two rings being 6·9 (4) and 60·4 (5)°. The γ-lactone ring is intermediate between envelope and half-chair conformations with torsion angles at the juncture with the seven-membered ring being −31·4 (3) and 72·4 (3)°. Several atoms exhibit relatively large thermal motion which may be rationalized in terms of a short O(2)···C(14) contact of 2·954 (6) Å. Librational corrections lead to increases in bond lengths of 0·006 to 0·009 Å.

Lactucin (Ruban, Zabel, Gensch & Smalla, 1978) is similar to (1) except for the substituent at C(8) being an α-hydroxyl with a second hydroxyl group attached to C(15). Lactucin has extensive hydrogen bonding in the crystal which leads to small but significant changes in torsion angles and in some distances compared to those observed in (1). The molecules of (1) are held together in the crystal by van der Waals forces leaving sufficient room for thermal motion of methyl groups and substituents.

We thank the Robert A. Welch Foundation [P-074 (WHW), F-130 (TJM)], the National Science Foundation [INT8214877 (WHW)] and the National Institutes of Health [GM-35710 (TJM)].

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Structure of 1-Thia-5-azacyclononan-6-one

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(*Received* 3 *February* 1987; *accepted* 29 *May* 1987)

Abstract. $2(C_7H_{13}NOS)$, $M_r = 318·50$, orthorhombic, *Pbca*, $a = 9·613\ (3)$, $b = 14·468\ (4)$, $c = 23·707\ (5)$ Å, $V = 3297·0\ (15)$ Å^3, $Z = 8$, $D_x = 1·283$ Mg m^{-3} at 130 K, $\lambda(\mathrm{Mo}\ K\alpha) = 0·71069$ Å, $\mu = 0·31$ mm^{-1}, $F(000) = 1376$, $T = 130\ (1)$ K, $R = 0·037$ for 1677 observed reflections. The asymmetric unit contains two molecules linked by a hydrogen bond. Values of bond lengths and angles are normal.

Introduction. Transannular interactions between functional groups in medium-sized ring systems can be manifested by the observation of unusual spectroscopic properties or enhanced chemical reactivity. An interaction between the amine and ketone functions in 6-hydroxy-1-methyl-1-azacyclononan-5-one was revealed by a lowering of its carbonyl stretching frequency (Leonard, Fox & Oki, 1954). An interaction between a thioether and a sulfoxide was revealed by X-ray analysis and manifested in the rapid reduction of 3-methoxy-1,5-dithiacyclooctane 1-oxide by HI to 3-methoxy-1,5-dithiacyclooctane (Doi, Kessler, DeLeeuw, Olmstead & Musker, 1983). In our recent studies of the oxidation of thioethers with aqueous iodine (Doi, Goodrow & Musker, 1986), we found that a transannular amide group in the nine-membered ring 1-thia-5-azacyclononan-6-one increased the rate of thioether oxidation by a factor of 100 relative to a simple thioether (Doi, Bharadwaj & Musker, 1987). To see whether a transannular interaction was partially responsible for this rate acceleration, the structure of this amide-thioether was determined.

Experimental. The title compound was obtained by Beckmann rearrangement (Nace & Watterson, 1966) of 5-thiacyclooctanone oxime *p*-toluenesulfonate (Wise, Morrison, Egan & Lattime, 1971) on alumina, m.p. 359–360 K. Suitable crystals were grown from toluene solution. Colorless rectangular plate 0·31 × 0·23 ×

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0·11 mm; Syntex $P2_1$ diffractometer; systematic absences: *hkl*, none; 0*kl*, $k=2n+1$; *h*0*l*, $l=2n+1$; *hk*0, $h=2n+1$; consistent with the space group *Pbca* (No. 61); cell constants from setting angles of 20 moderately intense reflections with $35 < 2\theta < 50°$; ω scan with 1·0° range in ω and a 1·0° offset for stationary background count; random fluctuations of < 0·1% in intensity of two standard reflections; data corrected for Lorentz, polarization and decay; 2158 unique reflections measured with $2\theta \leq 50°$; 1677 with $I \geq 2\sigma(I)$ used in refinement. Range of *h,k,l:* 0 to 10, 0 to 15, 0 to 25.

Structure solved by direct methods (*SHELXTL*; Sheldrick, 1981). All non-hydrogen atoms located in the map; full-matrix refinement on *F*; at isotropic convergence, all H atoms located in the difference map; all non-hydrogen atoms anisotropic; all H atoms isotropic; data:parameter ratio > 5·8; weights based on counting statistics. Final $R = 0{\cdot}037$, $wR_{(F)} = 0{\cdot}034$, $S = 1{\cdot}181$, $(\Delta/\sigma)_{\max} = 0{\cdot}120$ for *U* of HN(*b*), $(\Delta/\sigma)_{\text{ave}} = 0{\cdot}029$; $\Delta\rho_{\max}$ + 0·23, $\Delta\rho_{\min}$ −0·24 e Å^{-3}; large peaks are located near H atoms connected to C atoms; neutral-atom scattering factors including anomalous terms from *International Tables for X-ray Crystalllography* (1974). Final positional parameters are given in Table 1.*

Discussion. A view of the title compound showing the atom-numbering scheme is given in Fig. 1. The molecule forms an extended intermolecular H-bonded lattice in the crystalline state. In the asymmetric unit there are two molecules connected by a hydrogen bond between the carbonyl O and the H bonded to the amide N. The N(*b*)—H···O(*a*) distance is 2·06 (3) Å while N(*a*)—H···O(*b*) is 2·00 (5) Å. There is no transannular interaction between the two functional groups within this nine-membered ring. The sulfur atom S(*a*) is equidistant from the carbonyl C(4*a*) [3·657 (4) Å] and from the amide N(*a*) [3·616 (4) Å]. The carbonyl oxygen atom, O(*a*), is still further away [4·555 (5) Å]. In molecule *B* these distances are 3·653 (5), 3·659 (4) and 4·557 (5) Å, respectively. The amide plane in molecule *A* described by C(4*a*), C(5*a*), N(*a*) and O(*a*) is planar to within ±0·003 Å. Likewise, the amide plane in molecule *B* is planar to within ±0·002 Å. The amide plane in molecule *A* is tilted with respect to the transannular thioether such that the angle S(*a*)—C(4*a*)—O(*a*) is 129·6°. The amide plane in molecule *B* has a similar orientation.

Table 1. *Atomic coordinates* (×10^4) *and equivalent isotropic thermal parameters* (Å^2×10^3)

	x	*y*	*z*	U_{eq}/U_{iso}
S(*a*)	5841 (1)	1659 (1)	−4 (1)	26 (1)*
O(*a*)	6488 (2)	2356 (1)	1851 (1)	23 (1)*
N(*a*)	7921 (3)	1598 (2)	1266 (1)	20 (1)*
C(1*a*)	6505 (3)	610 (2)	336 (1)	25 (1)*
C(2*a*)	8062 (3)	584 (2)	425 (1)	24 (1)*
C(3*a*)	8676 (3)	1405 (2)	742 (1)	21 (1)*
C(4*a*)	7119 (3)	2320 (2)	1392 (1)	18 (1)*
C(5*a*)	6945 (3)	3107 (2)	977 (1)	22 (1)*
C(6*a*)	5448 (3)	3141 (2)	759 (1)	26 (1)*
C(7*a*)	4863 (3)	2225 (2)	560 (1)	24 (1)*
HN(*a*)	7954 (28)	1167 (18)	1507 (11)	21 (9)
S(*b*)	6760 (1)	4751 (1)	3811 (1)	27 (1)*
O(*b*)	6750 (2)	4962 (1)	1893 (1)	24 (1)*
N(*b*)	5527 (2)	3980 (2)	2429 (1)	21 (1)*
C(1*b*)	6934 (3)	3589 (2)	3516 (1)	26 (1)*
C(2*b*)	5586 (3)	3128 (2)	3337 (1)	26 (1)*
C(3*b*)	4713 (3)	3670 (2)	2913 (1)	23 (1)*
C(4*b*)	5960 (3)	4835 (2)	2305 (1)	17 (1)*
C(5*b*)	5501 (3)	5648 (2)	2654 (1)	22 (1)*
C(6*b*)	6725 (3)	6116 (2)	2950 (1)	23 (1)*
C(7*b*)	7652 (3)	5470 (2)	3289 (1)	26 (1)*
HN(*b*)	5808 (24)	3579 (16)	2205 (10)	24 (7)

* Equivalent isotropic *U* defined as one third of the trace of the orthogonalized U_{ij} tensor.

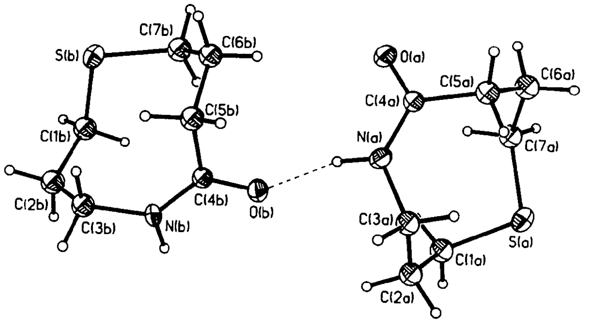

Fig. 1. View of the title compound showing the atom-numbering scheme. H atoms are not named for clarity. Thermal ellipsoids are drawn at the 50% probability level.

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* Lists of bond distances, bond angles, structure factors, H-atom coordinates and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44103 (22 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.