The molecular packing of sulfisomidine as seen down the c axis is shown in Fig. 2. The dotted lines indicate the hydrogen bonds. The amino nitrogen atom N(7) is a proton donor in a hydrogen bond with O(10) of the symmetry-related molecule [N(7)—H(71)···O(10) = 3·131 (5) Å, 165·5 (5)°] and the imino nitrogen atom N(11) forms a hydrogen bond with N(15) of the symmetry-related molecule [N(11)—H(11)···N(15) = 2·948 (3) Å, 149·3 (4)°]. There is one significantly short intermolecular contact distance of van der Waals type between O(9) and N(15) [O(9)···N(15) = 2·939 (4) Å]. The molecules are stacked together along the diagonals of the ab plane and cross-link each other at the centre of the cell.

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Structure of (7S)-N-Acetyldemecolcine, $C_{23}H_{27}NO_6$

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Abstract. $M_r = 413·47$, monoclinic, $P2_1$, $a = 11·629$ (1), $b = 11·748$ (1), $c = 15·782$ (2) Å, $\beta = 97·23$ (1)°, $V = 2138·9$ Å³, $D_m = 1·27$ (1), $D_x = 1·284$ g cm⁻³, $Z = 4$, $\lambda(\text{Cu }K\alpha) = 1·5418$ Å, $\mu = 6·12$ cm⁻¹, $F(000) = 880$, $T = 293$ K, $R = 0·044$ for 2998 reflections. The asymmetric unit of this relatively active colchinoid compound contains two independent molecules which do not differ significantly in bond lengths or angles and the molecular conformations are very similar to each other and to those of previously reported compounds of this type.

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Introduction. Colchicine and its analogs are thought to inhibit biological processes by blocking the polymerization of tubulin. While chemical manipulation of colchicine has indicated that aromatic substitution, proper chirality of the acetimido function and a carbonyl group at C(9) are necessary for effective tubulin binding (Capraro & Brossi, 1983), definite conclusions as to the role of these functionalities are lacking. The present structure represents the first of a series of investigations of the stereochemical characteristics of both relatively active and relatively inactive colchinoids.

The general formula of the colchinoids is given as (I) and N-acetyldemecolcine ($R1 = R2 = R3 = R4 =$ methyl, $R5 =$ acetyl) is a member of the demecolcine

Table 1. *Atomic parameters for the heavier atoms*

The equivalent isotropic temperature factor, U_{eq}, is the geometric mean of the diagonal terms of the vibration tensor. Positional parameters are multiplied by 10^4 and U_{eq} values by 10^3.

	Molecule (1)				Molecule (2)			
	x	y	z	$U_{eq}(\text{Å}^2)$	x	y	z	$U_{eq}(\text{Å}^2)$
N	4024 (2)	−1322 (3)	8555 (2)	56 (1)	9964 (2)	−7980 (3)	3984 (2)	51 (1)
C(1)	8322 (3)	−1905 (3)	9654 (2)	40 (1)	6013 (3)	−6867 (4)	4779 (2)	48 (1)
O(1)	8350 (2)	−2947 (2)	10051 (2)	52 (1)	6184 (2)	−5798 (3)	5129 (2)	62 (1)
C(1m)	7692 (5)	−3812 (5)	9571 (4)	83 (2)	6758 (6)	−5041 (5)	4638 (4)	97 (3)
C(2)	9210 (3)	−1644 (3)	9173 (2)	43 (1)	5003 (3)	−7073 (4)	4228 (2)	48 (1)
O(2)	10026 (2)	−2447 (2)	9036 (2)	58 (1)	4192 (2)	−6221 (3)	4069 (2)	61 (1)
C(2m)	10983 (4)	−2500 (6)	9685 (4)	89 (2)	4079 (4)	−5761 (5)	3219 (4)	80 (2)
C(3)	9245 (3)	−567 (3)	8795 (2)	45 (1)	4809 (3)	−8158 (4)	3888 (2)	52 (1)
O(3)	10181 (2)	−378 (3)	8376 (2)	61 (1)	3771 (2)	−8293 (3)	3372 (2)	65 (1)
C(3m)	10260 (4)	712 (5)	7984 (3)	75 (2)	3642 (4)	−9293 (5)	2855 (3)	73 (2)
C(4)	8358 (3)	185 (4)	8859 (2)	47 (1)	5606 (3)	−9019 (4)	4078 (3)	57 (1)
C(4a)	7456 (3)	−84 (3)	9323 (2)	44 (1)	6623 (3)	−8797 (4)	4631 (2)	53 (1)
C(5)	6485 (3)	755 (4)	9401 (3)	57 (2)	7534 (4)	−9702 (4)	4811 (3)	63 (2)
C(6)	5336 (3)	361 (4)	8894 (3)	60 (2)	8607 (3)	−9472 (4)	4353 (3)	59 (2)
C(7)	5130 (3)	−906 (4)	9006 (2)	46 (1)	8928 (3)	−8213 (4)	4391 (2)	45 (1)
C(7a)	5319 (2)	−1220 (3)	9957 (2)	39 (1)	9013 (2)	−7741 (3)	5304 (2)	41 (1)
C(8)	4371 (3)	−1257 (3)	10374 (2)	42 (1)	10080 (3)	−7614 (3)	5745 (2)	47 (1)
C(9)	4175 (3)	−1443 (3)	11249 (2)	44 (1)	10522 (3)	−7181 (4)	6577 (2)	51 (1)
O(9)	3177 (2)	−1332 (3)	11444 (1)	57 (1)	11581 (2)	−7202 (3)	6801 (2)	73 (1)
C(10)	5117 (3)	−1802 (3)	11902 (2)	44 (1)	9766 (3)	−6681 (3)	7154 (2)	50 (1)
O(10)	4690 (2)	−2071 (3)	12643 (1)	60 (1)	10411 (2)	−6224 (3)	7846 (2)	67 (1)
C(10m)	5481 (4)	−2499 (5)	13347 (3)	72 (2)	9841 (5)	−5579 (6)	8446 (3)	89 (2)
C(11)	6265 (3)	−1873 (4)	11832 (2)	48 (1)	8592 (3)	−6678 (4)	7062 (2)	51 (1)
C(12)	6881 (3)	−1652 (4)	11133 (2)	47 (1)	7786 (3)	−7065 (4)	6379 (2)	51 (1)
C(12a)	6514 (2)	−1352 (3)	10310 (2)	40 (1)	7911 (3)	−7492 (3)	5591 (2)	47 (1)
C(12b)	7440 (2)	−1119 (3)	9754 (2)	39 (1)	6829 (2)	−7726 (3)	4997 (2)	45 (1)
C(13)	3974 (3)	−2454 (5)	8392 (2)	69 (2)	10113 (3)	−6895 (5)	3731 (2)	61 (1)
O(13)	4767 (3)	−3094 (3)	8664 (2)	83 (1)	9403 (2)	−6155 (3)	3855 (2)	86 (1)
C(14)	2905 (5)	−2954 (8)	7860 (4)	107 (3)	11173 (4)	−6618 (7)	3316 (3)	89 (2)
C(15)	3090 (4)	−534 (6)	8242 (4)	89 (2)	10793 (5)	−8888 (6)	3852 (4)	98 (2)

(*N*-methylcolchicine) family of compounds with an activity comparable with colchicine itself (Capraro & Brossi, 1983).

(I)
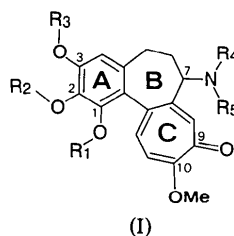
Experimental. (7S)-N-Acetyldemecolcine (crystallized from acetone): m.p. (uncorrected) (Fisher–Johns apparatus) 503 K; optical rotation (Perkin–Elmer 241 polarimeter): $[\alpha]_D^{20°C} = -252°$ ($c = 0.47$ g dm^{-3}, CHCl$_3$). Literature (Hufford, Capraro & Brossi, 1980): m.p. 506–508 K, $[\alpha]_D^{20°C} = -251°$ (CHCl$_3$). Composition calculated for C$_{23}$H$_{27}$NO$_6$: C, H, N 66·81, 6·58, 3·38%, respectively; found (Microanalytical Section, Laboratory of Chemistry, NIADDK, NIH): 66·69, 7·01, 3·44%.

X-ray experimental data: Enraf–Nonius CAD-4 diffractometer; colorless prismatic crystals 0·3 × 0·2 × 0·2 mm, max. $\sin\theta/\lambda = 0.6231$ Å^{-1}; 4553 reflections measured, statistical accuracy 3% (max. time 60 s); 1555 reflections with $I \leq \sigma(I)$. 20 reflections ($20° \leq \theta \leq 30°$) used for measuring lattice parameters. No absorption correction.

No interpretable E maps were produced by automatic and semi-automatic use of MULTAN78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) with up to 9 variable phases. The solution involved selection of planes occurring most frequently in negative quartets (Silverton, Kabuto & Akiyama, 1978) and use of 9 of these as magic-integer variables in the multisolution technique. The reliability of the solutions was assessed by both a weighted figure of merit and the NQEST test (DeTitta, Edmonds, Langs & Hauptman, 1975). The solution with the best value of NQEST, which was also one of the best on the combined figure of merit, gave an E map showing most of the heavier atoms. All subsequent work used the programs of XRAY72 (Stewart, Kruger, Ammon, Dickinson & Hall, 1972) in the sequence: isotropic least-squares refinement of the trial model, finding missing heavy atoms from difference maps, anisotropic refinement, location of all H atoms and final refinement using isotropic refinement of H atoms. There was some difficulty in refining certain H atoms and, in such cases, only the positional parameters were refined. Refinement: weighting scheme after Peterson & Levy (1957), function minimized: $\sum w\Delta^2$, final $R = 0.044$ (observed reflections only), $wR = 0.040$, $S = 1.36$, $\langle \Delta/\sigma \rangle = 0.05$, $|\Delta\rho_{max}| = 0.2$ e Å^{-3}; no correction for secondary extinction; scattering factors from XRAY72; anisotropic temperature factor: $\exp[-2\pi^2\sum_i\sum_j(U_{ij}h_ih_ja_i^*a_j^*)]$. The structure was refined by alternately holding the parameters of one independent molecule constant and

varying those of the other. The origin was effectively fixed by the unrefined atoms and thus e.s.d.'s are given for all y parameters.*

Discussion. The atomic parameters for the heavier atoms are given in Table 1 and crystal conformations, bond lengths and angles for both molecules are shown in Fig. 1. Differences in bond lengths and angles do not exceed 3 e.s.d.'s. (Some differences are numerically large but it can be seen from the thermal ellipsoids depicted in Fig. 1 that the atoms concerned have fairly high thermal parameters.) The molecular conformations of the two independent molecules of N-acetyldemecolcine can be seen to be very similar and it is apparent that the acetyl group is on the same side of both molecules. The only distinct conformational difference between the two independent molecules lies in the position of the O(2) methoxy group. The molecular packing is shown in Fig. 2 and, as might be expected, no hydrogen bonds are formed. The different packing forces acting on the independent molecules appear to account for the minor conformational differences indicated by the following distances (Å) of atoms from least-squares planes of the rings: N to ring A: 3·21, 3·33; N to ring C: 0·78, 0·71; O(9) to ring A: 0·12, 0·90; O(13) to ring A: 3·50, 3·70; O(13) to ring C: 2·69, 2·70.

The molecular conformation observed in the present structure is very similar to that observed for all colchinoids previously reported and all such compounds possessing an N-carbonyl group (for references see Table 2) have it disposed on the same side of the molecule as in the present case. The similarity of ring conformations is indicated in Table 2 which is an extension of the table of Miravitlles, Solans, Bladé-Font, Germain & Declercq (1982). It is pertinent to remark that neither molecular rotations nor absolute configurations are given in the original references for the compounds of the table. Some structures in the literature have atomic coordinates corresponding to the R configuration and some to the S and some show figures with the S configuration but list R coordinates. Miravitlles, Solans, Bladé-Font, Germain & Declercq (1982) show a figure and some torsion angles corresponding to (7S)-colchiceine acetate but their table inverts the values for comparison with the previous literature. Since unnatural colchicine is not readily available, it seems probable that all of the compounds have the 7S configuration which was established for colchicine itself by Corrodi & Hardegger (1955) and

this configuration, implied in the table, is that of the present compound.
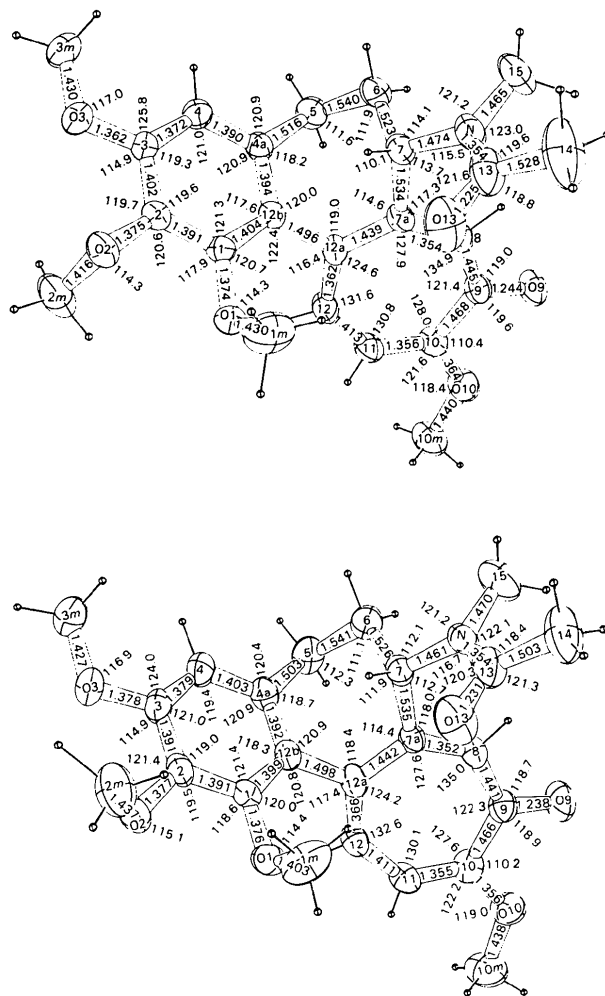

Fig. 1. Crystal conformations and dimensions of the two independent molecules of N-acetyldemecolcine. *ORTEP* drawing (Johnson, 1965); thermal ellipsoids are drawn at 40% probability and the H atoms are arbitrary. E.s.d.'s of bond lengths are ≤0·007 Å and e.s.d.'s of bond angles are ≤0·5°.
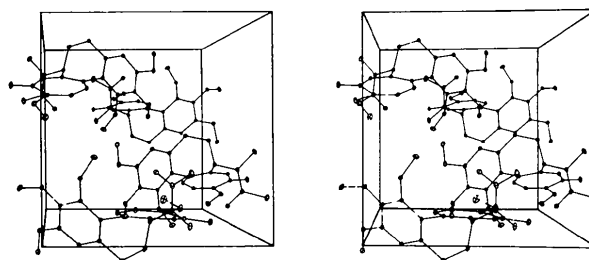

Fig. 2. Packing diagram; direction of projection **a**; coordinate origin at center of depicted **b** axis (horizontal).

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* Lists of factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39994 (23 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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 ≤1·0°.

			Torsion angle†				
Compound*	(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. $M_r = 161·18$, monoclinic, $C2/c$, $a = 14·892$ (3), $b = 7·593$ (4), $c = 12·991$ (4) Å, $\beta = 113·51$ (2)°, $V = 1347·0$ (4) Å^3, $Z = 8$, $D_m = 1·56$ (2), $D_x = 1·59$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0·71073$ Å, $F(000) = 672$, $\mu = 0·362$ mm^{-1}, room temperature, $R = 0·037$, 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. 0·015 (2) Å] from planarity. The molecule exists in the thione-oxo-amino tautomeric form. There are no unusual bond distances or angles.

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* 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–CCl$_4$ by flotation; systematic absences (from Weissenberg photographs); space group $C2/c$ (confirmed by subsequent analysis); crystal 0·35 × 0·52 × 0·22 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-