(Thiele, Rotter, Lietz & Ellermann, 1984) the P(1)—C(1)—C(4) angle is enlarged, and here has a value of 121·9 (5)°, whereas P(2)—C(2)—C(4) and P(3)—C(3)—C(4) are only 113·0 (4) and 114·0 (4)°, respectively.

The molecular arrangement in the crystal is shown in Fig. 2. There are close intermolecular P···P interactions [P(3)···P(3) 3·414 (3), P(2)···P(3) 3·711 (3) Å] across the inversion centres (000, $0\frac{1}{2}\frac{1}{2}$). These 'dimers' are stabilized in three dimensions by P···Se and Se···Se intermolecular contacts listed in Table 2.

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Diaquadiiodozinc(II)–18-Crown-6 Ether* Monohydrate

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Abstract. $[ZnI_2(H_2O)_2].C_{12}H_{24}O_6.H_2O$, $M_r = 637·6$, monoclinic, $P2_1/c$, $a = 9·386\ (1)$, $b = 13·612\ (2)$, $c = 20·058\ (2)$ Å, $\beta = 115·76\ (5)°$, $V = 2308·0\ (5)$ Å^3, $Z = 4$, $D_x = 1·835\ (1)$ Mg m^{-3}, $\lambda(\text{Mo}\ K\alpha) = 0·71073$ Å, $\mu = 3·770\ (1)$ mm^{-1}, $F(000) = 1240$, $T = 295$ K, $R = 0·037$, $wR = 0·040$ for 1732 reflexions $[I > 3\sigma(I)]$ and 220 variables. The structure is built up of stacks of crown ethers which are linked by weak hydrogen bonds, O···O = 2·79 (1) to 2·86 (1) Å, to a water molecule and to the $ZnI_2(H_2O)_2$ tetrahedra. The mean Zn—I and Zn—O distances are 2·543 (1) and 2·004 (5) Å, the I—Zn—I angle is 122·2 (5)°.

Introduction. The crystal structures have been determined for several 18-crown-6 ether complexes with cadmium or mercury halides (Paige & Richardson, 1984; Pears, Stoddart, Crosby, Allwood & Williams, 1986; Hazell, 1988). In these the metal atom is at the centre of the crown and is eight-coordinated. The attempted preparation of the corresponding zinc compound yielded the title compound in which the crown ethers are not coordinated to zinc but are weakly hydrogen-bonded to $ZnI_2(H_2O)_2$ tetrahedra and to a water molecule.

Experimental. The compound was prepared by dissolving equimolar amounts of ZnI_2 and 18-crown-6 ether in acetonitrile from which it was recrystallized. A crystal was mounted on a Huber four-circle diffractometer. The cell dimensions were determined from the setting angles of 60 reflexions with $18 \leq 2\theta \leq 26°$. Intensities were measured out to $2\theta_{\max} = 50°$ using an ω–2θ scan with Nb-filtered Mo $K\alpha$ radiation, the scan width was $(1·0 + 0·692\tan\theta)°$ which was divided into 50 steps, the counting time was 1 s step^{-1}. Reflexions with $-10 \leq h \leq 11$, $-16 \leq k \leq 0$, $-23 \leq l \leq 0$ were measured giving 3946 independent reflexions of which 1732 had $I > 3\sigma(I)$. Reflexions 080 and 400 were monitored every 100 reflexions, the overall fall off in intensity was 0·3%. Reflexions were integrated using the Nelmes (1975) method, the data were corrected for

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* 18-Crown-6 ether is 1,4,7,10,13,16-hexaoxacyclooctadecane.

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absorption (crystal 0·28 × 0·44 × 0·28 mm), transmission factors ranging from 0·20 to 0·47. The positions of the Zn and I atoms were determined from *MULTAN*80 (Main *et al.*, 1980), those of the C and O atoms from subsequent Fourier syntheses. The positions of the H atoms in the crown were calculated assuming C—H = 0·95 Å and were not refined. All non-H atoms were refined anisotropically, common isotropic thermal parameters were refined for the H atoms of each crystallographically independent crown, $U = 0{\cdot}16\,(2)$ Å^2 for *A* and $0{\cdot}19\,(2)$ Å^2 for *B*. An isotropic extinction factor was refined giving $g = 0{\cdot}36\,(3) \times 10^{-4}$, which corresponds to a minimum value of $I/I_{\rm corr}$ of 0·76. $w = 1/\sigma(F)$, where $\sigma(F) = [\sigma_c(F^2) + 1{\cdot}03F^2]^{1/2} - |F|$, where $\sigma_c(F^2)$ is the standard deviation of F^2. The final R* (on F) = 0·037, wR = 0·040, S = 1·282, $(\Delta/\sigma)_{\rm max} = 0{\cdot}15$, $\Delta\rho = -0{\cdot}6\,(1)$ to $0{\cdot}5\,(1)$ e Å^{-3}. Fractional coordinates are listed in Table 1, bond distances and angles in Table 2. Computations were carried out on a VAX 11/780 with the following programs: *INTEG* – integration; *DATAP* and *DSORTH* (State University of New York, Buffalo) – absorption correction and sorting; modified *ORFLS* (Busing, Martin & Levy, 1962) – least-squares refinement; *ORFFE* (Busing, Martin & Levy, 1964) – geometry and errors, *ORTEP* (Johnson, 1965) – drawings. Scattering curves: Cromer & Mann (1968) for Zn, I, N and C; Stewart, Davidson & Simpson (1965) for H; anomalous-dispersion corrections for Zn and I from Cromer & Liberman (1970).

Table 1. *Fractional coordinates* (× 10^5 *for* I *and* Zn, × 10^4 *for* H_2O, O *and* C), *equivalent isotropic thermal parameters* (Å^2 × 10^{-3}) *and the deviations* Δ (Å × 10^{-3}) *from the best planes through the* O *atoms*

	x	y	z	$U_{\rm eq}$*	Δ
I(1)	19636 (8)	31647 (5)	75549 (4)	73 (1)	
I(2)	72138 (9)	28977 (6)	86232 (4)	77 (1)	
Zn	46086 (13)	31728 (8)	96982 (6)	51 (1)	
H2O(1)	4894 (7)	4418 (4)	9287 (3)	66 (4)	
H2O(2)	4238 (9)	2168 (5)	9324 (4)	96 (6)	
H2O(3)	3777 (8)	6074 (5)	4651 (3)	81 (5)	
O(1)	−2014 (8)	5174 (6)	173 (4)	79 (5)	225 (8)
C(1)	−966 (13)	4920 (11)	921 (6)	85 (9)	−289 (13)
C(2)	−1377 (16)	3947 (10)	1120 (7)	94 (10)	348 (14)
O(2)	−2731 (10)	4041 (6)	1261 (4)	89 (6)	−221 (9)
C(3)	−3110 (19)	3175 (9)	1545 (7)	101 (10)	201 (15)
C(4)	−4652 (21)	3354 (11)	1552 (8)	112 (12)	−315 (17)
O(3)	−5832 (11)	3484 (6)	863 (4)	89 (6)	218 (9)
C(5)	−7313 (17)	3740 (9)	831 (9)	95 (11)	−312 (16)
C(6)	−8394 (15)	3917 (9)	32 (7)	89 (10)	341 (14)
O(4)	7971 (11)	5428 (6)	5056 (8)	111 (8)	−233 (11)
C(7)	7639 (28)	5898 (14)	4344 (17)	161 (22)	360 (27)
C(8)	6539 (35)	5287 (15)	3752 (14)	165 (23)	−229 (28)
O(5)	5040 (16)	5347 (7)	3688 (6)	108 (8)	244 (12)
C(9)	3865 (34)	4876 (14)	3048 (11)	151 (18)	−149 (25)
C(10)	2320 (37)	4967 (17)	2991 (13)	211 (21)	376 (28)
O(6)	2298 (13)	4426 (6)	3590 (6)	112 (7)	−232 (11)
C(11)	805 (27)	4505 (14)	3661 (13)	175 (18)	283 (24)
C(12)	975 (19)	4038 (12)	4339 (17)	181 (19)	−268 (23)

* $U_{\rm eq} = \frac{1}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

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

Symmetry code: (i) $-1-x$, $1-y$, $-z$; (ii) $1-x$, $1-y$, $1-z$.

Zn—I(1)	2·544 (1)	Zn—I(2)	2·542 (1)
Zn—H2O(1)	2·016 (6)	Zn—H2O(2)	1·987 (7)
O(1)—C(2)	1·43 (1)	O(4)—C(7)	1·47 (2)
O(1)—C(6$^{\rm i}$)	1·41 (1)	O(4)—C(12$^{\rm ii}$)	1·39 (2)
C(1)—C(2)	1·48 (2)	C(7)—C(8)	1·45 (3)
C(2)—O(2)	1·42 (2)	C(8)—O(5)	1·36 (2)
O(2)—C(3)	1·42 (1)	O(5)—C(9)	1·43 (2)
C(3)—C(4)	1·47 (2)	C(9)—C(10)	1·41 (4)
C(4)—O(3)	1·36 (2)	C(10)—O(6)	1·42 (3)
O(3)—C(5)	1·41 (1)	O(6)—C(11)	1·47 (3)
C(5)—C(6)	1·50 (2)	C(11)—C(12)	1·45 (3)

I(1)—Zn—I(2)	122·2 (1)	I(2)—Zn—H2O(1)	105·8 (2)
I(1)—Zn—H2O(1)	111·2 (2)	I(2)—Zn—H2O(2)	111·9 (2)
I(1)—Zn—H2O(2)	101·3 (2)	H2O(1)—Zn—H2O(2)	102·9 (3)
C(1)—O(1)—C(6$^{\rm i}$)	112 (1)	C(7)—O(4)—C(12$^{\rm ii}$)	113 (2)
O(1)—C(1)—C(2)	111 (1)	O(4)—C(7)—C(8)	109 (2)
C(1)—C(2)—O(2)	109 (1)	C(7)—C(8)—O(5)	112 (2)
C(2)—O(2)—C(3)	114 (1)	C(8)—O(5)—C(9)	115 (2)
O(2)—C(3)—C(4)	107 (1)	O(5)—C(9)—C(10)	113 (2)
C(3)—C(4)—O(3)	112 (1)	C(9)—C(10)—O(6)	106 (2)
C(4)—O(3)—C(5)	116 (1)	C(10)—O(6)—C(11)	114 (2)
O(3)—C(5)—C(6)	106 (1)	O(6)—C(11)—C(12)	110 (2)
C(5)—C(6)—O(1$^{\rm i}$)	108 (1)	C(11)—C(12)—O(4$^{\rm ii}$)	110 (2)

Discussion. The cell contains two crystallographically independent crown ether molecules (*A* and *B*) which are on symmetry centres. Both crown ether molecules have approximate D_{3d} symmetry. There is large anisotropic thermal motion for molecule *B* and its geometry is poorly determined. The molecules form stacks, *ABAB*..., along the **b** direction with the $ZnI_2(H_2O)_2$ tetrahedra and water molecules between the layers (Fig.

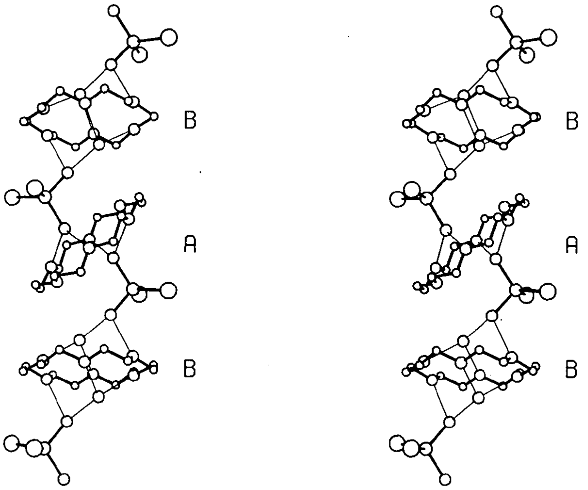

Fig. 1. A stereoscopic view of one of the stacks along **b**; the thin lines represent hydrogen bonds.

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* Lists of structure factors, anisotropic thermal parameters, H-atom coordinates, torsion angles and details of the hydrogen bonding have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44527 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

1), the water molecules apparently coming from the crown ether which was not completely dry. There is a strong hydrogen bond, O···O = 2·566 (9) Å, between the water of crystallization, H2O(3), and one of the waters, H2O(2), coordinated to the Zn atom. There are weak hydrogen bonds between the waters and the O atoms of the crowns linking the crowns together in the stacks. The O···O distances range from 2·79 (1) to 2·86 (1) Å which is rather longer than the 2·69 (5) Å found in triaquatetranitrothorium(IV)–18-crown-6 (Rogers, Kurihara & Benning, 1987). The angles at the O atoms involved range from 93 to 132°. This would appear to be the first $ZnI_2(H_2O)_2$ tetrahedron reported in the literature. The mean Zn—I distance, 2·543 (1) Å, is similar to that in other ZnI_2L_2 molecules, *e.g.* 2·552 (1) Å in diiodobis(pyridine)zinc (Le Querler, Borel & LeClaire, 1977), as is the large, 122·2 (5)°, I—Zn—I angle. The mean Zn—O distance is 2·004 (5) Å, which is in the range expected for tetrahedral zinc complexes (Brown & Lewis, 1984, and references therein).

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Structure of (2,2′-Bipyridyl)(*N*,*N*-carboxylatomethylanthranilato)chromium(III) Trihydrate

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Abstract. $[Cr(C_{11}H_8NO_6)(C_{10}H_8N_2)].3H_2O$, M_r = 512·4, monoclinic, $P2_1/n$, a = 9·679 (2), b = 12·831 (1), c = 17·770 (2) Å, β = 98·52 (1)°, V = 2182·60 Å^3, Z = 4, D_x = 1·556, D_m(by flotation) = 1·562 g cm^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, graphite monochromator, μ = 6·12 cm^{-1}, $F(000)$ = 1048, room temperature, R = 0·028 with unit weights for 2053 unique reflections. The two N atoms of bipyridyl and the N atom and the three carboxyl O atoms of the quadridentate anthranilic diacetate (atda) moiety give distorted octahedral coordination around Cr. The Cr—N(1) bond distance of the bipyridyl is shorter [2·037 (3) Å] than the other two Cr—N bond distances whereas the Cr—O distances are nearly equal [average 1·933 (3) Å]. The phenyl ring is effectively planar and the *o*-carboxyl group is rotated by 29·6 (2)°. The bipyridyl ring is twisted.

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