$O(2)$—$H(4)$—$O(1)^{ii}$	140 (4)
$O(1)$—Br	3·317 (2)	$H(1)$—Br	3·15 (4)	3·14	$O(1)$—$H(1)$—Br	95 (3)
$O(1)$—Br^{vi}	3·349 (2)	$H(2)$—Br^{vi}	2·58 (3)	2·41	$O(1)$—$H(2)$—Br^{vi}	163 (3)
$O(2)$—Br^{iv}	3·350 (3)	$H(3)$—Br^{iv}	2·42 (7)	2·39	$O(2)$—$H(3)$—Br^{iv}	171 (6)

Code for symmetry-related atoms: none x, y, z; (ii) $x, -y, z$; (iii) $-x, y, -z$; (iv) $x, y, 1 + z$; (v) $\frac{1}{2} - x, \frac{1}{2} - y, 1 - z$; (vi) $-\frac{1}{2} + x, \frac{1}{2} + y, z$; (vii) $\frac{1}{2} - x, -\frac{1}{2} + y, 1 - z$.
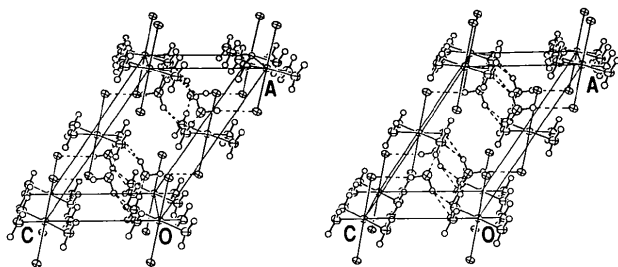

Fig. 1. Stereoview of a portion, including a unit cell, of the tetraaquacobalt(II) bromide dihydrate structure at 296 K drawn using *ORTEP*II (Johnson, 1976). Thermal ellipsoids are drawn at 50% probability for all atoms except hydrogen, for which they have been set artificially small. The H-atom positions illustrated are the neutron-adjusted positions (see text). While all hydrogen bonds involving the water molecule not coordinated to a cobalt have been indicated, many of those involving the water molecule coordinated to cobalt have been omitted for clarity. The hydrogen bonds are indicated by dashed lines.
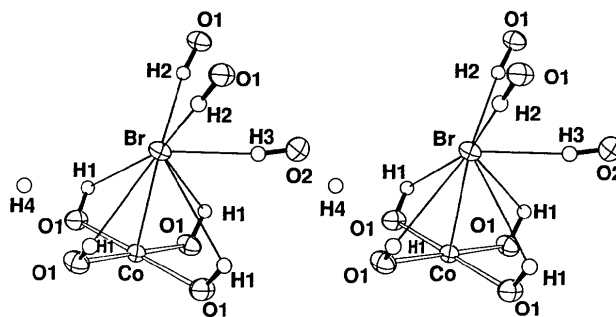

Fig. 2. Stereoview of the environment within 3·5 Å of bromine in the tetraaquacobalt(II) bromide dihydrate structure drawn using *ORTEP*II (Johnson, 1976). Thermal ellipsoids have been drawn at 50% probability for all atoms except hydrogen, for which they have been set artificially small. The orientation is the same as that shown in Fig. 1. The lighter lines are drawn to atoms lying within 3·2 Å of the bromine. It may be noted that no O atoms lie within 3·2 Å of the bromine.

hydrogen-bonding situation. Our analysis of these hydrogen bonds and their geometries is based partially on treatments of hydrogen bonding by Jeffrey (1987) and Chiari & Ferraris (1982).

To account for the foreshortening of the water O—H bond distances as determined by X-ray diffraction, the positions of H(1) and H(2) were adjusted along the corresponding O(1)—H bond direction (as determined by the X-ray data) until the O(1)—H distances were 0·972 Å, the mean value for the O—H bond distance as determined by neutron diffraction for water molecules of class *H*; similarly, the positions of H(3) and H(4) were so adjusted until the O(2)—H bond distances were 0·966 Å, the mean value from neutron-diffraction determinations for water molecules of class *E*. The resulting adjusted proton coordinates were then used to calculate distances and angles involving potential hydrogen-bond acceptor atoms. Since the adjusted O(water)—H—acceptor angles typically differed from the observed angles (calculated directly from the X-ray determined coordinates) by less than one e.s.d., only the observed angles were tabulated together with

observed O(water)—acceptor distances and observed and neutron-adjusted H—acceptor distances in Table 3. In the following material, the neutron-adjusted H—acceptor distance is taken to be the hydrogen-bond length.

As shown in Table 3, each of the H atoms H(1), H(2) and H(3) is involved in a single strong hydrogen bond: for H(1) the acceptor is O(2), while for H(2) and H(3) the acceptor is Br. Moreover, the fourth hydrogen, H(4), is involved in hydrogen bonding to two equidistant O(1) atoms. Two weaker hydrogen-bonding interactions [both involving H(1)] have also been tabulated. It should be noted that none of the tabulated O(water)—O(acceptor) distances for hydrogen bonds exceeds the 'maximum' value cited by Jeffrey (1987) and Chiari & Ferraris (1982) for hydrogen-bond formation in inorganic hydrates (3·15 Å).

The present data show that, contrary to the conclusion of Stroganov, Andreev, Kožina & Solov'ev (1961), cobalt bromide hexahydrate and cobalt chloride hexahydrate are indeed isomorphic. [That the cobalt chloride hexahydrate structure is also

$C2/m$ was established by Mizuno, Ukei & Sugawara (1959) and Mizuno (1960).]

Fig. 2 presents a view of the environment of a (central) bromine in the bromide structure up to 3·5 Å from bromine, with those atoms lying within 3·2 Å of bromine being connected to it by sight lines. This figure clearly shows that, contrary to Stroganov *et al.* (1961), there is no O atom in this structure within 2·84 Å of the bromine but that there are seven O atoms lying within 3·5 Å rather than two as stated by those authors. The pattern of the remaining bromine–oxygen distances reported there is also quite different. Moreover, their value for Co—O(1) (2·24 Å) is too large, while their value for Co—Br (2·49 Å) is too small.

So far as we are aware, the first documentation concerning the isomorphism of these cobalt bromide and chloride hexahydrates was provided in a private communication from G. C. Verschoor cited by Metselaar & De Klerk (1973). They reported that at room temperature the cell dimensions of the monoclinic bromide salt are $a = 11·029$, $b = 7·178$ and $c = 6·908$ Å (each estimated to be accurate to one part per thousand) with $\beta = 124·71$ (4)°. It will be noted that these values are in good agreement with the values reported here, deviating typically by slightly more than the combined uncertainties. Prior to this report of Metselaar & De Klerk (1973), and remarkably, after it as well, the isomorphism was apparently merely assumed or misderived from the report of Stroganov *et al.* (1961) notwithstanding their explicit assertion of the lack of isomorphism. As a consequence of the invariable assessment that the chloride and bromide are isomorphic, no alterations to the conclusions of the numerous papers on the magnetic properties of cobalt bromide hexa-hydrate (beginning with the report of antiferromagnetism in this salt by Forstat, Taylor & Spence, 1959) are required. This is rather surprising since the sole published structural study was incorrect.

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Polymerer Cadmium(II)-Komplex mit Di(pyrazolidino)thiophosphorsäure-O-phenylester als dreizähnigem *S,N,N*-Ligand

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Abstract. Poly-μ-chloro-{chloro[*O*-phenyl di-(pyrazolidino)thiophosphate-*S,N,N*]cadmium(II)}, [CdCl$_2$(C$_{12}$H$_{19}$N$_4$OPS)], $M_r = 481·65$, monoclinic, $P2_1/c$, $a = 12·387$ (3), $b = 7·523$ (2), $c = 18·585$ (3) Å, $\beta = 96·09$ (2)°, $V = 1722$ (3) Å^3, $Z = 4$, $D_x = 1·858$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 1·78$ mm^{-1}, $F(000) = 960$, $T = 293$ K, $R = 0·041$ ($wR = 0·024$) for 4114 unique contributing reflections. The title compound has been prepared from alcoholic solutions of the ligand and CdCl$_2$.H$_2$O in a