Phelps, D. W. & Cordes, A. W. (1974). *Acta Cryst.* B**30**, 2812–2816.
Puig-Torres, S., Martin, G. E., Smith, K., Cacioli, P. & Reiss, J. A. (1982). *J. Heterocycl. Chem.* **19**, 879–882.
Riley, P. E. & Davis, R. E. (1976). *Acta Cryst.* B**32**, 381–386.
Womack, C. H., Turley, J. C., Martin, G. E., Kimura, M. & Simonsen, S. H. (1981). *J. Heterocycl. Chem.* **18**, 1173–1178.

Acta Cryst. (1984). C**40**, 1486–1490

Structures of Morphine Methyl Iodide Monohydrate, $(C_{18}H_{22}NO_3)^+.I^-.H_2O$, and Di(morphine) Dihydrogensulfate Pentahydrate, $2(C_{17}H_{20}NO_3)^+.SO_4^{2-}.5H_2O$*

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(*Received* 4 *November* 1983; *accepted* 10 *May* 1984)

Abstract. Morphine methyl iodide monohydrate: $M_r = 445{\cdot}3$, orthorhombic, $P2_12_12_1$, $a = 9{\cdot}574\,(1)$, $b = 10{\cdot}614\,(2)$, $c = 17{\cdot}549\,(4)$ Å, $V = 1783{\cdot}3\,(6)$ Å^3, $Z = 4$, $D_x = 1{\cdot}658$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu = 17{\cdot}95$ cm^{-1}, $F(000) = 896$, room temperature, final $R = 6{\cdot}1\%$ for 2575 reflections. Morphine methyl iodide displays methyl bonding to nitrogen and an extensive hydrogen-bonding network involving a phenolic hydrogen with an oxygen of the monohydrate. (Morphine)$_2$ H_2SO_4 pentahydrate; asymmetric unit $(C_{17}H_{19}NO_3)$-$\frac{1}{2}H_2SO_4.2\frac{1}{2}H_2O$, orthorhombic, $P22_12_1$, $M_r = 379{\cdot}4$, $a = 8{\cdot}463\,(3)$, $b = 6{\cdot}825\,(2)$, $c = 30{\cdot}71\,(1)$ Å, $V = 1774\,(1)$ Å^3, $Z = 4$, $D_x = 1{\cdot}420$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu = 1{\cdot}58$ cm^{-1}, $F(000) = 808$, room temperature, final $R = 6{\cdot}2\%$ for 1754 reflections. (Morphine)$_2H_2SO_4$ pentahydrate shows similar detail in the framework structure but the hydrogen bonding involves OH and NH hydrogen atoms and sulfate oxygen atoms.

Introduction. The molecular chirality of the opium alkaloids has been exploited in the development of analytical procedures for their determination using circular dichroism (CD) spectropolarimetry. Spectra have been obtained for morphine in three different matrices, namely pressed in KBr pellets (Bowen & Purdie, 1980), dissolved in cholesteric liquid crystals (Bowen, Crone, Hermann & Purdie, 1980) and in aqueous media (Crone & Purdie, 1981). The third is the only quantitative procedure.

The CD spectra of morphine free base and its salts are similar in all the supporting media except the solid state. In this matrix only the spectra of the salts bear a qualitative resemblance to the spectra observed in aqueous solution. Typically two Cotton bands are observed over the wavelength range 230–350 nm. These have been attributed to the electronic transitions ^1L_a (positive maximum at 248 nm) and ^1L_b (negative maximum at 286 nm). For the ^1L_b transition an inversion to a weak positive Cotton band is observed for morphine free base in a KBr pellet.

In order to interpret these obvious changes in chirality in the solid state, single-crystal X-ray structures have been determined at room temperature for morphine methyl iodide monohydrate (I) and (morphine)$_2H_2SO_4$ pentahydrate (II). These results may be compared with previously published data for morphine free base (Bye, 1976).

Experimental. Crystals of (I) and (II) [$0{\cdot}1 \times 0{\cdot}2 \times 0{\cdot}2$ (I); $0{\cdot}2 \times 0{\cdot}2 \times 0{\cdot}4$ (II)] sealed in a capillary and mounted on Syntex *P*3 automated diffractometer. Unit-cell dimensions determined by least-squares refinement of the best angular positions for 15 independent reflections ($2\theta > 15°$) during normal alignment procedures. Data sets [(I) 8044 points, h 0–12, k 0–12, l 0–17; (II) 4268 points h 0–15, k 0–15, l 0–20] collected using variable scan rate, θ–2θ scan mode and scan width 1·2° below $K\alpha_1$ and 1·2° above $K\alpha_2$ to max. 2θ of 116°. Backgrounds measured at each side of the scan for combined time equal to total scan time. Systematic absences (I) $h00$, $h \neq 2n$; $0k0$, $k \neq 2n$, $00l$, $l \neq 2n$; (II) $0k0$, $k \neq 2n$, $00l$, $l \neq 2n$. Intensities of three standard reflections remeasured after every 97 reflections, <8% variation, no corrections for decomposition. Data corrected for Lorentz, polarization and background effects. After removal of redundant and space-group forbidden data, 2575 (I), 1754 (II) reflections considered observed [$I > 3{\cdot}0\sigma(I)$]. Structures solved by direct methods using *MULTAN*80 (Main *et al.*, 1980). Refinement of scale factor, positional and anisotropic thermal parameters for all nonhydrogen atoms with *XRAY* (Stewart, 1978).

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* 17-Methylmorphinium iodide and di(17H^+-morphinium) sulfate.

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Hydrogen positional parameters determined by difference Fourier synthesis. Hydrogen positional parameters and associated isotropic thermal parameters refined with nonhydrogen parameters in final cycles of refinement for (II). For (I) hydrogen positional parameters included in final cycles of refinement. Hydrogen atoms assigned isotropic thermal parameters of $U = 0·03$ Å^2. All parameters associated with hydrogen atoms held invariant. Function minimized $(|F_o| - |F_c|)^2$, $R = 6·1$ (I), 6·2% (II). Anomalous-dispersion corrections made for I in (I) and for S in (II). Scattering factors from Cromer & Mann (1968). Unit weights used throughout. Max. $\Delta/\sigma = 0·2$. Max., min. height in final difference Fourier map = 0·3, −0·3 e Å^{-3}. Final atomic coordinates for (I) and (II) are given in Tables 1 and 2.*

Discussion. Morphine methyl iodide monohydrate (I) and (morphine)$_2$ dihydrogensulfate pentahydrate (II) in the crystalline state display similar bond angles and distances (Table 3, Fig. 1 and 2). In (I) average bonding angles [109·5 (10)°] about nitrogen bound to four carbon atoms display no significant distortion from the 109° angle expected for sp^3 hybridization. In (II) where the nitrogen is bound to one hydrogen and three carbons, the C—N—C angles show a larger average [112·6 (7)°], which may be expected considering the relative volumes of carbon and hydrogen and the expected compression of C—N—H angles.

In both molecules ring *C* displays a distorted boat geometry and ring *E* (containing nitrogen) is in the chair conformation. In (II) the methyl group, C(17), occupies an equatorial position relative to ring *E*, the axial position being occupied by a hydrogen from H_2SO_4. Comparisons may be drawn between these two salts and the hydrochloride salt (Gylbert, 1973). While structural details for the rest of the carbon skeleton are similar to those of the title structures, those of ring *E* more closely resemble those of the sulfate salt (II) than the methyl iodide salt (I).

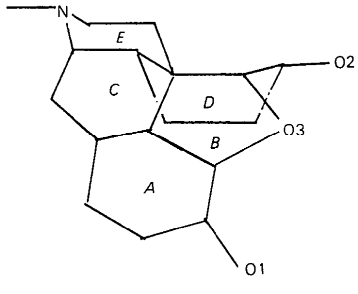

Table 1. *Positional parameters for* $C_{18}H_{22}NO_3I.H_2O$ (I)

U_{eq} = ⅓ the trace of the diagonalized matrix.

	x	*y*	*z*	U_{eq}(Å^2 × 10^3)
I(1)	−0·0021 (1)	0·0475 (1)	−0·10331 (4)	39·7
N(1)	−0·0120 (10)	0·4205 (6)	0·0414 (4)	27·0
O(1)	−0·1314 (11)	0·3350 (9)	0·4342 (4)	49·0
O(2)	0·3102 (12)	0·2273 (9)	0·3486 (4)	55·7
O(3)	0·1016 (8)	0·3857 (7)	0·3325 (4)	30·0
O(4)	−0·3310 (12)	0·2172 (9)	0·5114 (5)	61·0
C(1)	−0·2678 (11)	0·2522 (12)	0·2412 (7)	34·3
C(2)	−0·2637 (11)	0·2782 (11)	0·3211 (6)	33·3
C(3)	−0·1433 (12)	0·3208 (10)	0·3556 (6)	31·3
C(4)	−0·0291 (9)	0·3492 (8)	0·3106 (6)	26·0
C(5)	0·1954 (10)	0·3723 (9)	0·2663 (6)	27·3
C(6)	0·2831 (12)	0·2557 (12)	0·2695 (8)	33·0
C(7)	0·2227 (11)	0·1417 (9)	0·2280 (6)	34·7
C(8)	0·1537 (11)	0·1540 (9)	0·1634 (6)	31·3
C(9)	0·0017 (15)	0·2870 (7)	0·0766 (4)	26·3
C(10)	−0·1358 (11)	0·2423 (10)	0·1145 (6)	32·3
C(11)	−0·1504 (10)	0·2816 (9)	0·1972 (6)	26·0
C(12)	−0·0382 (9)	0·3343 (8)	0·2322 (5)	23·0
C(13)	0·0948 (9)	0·3757 (8)	0·1962 (5)	22·0
C(14)	0·1275 (10)	0·2845 (8)	0·1305 (6)	23·3
C(15)	0·0822 (10)	0·5093 (9)	0·1618 (6)	29·3
C(16)	−0·0314 (10)	0·5140 (8)	0·1042 (6)	30·7
C(17)	−0·1335 (14)	0·4272 (14)	−0·0129 (8)	45·0
C(18)	0·1151 (12)	0·4487 (14)	−0·0061 (7)	38·3

Table 2. *Positional parameters for* $C_{17}H_{19}NO_3.\frac{1}{2}H_2SO_4.2\frac{1}{2}H_2O$ (II)

U_{eq} = ⅓ the trace of the diagonalized matrix.

	x	*y*	*z*	U_{eq}(Å^2 × 10^3)
S(1)	0·6527 (3)	0·5000	0·5000	36·0
O(1)	0·4294 (7)	0·1036 (11)	0·2791 (2)	63·7
O(2)	0·3741 (7)	0·6331 (9)	0·3823 (2)	60·0
O(3)	0·3315 (6)	0·2453 (9)	0·3623 (2)	39·3
O(111)	0·7539 (7)	0·3284 (9)	0·4926 (2)	58·0
O(112)	0·5537 (8)	0·4730 (11)	0·5380 (2)	72·0
O(113)	0·5251 (8)	0·5064 (15)	0·3038 (2)	86·0
O(114)	0·6911 (8)	0·3011 (10)	0·0108 (2)	71·3
O(115)	0·9093 (10)	0·0000	0·0000	77·0
N(1)	−0·2470 (7)	0·1135 (9)	0·4185 (2)	31·3
C(1)	−0·0034 (10)	0·0153 (14)	0·2802 (3)	39·7
C(2)	0·1561 (10)	0·0129 (14)	0·2669 (3)	45·0
C(3)	0·2760 (10)	0·0841 (12)	0·2930 (3)	40·3
C(4)	0·2333 (9)	0·1507 (11)	0·3332 (3)	35·7
C(5)	0·2319 (9)	0·3281 (10)	0·3976 (3)	37·3
C(6)	0·2207 (11)	0·5536 (11)	0·3900 (3)	39·7
C(7)	0·1052 (11)	0·6083 (11)	0·3561 (3)	43·7
C(8)	−0·0372 (10)	0·5260 (10)	0·3567 (3)	35·3
C(9)	−0·2213 (8)	0·2444 (11)	0·3790 (3)	35·0
C(10)	−0·2102 (8)	0 1260 (13)	0·3366 (3)	43·3
C(11)	−0·0434 (8)	0·0858 (10)	0·3216 (3)	28·3
C(12)	0·0791 (8)	0·1382 (10)	0·3484 (3)	31·3
C(13)	0·0672 (8)	0·2301 (10)	0·3930 (3)	88·0
C(14)	−0·0746 (8)	0·3714 (9)	0·3900 (3)	33·3
C(15)	0·0405 (8)	0·0828 (10)	0·4299 (3)	35·7
C(16)	−0·1114 (9)	−0·0283 (10)	0·4252 (3)	37·3
C(17)	−0·4023 (10)	0·0080 (21)	0·4195 (5)	65·3

In (II), the two-carbon bridge, C(15)—C(16), shows no evidence of disorder; bond angles and distances involving the two atoms are normal and the anisotropic thermal parameters are of reasonable magnitude. Thus the observation of two peaks at the C(15) resonance position (Hexem, Frey & Opella, 1983) in the solid-state ^{13}C spectrum of this material appears not to be derived from the presence of two environments for this carbon in the solid state as has been suggested.

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters for (I) and (II) have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39467 (46 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 3. *Bond lengths* (Å) *and angles* (°) *for morphine methyl iodide*.H_2O (I), (*morphine*)$_2$*dihydrogensulfate*.-$5H_2O$ (II) *and morphine hydrate* (III)

	I	II	III
C(1)–C(2)	1·43 (2)	1·41 (1)	1·385 (6)
C(2)–C(3)	1·38 (2)	1·38 (1)	1·402 (5)
C(3)–C(4)	1·38 (1)	1·36 (1)	1·387 (5)
C(4)–O(3)	1·36 (1)	1·38 (1)	1·381 (4)
C(5)–C(6)	1·50 (2)	1·56 (1)	1·526 (6)
C(6)–O(2)	1·44 (2)	1·43 (1)	1·413 (5)
C(7)–C(8)	1·32 (1)	1·33 (1)	1·313 (6)
C(9)–C(10)	1·55 (2)	1·54 (1)	1·556 (6)
C(10)–C(11)	1·52 (1)	1·51 (1)	1·519 (6)
C(12)–C(13)	1·49 (1)	1·51 (1)	1·509 (5)
C(13)–C(15)	1·54 (1)	1·53 (1)	1·535 (6)
N(1)–C(9)	1·55 (1)	1·52 (1)	1·476 (5)
N(1)–C(17)	1·50 (2)	1·50 (1)	1·476 (6)
C(1)–C(11)	1·40 (1)	1·40 (1)	1·396 (6)
C(3)–O(1)	1·39 (1)	1·37 (1)	1·359 (5)
C(4)–C(12)	1·39 (1)	1·39 (1)	1·367 (5)
C(5)–O(3)	1·48 (1)	1·48 (1)	1·470 (4)
C(5)–C(13)	1·56 (1)	1·55 (1)	1·553 (5)
C(6)–C(7)	1·53 (2)	1·48 (1)	1·509 (6)
C(8)–C(14)	1·52 (1)	1·50 (1)	1·500 (6)
C(9)–C(14)	1·53 (2)	1·55 (1)	1·535 (5)
C(11)–C(12)	1·36 (1)	1·37 (1)	1·373 (5)
C(13)–C(14)	1·54 (1)	1·54 (1)	1·541 (5)
C(15)–C(16)	1·48 (1)	1·50 (1)	1·526 (7)
N(1)–C(16)	1·49 (1)	1·52 (1)	1·475 (5)
N(1)–C(18)	1·50 (1)		
S(1)–O(111)		1·469 (6)	
S(1)–O(112)		1·448 (7)	
C(1)–C(2)–C(3)	121 (1)	122·1 (8)	122·2 (4)
C(2)–C(3)–O(1)	123 (1)	123·2 (8)	120·9 (4)
C(3)–C(4)–O(3)	129 (1)	125·6 (7)	124·3 (4)
C(12)–C(4)–O(3)	112 (1)	122·2 (8)	113·0 (3)
C(13)–C(12)–C(4)	109 (1)	110·0 (6)	109·5 (3)
C(10)–C(11)–C(12)	118 (1)	118·4 (8)	117·9 (4)
C(2)–C(3)–C(4)	119 (1)	116·6 (8)	115·5 (4)
O(1)–C(3)–C(4)	118 (1)	120·1 (8)	123·3 (4)
C(3)–C(4)–C(12)	119 (1)	122·2 (8)	122·2 (4)
C(11)–C(12)–C(4)	123 (1)	121·6 (8)	122·4 (4)
C(11)–C(12)–C(13)	127 (1)	127·0 (7)	127·1 (4)
C(1)–C(11)–C(12)	118 (1)	116·8 (7)	116·9 (4)
C(1)–C(11)–C(10)	123 (1)	124·4 (7)	124·4 (4)
C(4)–O(3)–C(5)	108 (1)	108·0 (6)	106·3 (3)
C(13)–C(5)–O(3)	104 (1)	106·2 (6)	106·0 (3)
C(5)–C(13)–C(12)	101 (1)	101·6 (6)	100·3 (3)
C(5)–C(13)–C(15)	112 (1)	110·4 (6)	112·8 (4)
C(12)–C(13)–C(15)	112 (1)	114·1 (6)	112·5 (4)
C(5)–C(6)–C(7)	115 (1)	113·3 (7)	113·2 (3)
O(2)–C(6)–C(7)	111 (1)	112·9 (7)	109·3 (4)
C(7)–C(8)–C(14)	120 (1)	119·8 (8)	119·9 (4)
C(8)–C(14)–C(9)	112 (1)	114·4 (7)	113·5 (4)
C(9)–C(10)–C(11)	114 (1)	114·3 (6)	115·0 (4)
N(1)–C(9)–C(10)	112 (1)	112·1 (6)	114·9 (4)
C(13)–C(15)–C(16)	111 (1)	112·8 (7)	111·5 (4)
C(16)–N(1)–C(9)	109 (1)	112·0 (6)	111·9 (3)
C(17)–N(1)–C(16)	110 (1)	110·8 (7)	110·3 (4)
C(18)–N(1)–C(9)	110 (1)		
C(11)–C(1)–C(2)	118 (1)	119·9 (8)	120·6 (4)
O(3)–C(5)–C(6)	113 (1)	107·5 (6)	110·4 (3)
C(13)–C(5)–C(6)	113 (1)	110·9 (6)	113·3 (3)
C(5)–C(13)–C(14)	117 (1)	115·8 (5)	115·7 (3)
C(12)–C(13)–C(14)	108 (1)	104·9 (6)	106·0 (3)
C(14)–C(13)–C(15)	107 (1)	109·9 (6)	109·1 (3)
C(5)–C(6)–O(2)	108 (1)	110·2 (7)	113·3 (3)
C(6)–C(7)–C(8)	121 (1)	118·9 (8)	121·2 (4)
C(8)–C(14)–C(13)	109 (1)	108·4 (6)	109·6 (4)
C(13)–C(14)–C(9)	107 (1)	106·6 (5)	107·3 (3)
C(10)–C(9)–C(14)	113 (1)	115·4 (6)	112·6 (4)
N(1)–C(9)–C(14)	109 (1)	105·6 (6)	107·7 (3)
C(15)–C(16)–N(1)	113 (1)	109·8 (6)	111·3 (4)
C(17)–N(1)–C(9)	111 (1)	115·1 (7)	112·6 (4)
C(17)–N(1)–C(18)	105 (1)		
C(18)–N(1)–C(16)	112 (1)		

Table 3 (*cont.*)

	II
O(111)–S(1)–O(111′)	108·7 (4)
O(111)–S(1)–O(112)	111·1 (4)
O(111)–S(1)–O(112′)	108·4 (4)

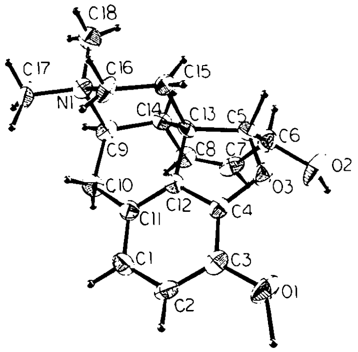

Fig. 1. Projection view of morphine methyl iodide monohydrate.

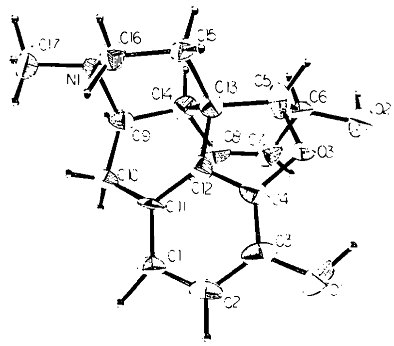

Fig. 2. Projection view of (morphine)$_2$.H_2SO_4 pentahydrate.

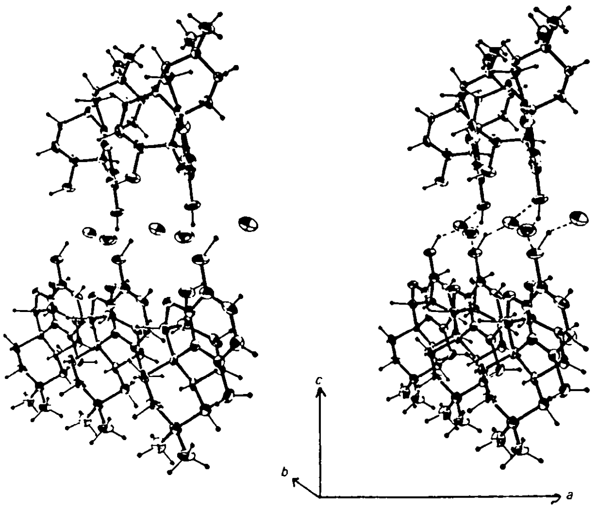

Fig. 3. Packing of morphine methyl iodide monohydrate in the unit cell.

In (I), a linear chain of hydrogen bonding extends through the crystal along the unit-cell axis *a*. The phenolic hydrogen, H(O1), is hydrogen bonded to the water oxygen molecule O(4) [H(O1)···O(4) 1·517 (11) Å] and the water oxygen O(4) appears to donate one hydrogen to the hydroxyl oxygen O(2′) on an adjacent morphine molecule [O(4)···O(2′) 2·86 (1) Å] and another hydrogen to the hydroxyl oxygen O(1′) of the same adjacent morphine molecule [O(4)···O(1′) 3·08 (2) Å]. Thus the hydrogen-bonding network links molecules with symmetry modifications x,y,z; $\frac{1}{2}+x$, $\frac{1}{2}-y$, $1-z$; $1+x$, y, z, *etc*., tracing a clockwise turn progressing from the origin along the *a* direction (Fig. 3). In (II) the oxygen, O(111), of the sulfate group accepts the hydrogen bound to nitrogen H(N1) of one morphine molecule in a hydrogen bond [O(111)···H(N1) 1·71 (6) Å] and a second sulfate oxygen, O(112), is within hydrogen-bonding distance of the hydroxyl hydrogen H(O2′) of an adjacent morphine molecule [O(112)···H(O2′) 2·39 (15) Å]. Thus a network of hydrogen bonding extends along the *a* axis linking in a clockwise spiral molecules of morphine with symmetry modifications x, y, z; $\frac{1}{2}+x$, $1-y$, $1-z$; $1+x$, y, z, *etc.* (Fig. 4). Two further hydrogen bonds occur between water oxygen O(113) and phenolic hydrogen H(O1) [O(113)···H(O1) 1·87 (15) Å] and between a hydrogen bonded to O(113) and O(2) of the same morphine molecule [O(113)···O(2) 2·862 (9) Å].

Thus in both (I) and (II) intermolecular hydrogen-bonding networks extend along an axis of the crystal linking molecules related by a 2_1 screw axis [x, y, z; $\frac{1}{2}+x$, $-y$, $-z$] and the chirality derived from the packing of the molecules in the unit cell is similar.

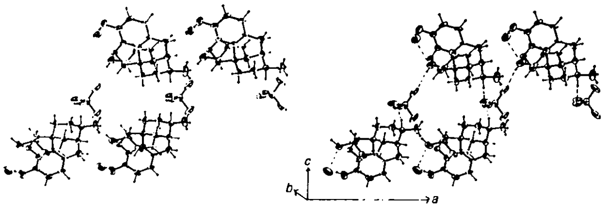

Fig. 4. Packing of (morphine)$_2$.H_2SO_4 pentahydrate in the unit cell.

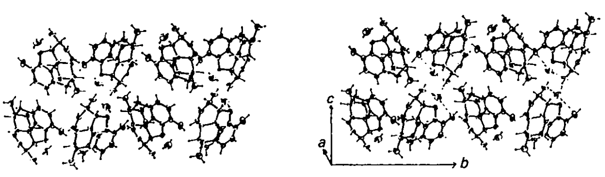

Fig. 5. Packing of morphine hydrate in the unit cell based on the atomic coordinates of Bye (1976). Primary network of hydrogen bonding designated by ...., secondary network designated by —·—·—·—·.

In the morphine free base, however, two networks of hydrogen bonding exist. One network consists of stronger hydrogen-bonded interactions forming a clockwise spiral along the *b* axis: the nitrogen on one morphine molecule attracts a phenolic hydrogen of a second morphine molecule [N(1)—H(O1′) 1·57 (3) Å], thus linking molecules with symmetry modifications x, y, z; $-x$, $\frac{1}{2}+y$, $\frac{1}{2}-z$; x, $1+y$, z, *etc*., *i.e.* molecules related by a 2_1 screw axis parallel to the *b* cell edge (Fig. 5).

A second and weaker network of hydrogen bonding involves hydrogens H(1O4) and H(2O4) of a single water molecule hydrogen bonding to O(2) and O(1), respectively, of the same morphine unit [H(1O4)···O(2) 1·97 (4) and H(2O4)···O(1) 2·27 (5) Å]. The oxygen of this water molecule, O(4), accepts a hydrogen H(O2′) of a second morphine molecule [O(4)···O(2′) 1·91 (3) Å] with symmetry modification $-\frac{1}{2}+x$, $\frac{1}{2}-y$, $1-z$. Thus a second network of hydrogen bonding links molecules related by a screw axis parallel to the *a* cell edge.

It is of interest to note that single-crystal X-ray examination of the white and yellow crystalline forms of 14-hydroxymorphinone (Chiang, DeCamp, Curtin, Paul, Shifrin & Weiss, 1978) showed the yellow forms to crystallize with an intermolecular hydrogen bond between hydroxyl O(3)—H(3) and a carbonyl oxygen atom and the hydroxyl hydrogen turned away from the ether oxygen, O(4), as in (I), whereas the white form crystallizes with that hydrogen attracted to the unshared pair on the hydroxyl oxygen of a neighboring molecule and turned towards O(4) as in (II). This difference appears not to influence the CD spectrum of the adjacent chromophore in (I) and (II), both of which are colorless.

From these structural observations, the following conclusions may be reached concerning the differences of the CD spectra of (I) and (II) from that of the morphine free base. The conformations of the morphine molecules are similar. This similarity excludes differences in the chirality of the N(1) atom. For (I) and (II), details of the hydrogen bonding are similar in that both display clockwise spirals of hydrogen bonding linking molecules related by a twofold screw axis parallel to one cell edge. However, this hydrogen-bonding network involves an intermediate water molecule in (I) and an intermediate sulfate molecule in (II). In (I) phenolic hydroxyl groups of the morphine are involved. In (II) a hydrogen bound to nitrogen is involved along with a hydroxyl OH. These differences appear to make insignificant contributions to the CD spectra. The period of the spiral is slightly different in the two salt structures as evidenced by the small differences in the *a* cell edge. It is known that the CD activity of cholesteric liquid crystals is affected by the pitch of the helical arrangement (Sackman & Mohwald, 1973). However, the *b* cell edge of morphine free base is

13·751 (3) Å and thus the periodicity of the spiral of strong hydrogen bonding is much greater. The observed differences in the solid-state CD spectra of the morphine methyl iodide and sulfate salts from that of morphine free base must arise from this difference in periodicity or from the superposition of a second chiral hydrogen-bonding network on the primary chiral hydrogen-bonding network in the solid-state structure of morphine free base.

References

Bowen, J. M., Crone, T. A., Hermann, A. O. & Purdie, N. (1980). *Anal. Chem.* **52**, 2436–2440.

Bowen, J. M. & Purdie, N. (1980). *Anal. Chem.* **52**, 573–575.

Bye, E. (1976). *Acta Chem. Scand. Ser. B*, **30**, 6–11.

Chiang, C. C., DeCamp, W. H., Curtin, D. Y., Paul I. C., Shifrin, S. & Weiss, U. (1978). *J. Am. Chem. Soc.* **100**, 6195–6201.

Cromer, D. T. & Mann, J. B. (1968). *Acta Cryst.* **A24**, 321–324.

Crone, T. A. & Purdie, N. (1981). *Anal. Chem.* **53**, 17–21.

Gylbert, L. (1973). *Acta Cryst.* **B29**, 1630–1635.

Hexem, J. G., Frey, M. H. & Opella, S. J. (1983). *J. Am. Chem. Soc.* **105**, 5717–5719.

Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1980). *MULTAN*80. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

Sackmann, E. & Mohwald, H. (1973). *J. Chem. Phys.* **58**(2), 5407–5416.

Stewart, J. M. (1978). Editor, the *XRAY* system – version of 1976. Tech. Rep. TR-446. Computer Science Center, Univ. of Maryland, College Park, Maryland.

Corrections and Additions

Acta Cryst. (1984). C**40**, 1490

Structure of methyl 8-isopropyl-3,3a,8,8a-tetrahydroindeno[2,1-*c*]pyrazole-8a-carboxylate, $C_{15}H_{18}N_2O_2$: erratum.

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(*Received* 22 *May* 1984)

In the abstract of the paper by Toupet & Messager [*Acta Cryst.* (1984). C**40**, 330–331], the cell parameters are incorrect. The correct values are: $a = 5{\cdot}791$ (4), $b = 15{\cdot}503$ (4), $c = 15{\cdot}954$ (5) Å, $\alpha = 82{\cdot}24$ (5), $\beta = 79{\cdot}35$ (6), $\gamma = 79{\cdot}13$ (5)°, $V = 1375$ (4) Å^3.

0108-2701/84/081490-01$01.50

Acta Cryst. (1984). C**40**, 1490

Structure of 6-chloro-4-phenyl-1,2,3-benzoxathiazine 2,2-dioxide, $C_{13}H_8ClNO_3S$: erratum.

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(*Received* 15 *June* 1984)

A printer's error is corrected. In the paper by Safia Mehdi & Rama Rao [*Acta Cryst.* (1984). C**40**, 1057–1059] two of the atom labels in Table 1 are incorrect. The correct labels with their corresponding fractional coordinates are given below:

	x	y	z
C(5)	9604 (9)	4384 (5)	3791 (4)
C(6)	9878 (9)	5304 (5)	3782 (4)
Cl	10979 (3)	5813 (2)	4392 (1)