

TRADITIONAL MEDICINAL PLANTS OF THAILAND, V.
ANCISTROTECTORINE, A NEW NAPHTHALENE-
ISOQUINOLINE ALKALOID FROM
*ANCISTROCLADUS TECTORIUS*¹

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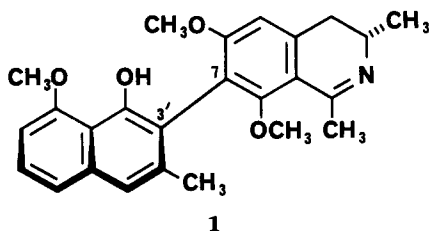
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ABSTRACT.—A new member of the naphthalene-isoquinoline series of alkaloids, ancistrotectorine (**2**), has been isolated from the leaves of the Thai medicinal plant *Ancistrocladus tectorius* (Ancistrocladaceae). The structure was deduced through single crystal X-ray crystallography and complete ¹H-nmr spectroscopic assignments were made. Ancistrotectorine (**2**) is the second member of the 7,3'-linked naphthalene-isoquinoline alkaloid group to be isolated.

Ancistrocladus is the only genus of the plant family Ancistrocladaceae and is composed of nearly 20 species distributed in tropical Asia, Malayasia, and West Africa (2). The genus is a source, together with two genera in the Dionchophyllaceae, of the naphthalene-isoquinoline group of alkaloids (3, 4). Eighteen alkaloids in this series have been isolated to date, and the compounds may be grouped according to the location of the σ -bond joining the two units. Most of the alkaloids (ten) are linked 5-1' (5-12), whilst seven others are linked 7-1' (9, 13-15). The remaining alkaloid, ancistrocladidine (**1**) from *Ancistrocladus beyneanus* Wall. (16), is linked 7-3', and the crystallographic analysis of this alkaloid was recently presented (17). We report here the structure determination of a second alkaloid having a 7-3' linkage, ancistrotectorine (**2**) from the leaves of *Ancistrocladus tectorius* (Lour.) Merr.



Previous work on the stem and twigs of *A. tectorius* has yielded ancistrocladine (10), ancistrocladine (12), hamatine (12), and ancistrocline (12), subsequent to a demonstration that the species contained alkaloids (18). The roots of *A. tectorius* have been used to treat dysentery and malaria.

After partition of the EtOH extract, the CHCl₃ fraction was chromatographed on alumina to afford ancistrotectorine (0.016% yield). From its uv spectrum, which displayed λ_{\max} 234, 286, 306, 323, and 337 nm, ancistrotectorine was a member of the

¹For the previous paper in this series see Mukhopadhyay *et al.* (1).

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naphthalene-isoquinoline series of alkaloids containing a phenolic hydroxy group (ν_{\max} 3375 cm^{-1}), even though no positive FeCl_3 test or shift in base (2N NaOH solution) in the uv spectrum was observed. No molecular ion was observed in the electron impact (ei) mass spectrum, but a clear ($M^+ + H^+$) was found at m/z 422 in the chemical ionization mass spectrum using methane as the reagent gas. Important fragments in the ei mass spectrum were observed at m/z 406 and 203.

The low-field ^1H -nmr spectrum of ancistroretorine firmly established the naphthalene-isoquinoline nature of the compound and indicated the presence of two doublet methyl groups, an aromatic methyl group, an *N*-methyl group, and three aromatic methoxy groups. The aromatic region was complex, although two singlets were apparent.

In order to evaluate the structure more explicitly, a detailed examination of the high-field ^1H -nmr spectrum of ancistroretorine was undertaken. Two doublet methyl groups were observed at δ 1.268 and δ 1.466 from the 3- CH_3 and the 1- CH_3 , respectively, and their methine protons were observed at δ 2.544 and δ 3.731. Irradiation at δ 1.27 collapsed the signal at δ 2.544 to a doublet of doublets ($J=3.0, 10.3$ Hz), clearly establishing the 3- CH_3 to be equatorial. The aromatic region was now clearly resolved with singlets observed at δ 6.518 and 7.232, doublets at δ 6.689 and 7.343, and a triplet at δ 7.266. The latter three signals were assigned to adjacent protons on the naphthalene nucleus, and on biogenetic grounds a methyl group (δ 2.169) was placed at C-2' and oxygenation at C-4' and C-5'. Irradiation of the methoxyl group singlet at δ 3.691 caused an 8.1% nOe effect in the singlet at δ 6.518, and the reverse irradiation produced an nOe (4%) only in the δ 3.691 singlet. A similar spatial relationship was established between the aromatic methoxy group at δ 3.985 and the doublet at δ 6.689, which could therefore be assigned to a 5'- OCH_3 and its adjacent 6'-H. There were a number of alternatives to explain the former nOe experiments: (a) a 7-1' linkage, with the OH at C-4' and the third OCH_3 at C-8, (b) a 7-3' linkage with the OH/ OCH_3 at C-8/C-4' or reverse, and (c) a 5-3' linkage with OH at C-8 and OCH_3 at C-4'. Potentially, these structures could be distinguished by the base-induced shift of a proton *ortho* or *para* to the hydroxy group and whether or not the proton shifted is *ortho* to a methoxy group. However, the failure of ancistroretorine to undergo a bathochromic shift in the uv spectrum on the addition of 2N NaOH did not augur well for the success of this experiment, and consequently, the single crystal X-ray crystallographic analysis was carried out.

Ancistroretorine crystallized in the monoclinic $P2_1$ space group with unit cell dimensions of $a=11.858(14)$, $b=7.043(7)$, $c=14.693(28)$ Å and $\beta=114.43(12)$. The crystal structure was elucidated, using the program MULTAN (19), and refined, using the program SHELX (20). The hydrogen bond lengths were restricted to 1.0 Å during refinement, and the largest peak on a final electron density difference map was 0.12 $\text{e}\cdot\text{\AA}^{-3}$. The final $R=8.0\%$ over 1087 independent reflexions using a unit weighting scheme.

Final positional parameters, bond lengths, and valency angles are listed in Tables 1-3, respectively. The crystallographic structure deduced for ancistroretorine (Figure 1)

TABLE 1. Fractional Atomic Co-ordinates ($\times 10^4$) with e. s. d.'s (in parenthesis)

Atom	x	y	z
O(1)	1240(8)	9594(20)	0433(6)
O(2)	4670(7)	6549(22)	2800(6)
O(3)	3753(9)	11387(23)	2826(7)
O(4)	3645(10)	13613(23)	4207(6)

TABLE I. *Continued*

Atom	x	y	z
C(1)	1943(12)	8794(27)	-1116(9)
N(2)	2331(11)	7931(24)	-1864(8)
C(3)	2868(13)	6042(27)	-1609(10)
C(4)	3963(11)	6199(29)	-0718(10)
C(4A)	3682(12)	7021(28)	0111(9)
C(5)	4374(11)	6427(28)	1072(10)
C(6)	4056(11)	7043(25)	1837(9)
C(7)	3025(12)	8244(29)	1655(9)
C(8)	2347(12)	8766(27)	0679(9)
C(8A)	2689(11)	8158(25)	-0056(9)
C(1')	1312(11)	7786(30)	3363(11)
C(2')	1769(11)	7380(27)	2658(10)
C(3')	2589(10)	8651(25)	2481(9)
C(4')	2962(11)	10278(23)	3052(9)
C(4A')	2490(13)	10752(27)	3776(9)
C(5')	2789(12)	12383(27)	4364(10)
C(6')	2339(18)	12833(30)	5069(13)
C(7')	1546(16)	11543(32)	5221(11)
C(8')	1207(11)	9955(35)	4686(10)
C(8A')	1656(13)	9490(26)	3945(9)
C(9)	2074(14)	10965(24)	-1185(11)
C(10)	1288(16)	7917(32)	-2780(12)
C(11)	3135(14)	5103(3)	-2472(10)
C(12)	5591(12)	5143(29)	2970(9)
C(13)	1163(16)	11377(31)	0861(11)
C(14)	1417(14)	5579(28)	2077(12)
C(15)	4087(14)	15272(29)	4814(11)
HO(3)	3799(102)	12787(37)	2937(80)
H(1)	1102(41)	8521(175)	-1144(71)
H(3)	2306(81)	5109(127)	-1480(79)
H(4A)	4436(81)	5016(86)	-0463(68)
H(4B)	4559(77)	7121(122)	-0795(77)
H(5)	5014(69)	5531(121)	1053(74)
H(1')	0662(71)	6984(134)	3440(73)
H(6')	2342(100)	14212(56)	5234(79)
H(7')	1116(75)	11783(176)	5685(55)
H(8')	0583(72)	9069(124)	4760(74)
H(9A)	1546(77)	11586(160)	-0873(64)
H(9B)	2922(39)	10879(185)	-0680(52)
H(9C)	2105(94)	11794(152)	-1731(60)
H(10A)	0896(106)	9195(78)	-2902(88)
H(10B)	0712(97)	6960(116)	-2689(90)
H(10C)	1516(101)	7534(144)	-3343(62)
H(11A)	3642(84)	3971(104)	-2491(95)
H(11B)	2300(38)	4926(158)	-2963(65)
H(11C)	3510(89)	6260(106)	-2646(91)
H(12A)	5926(88)	4500(150)	3619(40)
H(12B)	5199(96)	4183(126)	2448(56)
H(12C)	6272(70)	5770(154)	2829(70)
H(13A)	0396(52)	12051(147)	0480(69)
H(13B)	1102(88)	10847(161)	1476(52)
H(13C)	1854(67)	12306(135)	1050(79)
H(14A)	2230(49)	5140(165)	2110(79)
H(14B)	0997(82)	4533(126)	2288(81)
H(14C)	0919(74)	5983(177)	1399(43)
H(15A)	4807(59)	15680(158)	4660(75)
H(15B)	3619(94)	16424(107)	4869(83)
H(15C)	4383(87)	14600(147)	5464(49)

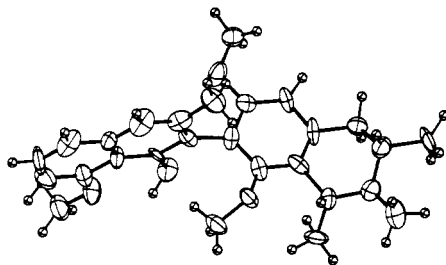
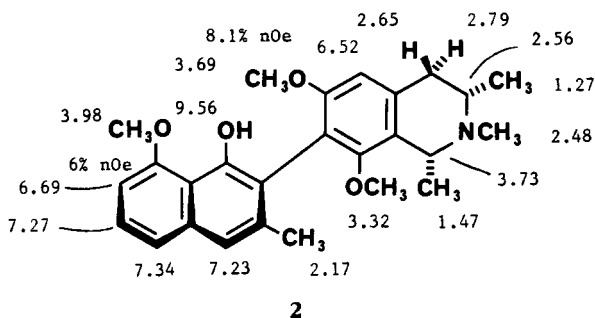


FIGURE 1. ORTEP Display of the Crystallographic Structure of Ancistrotorine (**2**).

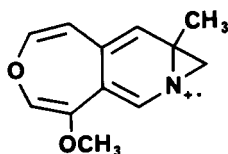
indicates that it is indeed a 7-3' linked naphthalene-isoquinoline alkaloid having the molecular array shown in **2** in which the C-1 and C-3 methyl groups are *cis* and the N-containing ring of the isoquinoline moiety adopts a sofa conformation with C-3 the out-of-plane atom. Steric hindrance about the diaryl linkage results in a dihedral angle of 56.4° between the two aromatic rings.

It now became possible to assign the ^1H spectrum completely. From the nOe experiments the singlets at δ 3.691 and δ 3.985 could be assigned to the 6- and 5'- OCH_3 , respectively, leaving the singlet at δ 3.323 to be a somewhat shielded methoxy group at C-8. Similarly, the aromatic singlet at δ 6.518 could be assigned to H-5 and at δ 7.232 to H-1'. Examination of the coupling constants permitted the doublets of doublets at δ 2.655 and δ 2.787 to be assigned to H-4 β and H-4 α respectively. The complete assignments are shown on structure **2**. Ancistrotorine (**2**) is therefore a member of the rare 7-3'-linked naphthalene-isoquinoline alkaloids of which ancistrocladidine (**1**) (16, 17), is the only other example.



2

The mass spectrum of ancistrotorine also merits some additional comment. A facile loss of 15 amu was apparent to give the stable species at m/z 406. The most interesting ion though is m/z 203 since this does not correspond to either half of the molecule as might be expected, although it clearly is derived from the isoquinoline unit. In our estimation this ion may have the structure **3** in which one of the aliphatic methyl groups has been lost together with one of the methyls from an aromatic methoxy group.



3 m/z 203

TABLE 2. Bond Lengths (Å) with eds's (in parentheses)

O(1)—C(8)	1.359(18)	C(5)—C(6)	1.380(22)
O(1)—C(13)	1.422(25)	C(6)—C(7)	1.429(22)
O(2)—C(6)	1.379(14)	C(7)—C(8)	1.413(17)
O(2)—C(12)	1.427(21)	C(7)—C(3')	1.510(21)
O(3)—C(4')	1.352(18)	C(8)—C(8A)	1.354(22)
O(4)—C(5')	1.416(22)	C(1')—C(2')	1.363(24)
O(4)—C(15)	1.447(23)	C(1')—C(8A')	1.443(25)
C(1)—N(2)	1.469(22)	C(2')—C(3')	1.414(23)
C(1)—C(8A)	1.550(17)	C(2')—C(14)	1.501(25)
C(1)—C(9)	1.544(25)	C(3')—C(4')	1.393(21)
N(2)—C(3)	1.463(24)	C(4')—C(4A')	1.411(22)
N(2)—C(10)	1.458(17)	C(4A')—C(5')	1.403(25)
C(3)—C(4)	1.473(16)	C(4A')—C(8A')	1.417(25)
C(3)—C(11)	1.562(25)	C(5')—C(6')	1.365(28)
C(4)—C(4A)	1.492(23)	C(6')—C(7')	1.383(31)
C(4A)—C(5)	1.413(18)	C(7')—C(8')	1.341(30)
C(4A)—C(8A)	1.370(21)	C(8')—C(8A')	1.414(23)

Ancistrocladidine (**1**)³ shows a loss of 15 amu from the molecular ion at m/z 405, but no stable species are observed below this mass.

The absolute configuration of naphthalene-isoquinoline alkaloids has been studied previously (8, 21). The cd spectrum of ancistrocladidine in MeOH showed a negative first Cotton effect ($[\theta]_{234} -34,179$) and a positive second Cotton effect ($[\theta]_{218} +70,167$) which clearly demonstrates the absolute configuration of ancistrocladidine to be that shown in **2**.

TABLE 3. Valency Angles (Å) with eds's (in parentheses)

C(13)—O(1)—C(8)	118.2(13)	C(7)—C(8)—O(1)	121.2(12)
C(8A)—C(8)—O(1)	117.7(12)	C(12)—O(2)—C(6)	116.0(11)
C(5)—C(6)—O(2)	123.9(14)	C(7)—C(6)—O(2)	115.9(11)
C(3')—C(4')—O(3)	115.0(11)	C(4A')—C(4')—O(3)	123.4(14)
C(15)—O(4)—C(5')	120.9(12)	C(4A')—C(5')—O(4)	115.8(13)
C(6')—C(5')—O(4)	118.9(16)	C(8A)—C(1)—N(2)	114.4(13)
C(9)—C(1)—N(2)	107.1(13)	C(3)—N(2)—C(1)	114.2(12)
C(10)—N(2)—C(1)	107.5(13)	C(9)—C(1)—C(8A)	108.5(12)
C(4A)—C(8A)—C(1)	119.1(12)	C(8)—C(8A)—C(1)	118.9(13)
C(10)—N(2)—C(3)	111.9(14)	C(4)—C(3)—N(2)	108.6(15)
C(11)—C(3)—N(2)	112.1(12)	C(11)—C(3)—C(4)	112.3(14)
C(4A)—C(4)—C(3)	111.6(13)	C(5)—C(4A)—C(4)	118.7(15)
C(8A)—C(4A)—C(4)	120.9(12)	C(8A)—C(4A)—C(5)	120.0(13)
C(6)—C(5)—C(4A)	119.3(14)	C(8)—C(8A)—C(4A)	122.0(13)
C(7)—C(6)—C(5)	120.2(12)	C(8)—C(7)—C(6)	118.3(13)
C(3')—C(7)—C(6)	118.9(12)	C(3')—C(7)—C(8)	122.1(14)
C(8A)—C(8)—C(7)	120.1(15)	C(2')—C(3')—C(7)	119.2(14)
C(4')—C(3')—C(7)	120.9(14)	C(8A')—C(1')—C(2')	121.3(16)
C(3')—C(2')—C(1')	119.9(16)	C(14)—C(2')—C(1')	120.3(16)
C(4A')—C(8A')—C(1')	118.8(13)	C(8')—C(8A')C(1')	123.0(16)
C(14)—C(2')—C(3')	119.8(13)	C(4')—C(3')—C(2')	119.9(12)
C(4A')—C(4')—C(3')	121.5(14)	C(5')—C(4A')—C(4')	125.3(15)
C(8A')—C(4A')—C(4')	118.6(15)	C(8A')—C(4A')—C(5')	116.2(13)
C(6')—C(5')—C(4A')	125.2(16)	C(8')—C(8A')—C(4A')	118.2(16)
C(7')—C(6')—C(5')	116.6(18)	C(8')—C(7')—C(6')	121.9(16)
C(8A')—C(8')—C(7')	121.9(17)		

³Kindly supplied by Dr. P. C. Parthasarathy, Hindustan CIBA-Geigy Ltd., Bombay, India.

EXPERIMENTAL

GENERAL EXPERIMENTAL PROCEDURES.—Melting points were determined using a Kofler hot-stage microscope and are uncorrected. Uv spectra were recorded on a Beckman model DB-G spectrophotometer and ir spectra on a Nicolet model MX-1 FT-IR interferometer. Mass spectra were obtained with Finnegan model 4500 quadrupole mass spectrometer. ¹H-nmr spectra were recorded on a Nicolet NT-360 instrument operating at 360 MHz at the NSF Regional NMR Facility at the University of Illinois at Urbana, Urbana-Champaign. X-Ray intensity data were measured with Mo-K_α radiation on a Nicolet P3 automated diffractometer. Alumina used for column chromatography was obtained from E. Merck, Darmstadt, W. Germany.

PLANT MATERIAL.—The leaf material of *A. tectorius* was collected at Sakae-Raj Environmental Research Station, Rachasima Province, Thailand, in October 1982. The plant material was identified by the Botany Section, Technical Division, Department of Agriculture, Ministry of Agriculture and Cooperatives, Thailand. A herbarium specimen is deposited in the herbarium of the Department of Pharmaceutical Botany, Faculty of Pharmaceutical Sciences, Chulalongkorn University, Bangkok, Thailand.

ISOLATION OF ANCISTROTECTORINE (2).—The dried powdered leaves of *A. tectorius* (800 g) were macerated twice for 3-day periods with 95% EtOH (8 liters and 5 liters) and the combined EtOH extracts pooled and evaporated under reduced pressure. The residue (125 g) was suspended in distilled H₂O (5 liters) and the filtrate extracted with CHCl₃ (2 × 5 liters). The combined CHCl₃ extracts were dried (Na₂SO₄), filtered, and evaporated under reduced pressure to afford a light brown powder (13 g).

The powder was divided into six portions, and each was separately chromatographed as follows. Each portion was dissolved in CHCl₃ (10 ml), absorbed onto neutral alumina (5 g), dried, and placed on top of a dry alumina column (6 × 20 cm) and eluted with Et₂O. Twenty fractions were collected, examined by tlc, and fractions of similar composition were combined. Pooled fractions 9-20 were homogeneous by tlc and yielded ancistrotectorine (2, 125 mg, 0.016% yield).

PHYSICAL PROPERTIES OF ANCISTROTECTORINE (2).—Ancistrotectorine (2) crystallized from Me₂CO as pale yellow needles, mp 134-140°; ir ν_{max} (KBr) 3375, 2960, 2925, 2840, 2770, 1640, 1600, 1555, 1458, 1400, 1362, 1320, 1115, 1095, 1085, 825, and 755 cm⁻¹; uv λ_{max} (EtOH) 234 (log ε 4.73), 286 (3.64), 306 (3.61), 323 (3.66), and 337 nm (3.69); ms *m/z* (ei, 70eV, 180°) no M⁺ observed, 420 (1%), 407 (28), 406 (100), 360 (17), 203 (72), 202 (5), 195 (6), 189 (4), 188 (9), 187 (5), 181 (8), 180 (36), 172 (28), 167 (9), 165 (28), 158 (31), 151 (33), 145 (8), 144 (8), 143 (8), 137 (7), 136 (8), 131 (7), 129 (10), 128 (8), 115 (12), and 107 (7); ms, *m/z* (ci, CH₄ gas, 70eV) 422 (M+H⁺, 100%) and 406 (9); ¹H-nmr (360 MHz, CDCl₃) δ 1.268 (d, 3H, *J*=6.1 Hz, 3-CH₃), 1.466 (d, 3H, *J*=6.4 Hz, 1-CH₃), 2.169 (s, 3H, 2'-CH₃), 2.484 (s, 3H, N-CH₃), 2.544 (ddq, 1H, *J*=3.0, 6.1, 10.3 Hz, 3-H), 2.655 (dd, 1H, *J*=3.0, 15.3 Hz, 4-Hβ), 2.787 (dd, 1H, *J*=10.3, 15.4 Hz, 4-Hα), 3.323 (s, 3H, 8-OCH₃), 3.691 (s, 3H, 6-OCH₃), 3.731 (q, 1H, *J*=6.4 Hz, 1-H), 3.985 (s, 3H, 5'-OCH₃), 6.518 (s, 1H, 5-H), 6.689 (d, 1H, *J*=7.5 Hz, 6'-H), 7.232 (s, 1H, 1'-H), 7.266 (t, 1H, *J*=7.8 Hz, 7'-H), 7.343 (d, 1H, *J*=8.0 Hz, 8'-H), and 9.565 (s, 1H, 4'-OH); [α]_D²⁶ 0° (CHCl₃); cd (MeOH) [θ]₂₁₈+70, 167, [θ]₂₃₄-34, 179, [θ]₂₈₃-10, 291, [θ]₃₀₈-5, 146, [θ]₃₂₁-6, 549 and [θ]₃₃₆-8, 420.

CRYSTALLOGRAPHIC ANALYSIS⁴.—Crystal data, C₂₆H₃₁NO₄, M=421.5, monoclinic, *a*=11.858(14), *b*=7.043(7), *c*=14.693(28) Å, β=111.43(12)°, *U*=1142.6 Å³, *Z*=2 *D*_c=1.22 g cm⁻³, space group P2₁, μ (MO-K_α)=0.46 cm⁻¹.

The crystal structure was elucidated using MULTAN (19) and refined using SHELX (20). In the final cycles of least-squares refinement, the positional parameters for all atoms, anisotropic thermal parameters for the C and O atoms and common isotropic thermal parameters for the methyl and non-methyl hydrogens were varied. Structure amplitudes and thermal parameters are listed in material available from the authors.

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We also thank Dr. R. A. Howie, Aberdeen University, for assistance in collecting the X-ray data and the SERC for a studentship (to HJC).

⁴Atomic coordinates for this structure have been deposited with the Cambridge Crystallographic Data Centre and can be obtained on request from Dr. Olga Kennard, University Chemical Laboratory, Lensfield Rd., Cambridge, CB2 EW, UK.

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