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Crystal Structure of Tetrakis(diacetamide) monoaquostrontium(II) Perchlorate

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A new compound, tetrakis(diacetamide)monoaquostrontium(II) perchlorate, $Sr(CH_3CONHCOCH_3)_4H_2O(ClO_4)_2$ has been synthesized and its structure determined from three dimensional X-ray diffraction data using monochromatic CuKa radiation. The unit cell is monoclinic, space group $P2_1/m$ with dimensions $a = 8.642(.9)$Å, $b = 21.833(2)$, $c = 8.652(.9)$, $\beta = 78°41'$ (1') and $Z = 2$. Three dimensional intensity data were collected on the Enraf Nonius CAD-4 automated diffractometer. The crystal structure was determined by the heavy atom method and refined anisotropically by block diagonal least squares to a conventional unweighted R-factor of 0.083.

The complex forms the nine-coordinate monocapped square antiprism ($C4_v$), rather than the lower energy symmetrically tricapped trigonal prism ($D3_h$). Eight of the coordinated oxygens are contributed by the four bidentate diacetamides and the ninth by the water. Two of the diacetamides bridge the equatorial planes while a third spans an edge of one equatorial plane and the fourth bridges the cap position and an equatorial plane. Two types of hydrogen bonding are present: (1) O–H···O between water and perchlorate and (2) N–H···O between amide and perchlorate. Strontium–oxygen bond distances fall within the range 2.54Å to 2.69Å.

Introduction

Based on stoichiometry and infrared studies, Gentile and Shankoff[1] reported a number of nine-coordinate complexes for the alkaline earth metals with multidentate ligands such as diacetamide (DA). One of the compounds, a barium complex, $[Ba(DA)_5](ClO_4)_2$, pentakis(diacetamide)barium(II) perchlorate, was later found[2] by X-ray crystallographic studies to have a higher coordination number (CN 10) than postulated. As a result, it was felt that $[Sr(DA)_4(H_2O)_2](ClO_4)_2$, tetrakis(diacetamide)diaquostrontium(II) perchlorate, might also be ten-coordinate which would be the highest coordination yet found for a strontium complex.

A three-dimensional X-ray analysis would, in addition to defining the CN unequivocally, yield structural parameters that could be used for better interpretation of infrared data on other diacetamide complexes.

Experimental

At the outset it was desired to prepare the $[Sr(DA)_4(H_2O)_2](ClO_4)_2$ complex as described in the literature.[1] However, the preparation yielded crystals unsuitable for X-ray analysis. While acetone was used as the solvent in the original preparation, methanol was substituted to decrease the rate of evaporation and allow larger crystals to form. This led to the formation of the monoaquo complex which has not previously been reported.

Preparation

Diacetamide was prepared by the acylation of acetamide with acetic anhydride as described by Davidson and Kartan,[3] and recrystallized from ethyl ether just prior to use. Tetrakis(diacetamide)monoaquostrontium(II) perchlorate was prepared by dissolving anhydrous strontium perchlorate and recrystallized diacetamide in stoichiometric quantities and twice the stoichiometric weight of water in the minimum amount of pure methyl alcohol. Upon very slow (8–10 days) evaporation of the solvent, small, colorless, well formed crystals of the complex were obtained. The crystals, immersed in their mother liquor, were sealed in quartz capillaries to avoid dissolution due to atmospheric moisture.

Crystallographie

A section of a single crystal was used for the determination of lattice parameters as well as the collection of intensity data. The crystal was mounted with its long axis [010] parallel to the goniometer head spindle. Another section of the same crystal was mounted with its long axis normal to the goniometer head spindle. Unit cell parameters were first determined from zero level Weissenberg photographs about the *a*-axis and *b*-axis at

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room temperature (25 ± 2 °C) with nickel filtered copper Kα radiation. The photographically determined lattice parameters were verified on an Enraf–Nonius CAD-4 computer controlled diffractometer with graphite monochromatized copper Kα radiation. Based on crystal volume and measured density (by the flotation technique) it was determined that there were two molecules per unit cell. All crystal data are presented in Table I.

A series of Weissenberg photographs including $h0l$, $h1l$, $0kl$ and $1kl$ were taken to determine the space group. None of the photographs about the b-axis showed any systematic absences. However, the $0k0$ reflections, with k odd, were absent on the $0kl$ film, but the $1kl$ film showed no absences indicating a 2-fold screw along the b-axis. Oscillation photographs showed the crystals to belong to the monoclinic system, therefore limiting the choice of space group to the non-centrosymmetric $P2_1$ or the centrosymmetric $P2_1/m$. These two space groups exhibit the same extinctions and cannot be differentiated by examination of the preliminary data.[4–6]

Intensity data were collected on the CAD-4 using two scans made through each reflection: a prescan to determine the speed with which the measuring scan should be made, and the actual measuring scan. During the prescan, if a statistical difference was not detected between the background count and the count for the calculated position of the reflection, the measuring scan was not carried out. For reflections with a net count statistically above background, two sweeps were made at the precalculated speed using the ω–2Θ scan. The scan range and the width of the receiving aperture were calculated as a function of Θ. The standard reflection was measured every 30 minutes. A total of 3031 reflections were collected within a Θ angle of 71° of which 32 percent were considered unobserved by the computer's statistical test. The reflections thus collected were corrected for Lorentz, polarization[7] and absorption errors. The absorption corrections were applied to the intensity and structure factor data using the m-point Gaussian integration formula.[8]

TABLE I. Crystallographic Data for $Sr(DA)_4H_2O(ClO_4)_2$.

M.W. = 708.96
Crystallographic System: Monoclinic
Space Group: $P2_1/m$
Unit Cell Parameters:
a = 8.642(.9) Å
b = 21.833(2) Å
c = 8.652(.9) Å
β = 78.685°(7)
V = 1600.783 Å^3
Z = 2
$d_{calcd.}$ = 1.471 gm/cm^{-3} F(000) = 362
$d_{obs.}$ = 1.528 gm/cm^{-3} λ = 1.54051 Å
$\mu_{CuK\alpha}$ = 47.56 cm^{-1}

Solution and Refinement

The strontium atom provided initial phases for obtaining the solution of the structure utilizing the heavy atom technique.

Since unit cell parameters indicated two molecules per unit cell, the non-centrosymmetric space group, $P2_1$, which has two asymmetric units per cell would require that each molecule be an asymmetric unit. However, if each molecule were centered on a special position of $P2_1/m$, two asymmetric units per molecule, or four asymmetric units per cell, would be generated.

The key to the resolution of the $P2_1$–$P2_1/m$ space group ambiguity was to be found in either the confirmation of a true or a pseudo mirror plane indicating $P2_1/m$ or $P2_1$ respectively. A sharpened, three dimensional Patterson map was calculated[9] from which the x/a and z/c coordinates of the strontium were determined. Since there was no natural origin on the b-axis in the $P2_1$ space group, the y/b coordinate was arbitrarily chosen as 1/4, which would place the strontium atom directly on the mirror plane of $P2_1/m$.

A 3-dimensional Fourier map was generated from the phases of the strontium atom coordinates which had been refined by three cycles of block diagonal least squares.[10] The unweighted residual, R, defined as $\Sigma(|F_o|-k|F_c|)/\Sigma|F_o|$, was 0,41 at this point. An examination of the electron density maxima of the map confirmed the mirror plane as a true mirror and hence, the $P2_1/m$ space group. Ten peaks of high density were located on this map which, when subjected to a bond distance and angle analysis, were identified as follows: three carbon atoms and a nitrogen atom of a single diacetamide group, a chlorine atom and two oxygen atoms of a perchlorate group and three oxygen atoms within the strontium coordination sphere.

Using an overall temperature factor, B of 3.97 (determined by a Wilson Plot[11]), structure factors were calculated giving an R-factor of 0.37. A new Fourier map was generated from which nine more atoms were identified and all the important features of the structure became apparent. A third Fourier map revealed the positions of the remaining symmetry independent atoms giving an R of 0.33. After five cycles of isotropic, block diagonal least squares, the R was 0.21. Ten further cycles of anisotropic least squares reduced the R to 0.083 and a difference electron density map showed no spurious peaks.

Mean atomic scattering factors were corrected for both real and imaginary dispersion effects.[12] The quantity minimized in the block diagonal least squares was $(\Sigma|F_o|-\Sigma|F_c|)^2$. The R rose to 0.11 when a weighting scheme based on counting statistics was attempted. To avoid wide oscillations in parameter shifts common to block diagonal least squares, a damping factor was applied as described by Hodgson and Rollett,[13] reducing the number of refinement cycles required. Unobserved

Table II. Fractional Coordinates of $Sr(C10_4)_2 \cdot 4DA \cdot H_2O$.

	Atom	x/a	y/b	z/c
	Sr	0.2052(2)[a]	0.2500(–)[b]	0.2376(2)
DA_1				
	N_1	0.3505(13)	0.0774(5)	0.2269(14)
	C_{11}	0.6170(17)	0.0931(7)	0.2635(20)
	C_{12}	0.4634(14)	0.1206(6)	0.2333(13)
	C_{13}	0.1097(24)	0.0325(8)	0.1734(37)
	C_{14}	0.2024(18)	0.0913(7)	0.1838(19)
	O_2	0.1538(10)	0.1402(4)	0.1631(11)
	O_9	0.4459(8)	0.1753(3)	0.2226(9)
DA_4				
	N_{1*}[c]	0.3505(13)	0.4225(5)	0.2269(14)
	C_{11*}	0.6170(17)	0.4069(7)	0.2635(20)
	C_{12*}	0.4634(14)	0.3793(6)	0.2333(13)
	C_{13*}	0.1097(24)	0.4674(8)	0.1734(37)
	C_{14*}	0.2025(18)	0.4086(7)	0.1838(19)
	$O_{2*}(O_4)$[d]	0.1538(10)	0.3597(4)	0.1631(11)
	$O_{9*}(O_8)$	0.4459(8)	0.3246(3)	0.2226(9)
DA_2				
	N_2	–0.2554(13)	0.2500(–)	0.2282(14)
	C_{21}	0.6473(19)	0.2500(–)	0.5105(18)
	C_{22}	–0.2466(21)	0.2500(–)	–0.0466(18)
	C_{23}	0.8408(17)	0.2500(–)	0.0793(16)
	C_{24}	0.7805(17)	0.2500(–)	0.3740(16)
	O_1	–0.0170(11)	0.2500(–)	0.0588(11)
	O_3	–0.0785(13)	0.2500(–)	0.3917(12)
DA_3				
	N_3	0.1251(16)	0.2500(–)	0.7071(14)
	C_{31}	0.1059(20)	0.1403(7)	0.7463(15)
	C_{32*}	0.1465(13)	0.3053(6)	0.6340(13)
	O_6	0.1904(11)	0.1874(4)	0.4945(8)
	$O_{6*}(O_7)$	0.1904(11)	0.3126(4)	0.4945(8)
H_2O				
	O_5	0.3484(12)	0.2500(–)	–0.0565(11)
ClO_4				
	Cl_1	0.5700(6)	0.0947(2)	0.7714(5)
	O_{11}	0.4902(19)	0.1451(5)	0.7886(23)
	O_{12}	0.6755(29)	0.0885(14)	0.6568(18)
	O_{13}	0.6149(44)	0.0820(17)	0.8942(21)
	O_{14}	0.5094(43)	0.0488(11)	0.7717(63)
ClO_4				
	Cl_{1*}	0.5700(6)	0.4052(2)	0.7714(5)
	O_{11*}	0.4902(19)	0.3548(5)	0.7886(23)
	O_{12*}	0.6755(29)	0.4114(14)	0.6568(18)
	O_{13*}	0.6149(44)	0.4179(17)	0.8942(21)
	O_{14*}	0.5094(43)	0.4511(11)	0.7717(63)

[a] The quantities enclosed in brackets are the estimated standard deviations (x 10^4). [b] (–) indicates an atom lying on the special position x, $^1/_4$, z so that the y coordinate is determined by definition. [c] * indicates an atom generated by reflection across the mirror plane at y = $^1/_4$. [d] Atom numbers in parentheses indicate numbering used in accordance with the numbering rules for coordination polyhedra.

reflections were not included in the least squares refinement. Observed and calculated structure amplitudes are available from the Editor on request and fractional coordinates are presented in Table II. Numbering of coordinated oxygens (Fig. 1) conforms to the rules for numbering coordination polyhedra.[15]

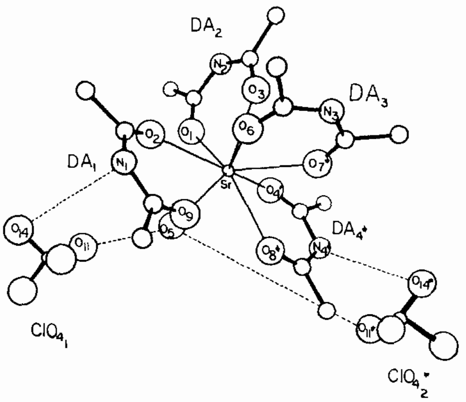

Figure 1. Three-dimensional view of the asymmetric unit[14].

Discussion

Based on the radius ratio test[16] a strontium–oxygen complex can reasonably be expected to exhibit nine and possibly ten-coordination. The strontium complex, tetrakis(diacetamide)monoaquostrontium(II) perchlorate is a nine-coordinate chelate with eight of the coordinated oxygen atoms contributed by the four bidentate diacetamide ligands and the ninth coordinated oxygen atom contributed by the water. This strontium complex is unique in that it is formed, in part, with four bidentate ligands and the coordinated oxygen atoms lie at the verteces of a monocapped square antiprism (MSAP) ($C4_v$) rather than the lower energy[15] tricapped trigonal (TCTP) ($D3_h$). Figure 2 shows a schematic diagram of the MSAP.

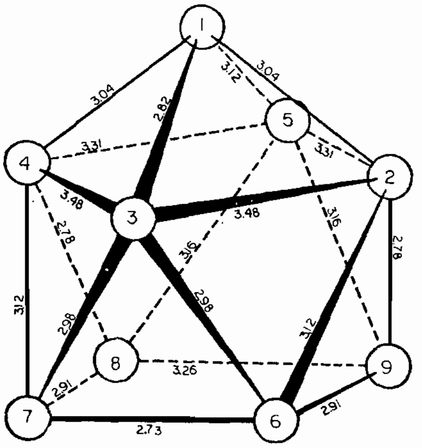

Figure 2. The idealised monocapped square antiprism (MSAP). The numbers refer to coordinated oxygen atoms and are in accordance with the rules for numbering coordination polyhedra[15].

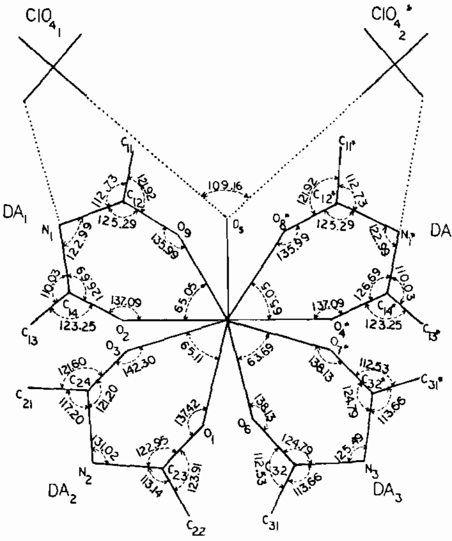

Figure 3. Bond angles (degrees).

With reference to Figures 1 and 2, a mirror plane is seen to include the water oxygen at position 5 and DA_2 which spans the 1–3 positions. DA_1 and DA_4 which are mirror images bridge positions 2–9 and 4–8 respectively. The mirror also bisects DA_3 which spans positions 6–7. Equatorial plane-2, O(6)–O(7)–O(8)–O(9), bound by symmetry constraints is perfectly planar, while equatorial plane-2, O(2)–O(3)–O(4)–O(5), shows a slight deviation from planarity (r.m.s. deviation = 5×10^{-2}Å). The dihedral angle between equatorial plane-1 and equatorial plane-2 is 0.43° which is within experimental error. The bridging from equatorial plane-1 to equatorial plane-2 of DA_1 and DA_4 is an unusual feature of the structure. Hoard and Silverton[19] in their discussion of the stereochemistry of eight-coordinate complexes and the geometry of the square antiprism, which is related to the MSAP, stated that all ligands should either span (111) the equatorial planes or be associated with only one plane (sss). A hybrid, that is, some ligands bridging and some not (11s) is not to be favored when dealing with chemically identical chelating groups.

The coordination polyhedron of the $[Ba(DA)_5](ClO_4)_2$ complex is the symmetrically bicapped square antiprism (SBSA) and is related to the MSAP of the strontium complex. In the barium complex, the bite of the five bidentate diacetamide ligands leads to distortion of the SBSA, whereas the presence of the water in an equatorial position of the strontium complex probably relieves strain resulting in less distortion of the geometry of the MSAP. Judging from structural data of other nine-coordinate compounds with monodentate ligands only, few restrictions are left and the formation of the TCTP polyhedron, which is at a lower energy level, is favored.

TABLE III. Bond Distances in $Sr(ClO_4)_2 \cdot 4DA \cdot H_2O$.

	Atoms	D(Å)		Atoms	D(Å)
DA_1			DA_2		
	$Sr–O_2$	2.544(08)[a]		$Sr–O_1$	2.692(10)
	$Sr–O_9$	2.626(07)		$Sr–O_3$	2.552(11)
	$O_2–C_{14}$	1.173(17)		$O_1–C_{23}$	1.206(18)
	$C_{14}–C_{13}(Me)$	1.526(24)		$C_{23}–C_{22}$	1.443(22)
	$O_9–C_{12}$	1.208(14)		$O_3–C_{24}$	1.256(19)
	$C_{12}–C_{11}(Me)$	1.526(19)		$C_{24}–C_{21}(Me)$	1.479(22)
	$N_1–C_{14}$	1.434(19)		$N_2–C_{23}$	1.390(18)
	$N_1–C_{12}$	1.366(16)		$N_2–C_{24}$	1.357(19)
DA_3			DA_4		
	$Sr–O_6$	2.591(08)		mirror image of DA_1	
	$Sr–O_7+$[b]	2.591(08)			
	$O_6–C_{32}$	1.203(13)			
	$C_{32}–C_{31}(Me)$	1.529(19)			
	$O_7+–C_{32}+$	1.203(13)			
	$C_{32}+–C_{31}+$	1.529(19)			
	$N_3–C_{32}$	1.360(14)			
	$N_3–C_{32}+$	1.360(14)			
H_2O					
	$Sr–O_5$	2.602(09)			
ClO_4					
	$Cl–O_{11}$	1.290(14)			
	$Cl–O_{12}$	1.215(20)			
	$Cl–O_{13}$	1.233(26)			
	$Cl–O_{14}$	1.131(29)			

[a] The quantities enclosed in parentheses are the estimated standard deviations (x 10^3). [b] + indicates an atom generated by reflection through the mirror plane at y = $^1/_4$.

TABLE IV. Intra Group Dihedral Angles (Planes Defined by Three Atoms) in $Sr(ClO_4)_2 \cdot 4DA \cdot H_2O$.

	Plane 1	Plane 2	Angle (°)
DA_1			
	$O_2–C_{14}–N_1$	$O_9–C_{12}–N_1$	7.99
DA_2			
	$O_1–C_{23}–N_2$	$O_3–C_{24}–N_2$	0.0
DA_3			
	$O_6–C_{32}–N_3$	$O_7+–C_{32}+–N_3$	1.34
DA_4+[a]			
	$O_8+–C_{12}+–N_1+$	$O_4+–C_{14}+–N_1+$	7.99
ClO_4			
	$O_{11}–Cl_1–O_{12}$	$O_{13}–Cl_1–O_{14}$	98.46

[a] + indicates group or atom generated by morror plane at y = $^1/_4$.

TABLE V. Hydrogen Bond Distances and Angles in $Sr(ClO_4)_2 \cdot 4DA \cdot H_2O$.

Type O–H···O			
H_2O	ClO_4	D(Å)	Angle[b]
O_5	O_{11}	2.810(15)[a]	109.16°
O_5	$O_{11}+$[c]	2.810(15)	
Type N–H···O			
ClO_4	DA	D(Å)	
O_{14}	N_1	3.012(53)	
O_{14}	N_1+	3.012(53)	

[a] The values in parentheses are the estimated standard deviations (x 10^3). [b] $O_{11}–O_5–O_{11}+$. [c] + indicates atom generated by reflection across mirror at y = $^1/_4$.

The unstable nature of the complex and its susceptibility to decomposition by atmospheric moisture indicated weak metal to oxygen bonding. This is supported by an examination of strontium–oxygen bond distances (2.54Å–2.69Å) which are slightly greater than the sum of the ionic radii (Sr^{+2} = 1.13Å, O^{-2} = 1.40Å).[20] The data are show in Table III. The strong C–O bond of the diacetamide indicated by a short average C–O bond dis-

TABLE VI. Anisotropic Thermal Parameters as Mean Square Amplitudes of Vibration[a] (Å^2) for $Sr(ClO_4)_2 \cdot 4DA \cdot H_2O$.

	Atom	$U_{11} \times 10^3$	$U_{22} \times 10^3$	$U_{33} \times 10^3$	$U_{13} \times 10^3$
	Sr	31(1)[c]	48(1)	34(1)	9(1)
DA_1+[b]	N_1	74(&)	51(5)	113(9)	−34(7)
	C_{11}	77(10)	68(10)	155(15)	53(10)
	C_{12}	62(7)	65(7)	56(6)	20(6)
	C_{13}	127(17)	55(10)	414(40)	155(22)
	C_{14}	88(11)	72(10)	112(13)	−37(10)
	O_2	72(6)	55(5)	111(7)	47(5)
	O_9	38(13)	60(7)	65(23)	14(14)
DA_2	N_2	27(6)	109(12)	49(7)	−14(6)
	C_{21}	42(9)	130(17)	43(9)	9(8)
	C_{22}	61(11)	114(17)	38(8)	−19(8)
	C_{23}	38(8)	80(12)	38(8)	−16(7)
	C_{24}	44(8)	60(10)	45(8)	−5(7)
	O_1	39(6)	77(7)	43(6)	−11(5)
	O_3	44(6)	116(10)	51(6)	0(5)
DA_3	N_3	57(8)	68(10)	45(7)	−11(6)
	C_{31}+	118(13)	80(10)	67(8)	−15(9)
	C_{32}+	44(6)	97(10)	50(6)	−16(5)
	O_6+	91(6)	85(10)	37(4)	−2(4)
H_2O	O_5	47(6)	65(7)	44(6)	−7(5)
ClO_4+	Cl_1	152(4)	65(22)	94(3)	−14(3)
	O_{11}	155(13)	60(7)	324(23)	4(14)
	O_{12}	340(30)	510(43)	128(12)	−33(16)
	O_{13}	663(63)	558(56)	132(14)	43(25)
	O_{14}	447(48)	167(22)	1028(109)	−450(63)

[a] The form of the anisotropic thermal parameter is: $\exp\{-2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}l^2c^{*2} + 2U_{12}hka^*b^* + 2U_{13}hla^*c^* + 2U_{23}klb^*c^*)\}$ and $U_{11} = \frac{\beta_{11}}{2\pi^2a^{*2}}$, $U_{22} = \frac{\beta_{22}}{2\pi^2b^{*2}}$, $U_{33} = \frac{\beta_{33}}{2\pi^2c^{*2}}$, $U_{13} = \frac{\beta_{13}}{4\pi^2a^*c^*}$. [b] Anisotropic thermal parameters are not calculated for mirror related atoms. [c] Quantities in parentheses are the estimated standard deviations (x 10^3).

tance (1.204Å) is considerably less than that found in crystalline urea,[21] and tends to weaken the Sr–O bonds.

All of the diacetamide ligands are close to the *trans–trans* configuration[22] (Table VII) and what little distortion exists is manifested as an out-of-plane bending along the strontium–nitrogen axis. This occurs in two of the four ligands and appears as a dihedral angle between the two planes difined by the N–C–O on each side of the ligand (Table IV). The largest such angle found was 8.0° and its presence if not its magnitude was predicted by infrared studies of the $[Sr(DA)_4(H_2O)_2](ClO_4)_2$.

While the positions of all the hydrogen atoms were not determined directly, those associated with the water were deduced by symmetry relationships and hydrogen bond distance and angle data (Table V). The oxygen atom of the water was on the mirror plane thus allowing only two possible configurations for the hydrogen atoms, *i.e.* both coplanar with the mirror, or mirror images of each other. An analysis of nonbonded distances revealed the possibility of hydrogen bonding between

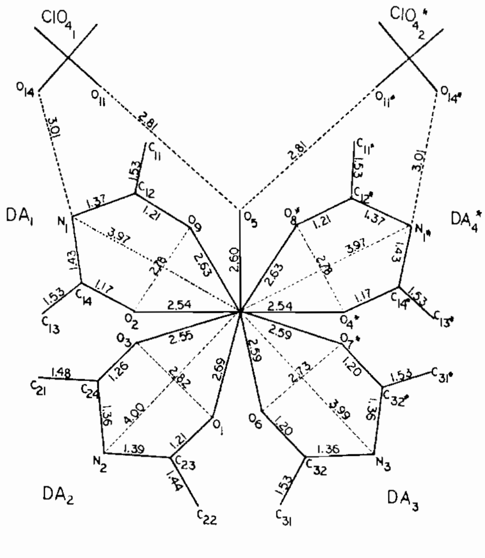

Figure 4. Interatomic distances (Å).

TABLE VII. Planes Defined by Diacetamide in $Sr(ClO_4)_2 \cdot 4DA \cdot H_2O$.

Group	Atoms	R.m.s. Deviation from Planarity (Å)
DA_1	$N_1–O_2–O_9–C_{11}–C_{12}–C_{13}–C_{14}$	0.0395
DA_2	$N_2–O_1–O_3–C_{21}–C_{22}–C_{23}–C_{24}$	0.0
DA_3	$N_3–O_6–O_{7+}–C_{31}–C_{31+}–C_{32}–C_{32+}$[a]	0.0190
DA_4	Mirror image of DA_1	

[a] + indicates atoms generated by reflection across the mirror plane at y = 1/4.

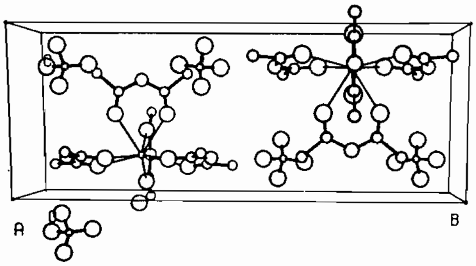

Figure 5. Packing diagram[14].

the water molecule and the two mirror related perchlorate groups (Figures 1, 3, 4), indicating that the hydrogen atoms were mirror images. Placing them along straight lines joining the coordinated oxygen of the water molecule, O(5), and the perchlorate oxygens, O(11) and O(11)* gave an H–O–H angle of 109.16° and an O–H···O distance of 2.81Å.

Previous infrared data[22] indicated the existence of hydrogen bonding between the amide and the perchlorate group. This was confirmed by the presence of a non-bonded distance of 3.012Å between the amide nitrogen, N(1), and O(14) of the perchlorate (Fig. 1).

As expected, the methyl carbons of the ligands exhibit relatively high thermal parameters as do the perchlorate oxygens. The coordinated carbonyl oxygens and the oxygen atom of the water show low thermal parameters (Table VI).

As in other instances reported,[23, 24] the perchlorate groups are ionic and show significant distortion from their idealized T_d geometry and bond distances.[25]

It is interesting to note that for the reported diaquostrontium complex $[Sr(DA)_4(H_2O)_2](ClO_4)_2$,[1] the second water molecule could coordinate at the uncapped position of the MSAP. This would yield a ten-coordinate strontium complex in the SBSA configuration.

A packing diagram is shown in Figure 5 and bond angles in Table VIII.

TABLE VIII. Bond Angles in $Sr(ClO_4)_2 \cdot 4DA \cdot H_2O$.

	Atoms	Angle		Atoms	Angle
DA_1			DA_2		
	$Sr–O_2–C_{14}$	137.09(97)[a]		$Sr–O_1–C_{23}$	137.42(75)
	$Sr–O_9–C_{12}$	135.99(73)		$Sr–O_3–C_{24}$	142.30(79)
	$O_2–C_{14}–N_1$	126.69(139)		$O_1–C_{23}–N_2$	122.95(106)
	$O_9–C_{12}–N_1$	125.29(109)		$O_3–C_{24}–N_2$	121.20(108)
	$O_2–C_{14}–C_{13}$	123.25(156)		$O_2–C_{23}–C_{22}$	123.91(111)
	$O_9–C_{12}–C_{11}$	121.92(111)		$O_3–C_{24}–C_{21}$	121.60(110)
	$C_{13}–C_{14}–N_1$	110.103(141)		$C_{21}–C_{24}–N_2$	117.20(106)
	$C_{11}–C_{12}–N_1$	112.73(108)		$C_{22}–C_{23}–N_2$	113.14(103)
	$C_{12}–N_1–C_{14}$	122.99(112)		$C_{23}–N_2–C_{24}$	131.02(101)
	$O_2–Sr–O_9$	65.05		$O_1–Sr–O_3$	65.11(26)
DA_3			DA_4		
	$Sr–O_6–C_{32}$	138.13(80)		Mirror image of DA	
	$Sr–O_{7+}–C_{32+}$[b]	138.13(80)			
	$O_6–C_{32}–N_3$	124.79(113)			
	$O_{7+}–C_{32+}–N_3$	124.79(113)			
	$O_6–C_{32}–C_{31}$	112.53(82)			
	$O_{7+}–C_{32+}–C_{31+}$	112.53(82)			
	$C_{31}–C_{32}–N_3$	113.66(88)			
	$C_{31+}–C_{32+}–N_3$	113.66(88)			
	$C_{32}–N_3–C_{32+}$	125.49(91)			

[a] Values in parentheses are the estimated standard deviations (x 10^2). [b] + indicates an atom generated by reflection across the mirror plane at y = 1/4.

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