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HAPLODIMERINE – A NEW TYPE OF DIMERIC QUINOLINE ALKALOIDS

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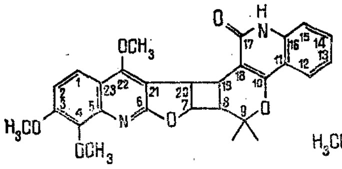
A base, which has been called haplodimerine, has been isolated from the fruit of the plant Haplophyllum foliosum Vved. It has been established by x-ray structural analysis that haplodimerine includes fragments of the known alkaloids skimmianine and flindersine attached to one another through the double bonds of furan and dimethylpyran rings with the formation of a four-membered ring.

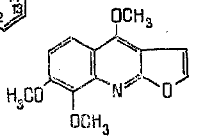
In an investigation of the fruit of the plant of Haplophyllum foliosum Vved. (family Rutaceae) growing in the environs of Alimtai (TadzhSSR), in addition to known alkaloids [1] we have isolated a minor base with the composition $C_{28}H_{26}N_2O_6$ (M^+ 486.1706 HRMS), mp 292-293°C, which we have called haplodimerine (I). The substance is optically inactive and sparingly soluble in organic solvents, water, and dilute alkali. It dissolves in mineral acids and gives the reaction for alkaloids with tungstosilicic acid.

The structure of the base isolated was first studied by the methods of IR, UV, NMR, and mass spectroscopy. The spectral material taken all together enabled us to determine the following fragments as components of the alkaloid (I): unsubstituted and ortho-substituted benzene rings, one of which formed part of a 2-quinolone nucleus, and three methoxy and two C–CH_3 groups [2, 3].

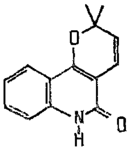
However, the spectral characteristics proved to be insufficient to determine the structure of haplodimerine. We performed an x-ray structural investigation which showed that the base isolated had structure (I), i.e., the haplodimerine molecule includes fragments of skimmianine (II), and flindersine (III) linked with one another through the double bonds of furan and dimethylpyran rings with the formation of a four-membered ring.



I



II



III

Institute of the Chemistry of Plant Substances, Academy of Sciences of the Uzbek SSR, Tashkent. Institute of Heteroorganic Compounds, Academy of Sciences of the USSR, Moscow. Translated from Khimiya Prirodynkh Soedinenii, No. 6, pp. 838-845, November-December, 1988. Original article submitted January 28, 1988.

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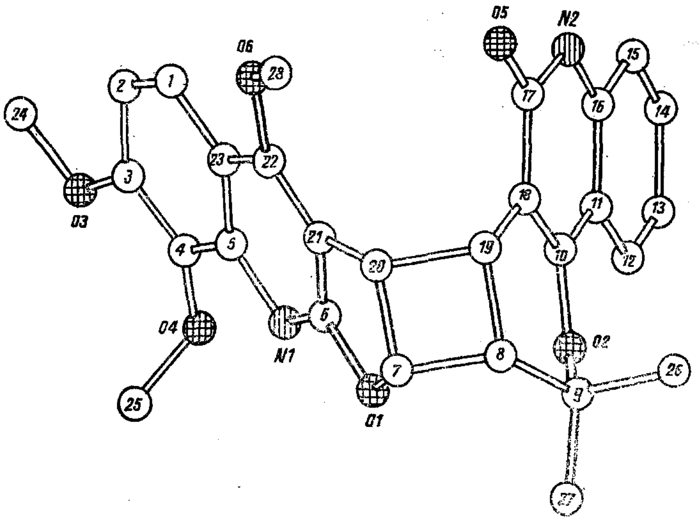

Fig. 1. Structure of one of the molecules (Ic) of haplodimerine.

TABLE 1. Characteristic Torsional Angles in the Structure of (I) [for comparison, the theoretical values of an unsubstituted cyclopentane ring (envelope) and of a cyclohexane ring (sofa) are given]

	Fragment	φ_{Ia}	φ_{Ib}	φ_{Ic}	φ_{theor}
Furan ring	O1C7C20C21	15,7	14,4	14,3	46,1
	C7C20C21C6	—11,0	—8,2	—10,2	—28,6
	C20C21C6O1	1,7	—1,4	2,4	0,0
	C21C6O1C7	8,9	10,9	7,1	28,6
	C6O1C7C20	—15,4	—15,7	—13,4	—46,1
Pyran ring	C8C9O2C10	44,0	48,3	48,9	60,0
	C9O2C10C18	—20,5	—30,8	—27,1	—30,0
	O2C10C18C19	—5,2	4,5	—2,7	0,0
	C10C18C19C8	3,6	1,1	6,4	0,0
	C18C19C8C9	22,9	18,9	18,9	30,0
	C19C8C9O2	—45,7	—42,0	—44,8	—60,0
Methyl groups	C24O3C3C2	12,3	6,2	—1,3	
	C24O3C3C4	—169,9	—171,8	—179,3	
	C25O4C4C3	—89,6	65,9	—89,4	
	C25O4C4C5	95 4	—122,4	92,1	
	C28O6C22C21	—11,5	—11,2	—14,5	
	C28O6C22C23	168,4	167,9	164,3	

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*The torsional angles are given for the inverted (Ib) molecule (i.e., that connected with the basic center of symmetry); the torsional angles φ_{Ia} and φ_{Ib} are given for the basic molecules (Table 4).

Analysis of literature information permitted us to conclude that haplodimerine is the first representative of a new type of dimeric quinoline alkaloids [4-6].

Alkaloid (I) crystallizes in the form of a racemate with three molecules (Ia, Ib, and Ic) in the independent part of the elementary cell which also contains one molecule of water, i.e., the crystals represent a 1/3-hydrate.

Figure 1 shows one of the independent molecules (Ic) in a projection on the mean-square plane of the cyclobutane ring. As can be seen from Fig. 1, the dihydrofuranoquinoline (C1-C7, C20-C25, C28, N1, O1, O3, O4, and O6 atoms) and the dihydropyrano-2-quinolone (the C8-C19, C26, C27, N2, O2, and O5 atoms) parts of the molecule have the mutual cis-arrangement relative to the plane of the cyclobutane ring (the C7, C8, C19, and C20 atoms), although the mutual trans arrangement would apparently be energetically favorable. In the structures of (I), the quinoline and quinolone nuclei have planar conformations, and, therefore, Table 1 gives the torsional angles of the dihydrofuran and dimethyldihydropyran rings, which determine the conformations of these rings, and also the angles characterizing the rotation of

TABLE 2. Bond Lengths (r, Å) in Structure (I) (the errors of determination do not exceed 0.012 Å)

Distance	r_{Ia}	r_{Ib}	r_{Ic}	Distance	r_{Ia}	r_{Ib}	r_{Ic}
C1—C2	1,35	1,37	1,37	C10—C11	1,44	1,43	1,44
C1—C23	1,42	1,37	1,39	C10—C18	1,34	1,35	1,35
C2—C3	1,38	1,40	1,42	C11—C12	1,39	1,40	1,43
C3—C4	1,38	1,39	1,39	C11—C16	1,41	1,39	1,38
C3—O3	1,39	1,36	1,38	C12—C13	1,37	1,36	1,38
C4—C5	1,42	1,42	1,40	C13—C14	1,37	1,41	1,39
C4—O4	1,39	1,35	1,38	C14—C15	1,38	1,39	1,38
C5—N1	1,39	1,38	1,40	C15—C16	1,40	1,39	1,38
C5—C23	1,39	1,42	1,42	C16—N2	1,40	1,40	1,39
N1—C6	1,29	1,30	1,31	N2—C17	1,37	1,37	1,38
C6—O1	1,40	1,38	1,36	C17—C18	1,44	1,46	1,46
C6—C21	1,40	1,42	1,44	C17—O5	1,26	1,26	1,25
O1—C7	1,48	1,47	1,46	C18—C19	1,49	1,51	1,52
C7—C8	1,53	1,53	1,54	C19—C20	1,57	1,58	1,57
C7—C20	1,54	1,53	1,54	C20—C21	1,51	1,50	1,49
C8—C9	1,50	1,50	1,52	C21—C22	1,34	1,35	1,36
C8—C19	1,53	1,57	1,54	C22—C23	1,42	1,44	1,45
C9—O2	1,47	1,48	1,49	C22—O6	1,37	1,37	1,37
C9—C26	1,53	1,52	1,51	O3—C24	1,39	1,36	1,44
C9—C27	1,53	1,51	1,53	O4—C25	1,44	1,39	1,41
O2—C10	1,37	1,37	1,36	O6—C28	1,44	1,44	1,44

TABLE 3. Valence Angles (ω, deg) in Structure (I) (errors not greater than 0.7°)

Angle	ω_{Ia}	ω_{Ib}	ω_{Ic}	Angle	ω_{Ia}	ω_{Ib}	ω_{Ic}
C2C1C23	120,4	121,5	122,6	C12C11C16	118,7	119,7	119,3
C1C2C3	121,6	120,9	117,2	C11C12C13	120,8	120,3	119,7
C2C3C4	119,9	119,4	122,2	C12C13C14	120,9	120,2	119,1
C2C3O3	125,3	124,4	123,1	C13C14C15	119,9	119,6	122,1
C4C3O3	114,8	116,2	114,7	C14C15C16	120,5	120,2	118,9
C3C4C5	119,5	119,5	118,7	C11C16C15	119,1	119,8	120,8
C3C4O4	120,3	121,7	120,1	C11C16N2	122,6	121,0	121,4
C5C4O4	118,9	118,3	121,2	C15C16N2	118,3	119,2	117,7
C4C5N1	116,5	118,6	116,6	C16N2C17	119,2	121,4	120,4
C4C5C23	120,0	119,5	119,8	N2C17C18	120,9	119,3	118,7
N1C5C23	123,3	121,9	123,6	N2C17O5	118,1	118,3	119,1
C5N1C6	112,1	114,4	114,4	C18C17O5	121,0	122,3	122,1
N1C6O1	116,8	116,5	117,9	C10C18C17	118,2	117,6	119,5
N1C6C21	131,1	131,1	128,9	C10C18C19	123,7	124,7	122,1
O1C6C21	112,2	112,4	113,1	C17C18C19	118,1	117,5	118,3
C6O1C7	108,9	107,8	108,1	C8C19C18	111,0	110,6	111,8
O1C7C8	115,4	113,6	113,8	C8C19C20	89,2	88,1	89,3
O1C7C20	104,6	106,3	106,7	C18C19C20	116,2	116,2	116,6
C8C7C20	90,3	91,7	90,4	C7C20C19	87,7	89,3	89,0
C7C8C9	122,6	122,9	123,5	C7C20C21	104,3	104,0	103,2
C7C8C19	89,7	89,5	89,9	C19C20C21	118,4	119,4	119,6
C9C8C19	117,8	117,5	117,3	C6C21C20	107,7	107,1	106,4
C8C9O2	110,1	110,5	109,1	C6C21C22	115,0	113,3	114,6
C8C9C26	110,9	111,7	111,6	C20C21C22	137,4	139,5	139,0
C8C9C27	113,3	111,3	113,3	C21C22C23	120,2	121,3	121,8
O2C9C26	106,6	103,7	107,3	C21C22O6	127,1	127,5	126,1
O2C9C27	105,2	106,4	102,7	C23C22O6	112,3	112,8	112,1
C26C9C27	110,5	112,8	112,3	C1C23C5	118,3	118,9	119,2
C9O2C10	117,0	117,6	116,2	C1C23C22	123,6	122,9	124,1
O2C10C11	113,3	114,4	114,2	C5C23C22	118,1	118,1	116,6
O2C10C18	123,2	121,5	124,3	C3O3C24	116,3	119,2	117,2
C11C10C18	123,4	123,5	121,5	C4O4C25	112,4	115,4	116,6
C10C11C12	125,9	123,5	122,6	C22O6C28	121,1	119,4	118,9
C10C11C16	115,3	116,5	118,0				

the method groups relative to the C—O bonds. It is apparent from the values of the torsional angles that in the three molecules (Ia), (Ib), and (Ic) the dihydrofuran ring assumes the form of a highly flattened envelope, while the dimethyldihydropyran ring has the rarely encountered "sofa" conformation (with the departure of the C9 atom from the plane of the other five atoms). Both rings retain their intrinsic C_S symmetry. A comparison of the majority of the torsional angles in the molecules of (Ia)-(Ic) shows differences by 4-5° on an average, which is apparently connected with packing effects. These effects are clearly pronounced in the molecule of (Ib) in the rotation of the methyl group relative to the O4—C4 bond.

TABLE 4. Coordinates of the Atoms (× 10^4) in the Structure of Haplodimerine Hydrate

Atom	Molecule (Ia)			Molecule (Ib)			Molecule (Ic)		
	x/a	y/b	z/c	x/a	y/b	z/c	x/a	y/b	z/c
O1	4613 (2)	2419 (2)	2427 (5)	1277 (2)	2434 (2)	1317 (5)	1825 (2)	—2104 (2)	9150 (5)
O2	5559 (2)	2010 (2)	2627 (5)	2192 (2)	1944 (2)	1096 (5)	1085 (2)	—1324 (2)	8106 (5)
O3	3156 (2)	617 (2)	5367 (5)	—484 (2)	1179 (3)	—2778 (6)	3736 (2)	—505 (2)	11667 (5)
O4	3808 (2)	1420 (2)	5503 (5)	191 (2)	1968 (2)	—2303 (5)	2901 (2)	—917 (2)	11863 (5)
O5	4840 (2)	710 (2)	—374 (5)	1686 (2)	434 (2)	3550 (5)	1940 (2)	—1194 (2)	4641 (5)
O6	3715 (2)	1039 (2)	—27 (5)	490 (2)	818 (2)	2712 (5)	2970 (2)	—1645 (2)	6526 (5)
N1	4153 (2)	1819 (3)	3418 (6)	741 (2)	1992 (3)	—76 (6)	2429 (2)	—1540 (3)	9962 (5)
N2	5180 (2)	359 (3)	1459 (6)	932 (2)	230 (3)	1696 (6)	1714 (2)	—375 (2)	5544 (6)
C1	3330 (3)	707 (3)	2039 (8)	—10 (3)	768 (4)	419 (8)	3427 (3)	—1015 (3)	8443 (8)
C2	3162 (3)	532 (4)	3093 (8)	—274 (3)	746 (4)	—718 (9)	3677 (2)	—733 (3)	9114 (8)
C3	3314 (3)	765 (3)	4247 (8)	—218 (3)	1153 (4)	—1649 (9)	3505 (2)	—745 (3)	10595 (8)
C4	3641 (3)	1195 (3)	4341 (8)	135 (3)	1551 (4)	—1455 (8)	3081 (2)	—983 (3)	10748 (8)
C5	3832 (3)	1376 (3)	3257 (8)	410 (3)	1573 (3)	—273 (8)	2840 (2)	—1277 (3)	9741 (7)
C6	4297 (3)	1969 (3)	2374 (8)	956 (3)	2003 (4)	1056 (8)	2220 (2)	—1821 (3)	8997 (8)
C7	4681 (3)	2585 (4)	1132 (8)	1415 (3)	2438 (4)	2671 (7)	1701 (2)	—2461 (4)	8048 (7)
C8	5157 (2)	2456 (4)	756 (8)	1919 (3)	2277 (3)	3036 (8)	1265 (2)	—2258 (3)	7221 (8)
C9	5584 (3)	2463 (4)	1679 (8)	2279 (3)	2345 (3)	2163 (7)	892 (3)	—1899 (3)	7707 (8)
C10	5403 (3)	1480 (3)	2206 (8)	2106 (3)	1381 (3)	1356 (7)	1323 (2)	—1047 (3)	7276 (7)
C11	5533 (3)	1022 (3)	3079 (7)	2227 (2)	980 (3)	437 (7)	1322 (2)	—428 (3)	7392 (7)
C12	5777 (3)	1084 (4)	4261 (8)	2465 (3)	1136 (3)	—568 (8)	1118 (3)	—137 (4)	8355 (9)
C13	5874 (3)	616 (4)	5023 (8)	2584 (3)	728 (3)	—1379 (7)	1129 (3)	456 (4)	8410 (9)
C14	5742 (3)	73 (3)	4627 (9)	2472 (3)	145 (3)	—1209 (8)	1334 (3)	760 (3)	7513 (9)
C15	5514 (3)	—7 (3)	3450 (9)	2245 (3)	—12 (3)	—201 (7)	1521 (3)	488 (3)	6559 (8)
C16	5403 (3)	465 (3)	2655 (8)	2136 (3)	402 (3)	647 (8)	1516 (3)	—111 (3)	6510 (8)
C17	5046 (3)	815 (4)	696 (7)	1823 (3)	621 (4)	2566 (8)	1749 (3)	—968 (3)	5498 (7)
C18	5153 (3)	1398 (3)	1092 (7)	1897 (3)	1232 (3)	2357 (8)	1543 (3)	—1311 (3)	6421 (7)
C19	4983 (3)	1874 (3)	236 (7)	1767 (3)	1647 (3)	3333 (7)	1566 (2)	—1961 (3)	6330 (7)
C20	4478 (3)	2081 (3)	317 (8)	1253 (3)	1866 (3)	3163 (7)	2000 (2)	—2259 (3)	7049 (8)
C21	4192 (3)	1753 (3)	1158 (7)	924 (3)	1638 (3)	2099 (7)	2361 (2)	—1900 (3)	7770 (7)
C22	3879 (3)	1331 (3)	1039 (7)	602 (3)	1221 (3)	1872 (8)	2760 (3)	—1630 (3)	7593 (7)
C23	3675 (3)	1140 (3)	2095 (7)	331 (3)	1171 (3)	671 (8)	3015 (3)	—1394 (3)	8571 (7)
C24	2885 (4)	127 (5)	5348 (11)	—799 (4)	758 (6)	—3092 (11)	4175 (3)	—246 (4)	11561 (9)
C25	3542 (3)	1900 (4)	5850 (9)	307 (3)	1775 (4)	—3449 (9)	3019 (3)	—1342 (4)	12786 (9)
C26	6008 (3)	2332 (4)	1032 (8)	2257 (3)	2935 (3)	1551 (8)	494 (3)	—1795 (4)	6701 (9)
C27	5650 (3)	3026 (4)	2422 (9)	2749 (3)	2202 (3)	2809 (8)	737 (3)	—2135 (3)	8910 (8)
C28	3812 (3)	1235 (4)	—1237 (8)	662 (3)	880 (4)	4013 (8)	2828 (3)	—2073 (4)	5588 (8)
O_w	1847 (2)	9000 (2)	2000 (5)						

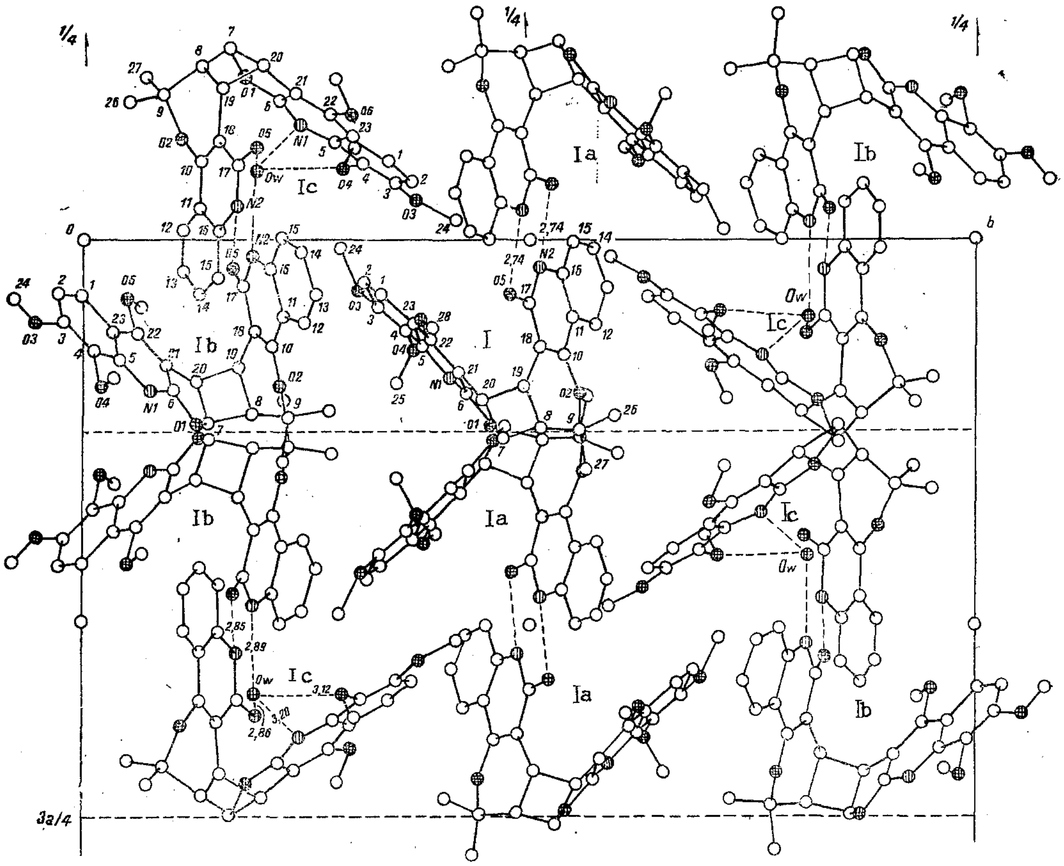

Fig. 2. Packing of the molecules.

The geometric parameters (bond lengths and valence angles) of structure (I) are given in Tables 2 and 3. A comparative analysis of chemically identical interatomic distances and valence angles of the (Ia)-(Ic) molecules reveals no anomalous deviations and shows good agreement of their averaged values with the standard values [8]. The observed tendency to a decrease in the length of the C17=O5 and C10=C18 bonds (1.26 Å and 1.35 Å, respectively) and to a decrease in the length of the C17–C18 and C10–C11 bonds (1.45 Å and 1.44 Å, respectively) indicates the existence of conjugation in the quinolone nucleus in structure (I).

The packing of the molecules in the structure of (I) is shown in Fig. 2 in a projection in the AB plane. As can be seen from Fig. 2, the molecules of (Ia), transformed by a (0, 1/2, 0) and (1/2, 1/2, 0) center of symmetry are linked by pairs of H bonds of the N–H···O type (the $N2_{Ia}...O5_{Ia}$ distance is 2.74 Å) forming chains. The initial molecules (Ib) and (Ic) interact by similar H-bonds and analogous sections (the $N2I_c...O5_{Ib}$ distance is 2.85 Å), but here the molecule of water of crystallization is an intermediate link for the establishment of these interactions (the $O_W...N2_{Ib}$ distance is 2.89 Å and the $O_W...O5_{Ic}$ distance is 2.86 Å). Furthermore, one of the H atoms of the water molecule possibly interacts weakly with the unshared pairs of electrons of $N1_{Ic}$ or $O4_{Ic}$ (the $O_W...N1_{Ic}$ and $O_W...O4_{Ic}$ distances are, respectively, 3.20 and 3.12 Å).

The structure of (I) determined by the x-ray structural method does not contradict the results of IR and UV spectroscopy and permits the four unresolved one-proton signals in the PMR spectrum of haplodimerine to be assigned to the protons of the cyclobutane ring. It also well explains the mass spectrum of haplodimerine, in which the fragmentation of the intensive peak of an ion with m/z 259 resembles the breakdown of the molecular ion of skimmianine (peaks of ions with m/z 258, 256, 244, 230, 216) [9]. These ion peaks are not shifted in the mass spectrum of the deutero analog of haplodimerine. Shifts of the peaks by 1 m.u. are observed for the fragments with m/z 227 and 212. The formation of the peak of an ion with m/z 212, one of the most intensive in the spectrum of (I) is typical for the breakdown of the molecular ion of flindersine [10].

EXPERIMENTAL

The spectra of the substances were obtained on the following instruments: Hitachi EPS-3T (ethanol) and UR-20 (KBr); MKh-1310; and JNM-4H-100/100 MHz (δ scale, CF_3COOH, 0 – HMDS).

Isolation of the Alkaloids. The extraction of the plant raw material and the working up of the combined alkaloids were described in [1]. The combined chloroform eluates (30 g) obtained on the separation of the chloroform-extracted nonphenolic alkaloids [1] were rechromatographed on alumina. Ethereal eluates yielded skimmianine, and chloroform eluates 150 mg of technical haplodimerine (0.001% on the weight of the dry raw material).

Haplodimerine, colorless prisms, mp 292-293°C (from ethanol) giving on TLC (silica gel) in the toluene–ethyl acetate–formic acid (5:4:1) system a spot having a bright blue fluorescence in UV light and being revealed by the Dragendorff reagent.

IR spectrum: $\nu_{\max}^{\mathrm{KBr}}$, 3165, 1645 cm^{-1} (NHCO), 1620, 1587, 1520, and 1490 cm^{-1} (aromatic system).

UV spectrum: $\lambda_{\max}^{C_2H_5OH}$ 219, 228, 243.5 inflection, 278, 286.5, 315, 325 nm (log ε 4.68, 4.69, 4.59, 3.94, 3.96, 3.94, 3.93). $\lambda_{\max}^{C_2H_5OH+H^+}$ 220, 252.5, 273, 285, 325 nm (log ε 4.67, 4.51, 3.90, 3.88, 4.06).

Mass spectrum: m/z (%) 486 (M^+ 1.1), 485 (1.6), 260 (20), 259 (100), 258 (20), 256 (1.6), 245 (17), 244 (75), 230 (40), 229 (14), 228 (14), 227 (15), 216 (10), 213 (25), 212 (50).

NMR spectrum (ppm): 7.81 (dd, 1H, J = 8 and 2 Hz, H-12); 7.42-6.87 (3H, m, H-13, 14, 15), 7.53 and 6.85 (d, 1H each, J = 9.5 Hz, H-1.2), 5.58, 4.85, 4.00, 3.25 (m, 1H each, H-y, 20, 19, 8), 4.20, 3.57, 3.21 ($3OCH_3$), 1.50 and 1.03 $[C(CH_3)_2]$.

The x-ray structural analysis was performed on a Hilger–Watts automatic four-circle diffractometer (λ MoK_α θ/2θ scanning, θ ≤ 24°), a = 29.363 (3), b = 23.234 (3), c = 10.765 (1) Å, β = 96.90 (1)°, d = 1.317 g/cm^3; space group $P2_1/c$; z = 12. In the interpretation and refinement of the structure 2875 reflections with $I \geq 2\sigma$ were used. The structure was deciphered by the direct method using the MULTAN program and was refined by the full-matrix least-square method in the anisotropic approximation for all the nonhydrogen atoms. In the refinement, the theoretically calculated positions of the H atoms attached to carbon atoms were included in the calculation. The final values of the divergence factors were $R_1 = 0.059$, $R_2 = 0.037$. All the calculations were performed on an Eclipse S/200 computer by means of INEXTL programs [11]. The coordinates of the nonhydrogen atoms of the molecule of (I) are given in Table 4.

CONCLUSIONS

A new dimeric alkaloid with the composition $C_{28}H_{26}N_2O_6$ has been isolated from Haplophyllum foliosum Vved. and has been called haplodimerine. It has been established by x-ray structural analysis that haplodimerine has structure (I), which includes fragments of the alkaloids skimmianine and flindersine linked with one another through the double bonds of the furan and dimethylpyrone rings with the formation of a four-membered ring.

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