polarization effects. The Laue group assignment, systematic absences and non-centrosymmetric intensity statistics indicated space group $I2$ or Im (No. 5 or 8). Initial preference was given to $I2$, since it occurs more commonly and since the homochirality of the sample (incompatible with Im) had not yet been independently established. Refinement proceeded well and $I2$ was adopted. Difference Fourier methods were used to locate initial H-atom positions, and the H atoms were then refined isotropically. Refined C—H distances ranged from 0.99 (2) to 1.02 (4) Å, with a mean value of 1.00 (3) Å. Subsequently, the methyl group H atoms were made canonical, with C—H = 0.98 Å and $U_{\rm iso} = 1.2U_{\rm eq}$ of the attached C atom. The refined O—H distance is given in Table 2. The extinction coefficient was predicted to be negative in the late stages of refinement, and was thus not included in the model. The maximum positive residual peak is located approximately midway between C1 and C2; the maximum negative peak is located ~1.3 and 1.4 Å from O2 and C1, respectively.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1995). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *TEXSAN*. Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *TEXSAN*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: FR1158). Services for accessing these data are described at the back of the journal.

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3-(3-Indolyl)acrylamide

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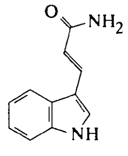
Abstract

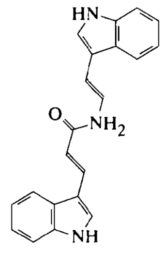
The title compound ($C_{11}H_{10}N_2O$) was isolated, together with other indole derivatives, from the acetone extract of the red algae *Chondria atropurpurea*. The acrylamide group deviates only slightly from the overall planarity of the entire molecule. The packing is stabilized by an intermolecular interaction between an amide H atom and the indole ring.

Comment

3-(3-Indolyl)acrylamide, (I), was isolated, together with other indole derivatives, from the acetone extract of the red algae *Chondria atropurpurea* Harvey (Rhodomelaceae). Chondriamide A, (II), is also found. The coexistence of both compounds in the abovementioned extract favours the hypothesis of the biosynthetic mechanism proposed by Palermo *et al.* (1992) for (II). Further structural research on (II) and other derivatives will be carried out, based on the results presented in this report.



(I)



(II)

As can be expected from the chemical structural diagram, the title compound is a molecule with a marked planarity. The least-squares equation of the molecular plane is $-9.268(5) \times x + 11.182(7) \times y + 2.996(3) \times z = 3.500(3)$, and the maximum deviation is 0.143 (2) Å, for O14. Slight deviation from this planarity can be described through the relative orientations of the two main planar moieties, namely, the acrylamide and the indole groups. The dihedral angle between the least-squares planes corresponding to these groups is 8.16 (13)°. The C2—C3—C10—C11 torsion angle has a value of $-167.9(3)°$ [*i.e.* 12.1 (3)°], which is much smaller than the equivalent angle in 3-(1-methyl-1,2,3,5-tetrahydropyrid-4-yl)indole, (III) (Bates *et al.*, 1988), that also contains the 3-substituted indole group with a conjugated double bond at the C10—C11 position. In this compound, the equivalent torsion angle is $-159.2(2)°$ [*i.e.* 20.8 (2)°]. This could be explained by steric effects. The C—C bond distances in the aliphatic chain are 1.442 (3), 1.329 (3) and 1.472 (3) Å, for C3—C10, C10—C11 and C11—C12, respectively. These distances demonstrate the presence of alternating single and double bonds. This behaviour is also observed in (III), where the corresponding bond distances have values of 1.467 (3), 1.333 (3) and 1.489 (4) Å, respectively. The C10—C11 double bond in both cases is in the *trans* configuration. Selected bond distances and angles are given in Table 1; Fig. 1 shows the molecule and the atom-labelling scheme.

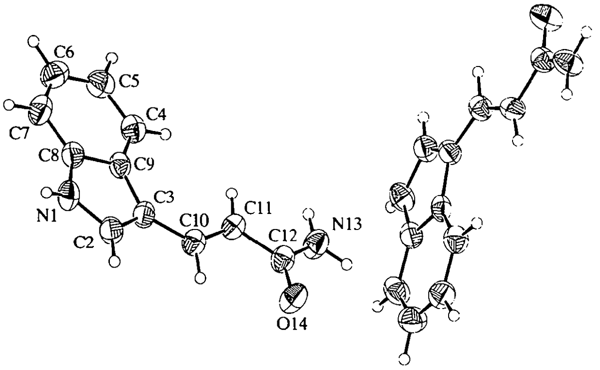


Fig. 1. A *ZORTEP* (Zsolnai & Pritzkow, 1995) drawing of (I), with the atom-labelling scheme. All displacement ellipsoids are drawn at 50% probability, except for H atoms, which are shown as circles of an arbitrary radius. The short intermolecular contact between the N13 atom and the indole ring of a related molecule at ($x - \frac{1}{2}$, $-y + \frac{1}{2}$, $1 - z$), as described in the text, is apparent.

There is a short intermolecular contact between the amide N13 atom and the six-membered ring of the indole moiety of an equivalent molecule at ($x - \frac{1}{2}$, $-y + \frac{1}{2}$, $1 - z$). This short contact also involves H13*B*. The distances between atoms N13 and H13*B* and the ring centroid are 3.391 and 2.693 Å, respectively, while the angles between the normal to the plane and the line connecting the ring centroid and each atom are 8.89° for N13 and 19.99° for H13*B*. The angle between the N13—H13*B* bond and the ring plane is 134.21°. Hydrogen bonds (Table 2) connect equivalent molecules in an infinite two-dimensional network normal to the [001] direction. The parallel arrays are connected along the *a* direction by the short contact previously described.

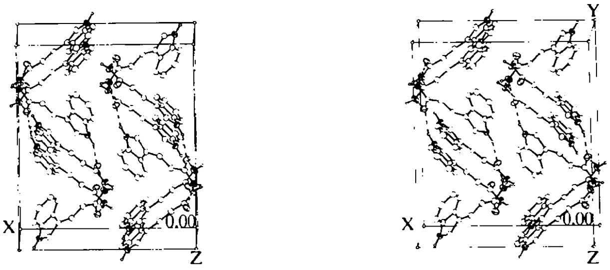


Fig. 2. A *ZORTEP* (Zsolnai & Pritzkow, 1995) stereo-packing diagram of (I). Intermolecular hydrogen bonds are marked as dashed lines. Most H atoms, except those involved in hydrogen bonds or contacts, have been excluded for clarity.

Experimental

Compound (I) was isolated from the acetone extract of the red algae *Chondria atropurpurea* Harvey (Rhodomelaceae). The extract was fractioned by column chromatography on silica gel using a gradient of *n*-hexane, EtOAc and MeOH. The fraction containing (I) was then purified by MPLC (medium-pressure liquid chromatography) over silica gel with EtOH, and was recrystallized by slow evaporation from acetone to yield yellow plates suitable for diffraction.

Crystal data

$C_{11}H_{10}N_2O$
$M_r = 186.21$
Orthorhombic
Pbca
$a = 14.283(2)$ Å
$b = 17.083(4)$ Å
$c = 7.723(3)$ Å
$V = 1884.5(8)$ Å^3
$Z = 8$
$D_x = 1.313$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 20.32$–$24.98°$
$\mu = 0.087$ mm^{-1}
$T = 293(2)$ K
Plate
0.7 × 0.4 × 0.2 mm
Yellow

Data collection

Rigaku AFC-7*S* diffractometer
$\theta/2\theta$ scans
Absorption correction: ψ-scan (*MSC/AFC Diffractometer Control Software*; Molecular Structure Corporation, 1993)
$T_{min} = 0.942$, $T_{max} = 0.983$
2542 measured reflections

2159 independent reflections
893 reflections with $I > 2\sigma(I)$
$R_{int} = 0.021$
$\theta_{max} = 27.5°$
$h = -10 \rightarrow 18$
$k = -12 \rightarrow 22$
$l = -5 \rightarrow 10$
3 standard reflections every 150 reflections intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.041$
$wR(F^2) = 0.132$
$S = 0.980$
2159 reflections
167 parameters
All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0495P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.158$ e Å^{-3}
$\Delta\rho_{min} = -0.192$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

N1—C2	1.357 (3)	C6—C7	1.372 (4)
N1—C8	1.381 (3)	C7—C8	1.391 (3)
C2—C3	1.375 (3)	C8—C9	1.408 (3)
C3—C10	1.442 (3)	C10—C11	1.329 (3)
C3—C9	1.447 (3)	C11—C12	1.472 (3)
C4—C5	1.382 (4)	C12—O14	1.238 (3)
C4—C9	1.397 (3)	C12—N13	1.336 (3)
C5—C6	1.393 (4)		
C2—N1—C8	108.9 (2)	N1—C8—C9	107.9 (2)
N1—C2—C3	110.6 (2)	C7—C8—C9	123.0 (2)
C2—C3—C10	122.6 (2)	C4—C9—C8	117.9 (2)
C2—C3—C9	106.0 (2)	C4—C9—C3	135.5 (2)
C10—C3—C9	131.2 (2)	C8—C9—C3	106.6 (2)
C5—C4—C9	119.4 (2)	C11—C10—C3	128.9 (3)
C4—C5—C6	121.3 (3)	C10—C11—C12	121.2 (2)
C7—C6—C5	121.1 (3)	O14—C12—N13	121.5 (2)
C6—C7—C8	117.4 (3)	O14—C12—C11	122.8 (2)
N1—C8—C7	129.1 (2)	N13—C12—C11	115.7 (2)

Table 2. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O14^{i}	0.90 (3)	2.09 (3)	2.945 (3)	157 (2)
N13—H13*A*···O14^{ii}	0.94 (3)	1.95 (3)	2.892 (3)	173 (2)

Symmetry codes: (i) $\frac{1}{2} - x, 1 - y, \frac{1}{2} + z$; (ii) $x, \frac{1}{2} - y, \frac{1}{2} + z$.

The data were collected using a 1.0 mm collimator. The structure was solved by direct methods, locating all non-H atoms. All H atoms were located on difference Fourier maps and freely refined.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1993). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *MSC/AFC Diffractometer Control Software*. Program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997*a*). Program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997*b*). Molecular graphics: *ZORTEP* (Zsolnai & Pritzkow, 1995). Software used to prepare material for publication: *PLATON* (Spek, 1990).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: SX1071). Services for accessing these data are described at the back of the journal.

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Taraxerone

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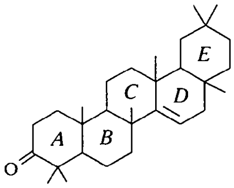
Abstract

The crystal structure of taraxerone (*D*-friedoolean-14-en-3-one), $C_{30}H_{48}O$, a pentacyclo-triterpenoid isolated from *Skimmia laureola*, is composed of independent molecules which are separated by normal van der Waals distances. The molecular dimensions are normal, with mean bond distances as follows: Csp^3—Csp^3 1.539 (17), Csp^3—Csp^2 1.519 (19), C═C 1.338 (3) and C═O 1.199 (4) Å. The structure is composed of five six-membered rings, with two rings in chair, one in twisted boat and two in slightly twisted boat conformations.

Comment

Skimmia laureola, a plant indigenous to the Western Himalayas, is abundant in the Bagh district, Azad Kashmir, Pakistan. It contains a large number of terpenoids, including the title compound, (I). In this article we report the molecular structure of (I), which has been determined unequivocally by the X-ray crystallographic method; (I) has also been reported as one of the constituents of *Myrica rubra* (Sakurai *et al.*, 1987).



(I)

The crystal structure of the title compound contains independent molecules (Fig. 1), which are separated by normal van der Waals distances. The molecular dimensions in (I) are normal, and lie within expected ranges for the corresponding bond distances and angles, with mean bond distances as follows: Csp^3—Csp^3 1.539 (17), Csp^3—Csp^2 1.519 (19), C═C 1.338 (3) and C═O 1.199 (4) Å.

The structure is composed of five six-membered rings. Rings *A* and *B* have chair conformation, with puckering