restriction causes the opposite O(4)—Cu—O(4) angle to open to 95·5 (1)°. The other bond angles are close to those expected for a distorted octahedron.

The structure of the $PF_2O_2^-$ anion is best described as a distorted tetrahedron. The four F—P—O bond angles are within 3° of the 109·5° tetrahedral angle. The angle formed by the two O atoms, O(4)—P—O(3), is significantly enlarged to 123·2 (1)° and the opposite F(1)—P—F(2) angle correspondingly contracted at 96·8 (2)°. These bond distances and angles are all very close to those reported for $PF_2O_2^-$ when not coordinated to a transition metal (Wells, 1984; Harrison & Trotter, 1969; Granier, Durand, Cot & Galigne, 1975).

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Crystal Phases of Dodecyl Sulfates Obtained from Aqueous Solutions: Structure of the Hexaaquamagnesium Salt

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Abstract. Hexaaquamagnesium dodecyl sulfate, $[Mg(H_2O)_6][C_{12}H_{25}O_4S]_2$, $M_r = 663{\cdot}2$, monoclinic, $A2$, $a = 6{\cdot}2860$ (4), $b = 7{\cdot}0234$ (6), $c = 42{\cdot}086$ (3) Å, $\beta = 92{\cdot}915$ (5)°, $V = 1855{\cdot}7$ (2) Å^3, $Z = 4$, $D_x = 1{\cdot}19$ g cm^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, μ(Cu $K\alpha$) = 18·9 cm^{-1}, $F(000) = 724$, room temperature, $R = 0{\cdot}074$ for 1614 independent reflections with $I \geq 1{\cdot}5\sigma(I)$. The crystal packing is characterized by a lamellar structure where polar and apolar regions alternate. In the polar region each magnesium ion is hydrated by six water molecules octahedrally coordinated and each water molecule forms two hydrogen bonds with different O atoms of the adjacent polar heads. Therefore, the Coulombic interactions in the polar region are partially shielded by the water molecules surrounding the cation. In contrast with previously determined crystal packings of Na and Rb dodecyl sulfates, in the apolar region the hydrocarbon chains of opposite bilayer halves interdigitate completely and form a common matrix. The intermolecular contacts among the C atoms of the adjacent hydrocarbon chains show, however, that weak van der Waals interactions contribute to the packing in this region.

Introduction. The current models of micellar aggregates in aqueous solutions do not account satisfactorily for the available data and often give rise to contradictions (Dill, Koppel, Cantor, Dill, Bendedouch & Chen, 1984; Menger & Doll, 1984). In order to obtain models of micellar aggregates formed from sodium dodecyl sulfate (SDS) and to verify them with various techniques in the study of micellar solutions, we are following a strategy successfully applied in the structural elucidation of deoxycholate micelles (Campanelli, Candeloro De Sanctis, Giglio & Petriconi, 1984; Conte, Di Blasi, Giglio, Parretta & Pavel, 1984; Esposito, Zanobi, Giglio, Pavel & Campbell, 1987; Esposito, Giglio, Pavel & Zanobi, 1987; Giglio, Loreti & Pavel, 1988). Therefore, crystal phases of sodium dodecyl sulfate,

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obtained from aqueous solutions at a concentration above the critical micellar concentration, have been investigated by X-ray crystallography (Coiro, Mazza & Pochetti, 1986, 1987; Coiro, Manigrasso, Mazza & Pochetti, 1987). Furthermore, since micellization is very sensitive to the properties of the counterion (*e.g.* its dimensions, electric charge and hydration number), which contributes to the average area of the polar head (Missel, Mazer, Carey & Benedek, 1982), we have also examined the crystal packings of the same amphiphile in the presence of different cations (Coiro & Mazza, 1989).

Magnesium salts of dodecyl sulfate also give rise to micellar aggregates in aqueous solutions (Hato & Shinoda, 1973). Here we report the crystal structure of hexaaquamagnesium dodecyl sulfate (MgDS).

Experimental. MgDS was prepared by mixing aqueous solutions of magnesium chloride and SDS in a 1:2 molar ratio; the precipitate was purified by repeated crystallizations from water. Colourless plate-like crystals were obtained by slow evaporation from an aqueous solution at 283 K (the same crystals can be obtained from an ethanol solution). A crystal of dimensions 0·6 × 0·4 × 0·05 mm was mounted on a Syntex $P2_1$ diffractometer equipped with graphite monochromator and Cu $K\alpha$ radiation. The unit-cell parameters were determined from a least-squares fit of the angular settings of 50 reflections in the range $20 \leq \theta \leq 42°$. The systematic absences were consistent with the three space groups *A*2, *Am*, *A*2/*m* (*hkl* with $k+l$ odd); *A*2 was confirmed after refinement, according to the Hamilton test.

Intensity data were recorded by the θ–2θ-scan technique, with a scan rate varying in the interval 29·3–1·0° min^{-1} up to a maximum 2θ value of 130°. Background counts were taken for a time equal to one third of the scan time. Intensity data were corrected for the anisotropy of absorption by means of φ-scan curves obtained for two reflections with $\chi \simeq 90°$. Three standard reflections (002, 113 and 302), monitored after every 100 reflections, showed a small steady decrease which was taken into account as X-ray decay. After merging equivalent reflections R_{int} was 0·03. Out of a total of 1901 measured reflections, 1615 with $I \geq 1·5\sigma(I)$ (h 0→7, k 0→8, l −50→50) were obtained and corrected for Lorentz and polarization. 286 unobserved reflections.

Structure solution was accomplished in all three space groups by direct methods using the program *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). The refinement was carried out using the *SHELX*76 program (Sheldrick, 1976) with the weighting scheme $w = k_1[\sigma^2F_o + k_2F_o^2]^{-1}$, k_1 and k_2 being two constants redetermined after each cycle. The function minimized was $\sum w(|F_o| - |F_c|)^2$. The atomic scattering factors were corrected for anomalous dispersion. Attempts to refine the positional parameters individually without constraints failed in all three space groups. In fact, in all cases, after refinement, the hydrocarbon chain showed C—C bond lengths and C—C—C angles respectively shorter and larger than the expected values. Therefore it was decided to carry out the refinement in the three space groups constraining all the C—C bond lengths to approximate the expected value of 1·54 Å. While an improvement of the bond lengths and angles of the hydrocarbon chain was obtained using the space groups *A*2 and *A*2/*m*, the same improvement was not achieved in the space group *Am* so it was abandoned at this point. Anisotropic refinement was continued in the remaining two space groups treating the hydrocarbon chain as a rigid group with the same geometry as that obtained in the previous constrained refinement. Convergence was reached at R, wR, R_g values of 0·074, 0·090, 0·122 and 0·080, 0·107, 0·153 for the *A*2 and *A*2/*m* space groups respectively.

In the noncentrosymmetric space group the magnesium ion lies on the twofold axis, all the remaining atoms being in general positions and the atoms of the chain have approximately the same y coordinate.

In the centrosymmetric space group only one water molecule is on a general position; the dodecyl sulfate anion and the other water molecule lie on the mirror plane and the magnesium ion is at the origin.

In order to decide whether the increased number of parameters used to refine the structure in the noncentrosymmetric space group led to a significant improvement, we applied the Hamilton test (Hamilton, 1965), which gave the probability of making an incorrect assignment smaller than 0·005 (ratio of residuals = 1·2508, dimensions = 63, degrees of freedom = 1445). *A*2 was therefore assumed. In this noncentrosymmetric space group the atoms are arranged in such a manner as to simulate a mirror plane; the difference with respect to *A*2/*m* is given mainly by the alkyl chain atoms C(6), C(7), C(8), C(9) and C(11) which deviate significantly from planarity. This can indicate disorder and/or large thermal vibration in the chain region.

Five of the six H atoms of the water molecules in the asymmetric unit were found in the final Fourier difference synthesis: their isotropic thermal parameters were made similar to those of the O atoms and were included, together with their coordinates, in the last structure-factor calculation. In the last stages of refinement the reflection 002 was excluded because it was judged to be severely influenced by extinction or absorption. $(\Delta/\sigma)_{\text{max}}$ was 1·8. The maximum and the minimum heights in the final Fourier difference synthesis were 0·4 and −0·4 e Å^{-3} respectively. Scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol.

Table 1. *Fractional atomic coordinates and equivalent isotropic temperature factors* (Å^2) *for non-*H *atoms with e.s.d.'s in parentheses*

$U_{eq} = (U_{11} + U_{22} + U_{33})/3.$

	x	y	z	U_{eq}
Mg(1)*	0·000	0·1601	0·0000	0·051 (1)
S(1)	0·4016 (2)	−0·3386 (9)	0·0563 (3)	0·058 (1)
O(1)	0·4455 (7)	−0·3335 (15)	0·0932 (1)	0·086 (3)
O(2)	0·4930 (12)	−0·5043 (12)	0·0451 (3)	0·076 (6)
O(3)	0·4953 (13)	−0·1626 (13)	0·0437 (3)	0·079 (6)
O(4)	0·1703 (5)	−0·3393 (16)	0·0543 (1)	0·078 (3)
C(1)	0·6739 (7)	−0·3147 (15)	0·1044 (1)	0·114 (7)
C(2)	0·6807 (7)	−0·3128 (15)	0·1401 (1)	0·123 (8)
C(3)	0·9047 (7)	−0·3152 (15)	0·1548 (1)	0·145 (9)
C(4)	0·9127 (7)	−0·3116 (15)	0·1910 (1)	0·153 (10)
C(5)	1·1373 (7)	−0·3152 (15)	0·2060 (1)	0·164 (11)
C(6)	1·1363 (7)	−0·3626 (15)	0·2417 (1)	0·151 (11)
C(7)	1·3610 (7)	−0·3636 (15)	0·2571 (1)	0·141 (11)
C(8)	1·3756 (7)	−0·3045 (15)	0·2923 (1)	0·135 (9)
C(9)	1·5890 (7)	−0·3584 (15)	0·3091 (1)	0·160 (11)
C(10)	1·6016 (7)	−0·3141 (15)	0·3445 (1)	0·137 (9)
C(11)	1·8237 (7)	−0·2878 (15)	0·3602 (1)	0·180 (14)
C(12)	1·8361 (7)	−0·3347 (15)	0·3954 (1)	0·182 (12)
O(11)	0·0972 (1)	0·3763 (14)	−0·0292 (3)	0·075 (5)
O(12)	0·2948 (5)	0·1661 (16)	0·0222 (1)	0·062 (2)
O(13)	0·1053 (13)	−0·0507 (13)	−0·0303 (2)	0·077 (5)

* The positional coordinates of this atom were kept fixed during the refinement.

IV). All the calculations were carried out on a DG ECLIPSE MV/8000 II computer.

Discussion. The final atomic parameters are given in Table 1* and bond lengths and angles are given in Fig. 1, together with the atomic numbering adopted.

Alternate polar and apolar regions characterize the crystal packing. In the polar region the Mg ion occupies the special position on the twofold axis and is surrounded by six octahedrally coordinated water molecules as shown in Fig. 2. Three water molecules are symmetrically related to the other three by the twofold axis perpendicular to the Mg···O(12) direction. As shown in Table 2 the contacts between the Mg ion and the water O atoms range between 2·033 and 2·083 Å, while the O(11)···Mg(1)···O(11^i) angle of 85·3° shows the largest deviation from 90°. Each water molecule forms two hydrogen bonds with the surrounding O atoms of the polar heads (Fig. 2). The relevant geometric parameters of these hydrogen bonds are reported in Table 3; the O···O contacts range between 2·72 (1) and 2·82 (1) Å; all the H···O contacts are shorter than the sum of their van der Waals radii. The O—H···O angles range between 148 (1) and 171 (1)°. The angle at each water oxygen between the two O atoms of the polar heads engaged in hydrogen bonding approximates to the valence angle of the water molecule.

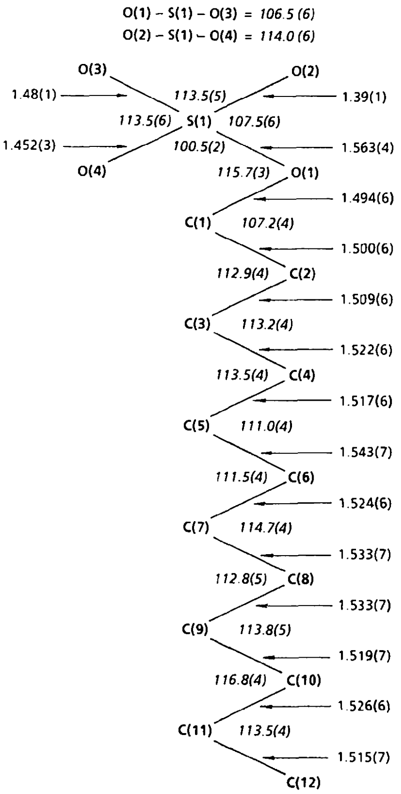

Fig. 1. Atomic numbering, bond lengths (Å) and angles (°) with e.s.d.'s in parentheses

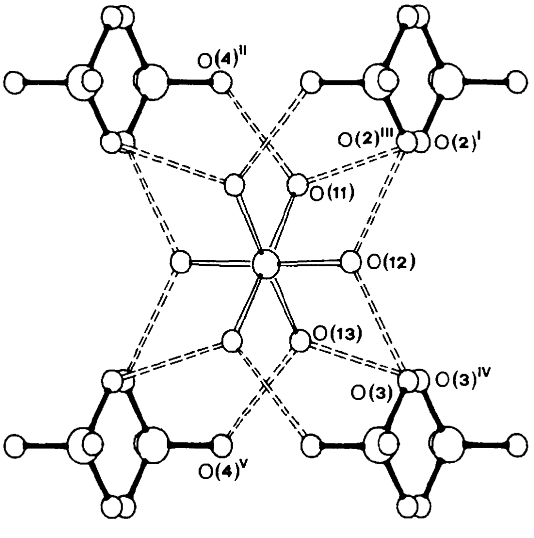

Fig. 2. The coordination of the magnesium ion (open bonds) and the hydrogen bonds (dashed bonds) connecting the water molecules to the O atoms of the surrounding polar heads. The symmetry code is that of Table 3.

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* Lists of structure factors, anisotropic thermal parameters and anion and water molecule bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53733 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Relevant contacts* (Å) *and angles* (°) *regarding the coordination of the magnesium ion with the water oxygens*

Mg(1)···O(11)	2·065 (9)	Mg(1)···O(13)	2·083 (9)
Mg(1)···O(12)	2·033 (3)		
O(11)···Mg(1)···O(11$^{\mathrm{i}}$)	85·3 (3)	O(12)···Mg(1)···O(12$^{\mathrm{i}}$)	177·6 (3)
O(11)···Mg(1)···O(12)	88·1 (3)	O(12)···Mg(1)···O(13)	89·2 (3)
O(11)···Mg(1)···O(12$^{\mathrm{i}}$)	90·1 (3)	O(12)···Mg(1)···O(13$^{\mathrm{i}}$)	92·5 (3)
O(11)···Mg(1)···O(13)	92·6 (3)	O(13)···Mg(1)···O(13$^{\mathrm{i}}$)	89·4 (3)
O(11)···Mg(1)···O(13$^{\mathrm{i}}$)	177·8 (3)		

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

Table 3. *Relevant geometric parameters* (Å,°) *of the hydrogen bonds between the water molecules and the* O *atoms of the polar heads*

D	*A*	O···O	O—H	H···O	O—H···O	*A*···*D*···Mg
O(11)	O(2$^{\mathrm{i}}$)	2·82 (1)	0·97 (1)	1·96 (1)	148 (1)	131·4 (4)
O(11)	O(4$^{\mathrm{ii}}$)	2·78 (1)	0·96 (1)	1·84 (1)	167 (1)	124·2 (3)
O(12)	O(2$^{\mathrm{iii}}$)	2·78 (1)	0·96 (1)	1·85 (1)	163 (1)	124·0 (4)
O(12)	O(3)	2·76 (1)	0·95 (1)	1·82 (1)	171 (1)	121·6 (5)
O(13)	O(3$^{\mathrm{iv}}$)	2·72 (1)				
O(13)	O(4$^{\mathrm{v}}$)	2·82 (1)	0·97 (1)	1·90 (1)	158 (1)	121·4 (4)

O(2)···O(11)···O(4)	103·7 (4)
O(2)···O(12)···O(3)	113·2 (3)
O(3)···O(13)···O(4)	106·0 (4)

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

Of the various crystal phases of SDS salts studied (Coiro, Maza & Pochetti, 1986, 1987; Coiro, Manigrasso, Mazza & Pochetti, 1987; Coiro & Mazza, 1989) the present salt possesses the largest number of water molecules coordinated by the metal. The water molecules play a double role in the crystal packing. They coordinate metal ions, partially shielding the strong Coulombic interactions between the counterions, and link the O atoms of the adjacent polar heads by hydrogen bonds along the **a** and **b** directions; small channels running parallel to the *a* axis are formed, in which the shortest contact between opposite O atoms is 4·44 (1) Å.

The polar head of the dodecyl sulfate anion shows that the three bonds S(1)—O(2), S(1)—O(3) and S(1)—O(4) are shorter than the ester bond S(1)—O(1). Also, the angles O(2)—S(1)—O(3), O(2)—S(1)—O(4) and O(3)—S(1)—O(4) are larger than those involving the ester O(1) atom (see Fig. 1). These effects, mainly due to the negative charge equally shared among the three non-ester O atoms, have already been noted in the structural investigations of the other dodecyl sulfate salts mentioned above. The three torsion angles (Klyne & Prelog, 1960), giving the orientation of the polar head with respect to the hydrocarbon chain, namely O(2)—S(1)—O(1)—C(1), O(3)—S(1)—O(1)—C(1) and O(4)—S(1)—O(1)—C(1), are 65·5 (9), −56·5 (9) and −175·0 (8)° respectively. The conformation of the hydrocarbon chain is almost *trans*-planar: the largest deviation from planarity being that of the atomic sequence C(5)—C(6)—C(7)—C(8) with a torsion angle of 149·2 (8)°. Views of the crystal packing of MgDS on the *ac* and *bc* planes are shown on Figs. 3(*a*) and 3(*b*) respectively. The chains lie on the *ac* plane making angles of 58 (1)° with the polar plane (*ac*) containing the S atoms of a monolayer.

In contrast with the other dodecyl sulfate crystal phases the most striking structural feature present in the crystal packing of MgDS is the interdigitation among the hydrocarbon chains. In the polar region the alkyl chains are extended, lie antiparallel to each other and fully interdigitate. This may be caused by the accommodation of the large number of water molecules of hydration which increase the average area per polar head with respect to the other dodecyl sulfate salts. Like the phospholipid arrangement in the bimolecular lamellar structure (Pascher, Sundell & Hauser, 1981), whenever the area per head group is equal or exceeds twice the cross section of the hydrocarbon chain, the chains will interdigitate with those of the opposite layer. For MgDS the average area per polar head is 44·1 Å^2, compared to the cross section normally required by a hydrocarbon chain in the solid state, that is in the range 19–21 Å^2. Average areas per polar head in the range 20·9–29·5 Å^2 have been obtained for the SDS crystal phases, whereas a value of 26·6 Å^2 has been obtained in the case of the non-hydrated rubidium salt (Coiro & Mazza, 1989).

Weak intermolecular van der Waals interactions occur between the C atoms of adjacent hydrocarbon

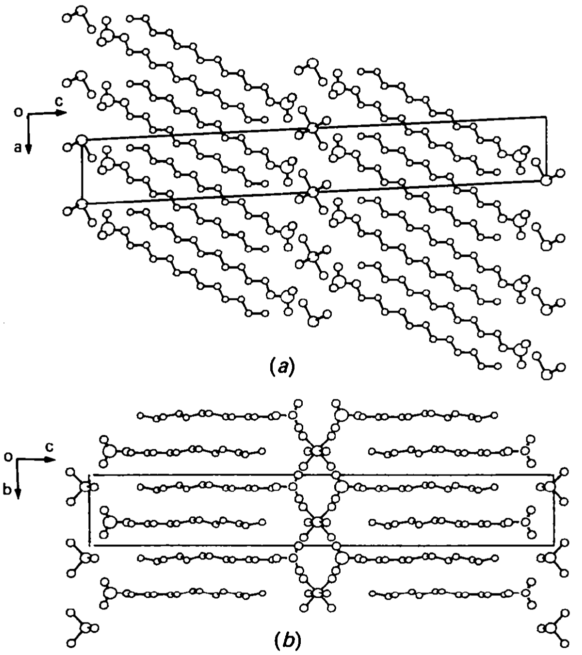

Fig. 3. Views of the crystal packing of MgDS (*a*) on the *ac* and (*b*) on the *bc* planes.

chains. The average non-bonded distance between C atoms of neighbouring chains is 4·3 Å. However, a number of C···C contacts within the range 3·71 (2)–4·15 (2) Å [mean value 3·98 (2) Å] contribute to stabilize the apolar region. The closest intermolecular contacts are that between C(5) and C(9) at $-x-3$, $y+\frac{1}{2}$, $z+\frac{1}{2}$ of 3·71 (2) Å, and that between C(3) and C(11) at $x-3$, $y-\frac{1}{2}$, $-z+\frac{1}{2}$ of 3·80 (2) Å.

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Structures of Diene–Tricarbonyl Iron Complexed *erythro* and *threo* Carbonates, Key Intermediates for the Preparation of 5,6-Dihetes and Lipoxin A4

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Abstract. (1) *erythro*-Tricarbonyl(η^4-methyl 4-{5-[5-(*tert*-butyldiphenylsiloxy)-1,3-pentadienyl]-2-oxo-1,3-dioxolan-4-yl}butanoate)iron(*erythro*-carbonate), $C_{32}H_{36}FeO_9Si$, $M_r = 648{\cdot}6$, monoclinic, $C2/c$, $a = 37{\cdot}14\,(1)$, $b = 8{\cdot}51\,(1)$, $c = 20{\cdot}732\,(6)$ Å, $\beta = 91{\cdot}84\,(7)°$, $V = 6549\,(1)$ Å^3, $Z = 8$, $D_x = 1{\cdot}32$ g cm^{-3}, $\lambda(\text{Mo }K\alpha) = 0{\cdot}71073$ Å, $\mu = 5{\cdot}43$ cm^{-1}, $F(000) = 2720$, $T = 293$ K, $R = 0{\cdot}049$ for 3491 reflections. (2) Major *threo*-carbonate, $C_{32}H_{36}FeO_9Si$, $M_r = 648{\cdot}6$, monoclinic, $C2/c$, $a = 37{\cdot}88\,(1)$, $b = 8{\cdot}175\,(5)$, $c = 20{\cdot}969\,(8)$ Å, $\beta = 91{\cdot}19\,(3)°$, $V = 6492\,(1)$ Å^3, $Z = 8$, $D_x = 1{\cdot}33$ g cm^{-3}, $\lambda(\text{Mo }K\alpha) = 0{\cdot}71073$ Å, $\mu = 5{\cdot}48$ cm^{-1}, $F(000) = 2720$, T 293 K, $R = 0{\cdot}051$ for 2133 reflections. (3) Minor *threo*-carbonate, $C_{32}H_{36}FeO_9Si$, $M_r = 648{\cdot}6$, monoclinic, $P2_1/c$, $a = 24{\cdot}96\,(2)$, $b = 10{\cdot}646\,(6)$, $c = 12{\cdot}232\,(3)$ Å, $\beta =$

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