Fig. 1. Molecular structure and numbering scheme.

α-Hydroxycarboxylate complexes of osmyls comprise a new class of osmyl-containing compounds. The general structural characteristics of the complex studied in this paper are consistent with osmyls in general, and spectroscopy (Hinckley & Kibala, 1986) indicates that this compound is broadly representative of the class. The six-membered chelate ring found in

this compound is unusual. The larger ring size is accommodated by an expansion of the interior bond angles within the chelate ring, accompanied by a flex in the ring to relieve strain. The supposed electronic stabilization of a planar unflexed molecule is evidently unimportant in this case. The structure of this single compound is consistent with the notion that the small chelate ring size which characterizes osmyl complexes of all classes is due to steric factors arising out of the size of the osmium atom, and to chemical constraints associated with their synthesis.

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Structure of Sodium *N*-(*p*-Aminobenzenesulfonyl)acetamide Monohydrate (Sodium Sulfacetamide Monohydrate)

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Abstract. $Na^+.C_8H_{10}N_2O_3S^-.H_2O$, $M_r = 255.24$, monoclinic, $P2_1/c$, $a = 6.617$ (2), $b = 23.771$ (4), $c = 7.014$ (1) Å, $\beta = 103.12$ (3)°, $V = 1074.2$ (2) Å^3, $Z = 4$, $D_m = 1.580$ (1), $D_x = 1.578$ Mg m^{-3}, λ(Cu $K\alpha$) $= 1.5418$ Å, $\mu = 2.48$ mm^{-1}, $F(000) = 532$, $T = 293$ K, final $R = 0.069$ for 2161 observed reflections. The asymmetric unit contains one sulfacetamide molecule, one Na atom and one water molecule. The Na atom has fivefold coordination in the structure. The bonding around the S atom is slightly distorted from the ideal tetrahedral symmetry. The sulfacetamide molecule shows a *gauche* conformation about the S—N bond with a torsion angle of 81.3 (5)°. The crystal structure is stabilized by an extended network of interdigitating hydrogen bonds, van der Waals forces and intermolecular coordinations through Na atoms.

Introduction. Recent studies on the action of sulfonamides on enzymatic extracts obtained from different bacterial species (McCullough & Maren, 1973; Ho, Corman, Morse & Schneider, 1975) have definitely shown that the target enzyme catalyses the formation of dihydropteroate from *p*-aminobenzoate (PAB) and (hydroxymethyl)dihydropteridine pyrophosphate

(Brown, 1970). Sulfonamides exert their antibacterial action by inhibiting this reaction competitively with PAB. This led to the general theory of sulfonamides acting as competitive inhibitors of the enzymatic incorporation of PAB in folic acid synthesis. As part of our programme of X-ray crystallographic investigations on sulfonamides and the enzyme dihydropteroate synthase (DHPS), the crystal structure of sodium sulfacetamide is reported here.
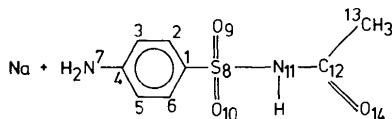
Experimental. Sodium sulfacetamide samples obtained from Indian Drugs and Pharmaceuticals Ltd, Hyderabad (India) crystallized from solution in methanol at 288 K. Colourless cylindrical crystals: one $0.80 \times 0.50 \times 0.40$ mm used for data collection. Unit-cell dimensions and space group from precession photographs. Cell dimensions refined for 25 independent high-angle reflections on diffractometer. Density by flotation in benzene and carbon tetrachloride. Intensity data collected on Enraf–Nonius CAD-4 automatic diffractometer in the ω–2θ-scan mode using Ni-filtered Cu $K\alpha$ radiation. Total number of reflections measured 2509, number of independent reflections 2259, observed reflections $[I \geq 2\sigma(I)]$ 2161, for $(\sin\theta)/\lambda \leq 0.56$ Å^{-1} with $h = -8$ to 7, $k = 0$ to 29 and $l = 0$ to 8. Corrections for Lorentz and polarization effects but not for absorption ($\mu r = 1.09$). Intensity measurements of standard reflections 132 and $\overline{2}40$ repeated at arbitrary intervals; variation in intensity throughout the experiment $\leq 4.3\%$. R_{int} (for merged data) $= 3.5\%$. The structure was solved by *MULTAN*80 (Main *et al.*, 1980) and the refinement was by block-diagonal least-squares (*SFLS*) procedure. Positions of H atoms by successive $\Delta\rho$ maps. Parameters refined using anisotropic temperature factors for non-H atoms and isotropic temperature factors for H atoms. Final $R = 0.069$ for 2161 observed reflections, $wR = 0.092$, $S = 2.56$. 18 strongest low-angle reflections corrected for secondary-extinction effect with empirical extinction-correction coefficient $g = 0.00006$ where $F_{corr} = |F_o|(1 + 2g|F_c|^2)^{1/2}$ (Stout & Jensen, 1968). Final $\Delta\rho$ excursions -0.31 to 0.28 e Å^{-3}. $(\Delta/\sigma)_{av} = 0.10$, $(\Delta/\sigma)_{max} = 0.23$ for non-H atoms. Weighting function $w = 1/(2.00 + 0.018|F_o| + 0.0004|F_o|^2)$ (Cruickshank, 1961) adjusted to make the average independent of F_o. $\sum w(\Delta F)^2$ used in the block-diagonal least-squares refinement. *SFLS* program originally written by Shiono (1968/1971) and extensively modified by the authors. Atomic scattering factors of Cromer & Mann (1968) for C, N, O and S and of Stewart, Davidson & Simpson (1965)

for H. All calculations on the HP computers at the All India Institute of Medical Sciences.

Discussion. The asymmetric unit contains one sodium sulfacetamide and one water molecule. The final positional and equivalent isotropic temperature factors of non-H atoms are given in Table 1.* The bond lengths and valence angles involving non-H atoms are listed in Table 2. The perspective view with numbering scheme is illustrated in Fig. 1. The crystal structure, the sodium coordination and the hydrogen-bonding scheme are shown in Fig. 2. The average C–H and N–H distances are 0.99 (8) and 0.95 (6) Å respectively. The bond lengths in the benzene ring vary from 1.382 (9) to 1.400 (10) Å while the valence angles have characteristic values. The C(4)–N(7), C(1)–S(8), S(8)–N(11) and N(11)–C(12) distances of 1.394 (8), 1.763 (6), 1.603 (5) and 1.349 (9) Å, respectively, are shorter than the corresponding theoretical single-bond lengths but agree well with those found in other sulfonamides (Haridas & Singh, 1986). The carbonyl distance C(12)–O(14) of 1.247 (7) Å is equivalent to that of a partial double bond as the O atom is involved in the coordination with the Na atom as well as in the intermolecular hydrogen bond. The valence angles around S(8) range from 106.2 (3) to 115.4 (3)°. There is, therefore, a significant departure from the ideal tetrahedral symmetry around the S atom. As has been observed in other structures (Singh, Patel & Haridas, 1984), the O(9)–S(8)–O(10) angle at the S atom with the value of 115.4 (3)° is the largest.

Sodium coordination. The coordination around Na is fivefold. When viewed down the a axis in the coordination sphere of Na, the atoms O(9) and O(14) are above, while O(10), N(7) and OW(16) are below the Na atom. The Na–O(10), Na–OW(16), Na–O(9), Na–O(14) and Na–N(7) distances are 2.273 (6), 2.296 (6), 2.349 (6), 2.440 (5) and 2.613 (6) Å, respectively. The angles around Na vary from 73.1 (2) to 146.0 (2)°. These values show that the coordination about the Na atom is not symmetrical. The molecular dimensions of the sulfacetamide moiety in sulfacetamide (Basak & Mazumdar, 1982), the sulfacetamide and caffeine complex (Leger, Alberola & Carpy, 1977), phthalylsulfacetamide (Singh *et al.*, 1984) and those in the present structure are, in general, in good agreement. The most prominent differences are found in S–N and C–O distances. The S–N distance in sodium sulfacetamide is 1.603 (5) Å while in sulfacetamide it is

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* Lists of structure factors, anisotropic temperature factors of non-H atoms, H-atom positional parameters, bond lengths and angles involving H atoms, intermolecular contact distances and some important least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43558 (15 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

1·653 (3) Å, in phthalylsulfacetamide 1·67 (1) Å and in the sulfacetamide and caffeine complex it is 1·652 (3) Å. In all these structures the imino N atoms are hydrogen-bonded. The C—O distance in the acetamido group of sodium sulfacetamide is 1·247 (7) Å while the corresponding distances in sulfacetamide, phthalylsulfacetamide and the sulfaceta-mide and caffeine complex are 1·218 (3), 1·22 (2) and 1·203 (4) Å, respectively. In sodium sulfacetamide the carbonyl O atoms are coordinated to the Na atom. As a result of this coordination the C—O distance in the sodium sulfacetamide is longer than those found in free sulfacetamide, in its derivative phthalylsulfacetamide and in its complex with caffeine. There is yet another very interesting observation from these structures. The torsion angles about the S—N bonds in sulfacetamide, phthalylsulfacetamide, the sulfacetamide and caffeine complex and sodium sulfacetamide are 55·9 (4), 64·6 (8), 70·0 (3) and 81·3 (6)°, respectively. This shows that the different levels of interaction with the sulfacetamide moiety introduce considerable con-formational changes in the sulfacetamide molecule. The O atoms O(9) and O(10) are displaced from the plane C(1)—S(8)—N(11) in sulfacetamide, phthalylsulfacet-amide, sodium sulfacetamide and the sulfacetamide and caffeine complex by −1·188 (2), 1·248 (2); −1·18 (1), 1·27 (1); −1·165 (5), 1·272 (5); and −1·179 (3), 1·278 (3) Å, respectively. This observa-tion is interesting because the intermolecular inter-actions through the sulfonyl O atoms are not similar.

Hydrogen bonding and crystal packing. The crystal structure as viewed down the *a* axis is shown in Fig. 2. The molecules are stacked along the *c* axis and held firmly through interdigitating hydrogen bonds, sodium coordination and van der Waals forces. There are five hydrogen bonds of varying strengths. The amino nitrogen N(7) participates in three hydrogen bonds. This atom acts as a proton donor in two hydrogen

Table 1. *Fractional coordinates* ($\times 10^4$) *of the non-H atoms and equivalent isotropic thermal parameters*

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

	x	y	z	$B_{eq}(\text{Å}^2)$
C(1)	8123 (9)	4467 (2)	7062 (8)	89 (8)
C(2)	6227 (10)	4671 (3)	7311 (10)	128 (11)
C(3)	6045 (11)	5228 (3)	7809 (10)	124 (11)
C(4)	7728 (10)	5590 (2)	8048 (8)	99 (9)
C(5)	9625 (10)	5384 (3)	7779 (9)	107 (10)
C(6)	9826 (10)	4826 (3)	7302 (9)	101 (9)
N(7)	7539 (10)	6159 (2)	8446 (9)	131 (10)
S(8)	8384 (2)	3756 (1)	6448 (2)	72 (2)
O(9)	6575 (7)	3605 (2)	4935 (6)	101 (7)
O(10)	10352 (8)	3705 (2)	5900 (8)	131 (8)
N(11)	8615 (8)	3396 (2)	8418 (7)	89 (7)
C(12)	6945 (9)	3246 (2)	9101 (9)	84 (8)
C(13)	7453 (12)	2905 (3)	10944 (10)	122 (11)
O(14)	5100 (8)	3367 (2)	8366 (8)	121 (8)
Na	3158 (4)	3344 (1)	4964 (4)	102 (4)
OW(16)	2254 (9)	2430 (2)	4128 (10)	193 (11)

Table 2. *Bond lengths* (Å) *and bond angles* (°) *involving non-H atoms*

C(1)—C(2)	1·392 (10)	S(8)—N(11)	1·603 (5)
C(1)—C(6)	1·303 (9)	N(11)—C(12)	1·349 (9)
C(1)—S(8)	1·763 (6)	C(12)—C(13)	1·498 (9)
C(2)—C(3)	1·382 (9)	C(12)—O(14)	1·247 (7)
C(3)—C(4)	1·387 (9)	Na—O(9)	2·349 (6)
C(4)—C(5)	1·400 (10)	Na—O(10^i)	2·273 (6)
C(5)—C(6)	1·382 (9)	Na—O(14)	2·440 (5)
S(8)—O(9)	1·453 (4)	Na—OW(16)	2·296 (6)
S(8)—O(10)	1·444 (6)	Na—N(7^{ii})	2·613 (6)
N(7)—C(4)	1·394 (8)		

C(2)—C(1)—C(6)	120·0 (6)	O(10)—S(8)—N(11)	105·6 (3)
C(2)—C(1)—S(8)	120·4 (5)	S(8)—N(11)—C(12)	121·5 (4)
C(6)—C(1)—S(8)	119·7 (5)	N(11)—C(12)—C(13)	114·1 (5)
C(1)—C(2)—C(3)	119·9 (6)	C(13)—C(12)—O(14)	119·3 (6)
C(2)—C(3)—C(4)	120·7 (6)	O(10)—Na—OW(16)	104·4 (2)
C(3)—C(4)—C(5)	119·1 (6)	O(10)—Na—O(9)	138·9 (2)
C(3)—C(4)—N(7)	121·4 (6)	O(10)—Na—O(14)	89·6 (2)
C(5)—C(4)—N(7)	119·4 (6)	O(10)—Na—N(7)	96·2 (2)
C(6)—C(5)—C(4)	120·5 (6)	OW(16)—Na—O(9)	116·4 (2)
C(5)—C(6)—C(1)	119·8 (6)	OW(16)—Na—O(14)	108·7 (2)
C(1)—S(8)—O(9)	107·7 (3)	OW(16)—Na—N(7)	102·3 (2)
C(1)—S(8)—N(11)	107·3 (3)	O(9)—Na—O(14)	73·1 (2)
O(9)—S(8)—O(10)	115·4 (3)	O(9)—Na—N(7)	81·0 (2)
O(9)—S(8)—N(11)	113·5 (3)	O(14)—Na—N(7)	146·0 (2)

Symmetry code: (i) x−1, y, z; (ii) −x+1, −y+1, −z+1.

Fig. 1. A perspective view of the molecules along with the sodium coordination as seen down the *a* axis and the numbering scheme.
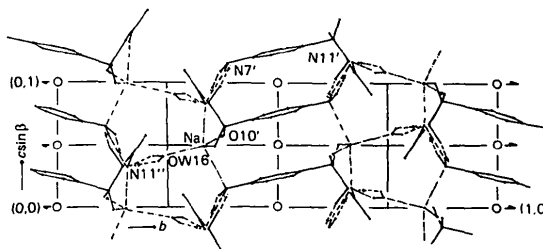

Fig. 2. The crystal structure as viewed along the *a* axis. The dashed lines indicate hydrogen bonds and the dotted-cum-dashed lines illustrate the sodium coordination. The atoms O(10′), N(7′), N(11′) and N(11″) correspond to the equivalent positions of x−1, y, z; −x+2, −y+1, −z+2; −x+2, −y+1, −z+2 and x−1, −y+½, z−½, respectively.

bonds while as an acceptor in the third with the imino nitrogen N(11). There is one water molecule in the asymmetric unit which acts as a strong stabilizing force since the water O atom is coordinated to the Na atom and it also acts as a proton donor in the hydrogen bond with the carbonyl oxygen O(14) of the acetamido moiety and the imino nitrogen N(11) of the symmetry-related molecule. Though all the sulfacetamide molecules, water molecules and Na atoms are linked strongly through hydrogen bonds and coordinating bonds, the non-polar ring moieties are stacked in such a way that they also generate significant van der Waals interactions between them.

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Structure of Tetracarbonyl(5,7,12,14-tetramethyldibenzo[b, i][1,4,8,11]-tetraazacyclotetradeca-2,4,6,9,11,14-hexaene)molybdenum(0)

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Abstract. [Mo(CO)$_4$(C$_{22}$H$_{24}$N$_4$)], $M_r = 552 \cdot 4$, triclinic, $P\bar{1}$, $a = 8 \cdot 909$ (6), $b = 9 \cdot 361$ (6), $c = 16 \cdot 544$ (6) Å, $\alpha = 89 \cdot 36$ (4), $\beta = 78 \cdot 03$ (5), $\gamma = 67 \cdot 77$ (5)°, $V = 1246$ Å^3, $Z = 2$, $D_x = 1 \cdot 473$ Mg m^{-3}, λ(Mo $K\alpha$) = 0 \cdot 71069 Å, $\mu = 0 \cdot 521$ mm^{-1}, $F(000) = 564$, $T = 298$ K, $R = 0 \cdot 0502$ for 2958 observed reflections. Two of the four N atoms of the cyclic ligand are coordinated in a *cis* configuration to the Mo(CO)$_4$ fragment, giving a distorted octahedral environment at Mo with the macrocycle folded away from the metal centre.

Introduction. The tetradentate macrocyclic ligand 5,7,12,14-tetramethyldibenzo[b,i][1,4,8,11]tetraazacyclotetradeca-2,4,6,9,11,13-hexaene ($L = $ H$_2$L') exhibits a range of conformations when bound to metal ions as the dianion (L'^{2-}). Compounds that have been prepared include square planar complexes with bivalent and trivalent metal centres (Jäger, 1964; Goedken, Pluth, Peng & Burnsten, 1976), five-coordinate square-based pyramidal species (Weiss, Burnsten, Peng & Goedken, 1976; Warren & Goedken, 1978) and octahedral complexes incorporating two mutually *cis* (Goedken & Ladd, 1982) and *trans* (Weiss & Goedken, 1976) ligands.

L'

L

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† Deceased.