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The Crystal Structure of the *epi*-Inositol–Strontium Chloride Complex

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The crystal structure of the *epi*-inositol–strontium chloride complex, $C_6H_{12}O_6.SrCl_2.5H_2O$, has been determined by a three-dimensional X-ray analysis. The space group is $P2_12_12_1$, with $Z = 4$, $a = 13{\cdot}561$ (5), $b = 15{\cdot}763$ (5), $c = 7{\cdot}364$ (5) Å. Structure determination was by the Patterson method, and full-matrix least-squares refinement of the positional and thermal parameters yielded a final unweighted R value of 0·020. The Sr ion is coordinated, with a mean Sr···O distance of 2·63 (5) Å, to nine O atoms, three of the oxygens being from the axial–equatorial–axial sequence of hydroxyls in the *epi*-inositol molecule. The position of the Sr ion in relation to these ax.–eq.–ax. oxygens confirms the structure of the complex postulated from NMR results.

Introduction

Mills (1961) (using paper ionophoresis) found that *epi*-inositol, in supporting electrolytes containing acetates of the alkali and alkaline-earth metals, revealed considerable movement towards the cathode. This was interpreted as being due to the formation of a complex with the cation. The crystal structure of *epi*-inositol has been determined by Jeffrey & Kim (1971), and this study showed the presence of an axial–equatorial–axial sequence of three hydroxyl groups in the molecule. It was proposed by Angyal & Davies (1971) that the ax.–eq.–ax. sequence was the site of complex formation. Subsequent observation of the NMR spectra of various *epi*-inositol complexes with cations (Angyal & Hickman, 1975) tended to confirm this proposal; it was to verify the NMR results that the X-ray study of the *epi*-inositol–strontium chloride complex was carried out.

Experimental

Colourless, striated, needle-shaped crystals were obtained by crystallization of the complex from aqueous methanol (Angyal & Hickman, 1975). Because of the striations, the crystal was 'rounded' in a crystal spherizer to an approximately cylindrical shape of diameter 0·37 mm and length 0·41 mm.

The space group was determined from zero- and first-level Weissenberg photographs about **a** and **c** with Ni-filtered Cu $K\alpha$ radiation. The absence of reflexions

0*k*0 with *k* odd, 00*l* with *l* odd, and *h*00 with *h* odd indicated that the space group was $P2_12_12_1$.

The crystal density was measured by flotation in carbon tetrachloride and 1,2-dibromoethane. The crystal data are: a = 13·561 (5), b = 15·763 (5), c = 7·364 (5) Å, V = 1574·1 Å^3; $D_{\text{exp.}}$ = 1·82, $D_{\text{calc.}}$ = 1·81 g cm^{-3}, Z = 4.

The intensities of 1730 independent reflexions with $\theta < 70°$ were measured on a Siemens automatic single-crystal diffractometer with Ni-filtered Cu $K\alpha$ radiation and a θ–2θ scan. Lorentz, polarization and absorption corrections were applied to the data set.

Structure determination and refinement

A three-dimensional Patterson map revealed the positions of the Sr ion and one chloride ion. Subsequent Fourier maps revealed the remaining nonhydrogen atoms. With isotropic temperature factors, the nonhydrogen atoms were partially refined by block-diagonal least-squares refinement. Structure factors with $\theta > 30°$ were calculated and the resulting Fourier-difference map yielded the locations of the H atoms. Anomalous-dispersion and secondary-extinction corrections were applied and refinement was completed with full-matrix least squares, the nonhydrogen atoms being given anisotropic, and the H atoms, isotropic temperature factors. The final unweighted R value was 0·020.*

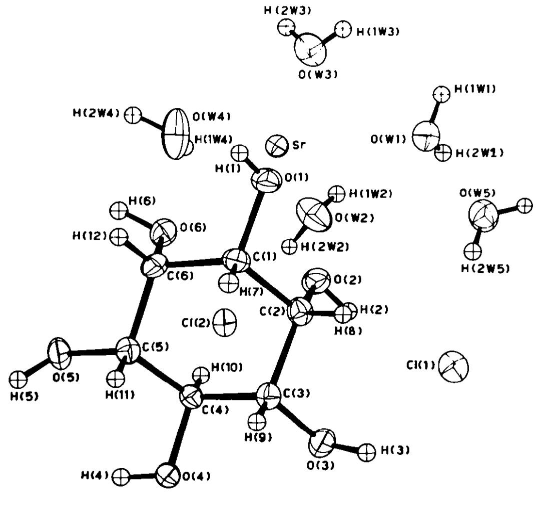

Fig. 1. The asymmetric unit for the *epi*-inositol–strontium chloride complex viewed parallel to −**a**.

The atomic positional parameters are given in Table 1. The asymmetric unit, as seen parallel to −**a**, is shown in Fig. 1, and projections of the structure on to the *ab* plane are shown in Fig. 2(*a*) and (*b*). The bond lengths, bond angles and torsional angles are given in Table 2.

Discussion

Each Sr ion is coordinated to nine O atoms comprising: O(*W*1), O(*W*2), O(*W*3), O(*W*4); O(1), O(2) and O(6) – which constitute the ax.–eq.–ax. sequence – from one *epi*-inositol molecule; and O(3) and O(4) from another molecule which corresponds to the previous molecule displaced one unit cell in the +**z** direction.

Table 1. *Fractional atomic coordinates* (×10^4 *for non-hydrogen atoms,* ×10^3 *for* H)

The estimated standard deviations are given in parentheses.

	x	y	z
Sr	1325 (0)	3228 (0)	5662 (0)
Cl(1)	1957 (1)	1149 (1)	141 (1)
Cl(2)	3931 (1)	3924 (0)	974 (1)
C(1)	−633 (2)	3590 (2)	2901 (5)
C(2)	−155 (2)	2927 (2)	1696 (5)
C(3)	−162 (2)	3248 (2)	−274 (4)
C(4)	369 (2)	4098 (2)	−406 (4)
C(5)	−166 (2)	4752 (2)	742 (5)
C(6)	−185 (2)	4482 (2)	2735 (4)
O(1)	−576 (2)	3294 (2)	4737 (3)
O(2)	822 (2)	2762 (2)	2343 (3)
O(3)	314 (2)	2669 (1)	−1460 (4)
O(4)	373 (2)	4334 (2)	−2284 (3)
O(5)	324 (2)	5548 (1)	587 (4)
O(6)	790 (2)	4469 (1)	3479 (3)
O(*W*1)	1296 (2)	1531 (2)	5943 (4)
O(*W*2)	2888 (2)	2810 (2)	3961 (5)
O(*W*3)	2516 (2)	2890 (2)	8274 (4)
O(*W*4)	2553 (2)	4476 (2)	6027 (7)
O(*W*5)	2847 (2)	763 (2)	3984 (4)
H(1)	−89 (4)	357 (3)	535 (8)
H(2)	100 (5)	239 (4)	161 (9)
H(3)	−15 (4)	218 (4)	−157 (9)
H(4)	62 (4)	487 (4)	−241 (7)
H(5)	0 (3)	595 (3)	2 (7)
H(6)	85 (4)	497 (4)	401 (8)
H(7)	−138 (3)	363 (2)	244 (5)
H(8)	−51 (3)	246 (3)	169 (6)
H(9)	−83 (3)	334 (2)	−73 (5)
H(10)	98 (3)	404 (2)	1 (6)
H(11)	−87 (3)	481 (2)	21 (6)
H(12)	−62 (3)	482 (2)	341 (6)
H(1*W*1)	132 (4)	136 (4)	699 (8)
H(2*W*1)	163 (5)	132 (4)	556 (10)
H(1*W*2)	335 (5)	254 (4)	452 (9)
H(2*W*2)	322 (5)	311 (5)	310 (12)
H(1*W*3)	245 (4)	250 (4)	880 (8)
H(2*W*3)	289 (4)	319 (4)	892 (8)
H(1*W*4)	306 (4)	441 (4)	572 (9)
H(2*W*4)	239 (4)	497 (4)	650 (7)
H(1*W*5)	291 (5)	26 (5)	425 (11)
H(2*W*5)	267 (5)	90 (4)	304 (10)

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* Lists of structure factors and thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32447 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

[Note that the numbering of *epi*-inositol used in this paper is the same as that employed by Jeffrey & Kim (1971), in order to allow direct comparison of our results with theirs. This numbering is not in accordance with the accepted system of cyclitol nomenclature (IUPAC-IUB Commission on Biochemical Nomenclature, 1974).]

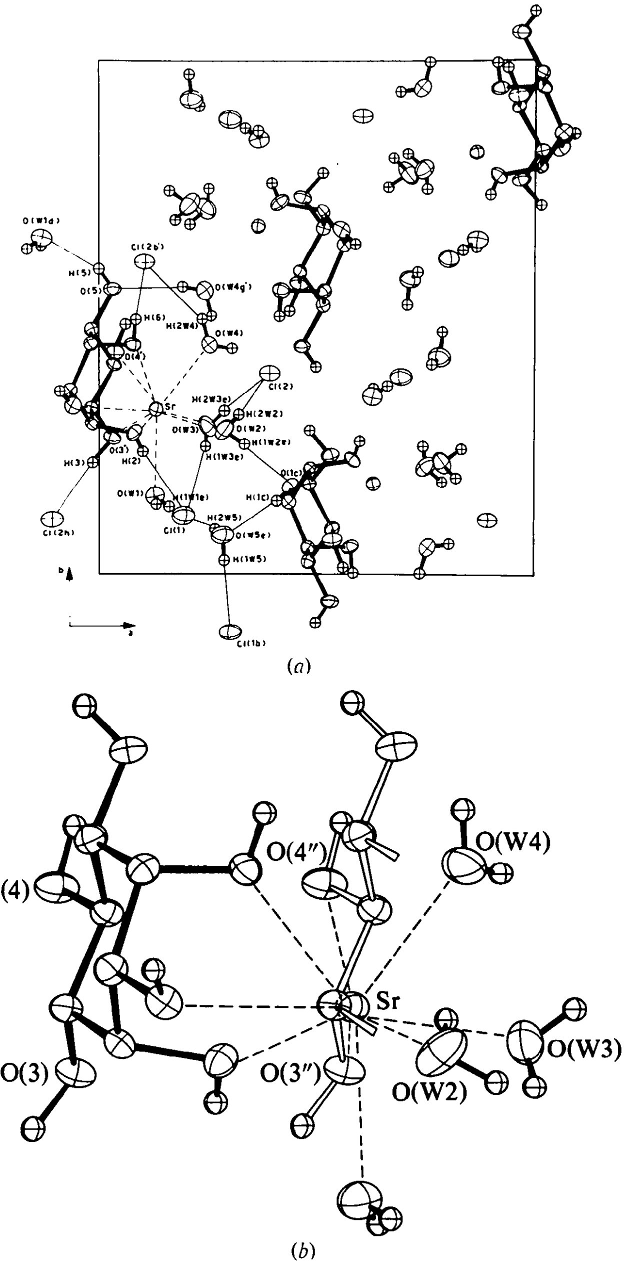

Fig. 2. (*a*) The crystal structure projected on to the *ab* plane. The solid lines represent hydrogen bonds and the dotted lines show Sr ion coordination. [Note: ring hydrogens are not included. The molecule containing O(3″) and O(4″) is displaced one unit cell along +**c**.] (*b*) Projection on to the *ab* plane of parts of the two overlying cells of Fig. 2(*a*), rotated 30° anticlockwise about **b**, with the unshaded bonds relating to the upper cell.

The mean Sr···O distance is 2·63 (5) Å, the individual distances being given in Table 3. The *epi*-inositol molecule in the complex has the chair conformation found by Jeffrey & Kim for the free *epi*-inositol molecule, with atoms C(2), C(3), C(5) and C(6) being less than 0·01 Å from the best least-squares plane, and

Table 2. *Bond lengths, bond angles and torsional angles*

The estimated standard deviations are given in parentheses and refer to the last decimal positions. The mean values and their estimated standard deviations are calculated from:

$$\bar{l} = \sum_{i=1}^{N} \frac{l_i}{\sigma_i^2} \Big/ \sum_{i=1}^{N} \frac{1}{\sigma_i^2}\ ; \sigma_{\text{mean}} = [\sum_{i=1}^{N} (\bar{l} - l_i)^2/(N-1)]^{1/2}.$$

$\bar{l}$(C–C) = 1·527 (8) Å ∠(C–C–C) = 109·9 (2·2)°
$\bar{l}$(C–O) = 1·429 (5) ∠(C–C–O) = 109·2 (1·7)

Bond lengths (Å)

i	*j*	*D*(*ij*)	*i*	*j*	*D*(*ij*)
C(1)	C(2)	1·517 (5)	O(6)	H(6)	0·88 (6)
C(2)	C(3)	1·536 (4)	C(1)	H(7)	1·07 (4)
C(3)	C(4)	1·524 (4)	C(2)	H(8)	0·87 (4)
C(4)	C(5)	1·518 (4)	C(3)	H(9)	0·97 (4)
C(5)	C(6)	1·529 (5)	C(4)	H(10)	0·89 (4)
C(6)	C(1)	1·535 (4)	C(5)	H(11)	1·03 (4)
C(1)	O(1)	1·433 (4)	C(6)	H(12)	0·94 (4)
C(2)	O(2)	1·432 (4)	O(*W*1)	H(1*W*1)	0·81 (6)
C(3)	O(3)	1·418 (4)	O(*W*1)	H(2*W*1)	0·63 (7)
C(4)	O(4)	1·432 (4)	O(*W*2)	H(1*W*2)	0·86 (6)
C(5)	O(5)	1·425 (4)	O(*W*2)	H(2*W*2)	0·91 (8)
C(6)	O(6)	1·431 (4)	O(*W*3)	H(1*W*3)	0·74 (6)
O(1)	H(1)	0·76 (6)	O(*W*3)	H(2*W*3)	0·84 (6)
O(2)	H(2)	0·83 (7)	O(*W*4)	H(1*W*4)	0·74 (6)
O(3)	H(3)	0·99 (6)	O(*W*4)	H(2*W*4)	0·89 (5)
O(4)	H(4)	0·92 (5)	O(*W*5)	H(1*W*5)	0·81 (8)
O(5)	H(5)	0·87 (5)	O(*W*5)	H(2*W*5)	0·76 (8)

Bond angles (°)

i	*j*	*k*	∠(*ijk*)
C(1)	C(2)	C(3)	108·8 (2)
C(2)	C(3)	C(4)	110·3 (2)
C(3)	C(4)	C(5)	109·6 (2)
C(4)	C(5)	C(6)	110·7 (2)
C(5)	C(6)	C(1)	109·8 (3)
C(6)	C(1)	C(2)	114·5 (3)
C(2)	C(1)	O(1)	107·7 (3)
C(6)	C(1)	O(1)	110·6 (3)
C(1)	C(2)	O(2)	109·0 (3)
C(3)	C(2)	O(2)	112·3 (3)
C(2)	C(3)	O(3)	111·5 (3)
C(4)	C(3)	O(3)	108·1 (2)
C(3)	C(4)	O(4)	107·0 (2)
C(5)	C(4)	O(4)	111·3 (3)
C(4)	C(5)	O(5)	109·3 (3)
C(6)	C(5)	O(5)	109·3 (3)
C(1)	C(6)	O(6)	108·8 (3)
C(5)	C(6)	O(6)	110·8 (3)
H(1*W*1)	O(*W*1)	H(2*W*1)	103 (7)
H(1*W*2)	O(*W*2)	H(2*W*2)	103 (6)
H(1*W*3)	O(*W*3)	H(2*W*3)	104 (6)
H(1*W*4)	O(*W*4)	H(2*W*4)	119 (6)
H(1*W*5)	O(*W*5)	H(2*W*5)	121 (7)

Table 2 (*cont.*)

Torsional angles (°)

i	*j*	*k*	*l*	(*ijk*)^(*jkl*)
C(6)	C(1)	C(2)	C(3)	−53·8 (3)
C(1)	C(2)	C(3)	C(4)	57·4 (3)
C(2)	C(3)	C(4)	C(5)	−61·9 (3)
C(3)	C(4)	C(5)	C(6)	60·5 (3)
C(4)	C(5)	C(6)	C(1)	−54·8 (3)
C(5)	C(6)	C(1)	C(2)	52·8 (4)
O(1)	C(1)	C(2)	O(2)	−54·5 (3)
C(1)	C(2)	O(2)	C(3)	59·3 (3)
O(2)	C(2)	C(3)	O(3)	56·7 (3)
C(2)	C(3)	O(3)	C(4)	58·6 (3)
O(3)	C(3)	C(4)	O(4)	55·1 (3)
C(3)	C(4)	O(4)	C(5)	−60·3 (3)
O(4)	C(4)	C(5)	O(5)	−60·8 (3)
C(4)	C(5)	O(5)	C(6)	58·7 (3)
O(5)	C(5)	C(6)	O(6)	−55·0 (3)
C(5)	C(6)	O(6)	C(1)	59·2 (4)
O(6)	C(6)	C(1)	O(1)	53·4 (3)

the out-of-plane carbons, C(1) and C(4), −0·63 and 0·72 Å from the plane respectively. The mean C—C bond length of 1·527 (8) Å and the mean C—O bond length of 1·429 (5) Å are the same as those of the free *epi*-inositol molecule (to within 0·001 Å). There are, however, some variations in the intramolecular O···O distances. In particular, the distances between O(1), O(2) and O(6), which constitute the ax.–eq.–ax. sequence, are shorter by 0·13–0·16 Å. The distance between the two axial O atoms has been reduced from 2·96 to 2·82 Å. The distance between O(3) and O(4), which are coordinated to the same Sr ion, is also shorter than in *epi*-inositol. These results show that coordination with a cation reduces the non-bonded interactions between the participating atoms and thereby stabilizes the complex.

There is extensive hydrogen bonding throughout the crystal. The hydrogen-bond distances and angles are given in Table 3 and the bonding pattern is shown in Fig. 2(*a*) and (*b*).

Each chloride ion is involved in hydrogen bonding with five atoms: Cl(1) is bonded to OH(2) and four water molecules; Cl(2) is bonded to OH(3), OH(6) and three water molecules.

The *epi*-inositol molecule itself is hydrogen bonded through OH(1) to two water molecules and through OH(5) to another two water molecules.

In terms of the water molecules, O(*W*1), O(*W*2), O(*W*3) and O(*W*4) (as well as being coordinated to the Sr ion) are each involved in two hydrogen bonds, and O(*W*5) is involved in three hydrogen bonds.

Table 3. *Hydrogen-bond distances and angles in the epi-inositol–strontium chloride complex*

The estimated standard deviations in parentheses refer to the last decimal positions.

i	*j*	*k*	*D*(*ij*)	*D*(*ik*)	∠(*ikj*)
O(1*c*)	O(*W*2*e*)	H(1*W*2*e*)	2·879 (4) Å	2·04 (6) Å	165 (6)°
O(1*c*)	O(*W*5*e*)	H(1*c*)	2·769 (4)	2·06 (6)	154 (6)
O(5)	O(*W*1*d*)	H(5)	2·914 (4)	2·11 (5)	154 (4)
O(5*f*)	O(*W*4*g*)	H(1*W*4*g*)	2·897 (4)	2·19 (6)	161 (7)
Cl(1)	O(2)	H(2)	3·386 (3)	2·59 (7)	161 (6)
Cl(1)	O(*W*1*e*)	H(1*W*1*e*)	3·274 (3)	2·50 (6)	159 (5)
Cl(1)	O(*W*3*e*)	H(1*W*3*e*)	3·162 (4)	2·44 (6)	169 (5)
Cl(1)	O(*W*5)	H(2*W*5)	3·136 (3)	2·38 (8)	172 (6)
Cl(1*b*)	O(*W*5)	H(1*W*5)	3·143 (3)	2·33 (8)	177 (8)
Cl(2)	O(*W*2)	H(2*W*2)	3·149 (3)	2·24 (8)	175 (7)
Cl(2)	O(*W*3*e*)	H(2*W*3*e*)	3·208 (3)	2·37 (6)	174 (5)
Cl(2*b*)	O(6*f*)	H(6*f*)	3·153 (2)	2·28 (6)	167 (5)
Cl(2*b*)	O(*W*4*f*)	H(2*W*4*f*)	3·228 (3)	2·52 (6)	137 (4)
Cl(2*h*)	O(3)	H(3)	3·154 (3)	2·19 (6)	163 (5)

Sr···O distances

i	*j*	*D*(*ij*)
Sr	O(1)	2·669 (3) Å
Sr	O(2)	2·642 (3)
Sr(*e*)	O(3)	2·674 (3)
Sr(*e*)	O(4)	2·645 (3)
Sr	O(6)	2·634 (2)
Sr	O(*W*1)	2·684 (3)
Sr	O(*W*2)	2·548 (3)
Sr	O(*W*3)	2·568 (3)
Sr	O(*W*4)	2·591 (3)

Non-bonded O···O distances less than 3·2 Å

i	*j*	*D*(*ij*)
O(1)	O(2)	2·722 (4) Å
O(1)	O(6)	2·779 (3)
O(2)	O(3)	2·887 (4)
O(2)	O(6)	2·819 (4)
O(3)	O(4)	2·694 (3)
O(4)	O(5)	2·852 (4)
O(5)	O(6)	2·798 (4)

Symmetry code

(*a*)	x,	y,	z
(*b*)	$\frac{1}{2}-x$,	$-y$,	$\frac{1}{2}+z$
(*b*′)	$\frac{1}{2}-x$,	$1-y$,	$\frac{1}{2}+z$
(*c*)	$\frac{1}{2}+x$,	$\frac{1}{2}\ y$,	$-z$
(*d*)	$-x$,	$\frac{1}{2}+y$,	$\frac{1}{2}-z$
(*e*)	x,	y,	$-1+z$
(*f*)	x,	$-1+y$,	z
(*g*)	$\frac{1}{2}-x$,	$-y$,	$-\frac{1}{2}+z$
(*g*′)	$\frac{1}{2}-x$,	$1-y$,	$-\frac{1}{2}+z$
(*h*)	$-\frac{1}{2}+x$,	$\frac{1}{2}-y$,	$-z$
(″)	x,	y,	$1+z$

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