Table 2. *Torsion angles (°) for ring B in the solid-state conformations of colchinoids*

When a compound number is repeated there are two molecules in the crystallographic asymmetric unit. E.s.d.'s for torsion angles in the present work are $\leq 1\cdot0°$.

Compound*	Torsion angle†						
	(1)	(2)	(3)	(4)	(5)	(6)	(7)
(1)	52	2	−78	49	39	−1	−71
(2)	53	6	−78	44	44	−7	−68
(2)	57	7	−79	46	42	−9	−70
(3)	53	5	−79	48	43	−4	−73
(3)	53	5	−81	49	42	−5	−70
(4)	56	−4	−75	54	36	−2	−69
(5)	53	3	−80	49	43	−3	−73
(5)	54	1	−79	50	41	−3	−72

* The compounds and references are: (1) colchicine acetate (Miravitlles, Solans, Bladé-Font, Germain & Declercq 1982); (2) isocolchicine (Lessinger & Margulis, 1978a); (3) colchicine (Lessinger & Margulis, 1978b); (4) colchiceine (Silverton, 1979); (5) N-acetyldemecolcine (this paper).

† Torsion angles are: (1) C(4a)–C(12b)–C(12a)–C(7a); (2) C(12b)–C(12a)–C(7a)–C(7); (3) C(12a)–C(7a)–C(7)–C(6); (4) C(7a)–C(7)–C(6)–C(5); (5) C(7)–C(6)–C(5)–C(4a); (6) C(5)–C(4a)–C(12b)–C(12a); (7) C(6)–C(5)–C(4a)–C(12b).

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Structure of 4-Amino-2-thioxo-6-pyrimidinone* Monohydrate, C4H5N3OS.H2O

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Abstract. $M_r = 161\cdot18$, monoclinic, $C2/c$, $a = 14\cdot892$ (3), $b = 7\cdot593$ (4), $c = 12\cdot991$ (4) Å, $\beta = 113\cdot51$ (2)°, $V = 1347\cdot0$ (4) Å³, $Z = 8$, $D_m = 1\cdot56$ (2), $D_x = 1\cdot59$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0\cdot71073$ Å, $F(000) = 672$, $\mu = 0\cdot362$ mm⁻¹, room temperature, $R = 0\cdot037$, 1117 unique observed reflections. The asymmetric unit consists of one heterocycle and one water molecule. A network of hydrogen bonds involves the water molecules, the ketonic O atom and the imido N atoms. The heterocyclic ring shows small deviations [max. $0\cdot015$ (2) Å] from planarity. The molecule exists in the thione-oxo-amino tautomeric form. There are no unusual bond distances or angles.

* IUPAC name: 6-amino-2-thioxo-2,3-dihydro-4(1H)-pyrimidinone.

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Introduction. As part of a study of heterocyclic thiones a series of structure determinations has been undertaken (Raper, Oughtred & Nowell, 1983; Raper, Creighton, Oughtred & Nowell, 1983). The structure of the title compound is reported herein.

Experimental. Material supplied by Aldrich Chemical Company, England, was recrystallized from water and then from absolute alcohol to give pale-yellow prisms; density measured in toluene–CCl₄ by flotation; systematic absences (from Weissenberg photographs); space group $C2/c$ (confirmed by subsequent analysis); crystal $0\cdot35 \times 0\cdot52 \times 0\cdot22$ mm; Enraf–Nonius CAD-4 diffractometer, Mo $K\alpha$ radiation; 2θ–ω scan, $4 < \theta < 50°$; lattice parameters measured using 25 reflections, θ range 1–48°; 1233 unique reflections, 1117 with $|F_o| > 2\sigma(F)$ used in structure determination; $-17 < h < 16, 0 < k < 8, 0 < l < 15$; intensities correc-

ted for Lorentz and polarization effects but not for absorption; reflections $6\bar{2}1$, $13\bar{2}$ and $44\bar{1}$ used as standards, no significant variation; R_{int} from merging equivalent reflections $= 0.032$; structure solved by direct methods using atomic scattering factors from *International Tables for X-ray Crystallography* (1974); E map gave positions of S and attached C atom, successive difference Fourier syntheses revealed positions of all remaining atoms; isotropic thermal parameters and positional parameters of the H atoms were not refined; final least-squares refinement of non-H atoms using F magnitudes and anisotropic temperature factors gave $R = 0.037$, $wR = 0.069$, $w = [\sigma^2 F_o + 0.006123(F_o)^2]^{-1}$; max. least-squares-shift to error in final refinement cycle 0.86 [$S(2) - U_{33}$]; largest peak in final difference map 0.27, largest hole -0.36 e Å^{-3}; numerical calculations performed on the NUMAC, IBM 370/168 computer at the University of Newcastle upon Tyne with *SHELX76* (Sheldrick, 1976).

Discussion. The final atomic parameters are listed in Table 1.* The molecular structure and atomic numbering are shown in Fig. 1. Table 2 lists bond lengths and angles and in Table 3 (deposited) they are compared with those of related structures. The heterocyclic ring is nearly planar with a maximum deviation of 0.015 (2) Å by atom N(3). The corresponding value for a similar structure, thiocytosine, is 0.043 Å by atom N(1) (Furberg & Jensen, 1970).

The heterocycle can, potentially, exist in thiol-thione, amino-imino and oxo-hydroxy tautomeric forms. This work shows that the thione-oxo-amino form predominates in the solid state.

The asymmetric units are linked by a network of hydrogen bonds (Table 4 and Fig. 2) involving the water molecules, the ketonic O atom and the imido-N atoms forming ribbon structures parallel to the c axis.

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* Lists of structure factors, anisotropic temperature factors, H-atom coordinates, bond distances involving H atoms, least-squares planes and Table 3 have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42012 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
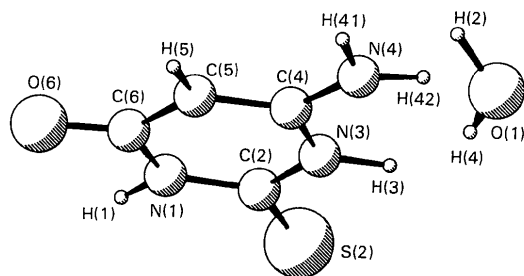

Fig. 1. Perspective view of the title compound.

The amino group is not involved in this type of contact, although this has been reported in the case of other aminopyrimidines (Clews & Cochran, 1949).

Table 1. *Final fractional coordinates* ($\times 10^4$, *for* S $\times 10^5$) *and* B_{eq} *values* (*Willis & Pryor*, 1975) *for non-*H *atoms, with e.s.d.'s in parentheses*

	x	y	z	$B_{eq}(\text{Å}^2)$
N(1)	2289 (1)	4861 (2)	5501 (1)	1.86 (4)
C(2)	2113 (1)	4631 (2)	4408 (1)	1.71 (5)
S(2)	10964 (3)	36632 (6)	35038 (3)	2.61 (2)
N(3)	2815 (1)	5260 (2)	4082 (1)	1.93 (4)
C(4)	3633 (1)	6118 (2)	4778 (1)	1.81 (4)
N(4)	4236 (1)	6724 (2)	4324 (1)	2.70 (5)
C(5)	3801 (1)	6318 (2)	5897 (1)	1.85 (4)
C(6)	3118 (1)	5664 (2)	6283 (1)	1.91 (4)
O(6)	3182 (1)	5762 (2)	7272 (1)	2.89 (4)
O(1)	910 (1)	6490 (1)	1315 (1)	2.61 (3)

Table 2. *Bond lengths* (Å) *and angles* (°) *involving non-*H *atoms, with e.s.d.'s in parentheses*

N(1)–C(2)	1.349 (2)	C(6)–N(1)–C(2)	124.8 (2)
C(2)–S(2)	1.671 (1)	S(2)–C(2)–N(1)	122.5 (1)
C(2)–N(3)	1.361 (2)	S(2)–C(2)–N(3)	121.9 (1)
N(3)–C(4)	1.359 (2)	N(1)–C(2)–N(3)	115.5 (1)
C(4)–N(4)	1.337 (3)	C(2)–N(3)–C(4)	123.9 (1)
C(4)–C(5)	1.382 (2)	N(3)–C(4)–N(4)	117.0 (2)
C(5)–C(6)	1.394 (3)	N(4)–C(4)–C(5)	123.7 (1)
C(6)–O(6)	1.252 (2)	N(3)–C(4)–C(5)	119.4 (2)
C(6)–N(1)	1.388 (2)	C(4)–C(5)–C(6)	119.2 (1)
		O(6)–C(6)–C(5)	125.5 (1)
		O(6)–C(6)–N(1)	117.4 (2)
		C(5)–C(6)–N(1)	117.1 (1)

Table 4. *Hydrogen-bond distances* (Å) *and angles* (°)

Symmetry code: none x, y, z; (i) $\frac{1}{2} - x$, $1\frac{1}{2}$ y, $1 - z$; (ii) x, $1 - y$, $\frac{1}{2} + z$; (iii) x, $1 - y$, $z - \frac{1}{2}$.

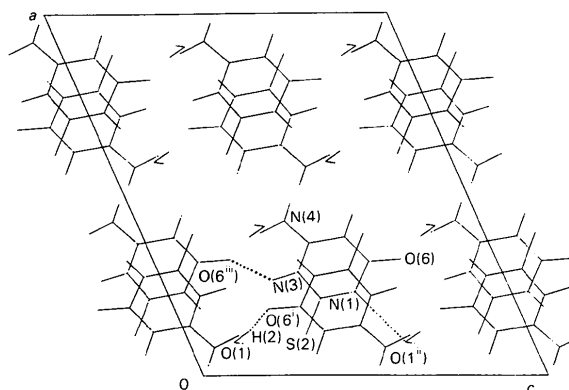| Donor–H···Acceptor (D–H···A)	D–H	H···A	D···A	$\angle DHA$
O(1)–H(2)···O(6^i)	1.039	1.722	2.751 (2)	169.9
N(1)–H(1)···O(1^{ii})	0.920	1.930	2.850 (2)	180.0
N(3)–H(3)···O(6^{iii})	1.039	1.884	2.730 (2)	136.2

Fig. 2. Unit-cell contents viewed down **b**. Hydrogen bonds are indicated by dotted lines. For clarity only a unique set is shown.

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Structure of Modification I of Cortisone Acetate, 21-Acetoxy-17α-hydroxy-4-pregnene-3,11,20-trione,* $C_{23}H_{30}O_6$

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Abstract. $M_r = 402.49$, orthorhombic, $P2_12_12_1$, $a = 8.018$ (4), $b = 12.579$ (7), $c = 20.963$ (1) Å, $Z = 4$, $V = 2114$ (2) Å^3, $D_x = 1.264$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0.71069$ Å, $\mu = 0.53$ cm^{-1}, $F(000) = 864$, $T = 294$ K, $R = 0.049$ for 1302 observed reflections with $I > 2.5\sigma(I)$. Rings *A*, *B* and *C* have distorted 1α-sofa (*A*) and distorted chair (*B* and *C*) conformations in both modifications I and II [Declercq, Germain & Van Meerssche (1972). *Cryst. Struct. Commun.* **1**, 59–62]. The differences between the two forms relate to ring *D* which is a 13β-envelope in I and a 13β,14α-half chair in II, and to the orientation of the side chains, where the average differences of the torsion angles about C(17)—C(20) and C(20)—C(21) are 16 (1) and 14 (1)° respectively. In I the molecules are connected by an intermolecular head-to-tail H bond [O(17)—H···O(3)], while in II there is an O(17)—H···O(22) head-to-head H bond. The packing mode of I is 221, of II 212.

Introduction. Three anhydrous polymorphs of cortisone acetate (CA), hereafter called mod I, mod II and mod III, have been reported in the literature (Callow & Kennard, 1961; Kuhnert-Brandstätter & Grimm, 1968*a,b*; Carless, Moustafa & Rapson, 1966; Shirotani & Sekiguchi, 1981). In pharmaceutical literature the high-melting polymorph is called mod I (Kuhnert-

Brandstätter & Grimm, 1968*a,b*) which is identical to modification III of Callow & Kennard (1961). In this paper the pharmaceutical nomenclature will be followed.

Both mod I and mod II were shown to be orthorhombic with space group $P2_12_12_1$, $Z = 4$ (Callow & Kennard, 1961), but with considerably different cell dimensions (for mod II: $a = 11.048$, $b = 7.102$, $c = 27.095$ Å), indicating a different packing of CA molecules. A sample manufactured by Laboratoires Français de Chimiothérapie (Roussel) was also analysed by Callow & Kennard (1961) and this form (mod III) was reported to be monoclinic ($P2_1$) with three independent molecules in the unit cell.

A structure analysis of mod II, which can easily be prepared from solvents like benzene or chloroform, was performed by Declercq, Germain & Van Meerssche (1972). The present paper reports the analysis of mod I.

Experimental. Crystallization of CA from absolute ethanol affords the mono-ethanolate from which solvent-free CA was obtained by heating at 373 K. The resulting powder, consisting of mod I, was used to seed a solution of CA in hot toluene. Slow cooling of this solution yields a mixture of single crystals of mod I, which appear as prisms, and of mod II, which are in the form of fine needles. A crystal of mod I with dimensions $0.3 \times 0.4 \times 1.0$ mm was selected for the analysis. D_m not determined.

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* IUPAC name: 17α-hydroxy-3,11,20-trioxo-4-pregnen-21-yl acetate.