carboxyl group and the peptide O atom protrude from the main body of the molecule and the indole NH is exposed at the outer region of the molecule; this emphasizes the amphipatic character of the molecule. The conformation of the peptide bond is *trans*, and there is no intramolecular hydrogen bond.

The crystal packing of *N*-IAA-L-Nle is determined by intermolecular hydrogen bonds (Table 4, Fig. 3). The molecular conformations and crystal packing of *N*-(IAA)-L-Nva (Kojić-Prodić *et al.*, 1991) and the title compound *N*-(IAA)-L-Nle, the amino-acid moieties of which are members of a series of straight-chain homologues (they differ by a CH_2 group), exhibit a similar pattern of hydrogen bonds. The indole NH acts as a donor to the carboxylic O212 group joining molecules along **c**. The carboxylic group is a donor to the peptide O atom forming the hydrogen bonds between molecules related by a 2_1 axis.

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Structure of Lansimide 2, a Product from *Clausena lansium*

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Abstract. The natural product lansimide 2 is a 1:1 mixture of two different cyclic amides, $C_{18}H_{17}NO_2.C_{18}H_{19}NO_3$. The mixture crystallizes as a molecular pair in the centrosymmetric space group $P2_1/n$. $M_r = 576.69$, monoclinic, $a = 20.151$ (2), $b = 6.2984$ (4), $c = 24.051$ (2) Å, $\beta = 104.339$ (8)°, $V = 2957.4$ Å^3, $Z = 4$, $D_x = 1.30$ g cm^{-3}, Cu $K\alpha$, $\lambda = 1.54178$ Å, $\mu = 6.1$ cm^{-1}, $F(000) = 1224$, $T = 163$ (1) K, $R = 0.033$, $wR = 0.034$ for 5002 observed reflections.

Introduction. A number of amides have been isolated from the roots and leaves of *Clausena lansium*. In folk medicine the leaf extract has been used for the treatment of dermatological diseases, viral hepatitis, asthma and gastro-intestinal diseases. The molecular structures of a number of these amides have been determined. These are lansimide 1 (Prakash, Raj, Kapil & Popli, 1980), the isomeric clausenamide and neoclausenamide together with cycloclausenamide (Yang, Chen & Huang, 1988), several cinnamamides (Lin, 1989), and lansimide 3, which is identical to molecule (II) in lansimide 2 (Lakshmi & Kapil, 1992). The present communication reports the structure of lansimide 2 which proves to be a 1:1 mixture of two cyclic amides. The lansimides have been

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found to have marked spasmolytic activity. It also should be noted that all the amides are found to be racemic mixtures. The single-crystal structures of clausenamide and cycloclausenamide have been reported (Yang *et al.*, 1988).

Experimental. Lansimide 2, $C_{18}H_{17}NO_2.C_{18}H_{19}NO_3$, was dissolved in a mixture of methanol and benzene (2:1 by volume). The solution was then kept at 277 K. Colorless plate-shaped crystals formed within 5 days. A crystal 0.09 × 0.16 × 0.45 mm was used for X-ray diffraction studies. The X-ray diffraction data were collected at 163 (1) K on an Enraf–Nonius CAD-4 diffractometer using Ni-filtered Cu $K\alpha$ radiation. 72 reflections ($39.7 > \theta > 20.1°$) and Cu $K\alpha_1$ wavelength (1.54051 Å) were used for lattice constants. Systematic absences were $0k0$ ($k = 2n + 1$), $h0l$ ($h + l = 2n + 1$). Diffraction data with $1.0 \le 2\theta \le 150.0°$ in $-25 \le h \le 25$, $0 \le k \le 7$ and $0 \le l \le 30$ were collected using ω–2θ scan techniques and a variable scan width calculated as $(0.80 + 0.20\tan\theta)°$. The maximum scan time for a single reflection was 60 s. The receiving aperture, located 173 mm from the data crystal, had a variable width which was calculated as $(2.40 + 0.86\tan\theta)$ mm, while the height of the aperture remained constant at 6 mm. Three orientational control monitors were checked every 200 reflections. Three intensity control monitors were measured every 7200 s of X-ray exposure time and they showed a maximum difference of 0.016 and an e.s.d. of 0.003. The profiles for all the reflections were observed and stored.

The data were processed by using the profile-analysis technique with the program *DREAM* (Blessing, 1987). Lorentz and polarization corrections were applied. No absorption correction was made. Among 6069 unique data, there were 5002 observed data [$I \ge 2\sigma(I)$]. The structure was solved by direct methods using the program *SHELXS*86 (Sheldrick, 1986). The 43 non-H atoms were refined anisotropically. The coordinates of the 36 H atoms were determined from successive difference Fourier syntheses and refined isotropically. *SHELX*76 (Sheldrick, 1976) was employed to carry out the refinement and Fourier calculations. A final R of 0.033 and wR of 0.034 were obtained by the full-matrix least-squares minimization of $\sum w(|F_o| - |kF_c|)^2$, where $w = 1/\sigma^2(F)$. The maximum shift/e.s.d. = 0.049 for non-H atoms and 0.062 for H atoms. The largest and the smallest peaks in the final difference Fourier map were +0.25 and −0.18 e Å^{-3}. The GOF = $[\sum w(F_o - F_c)^2/(N - NP)]^{1/2} = 1.8$, where N was the number of observed reflections used in the least-squares refinement and NP was the number of least-squares parameters which was 532. Scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV).

Table 1. *Atomic parameters for non-*H *atoms with e.s.d.'s in parentheses*

$U_{eq} = \frac{1}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
C(1)	0.03920 (6)	−0.2009 (2)	0.17611 (5)	0.0243 (3)
C(11)	−0.00528 (6)	−0.0220 (2)	0.14944 (5)	0.0226 (3)
C(12)	−0.02675 (7)	−0.0286 (2)	0.08909 (5)	0.0303 (4)
C(13)	−0.06390 (7)	0.1338 (3)	0.05769 (6)	0.0341 (4)
C(14)	−0.08267 (6)	0.3077 (2)	0.08573 (6)	0.0306 (4)
C(15)	−0.06447 (6)	0.3125 (2)	0.14517 (5)	0.0253 (4)
C(16)	−0.02582 (5)	0.1508 (2)	0.17806 (5)	0.0213 (3)
C(2)	0.10357 (6)	−0.1882 (2)	0.20732 (5)	0.0242 (3)
N(3)	0.13729 (5)	0.0059 (2)	0.22606 (4)	0.0227 (3)
C(3)	0.20183 (7)	0.0510 (3)	0.21057 (7)	0.0337 (4)
C(4)	0.11448 (6)	0.1442 (2)	0.25976 (5)	0.0221 (3)
O(4)	0.14336 (4)	0.3151 (1)	0.27415 (4)	0.0301 (3)
C(5)	0.05191 (6)	0.0790 (2)	0.28107 (5)	0.0211 (3)
O(5)	0.06052 (4)	0.1566 (2)	0.33790 (3)	0.0256 (3)
C(6)	−0.01384 (6)	0.1767 (2)	0.24291 (5)	0.0204 (3)
C(61)	−0.07568 (6)	0.1033 (2)	0.26359 (5)	0.0227 (3)
C(62)	−0.10069 (6)	−0.1025 (2)	0.25375 (6)	0.0285 (4)
C(63)	−0.15749 (7)	−0.1672 (3)	0.27267 (6)	0.0352 (4)
C(64)	−0.18907 (7)	−0.0261 (3)	0.30201 (7)	0.0399 (5)
C(65)	−0.16380 (8)	0.1769 (3)	0.31314 (8)	0.0445 (5)
C(66)	−0.10721 (7)	0.2423 (2)	0.29393 (6)	0.0343 (4)
C(1′)	0.16258 (6)	0.9070 (2)	0.48196 (5)	0.0223 (3)
C(11)′	0.19732 (6)	0.7497 (2)	0.52756 (5)	0.0236 (3)
C(12′)	0.17068 (7)	0.5495 (2)	0.53470 (5)	0.0275 (4)
C(13′)	0.20526 (7)	0.4133 (2)	0.57776 (6)	0.0328 (4)
C(14′)	0.26761 (7)	0.4727 (3)	0.61373 (6)	0.0354 (4)
C(15′)	0.29456 (7)	0.6704 (3)	0.60711 (6)	0.0363 (4)
C(16′)	0.25948 (6)	0.8081 (2)	0.56487 (5)	0.0299 (4)
C(2′)	0.08417 (6)	0.9093 (2)	0.47101 (5)	0.0233 (3)
O(2′)	0.05636 (5)	1.1110 (1)	0.45175 (4)	0.0284 (3)
N(3′)	0.05051 (5)	0.7460 (2)	0.43044 (4)	0.0241 (3)
C(3′)	−0.02322 (7)	0.7155 (3)	0.42519 (6)	0.0358 (5)
C(4′)	0.07669 (6)	0.6646 (2)	0.38870 (5)	0.0224 (3)
O(4′)	0.04008 (4)	0.5681 (2)	0.34770 (4)	0.0282 (3)
C(5′)	0.15384 (6)	0.6809 (2)	0.39453 (5)	0.0225 (3)
O(5′)	0.16809 (5)	0.6747 (2)	0.34010 (4)	0.0299 (3)
C(6′)	0.18283 (6)	0.8865 (2)	0.42386 (5)	0.0210 (3)
C(61′)	0.25883 (6)	0.9131 (2)	0.42840 (5)	0.0220 (3)
C(62′)	0.30619 (6)	0.7497 (2)	0.44460 (6)	0.0277 (4)
C(63′)	0.37499 (7)	0.7822 (2)	0.44607 (6)	0.0312 (4)
C(64′)	0.39751 (7)	0.9783 (2)	0.43221 (6)	0.0317 (4)
C(65′)	0.35105 (7)	1.1430 (2)	0.41673 (6)	0.0317 (4)
C(66′)	0.28209 (7)	1.1098 (2)	0.41456 (5)	0.0272 (4)

Discussion. The final positional parameters and equivalent isotropic temperature factors for non-H atoms are listed in Table 1.* A stereoview (Johnson, 1965) and the numbering scheme of a molecular pair are shown in Fig. 1. Bond distances and bond angles are presented in Table 2. Selected torsional angles can be found in Table 3.

The structure proves to be a 1:1 mixture of two different compounds (Figs. 1, 2), both of which are cyclic lactams. One is an unsaturated eight-membered cyclic lactam fused to an aromatic ring and substituted by a phenyl group and a hydroxyl group (I) with relative configuration 5*S*,6*R*. It is an isomer of cycloclausenamide (Yang *et al.*, 1988). The

* Tables of anisotropic thermal parameters, H-atom parameters, and calculated and observed structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54834 (35 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Bond distances* (Å) *and bond angles* (°) *with e.s.d.'s in parentheses*

	$C_{18}H_{17}NO_2$	$C_{18}H_{19}NO_3$
C(1)—C(11)	1.483 (2)	1.515 (2)
C(1)—C(2)	1.331 (2)	1.536 (2)
C(1)—C(6)	—	1.555 (1)
C(11)—C(12)	1.409 (1)	1.397 (2)
C(11)—C(16)	1.404 (2)	1.397 (2)
C(12)—C(13)	1.378 (2)	1.392 (2)
C(13)—C(14)	1.387 (2)	1.389 (2)
C(14)—C(15)	1.385 (2)	1.383 (2)
C(15)—C(16)	1.402 (2)	1.389 (2)
C(16)—C(6)	1.527 (1)	—
C(2)—O(2)	—	1.419 (2)
C(2)—N(3)	1.417 (2)	1.464 (2)
N(3)—C(3)	1.467 (2)	1.472 (2)
N(3)—C(4)	1.346 (1)	1.346 (1)
C(4)—O(4)	1.232 (2)	1.235 (1)
C(4)—C(5)	1.530 (2)	1.529 (2)
C(5)—O(5)	1.421 (1)	1.409 (1)
C(5)—C(6)	1.541 (2)	1.520 (2)
C(6)—C(61)	1.524 (2)	1.518 (2)
C(61)—C(62)	1.390 (2)	1.392 (2)
C(61)—C(66)	1.390 (2)	1.393 (2)
C(62)—C(63)	1.393 (2)	1.393 (2)
C(63)—C(64)	1.384 (2)	1.385 (2)
C(64)—C(65)	1.378 (3)	1.385 (2)
C(65)—C(66)	1.394 (2)	1.393 (2)
C(1)—C(11)—C(12)	114.9 (1)	123.8 (1)
C(1)—C(11)—C(16)	126.6 (1)	118.2 (1)
C(1)—C(2)—O(2)	—	111.8 (1)
C(1)—C(2)—N(3)	123.7 (1)	113.2 (1)
C(1)—C(6)—C(5)	—	109.4 (1)
C(1)—C(6)—C(61)	—	114.3 (1)
C(11)—C(1)—C(2)	126.8 (1)	113.7 (1)
C(11)—C(1)—C(6)	—	114.8 (1)
C(11)—C(12)—C(13)	122.0 (1)	120.7 (1)
C(11)—C(16)—C(15)	118.5 (1)	121.2 (1)
C(11)—C(16)—C(6)	126.3 (1)	—
C(12)—C(11)—C(16)	118.4 (1)	118.0 (1)
C(12)—C(13)—C(14)	119.8 (1)	120.4 (1)
C(13)—C(14)—C(15)	119.0 (1)	119.4 (1)
C(14)—C(15)—C(16)	122.3 (1)	120.2 (1)
C(15)—C(16)—C(6)	115.1 (1)	—
C(2)—C(1)—C(6)	—	109.5 (1)
C(16)—C(6)—C(5)	117.4 (1)	—
C(16)—C(6)—C(61)	111.5 (1)	—
C(2)—N(3)—C(3)	118.2 (1)	116.1 (1)
C(2)—N(3)—C(4)	122.8 (1)	124.2 (1)
O(2)—C(2)—N(3)	—	109.3 (1)
N(3)—C(4)—O(4)	121.8 (1)	121.1 (1)
N(3)—C(4)—C(5)	117.4 (1)	118.9 (1)
C(3)—N(3)—C(4)	119.0 (1)	117.4 (1)
C(4)—C(5)—O(5)	108.4 (1)	110.5 (1)
C(4)—C(5)—C(6)	110.6 (1)	111.4 (1)
O(4)—C(4)—C(5)	120.8 (1)	119.9 (1)
C(5)—C(6)—C(61)	109.7 (1)	113.1 (1)
O(5)—C(5)—C(6)	108.5 (1)	108.2 (1)
C(6)—C(61)—C(62)	121.3 (1)	123.2 (1)
C(6)—C(61)—C(66)	119.8 (1)	118.3 (1)
C(61)—C(62)—C(63)	120.6 (1)	120.5 (1)
C(61)—C(66)—C(65)	120.4 (1)	121.1 (1)
C(62)—C(61)—C(66)	118.9 (1)	118.4 (1)
C(62)—C(63)—C(64)	119.8 (1)	120.5 (1)
C(63)—C(64)—C(65)	120.0 (1)	119.5 (1)
C(64)—C(65)—C(66)	120.2 (1)	119.9 (1)

Table 3. *Selected torsional angles* (°) *with e.s.d.'s in parentheses*

		$C_{18}H_{17}NO_2$* (I)	C_8H_8	$C_{18}H_{19}NO_3$ (II)
	C(1)—C(11)—C(16)—C(6)	−8.0 (2)	1.1 (3)	—
	C(1)—C(2)—N(3)—C(4)	59.7 (1)	55.4 (3)	−27.4 (1)
	C(1)—C(6)—C(5)—C(4)	—	—	53.7 (1)
	C(11)—C(1)—C(2)—N(3)	8.3 (2)	1.1 (3)	−84.5 (1)
	C(16)—C(11)—C(1)—C(2)	−60.6 (2)	−57.9 (3)	−142.2 (1)
	C(16)—C(6)—C(5)—C(4)	49.4 (1)	0.3 (5)	—
	C(2)—C(1)—C(6)—C(5)	—	—	−60.1 (1)
ω_4	C(2)—N(3)—C(4)—C(5)	3.8 (1)	0.3 (5)	21.6 (2)
	N(3)—C(2)—C(1)—C(6)	—	—	45.4 (1)
	N(3)—C(4)—C(5)—C(6)	−97.6 (1)	−56.6 (3)	−34.7 (1)
	C(11)—C(16)—C(6)—C(5)	32.2 (2)	55.4 (3)	—
ω_1	C(3)—N(3)—C(4)—C(5)	−172.6 (1)	—	−176.2 (1)
ω_2	O(4)—C(4)—N(3)—C(2)	−177.7 (1)	—	−162.2 (1)
ω_3	O(4)—C(4)—N(3)—C(3)	5.8 (2)	—	0.0 (1)
χ_C	$= \omega_1 - \omega_3 + \pi$	1.6 (2)	—	3.8 (2)
χ_N	$= \omega_2 - \omega_3 + \pi$	−3.5 (2)	—	17.8 (2)
τ_1	$= \omega_1 + \omega_2$	9.7 (2)	—	21.6 (2)

*$C_{18}H_{17}NO_2$ and $C_{18}H_{19}NO_3$, present work; C_8H_8, 1,3,5,7-cyclooctatetraene (Claus & Krüger, 1988).

other contains a six-membered lactam ring, substituted by two phenyl rings and two hydroxyl groups (II) with relative configuration 1*R*,2*R*,5*S*,6*S*. It is an isomer of clausenamide (Yang *et al.*, 1988) and identical to lansimide 3 (Lakshmi & Kapil, 1992). The formal structures are shown in Fig. 1. The space group is centrosymmetric and the structure, therefore, contains racemic mixtures of both molecules. The 1:1 mixture involves a total of six potential chiral centers for the two molecules. The racemic nature of all amides reported from *Clausena lansium*

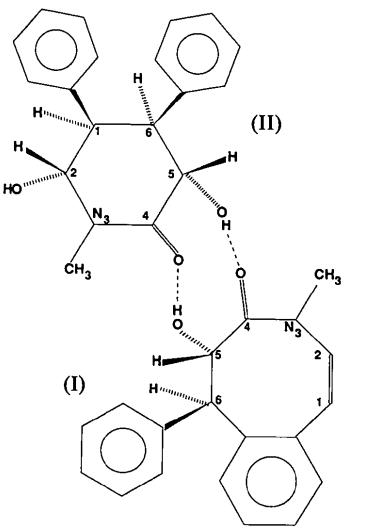

Fig. 1. A formal representation of both molecules (I) and (II) forming the 1:1 mixture in lansimide 2. Also shown is the relative stereochemistry.

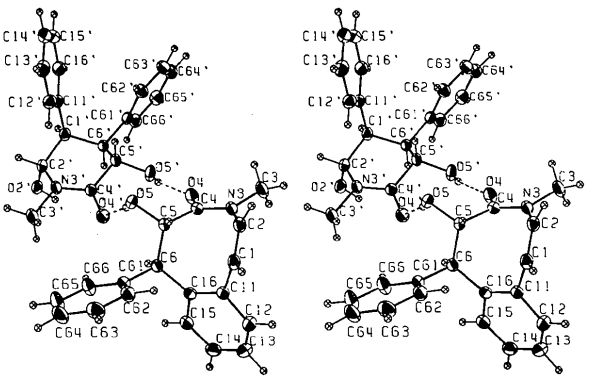

Fig. 2. A stereoview of the molecular pair in lansimide 2 (Johnson, 1965). The intermolecular hydrogen bonds O(4)···H(O5′)—O(5′) and O(4′)···H(O5)—O(5) are shown.

and the occurrence of isomers may indicate that the products are artifacts caused by the isolation of the compounds and that the true precursors still have to be found.

The two molecules form a molecular pair through two intermolecular hydrogen bonds (Fig. 2), O(4)···H(O5′)—O(5′) (2.739 Å, 159°) and O(4′)···H(O5)—O(5) (2.644 Å, 164°). The pair remains intact during purification. The molecular pairs form chains along the crystallographic *b* axis *via* a third intermolecular hydrogen bond, O(5)···H(O2′)—O(2′)(x, $y-1$, z) (2.775 Å, 174°).

No unusual bond distances and bond angles were observed (Table 2).

The eight-membered ring in $C_{18}H_{17}NO_2$ (I) assumes a twisted tub-shaped boat conformation, which can be compared to the ideal D_{2d} conformation in 1,3,5,7-cyclooctatetraene (Claus & Krüger, 1988). The D_{2d} symmetry in (I) is disturbed by the sp^3 hybridization of the C(5) and C(6) atoms (Table 3). The amide ring in $C_{18}H_{19}NO_3$ (II) is six-membered and can be described equally well as a (3′)-sofa or a (3′–4′)-half-chair. The asymmetry parameters are: $\Delta C_s(3') = 7.94°$ and $\Delta C_2(3'–4') = 7.82°$. For an ideal sofa conformation, $\Delta C_s = 0.0°$, and for an ideal half-chair, $\Delta C_2 = 0.0°$ (Duax & Norton, 1975).

The planarity of amide groups can be described with three parameters (Winkler & Dunitz, 1971): χ_C, describing the planarity of the three bonds around the carbonyl C atom, χ_N the planarity of the three bonds around the amide N atom and τ the rotation around the C—N bond. All three parameters are 0° for the planar amide group. For a non-planar amide group χ_C is often still close to 0°. It can be seen that the amide groups in both (I) and (II) are distinctly non-planar, but much more so in compound (II) which has a six-membered amide ring.

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Structure of 4′-(9-Acridinylamino)-2′-methoxymethanesulfonanilide (*o*-AMSA) Methanol Solvate, an Inactive Isomer of the Anti-Cancer Drug Amsacrine (*m*-AMSA)

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Abstract. $C_{21}H_{19}N_3O_3S.CH_4O$, $M_r = 425.51$, triclinic, $P\bar{1}$, $a = 9.545$ (2), $b = 14.338$ (1), $c = 8.3748$ (7) Å, $\alpha = 106.032$ (6), $\beta = 103.230$ (9), $\gamma = 70.96$ (1)°, $V = 1029.0$ (3) Å^3, $Z = 2$, $D_x = 1.373$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0.71069$ Å, $\mu = 1.94$ cm^{-1}, $F(000) = 448$, $T = 293$ (1) K, $R = 0.039$ for 2169 observed reflections ($I > 2.5\sigma$). The crystal packing is enhanced by intermolecular hydrogen bonds, by hydrogen bonds with the methanol solvate molecule, and by stacking interactions between the acridine rings. There are no obvious structural features which explain the lack of anti-tumour activity.

Introduction. The anti-cancer drug amsacrine (1) (*m*-AMSA) and a wide range of related compounds have been extensively studied in an attempt to understand the complex relationships between cytotoxicity, anti-tumour activity and DNA intercalating ability

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