

O...N, O...H and H—N interatomic distances are 3.087, 2.30, and 0.86 Å, respectively, and the O...H—N angle is 152° (Simonov, Malinovskii, Ganin, Dvorkin, Bogatskii, Lukyanenko & Popkov, 1981). The individual molecules are linked together by means of C=O...H—N hydrogen bonds. The O(5)...N(2'), O(5)...H(N2'), and H(N2)—N(2) bond distances are 2.850 (3), 2.06 (2), and 0.79 (2) Å, respectively. The O(5)...H(N2')—N(2') angle is almost linear with a value of 176 (2)°. These values fall in the ranges (N...O, 2.859–3.094; O...H, 1.92–2.36 Å; O...H—N, 161–177°) found for other macrocyclic amides (Northolt & Alexander, 1968; Simonov, Malinovskii, Ganin, Dvorkin, Bogatskii, Lukyanenko & Popkov, 1981).

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***trans*-3-(3,4-Dimethoxyphenyl)-6,7,8-trimethoxy-1-methyl-1,2,3,4-tetrahydroisoquinoline—Methanol (1/1)**

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Abstract. C₂₁H₂₇NO₅·CH₄O, *M_r* = 405.49, triclinic, *P* $\bar{1}$, *a* = 8.918 (2), *b* = 11.259 (1), *c* = 11.561 (3) Å, α = 101.37 (1), β = 94.37 (2), γ = 103.30 (1)°, *V* =

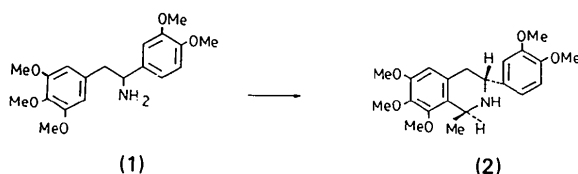
1098.5 (4) Å³, *Z* = 2, *D_x* = 1.226 Mg m⁻³, m.p. = 359 K, $\lambda(\text{Mo } K\alpha)$ = 0.7107 Å, μ = 0.095 mm⁻¹, *T* = 295 K, *F*(000) = 436, final *R* = 0.068 for 2047 observed reflections. The heterocyclic fragment of the molecule exhibits a half-chair conformation. The

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structural data also confirm the earlier configurational assignments and show that the solid-state conformation has the methyl and aryl substituents in pseudo-axial and equatorial positions respectively. The cohesion of the crystal is the result of van der Waals interactions and possible C—H...O hydrogen bonds. There are no unusual bond lengths or angles.

Introduction. In connection with our studies on the synthesis and stereochemistry of 3-arylisoquinolines, we have recently reported the preparation and preferred conformation in CDCl₃ solution of the isoquinoline (2), on the basis of ¹H NMR data (NOE difference measurements) (Dominguez, Badia, Castedo & Dominguez, 1988). In the present paper, we wish to report unequivocal evidence for the validity of our conclusions.

Thus, X-ray crystallographic investigations of the compound (2), obtained by diastereoselective cyclization of the 1,2-diarylethylamine (1) with acetaldehyde in acidic media, define the structure of the tetrahydroisoquinoline so obtained as *trans*-3-(3,4-dimethoxyphenyl)-6,7,8-trimethoxy-1-methyl-1,2,3,4-tetrahydroisoquinoline.



To the best of our knowledge, this work represents the first crystallographic study carried out on 3-aryltetrahydroisoquinoline derivatives.

Experimental. Colourless needle-like crystals grown from methanol. Data collected from crystal approximately 0.3 × 0.3 × 0.5 mm. Cell parameters determined by least squares from the setting angles of 25 reflections (30 ≤ 2θ ≤ 50°); 5085 intensities collected (4 ≤ 2θ ≤ 50°); Phillips PW 1100 four-circle diffractometer, graphite-monochromated Mo Kα radiation, ω scans; three standard reflections (200, 040, 005) measured every 60 showed only random deviation from mean intensity; 3084 unique reflections; Lp applied but no absorption correction; 2047 reflections [I ≥ 2.5σ(I)] considered observed and included in refinement; index range h -10→10, k -13→11, l 0→11; structure solved

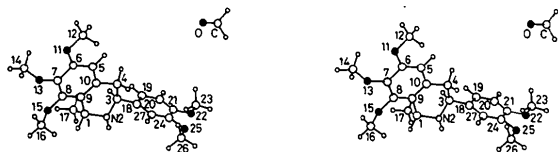


Fig. 1. Stereoscopic view of the molecule with the atom numbering.

Table 1. Fractional atomic coordinates (× 10⁴ for C, N and O) and equivalent isotropic temperature factors, calculated according to Willis & Pryor (1975)

	x	y	z	B _{eq} (Å ²)
C(1)	115 (10)	2604 (8)	-3927 (7)	4.32
N(2)	756 (10)	1605 (7)	-3571 (7)	5.02
C(3)	1643 (10)	2009 (8)	-2407 (8)	4.39
C(4)	606 (10)	2254 (8)	-1448 (7)	4.13
C(5)	-1361 (9)	3471 (7)	-925 (7)	3.78
C(6)	-2465 (9)	4077 (7)	-1224 (7)	3.74
C(7)	-2771 (9)	4183 (7)	-2397 (8)	3.91
C(8)	-1927 (9)	3694 (7)	-3256 (7)	3.74
C(9)	-794 (9)	3110 (7)	-2955 (7)	3.60
C(10)	-529 (9)	2973 (7)	-1805 (7)	3.53
O(11)	-3360 (7)	4590 (5)	-455 (5)	4.70
C(12)	-3222 (11)	4380 (9)	716 (8)	4.98
O(13)	-3973 (6)	4698 (6)	-2702 (5)	4.85
C(14)	-3517 (12)	6006 (10)	-2617 (11)	7.39
O(15)	-2168 (7)	3829 (6)	-4407 (5)	4.99
C(16)	-3628 (13)	3175 (12)	-5071 (9)	7.91
C(17)	1332 (11)	3665 (9)	-4237 (8)	5.35
C(18)	2601 (10)	1096 (8)	-2188 (7)	3.85
C(19)	4179 (10)	1556 (8)	-1860 (8)	4.69
C(20)	5100 (10)	755 (9)	-1643 (8)	4.85
C(21)	4436 (10)	-504 (8)	-1745 (7)	4.07
O(22)	5250 (7)	-1355 (6)	-1538 (6)	5.32
C(23)	6871 (10)	-889 (9)	-1123 (9)	5.86
C(24)	2832 (10)	-974 (8)	-2078 (7)	3.86
O(25)	2290 (7)	-2228 (6)	-2147 (6)	5.41
C(26)	676 (12)	-2743 (9)	-2447 (10)	6.58
C(27)	1922 (9)	-180 (8)	-2305 (7)	3.82
C	3140 (13)	262 (11)	4325 (11)	8.47
O	1772 (9)	611 (7)	4056 (7)	7.31

with MULTAN80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980); refinement by least squares with SHELX76 (Sheldrick, 1976), unit weights, Σ(ΔF)² minimized. Non-H atoms refined anisotropically, H atoms calculated, except H(2), and refined isotropically. Final R = 0.068. Max. Δ/σ (for non-H atoms) < 0.343. Max. and min. electron densities in final difference map 0.29 and -0.30 e Å⁻³. Atomic scattering factors from SHELX76.*

Discussion. Final atomic parameters with their B_{eq} values (Willis & Pryor, 1975) are given in Table 1. Other parameters of interest, such as intermolecular contacts and mean planes, were calculated with PARST (Nardelli, 1983). Fig. 1 shows a stereoscopic view of the molecule and the atom numbering (Motherwell & Clegg, 1978).

The molecule consists of a hydrogenated heterocyclic ring condensed with an aromatic ring and, as substituents, a methyl group and an aryl group bonded to the asymmetric atoms C(1) and C(3) respectively, and five methoxy groups bonded to C(6), C(7), C(8), C(21) and C(24).

* Lists of structure factors, anisotropic thermal parameters, bond lengths and bond angles, least-squares planes, and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51134 (29 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

The condensed aromatic ring is planar within experimental error, $\sum(\Delta/\sigma)^2 = 7.852$ vs. 7.810 max. for three degrees of freedom. A description of the orientation of the methoxy groups can be found in the relevant torsion angles: C(5)–C(6)–O(11)–C(12) -6 (1), C(6)–C(7)–O(13)–C(14) 92 (1) and C(7)–C(8)–O(15)–C(16) -67 (1) $^\circ$. It is noteworthy that the C(6) substituent is nearly coplanar while the C(7) substituent adopts a normal conformation relative to the aromatic ring already mentioned (Bellard, Elliot & McDonald, 1982). On the other hand, the plane of the C(8) methoxy group makes an angle of 113.3 (5) $^\circ$ with the condensed aromatic ring (Miravittles, Solans, Bladé-Font, Germain & Declercq, 1982).

The heterocyclic fragment of the molecule exhibits a half-chair conformation. The C(1)–C(9)–C(10)–C(4) angle is equal to -4 (1) $^\circ$ and the remaining torsion angles in the heterocyclic ring are consistent with the proposed conformation (Urbaniak, Jaskólski, Rozwadowska & Kosturkiewicz, 1986; Ammon, Prasad, Barnhart, Syal, El-Sayed & Wassel, 1987). The sum of valence angles around the N atom is 338 (10) $^\circ$ showing its considerable pyramidization. The structural data confirm the early configurational assignments (Dominguez, Badía, Castedo & Dominguez, 1988) and show that the solid-state conformation has the C(1) and C(3) substituents in pseudoaxial and equatorial positions respectively, torsion angles C(17)–C(1)–C(9)–C(10) 106 (1), H(1)–C(1)–C(9)–C(10) 139 (1), C(18)–C(3)–C(4)–H(4a) 70 (1), C(18)–C(3)–C(4)–H(4b) -50 (1), H(3)–C(3)–C(4)–H(4a) -46 (1), H(3)–C(3)–C(4)–H(4b) -166 (1) $^\circ$.

Bond lengths and angles in the heterocyclic ring do not deviate significantly from standard values reported for other compounds containing a similar ring system (Pavkovic, Glowinski, Feng & Brown, 1981, and

references therein; Ammon, Prasad, Barnhart, Syal, El-Sayed & Wassel, 1987).

In the aryl group at C(3), the methoxy groups are approximately coplanar with the benzene nucleus, as can be seen from the torsion angles C(20)–C(21)–O(22)–C(23) 4 (1), C(27)–C(24)–O(25)–C(26) -1 (1) $^\circ$. This behaviour is typical of *o*-dimethoxybenzene derivatives (Caillet, 1982; Bellard, Elliot & McDonald, 1982; Pavkovic, Glowinski, Feng & Brown, 1981). The aryl group at C(3) is in a perpendicular position with respect to the isoquinoline system.

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Structures of Four *cis* and *trans* Tricyclic Mono- and Dithioacetals

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Abstract. 1,10-*trans*-1,2-*trans*-2,7-*X*-Tricyclo[8.4.0.0^{2,7}]tetradecane: $X = \textit{trans}$ -6-thia-8-oxa, C₁₂H₂₀OS (I), $X = \textit{trans}$ -6-oxa-8-thia, C₁₂H₂₀OS (II), $X = \textit{trans}$ -6,8-dithia, C₁₂H₂₀S₂ (III), $X = \textit{cis}$ -6,8-dithia,

C₁₂H₂₀S₂ (IV). $M_r = 212.4$ (I) (II), 228.4 (III) (IV). Monoclinic, $P2_1/c$ (I) (II) (III); orthorhombic, $P2_12_1$ (IV). $Z = 4$, $a = 5.769$ (3), $b = 12.634$ (4), $c = 15.921$ (12) Å, $\beta = 94.61$ (5) $^\circ$, $V = 1156.6$ (8) Å³, final $R = 0.069$ for 1167 observed reflections (I); $a = 12.459$ (5), $b = 10.001$ (2), $c = 9.331$ (3) Å, $\beta =$

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