Discussion. Fig. 1 is an *ORTEP* drawing (Johnson, 1971) of solamaladine while Fig. 2 presents a side view of the molecule showing the extended conformation and indicates little bending of the steroid skeleton. All rings are *trans*-fused with the six-membered rings exhibiting chair conformations and the five-membered rings envelope conformations as indicated by the torsion angles. An $O(3)\cdots O(27)(x-1,y,z)$ distance of $3\cdot178$ (5) Å may indicate a weak hydrogen bond; however, the hydroxyl H atom could not be located in a difference Fourier map. All interatomic distances and valence angles are consistent with the structures of other steroids. The present analysis confirms the side-chain structure in solamaladine as being identical to that in the revised structure of tomatillidine and its derivatives.

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The Structure of 2-Chloro-8,9,10,11-tetrahydro-6*H*,11a*H*-pyridazino[1,2-*a*]cinnoline-1,4-dione

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Abstract. $C_{12}H_{13}ClN_2O_2$, $M_r = 252\cdot70$, monoclinic, $P2_1/a$, $a = 13\cdot340$ (2), $b = 18\cdot769$ (4), $c = 4\cdot7181$ (5) Å, $\beta = 99\cdot53$ (2)°, $U = 1165\cdot0$ (3) Å^3, $Z = 4$, $D_c = 1\cdot44$ Mg m^{-3}, $F(000) = 528$. The final $R = 0\cdot072$ for 1529 reflexions. The molecules are held together by van der Waals forces. The dihydropyridazine ring has a half-chair conformation with a maximum torsion angle of 37·4 (6)° at the N—N bond. The diazaquinone ring has an envelope conformation.

Introduction. A study is being carried out in the Departamento de Química Orgánica of the Universidad Complutense in Madrid to obtain diazapolycyclic compounds with quinone structure, which could have biological activity, by introduction of substituents in the diazaquinonic ring (Lora-Tamayo, Pardo, Soto, Verde & Gonzalez, 1980), leading to the synthesis of antibacterial compounds (Gomez-Contreras, Lora-Tamayo & Navarro, 1977; Gomez-Contreras & Lora-Tamayo, 1979). As part of the study, the structure of the title compound (I) has been determined.

(I)
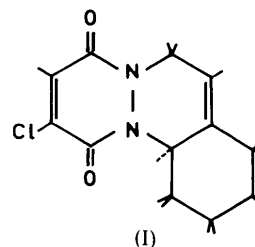
A crystal of dimensions $0\cdot3 \times 0\cdot2 \times 0\cdot35$ mm was used to collect the data on a PW 1100 four-circle diffractometer, with graphite-monochromated Mo $K\alpha$ radiation ($\lambda = 0\cdot71069$ Å). 3454 reflexions were measured in the $\omega/2\theta$ scan mode. The intensities were not corrected for absorption. By the criterion $I < 2\sigma(I)$,

$C_{12}H_{13}ClN_2O_2$

1529 reflexions were considered as unobserved. Scattering factors for neutral atoms and anomalous-dispersion coefficients for Cl were taken from *International Tables for X-ray Crystallography* (1974).

The structure was solved by *MULTAN* (Main, Lessinger, Woolfson, Germain & Declercq, 1977).

Table 1. *Final atomic coordinates* ($\times 10^4$; $\times 10^5$ *for y of* Cl) *and isotropic thermal parameters* ($\text{Å}^2 \times 10^4$)

$$U_{eq} = \frac{1}{3} \sum |U_{ij} a_i^* a_j^* a_i a_j \cos(a_i, a_j)|.$$

	x	y	z	U_{eq}
C(1)	1659 (3)	8055 (2)	5485 (10)	489 (15)
C(2)	1765 (4)	8804 (2)	4295 (13)	636 (19)
C(3)	0810 (4)	9243 (3)	4338 (14)	735 (24)
C(4)	−0135 (4)	8856 (3)	2754 (14)	737 (22)
C(4a)	−0198 (3)	8116 (2)	3904 (10)	506 (15)
C(5)	−0962 (3)	7870 (3)	5072 (12)	610 (18)
C(6)	−0977 (3)	7160 (3)	6362 (13)	703 (22)
N(7)	−0008 (3)	6771 (2)	6614 (8)	501 (13)
C(8)	0036 (4)	6124 (3)	7976 (11)	635 (19)
O(1)	−0581 (3)	5986 (2)	9560 (9)	891 (19)
C(9)	0808 (4)	5627 (3)	7369 (12)	666 (20)
C(10)	1388 (4)	5783 (2)	5467 (12)	588 (18)
Cl	2271 (1)	51935 (7)	4579 (4)	920 (9)
C(11)	1359 (3)	6476 (2)	3998 (11)	499 (15)
O(2)	1939 (3)	6649 (2)	2356 (8)	654 (14)
N(12)	0644 (3)	6939 (2)	4662 (8)	450 (12)
C(12a)	0731 (3)	7686 (2)	3764 (9)	404 (13)

Table 2. *Bond lengths* (Å) *and angles* (°) *for* $C_{12}H_{13}ClN_2O_2$

The results involving H atoms have been omitted. E.s.d.'s are given in parentheses.

C(1)–C(2)	1·528 (7)	C(2)–C(1)–C(12a)	109·8 (4)
C(1)–C(12a)	1·530 (6)		
C(2)–C(3)	1·521 (8)	C(1)–C(2)–C(3)	111·4 (5)
C(3)–C(4)	1·536 (8)	C(2)–C(3)–C(4)	111·0 (5)
C(4)–C(4a)	1·500 (7)	C(3)–C(4)–C(4a)	110·7 (5)
C(4a)–C(12a)	1·490 (6)	C(4)–C(4a)–C(12a)	112·9 (4)
C(4a)–C(5)	1·319 (7)	C(4)–C(4a)–C(5)	124·7 (5)
		C(5)–C(4a)–C(12a)	122·4 (5)
C(5)–C(6)	1·467 (8)	C(4a)–C(5)–C(6)	123·7 (5)
C(6)–N(7)	1·471 (6)	C(5)–C(6)–N(7)	114·3 (5)
N(7)–C(8)	1·372 (7)	C(6)–N(7)–C(8)	116·5 (4)
N(7)–N(12)	1·403 (6)	C(6)–N(7)–N(12)	117·9 (4)
		C(8)–N(7)–N(12)	121·6 (4)
C(8)–C(9)	1·453 (8)	N(7)–C(8)–C(9)	116·9 (5)
C(8)–O(1)	1·227 (7)	N(7)–C(8)–O(1)	119·5 (5)
		O(1)–C(8)–C(9)	123·6 (5)
C(9)–C(10)	1·311 (8)	C(8)–C(9)–C(10)	120·7 (5)
C(10)–C(11)	1·470 (7)	C(9)–C(10)–C(11)	123·2 (5)
C(10)–Cl	1·718 (5)	C(9)–C(10)–Cl	121·9 (4)
		Cl–C(10)–C(11)	114·8 (4)
C(11)–O(2)	1·226 (6)	C(10)–C(11)–N(12)	114·8 (4)
C(11)–N(12)	1·365 (6)	C(10)–C(11)–O(2)	124·2 (5)
		O(2)–C(11)–N(12)	121·0 (5)
N(12)–C(12a)	1·474 (5)	N(7)–N(12)–C(11)	122·7 (4)
		N(7)–N(12)–C(12a)	119·3 (4)
		C(11)–N(12)–C(12a)	116·6 (4)
		N(12)–C(12a)–C(4a)	113·3 (4)
		N(12)–C(12a)–C(1)	112·0 (4)
		C(4a)–C(12a)–C(1)	109·7 (4)

Positional and thermal parameters for C, N, O and Cl were refined. The H atoms were located in a further difference map and kept invariant during the last refinement. The weights, assigned by *PESOS* (Martínez-Ripoll & Cano, 1975), were $w = 0·42/\sigma^2(F)$ with $\sigma(F) = 0·58 + 0·17|F_o|$ if $|F_o| < 3·3$, $\sigma(F) = 1·86 - 0·25 |F_o|$ if $3·3 < |F_o| < 5·3$, $\sigma(F) = 0·46 + 0·011|F_o|$ if $5·3 < |F_o| < 12·5$ and $\sigma(F) = 0·17 + 0·04|F_o|$ if $|F_o| > 12·5$. A mixed refinement converged to $R = 0·072$ and $R_w = 0·088$ for observed reflexions only.*

Discussion. Atomic parameters and molecular dimensions are listed in Tables 1–3 and the shape of the molecule is represented in Fig. 1.

The puckering of all the rings has been calculated following Cremer & Pople (1975). Ring *C* has a chair conformation $|\varphi = 270$ (11)°, $\theta = 176·6$ (6)° and $q = 0·574$ (8) Å, origin at C(1)] with C(2) and C(4a) at the flaps. Their deviations from the C(1)–C(3)–C(4)–C(12a) plane are 0·677 (6) and −0·665 (5) Å respectively. Ring *B* is in a half-chair conformation $|\varphi = 209$ (2)°, $\theta = 130$ (1)° and $q = 0·259$ (10) Å, origin at
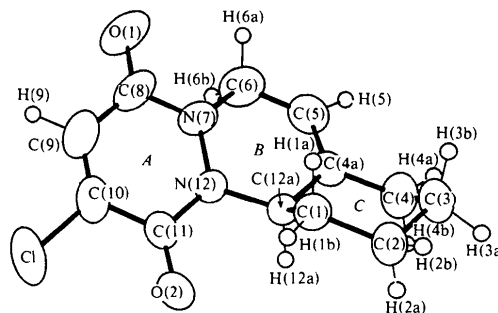
Fig. 1. Drawing of the molecule with the numbering as in Table 1. (Note that the numbering differs from that used to name the compound.)

Table 3. *Selected torsion angles* (°) *for* $C_{12}H_{13}ClN_2O_2$

Ring A		Ring B	
N(7)–C(8)–C(9)–C(10)	−4·0 (8)	N(12)–N(7)–C(6)–C(5)	−26·5 (6)
C(8)–C(9)–C(10)–C(11)	4·7 (8)	N(7)–C(6)–C(5)–C(4a)	8·0 (8)
C(9)–C(10)–C(11)–N(12)	−2·9 (7)	C(6)–C(5)–C(4a)–C(12a)	0·5 (8)
C(10)–C(11)–N(12)–N(7)	0·5 (6)	C(5)–C(4a)–C(12a)–N(12)	8·3 (6)
C(11)–N(12)–N(7)–C(8)	−0·2 (7)	C(4a)–C(12a)–N(12)–N(7)	−27·3 (5)
N(12)–N(7)–C(8)–C(9)	1·8 (7)	C(12a)–N(12)–N(7)–C(6)	37·4 (6)

Ring C	
C(12a)–C(1)–C(2)–C(3)	57·1 (6)
C(1)–C(2)–C(3)–C(4)	−54·5 (6)
C(2)–C(3)–C(4)–C(4a)	52·9 (6)
C(3)–C(4)–C(4a)–C(12a)	−56·1 (6)
C(4)–C(4a)–C(12a)–C(1)	58·9 (5)
C(4a)–C(12a)–C(1)–C(2)	−58·2 (5)

C(4a)|, the maximum torsion angle $|37\cdot4\ (6)°|$ corresponding to the N(7)—N(12) bond. Finally, ring A has an envelope conformation $|\varphi = 72\ (11)°$, $\theta = 50\ (9)°$ and $q = 0\cdot03\ (3)$ Å, origin at N(7)|, C(9) being at the flap with a deviation of $-0\cdot043\ (6)$ Å from the least-squares plane defined by the five remaining atoms. O(1), O(2) and Cl deviate $0\cdot063\ (5)$, $-0\cdot048\ (4)$ and $0\cdot031\ (2)$ Å respectively from this plane. The least-squares planes for rings A and B form an angle of $13\cdot6\ (6)°$.

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Structure of N-{2-[2-Methyl-3-(p-nitrobenzyl)oxy-1-isothioureido]ethyl}-p-toluenesulfonamide

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Abstract. $C_{18}H_{22}N_4O_5S_2$, $M_r = 438\cdot53$, triclinic, $P\bar{1}$, $a = 8\cdot127\ (2)$, $b = 7\cdot128\ (2)$, $c = 18\cdot040\ (3)$ Å, $\alpha = 86\cdot50\ (2)$, $\beta = 97\cdot13\ (2)$, $\gamma = 94\cdot30\ (2)°$; $V = 1032\cdot6$ Å^3; $Z = 2$, $d_c = 1\cdot43$ Mg m^{-3}, $F(000) = 460$; $\mu(\text{Mo } K\alpha) = 0\cdot247$ mm^{-1}. The structure was solved by direct methods and refined by full-matrix least squares to $R = 0\cdot043$ for 2973 independent reflections. The molecule has an open-chain conformation with an intramolecular hydrogen bond of $-$NH$\cdots$O type involving the amidine system and benzyloxy O atom. Steric effects of substituents at the C=N double bond, the conjugation in the amidine system and probably also the packing forces cause a curved conformation of the chain. A short van der Waals N$\cdots$N contact $|2\cdot947\ (6)$ Å| is found in the molecule.

Introduction. The total compound was synthesized (Wolski, 1980) in the course of research on new analogs of sulfamides having a hypoglycemic activity. The analogs generally show a hypoglycemic activity lower than that of the commercially available drugs. The investigations of these analogs by chemical and other methods (IR, ^{1}H-NMR, ^{13}C-NMR, MS) gave no clear indication as to their molecular structure. Among the three possible structures, those with an open chain (II or III) were more likely; it was impossible, however, to exclude completely the cyclic form (I) with the chiral C atom bonded to four heteroatoms. Analysis by X-ray diffraction was undertaken to solve the problem adequately.
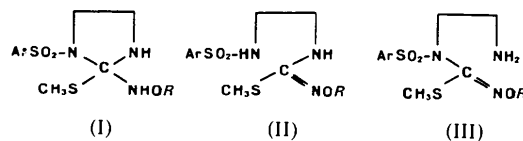
3367 independent reflections were measured on a four-circle automatic CAD-4 diffractometer (Jagiellonian University, SLAF & BS, Kraków) by the $\omega/2\theta$ technique up to $2\theta_{\text{max}} = 48°$ with graphite-monochromatized Mo $K\alpha$ radiation ($\lambda = 0\cdot71069$ Å). 2973 of these having $I > 2\sigma_I$ were considered as observed and after Lp correction were used in the refinement process.

The structure was solved by direct methods using the *SHELX* system (Sheldrick, 1976). The atomic parameters were refined with isotropic and then anisotropic thermal parameters (XRAY system,