

Fig. 1. Thermal ellipsoid plot of the title compound with ellipsoids drawn at the 20% probability level for nondisordered atoms. The disordered atoms are drawn as fixed-radius spheres. The open bonds represent the lower-occupancy conformation for the acetoxy group. The C(8) methylene H atoms for the lower-occupancy conformation are omitted for clarity.

Secondary-extinction parameter $p = 0.0010(2)$ in $F_c^* = F_c/[1.0 + 0.002(p)F_o^2/\sin(2\theta)]^{0.25}$. There were 256 parameters refined: atom coordinates, anisotropic thermal parameters for all non-H atoms, H atoms included using riding model in *SHELXTL*, H riding on C, C–H = 0.96 Å. $U(H) = 1.2U_{eq}(C)$. Amine hydrogens refined isotropically. Site occupation for the two conformations of the acetoxy group was variable and constrained to sum to unity, restraint applied to next-nearest-neighbor C...O distance (2.405 ± 0.008 Å) from each disordered terminal methyl C to respective acetyl O, C(8) treated as a pivot atom with methylene hydrogen pairs idealized for the two conformations and with the previously stated constraint on site occupation. $(\Delta/\sigma)_{max} = 0.12$, $R = 0.066$, $wR = 0.067$, $S = 1.825$. Final difference Fourier excursions 0.38 and -0.34 e Å⁻³. Atomic scattering factors from *International Tables for X-ray*

Crystallography (1974).^{*} Atom numbering for Table 1, atom coordinates, and Table 2, bond distances, bond angles and selected torsion angles, follows that shown in Fig. 1.

Related literature. For the structure of a similar compound, 2,4,6-trinitro-2,4,6-triaza-1,7-heptanediyldiacetate, see Cobbleddick & Small (1973a–c). For a similarly substituted pentaazanonane, see George & Gilardi (1989).

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^{*} Lists of structure factors, anisotropic thermal parameters and hydrogen coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51806 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of 4,7,8-Trimethoxyfuro[2,3-*b*]quinoline

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Abstract. Skimmianine, C₁₄H₁₃NO₄, $M_r = 259.3$, monoclinic, $P2_1/c$, $a = 7.390(1)$, $b = 10.458(1)$, $c = 15.584(4)$ Å, $\beta = 94.92(1)^\circ$, $V = 1199(1)$ Å³, $Z = 4$, $D_x = 1.435$ g cm⁻³, $\lambda(\text{Cu } K\alpha) = 1.5418$ Å, $\mu = 8.4$ cm⁻¹, $F(000) = 544$, $T = 297$ K, $R = 0.043$ for 2408 observations with $I > 3\sigma(I)$ (of 2656 unique

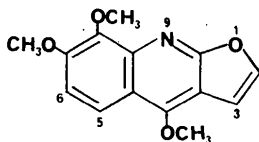
data). The orientation of the C4-methoxy group towards the C3 hydrogen corresponds to that predicted in solution by NMR studies. No unusual bond distances or angles are seen in the structure. Intermolecular distances correspond to van der Waals contacts.

Experimental. The title compound was isolated from the chloroform extract of the dried fruits of *Fagara rhoifolia*. The concentrated chloroform extract was subjected to column chromatography on silica gel using

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chloroform : acetone (6 : 1) as eluent. The crude product was recrystallized from a chloroform : acetonitrile (1 : 1) solution to afford 200 mg of (1): m.p. 452–453 K. Crystals were grown by slow evaporation from an acetonitrile : acetone (1 : 1) solution. The spectral data of this sample (IR, UV–visible and ¹H NMR) are identical to those reported in the literature.



A transparent orange rectangular plate crystal of dimensions 0.50 × 0.45 × 0.30 mm was mounted on a glass fiber, on an Enraf–Nonius CAD-4 diffractometer, data measured with graphite-monochromated Cu K α radiation. Unit-cell dimensions from setting angles for 25 reflections having 30 < θ < 39°, ω -2 θ scan mode at 2.4–16.5° min⁻¹, scan width (°) of 1.0 + 0.14tan(θ). Data having 0 < θ < 75°, for 0 < h < 9, 0 < k < 13, -19 < l < 19 measured. Negligible drift in three standards (181, 219, 441) measured every 3600 s, 2656 unique data, 2408 observations with $I > 3\sigma(I)$, from counting statistics. Empirical extinction [$F_c = F_o / (1.0 + 1 \times 10^{-6} I_o)$] and Lorentz–polarization corrections, but absorption ignored ($\mu = 8.4 \text{ cm}^{-1}$). Structure solved by direct methods using MULTAN 11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) and Fourier methods. Full-matrix least-squares refinement, $w(F_o - F_c)^2$ minimized with $w = 4F_o^2[\sigma^2(F) + (0.04F_o^2)^2]^{-1}$. Anisotropic thermal parameters for non-H atoms, isotropic for all H atoms, which were located from difference Fourier map. Final $R = 0.043$, $wR = 0.080$, $S = 2.42$ for observed data and 224 parameters. Maximum density fluctuation in final difference map in range 0.318–0.189 e Å⁻³, $(\Delta/\sigma)_{\text{max}} = 0.07$. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974) and programs used were those of Enraf–Nonius (1982) SDP. Fig. 1 (ORTEPII, Johnson, 1976) shows the

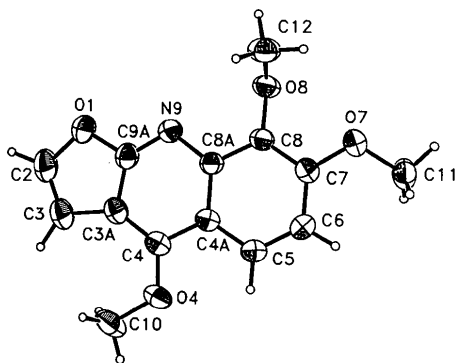


Fig. 1. Perspective view of the molecule with numbering scheme. Thermal ellipsoids are drawn at the 50% probability level.

Table 1. Fractional atomic coordinates and equivalent isotropic thermal parameters

Anisotropically refined atoms are given in the form of the isotropic equivalent displacement parameter defined as:
 $\frac{1}{3}[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)]$.

	x	y	z	B(Å ²)
O1	0.0758 (2)	0.0657 (1)	0.25603 (6)	4.41 (2)
O4	0.2162 (1)	0.24702 (8)	0.53140 (6)	4.13 (2)
O7	0.3624 (2)	-0.34087 (8)	0.59920 (6)	4.24 (2)
O8	0.2264 (1)	-0.30955 (7)	0.43624 (5)	3.48 (2)
N9	0.1672 (2)	-0.07255 (9)	0.36662 (6)	3.19 (2)
C2	0.0505 (2)	0.1965 (1)	0.2485 (1)	4.59 (3)
C3a	0.1438 (2)	0.1609 (1)	0.38682 (8)	3.22 (2)
C3	0.0877 (2)	0.2573 (1)	0.32281 (9)	4.12 (3)
C4	0.1998 (2)	0.1513 (1)	0.47379 (8)	3.05 (2)
C4a	0.2452 (2)	0.0278 (1)	0.50784 (7)	2.80 (2)
C5	0.3085 (2)	0.0088 (1)	0.59531 (7)	3.27 (2)
C6	0.3513 (2)	-0.1104 (1)	0.62629 (7)	3.46 (2)
C7	0.3275 (2)	-0.2186 (1)	0.57266 (7)	3.10 (2)
C8a	0.2250 (2)	-0.0807 (1)	0.45216 (7)	2.73 (2)
C8	0.2641 (2)	-0.2039 (1)	0.48721 (7)	2.84 (2)
C9a	0.1330 (2)	0.0438 (1)	0.34081 (7)	3.30 (2)
C10	0.1715 (2)	0.3738 (1)	0.5039 (1)	4.70 (3)
C11	0.4074 (2)	-0.3620 (1)	0.68844 (8)	4.28 (3)
C12	0.3701 (2)	-0.3540 (1)	0.39087 (9)	4.46 (3)

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

O1–C2	1.384 (2)	C4a–C5	1.417 (2)
O1–C9a	1.371 (1)	C4a–C8a	1.429 (1)
O4–C4	1.343 (1)	C5–C6	1.364 (2)
O4–C10	1.423 (2)	O7–C7	1.362 (1)
C6–C7	1.409 (2)	O7–C11	1.419 (2)
O8–C8	1.375 (1)	C7–C8	1.382 (1)
O8–C12	1.405 (2)	C8a–C8	1.419 (1)
N9–C8a	1.367 (1)	N9–C9a	1.299 (2)
C2–C3	1.328 (2)	C3a–C3	1.453 (2)
C3a–C4	1.386 (2)	C3a–C9a	1.418 (2)
C4–C4a	1.425 (1)		
C2–O1–C9a	105.9 (1)	C4–O4–C10	119.2 (1)
O7–C7–C6	124.3 (1)	C7–O7–C11	117.80 (9)
O7–C7–C8	115.9 (1)	C8–O8–C12	115.6 (1)
C6–C7–C8	119.8 (1)	C8a–N9–C9a	113.4 (1)
N9–C8a–C4a	123.4 (1)	O1–C2–C3	112.5 (1)
N9–C8a–C8	117.94 (9)	C4a–C8a–C8	118.7 (1)
O8–C8–C7	120.13 (9)	C3–C3a–C4	139.9 (1)
O8–C8–C8a	118.84 (9)	C3–C3a–C9a	104.6 (1)
C7–C8–C8a	120.9 (1)	C4–C3a–C9a	115.5 (1)
O1–C9a–N9	119.4 (1)	C2–C3–C3a	107.0 (1)
O1–C9a–C3a	110.0 (1)	N9–C9a–C3a	130.6 (1)
O4–C4–C3a	127.0 (1)	O4–C4–C4a	114.9 (1)
C3a–C4–C4a	118.1 (1)	C4–C4a–C5	122.2 (1)
C4–C4a–C8a	119.00 (9)	C5–C4a–C8a	118.8 (1)
C4a–C5–C6	121.1 (1)	C5–C6–C7	120.7 (1)
C2–O1–C9a–N9	179.83 (12)	C10–O4–C4–C3a	0.57 (19)
C3–C3a–C9a–N9	-179.69 (14)	C11–O7–C7–C6	6.90 (19)
C12–O8–C8–C8a	92.63 (13)		

molecular structure and its numbering scheme. Atomic coordinates are given in Table 1,* and molecular geometry in Table 2.

* Lists of H-atom parameters, anisotropic thermal parameters and structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51802 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Related literature. Two-dimensional NMR analysis of this compound shows a preferred orientation of the C4-methoxy group towards the H atom of C3 (Cox, Prieto, Retamozo & Rodriguez, 1989). Details for the isolation are given by Takeda (1941), Diment, Ritchie & Taylor (1967), Calderwood & Fish (1966), Fish & Waterman (1971), Benages, Juarez, Albonico, Urzua & Cassels (1974) and Torres & Cassels (1978). For other spectroscopic data see Mitscher, Bathala, Clark & Beal (1975).

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Crystal Studies of Heterocyclic Compounds Containing One Oxygen and Two Nitrogen Atoms. XIII. 5,6,7,8,9,10-Hexahydro-4*H*-*N,N'*-ditosyl-1-oxa-5,9-diazadibenzo[*b,k*]cyclododecene

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Abstract. $C_{31}H_{32}N_2O_5S_2$, $M_r = 576.7$, monoclinic, $P2_1/c$, $a = 11.358$ (4), $b = 21.199$ (6), $c = 12.23$ (3) Å, $\beta = 96.14$ (2)°, $V = 2928.4$ (1.6) Å³, $Z = 4$, $D_x = 1.307$ (1) g cm⁻³, $\lambda(\text{Cu } K\alpha) = 1.54178$ Å, $\mu = 18.83$ cm⁻¹, $F(000) = 1216$. Diffractometer data at room temperature, $R = 0.044$ for 3760 reflections with $I > 2.5\sigma(I)$. The 12-membered heterocycle may be considered as consisting of two parts, in chair-like and boat-like conformations respectively, while the tosyl groups are in an *exo,exo* conformation. The heterocycles are arranged along the *x* axis.

Experimental. The title compound was obtained by condensation of the disodium salt of bis[2-(ditosylaminomethyl)phenyl] ether with 1,3-bis(tosyloxy)propane. The formula was confirmed by MS, IR and NMR spectra.

Colourless crystals from ethanol at room temperature; crystal size 0.1 × 0.2 × 0.3 mm, Stoe diffractometer using θ – 2θ scan technique; unit-cell parameters from 21 reflections, $\theta_{\text{max}} = 20^\circ$, Cu $K\alpha$ radiation, range of *h*, *k* and *l* 0→12, 0→23, –13→13 respectively. Total of 4345 unique reflections measured to $(\sin\theta)/\lambda$