Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.052$
$wR(F^2) = 0.140$
$S = 1.043$
2797 reflections
213 parameters
H atoms constrained
$w = 1/[\sigma^2(F_o^2) + (0.0666P)^2 + 0.2075P]$
where $P = F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.22$ e Å^{-3}
$\Delta\rho_{\min} = -0.24$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

O1—C9	1.359 (3)	O5—C17	1.376 (3)
O1—C2	1.459 (3)	O5—C7	1.394 (2)
O2—C13	1.355 (3)	O6—C17	1.197 (3)
O2—C3	1.455 (3)	C2—C3	1.529 (3)
O3—C13	1.201 (3)	C3—C4	1.515 (3)
O4—C15	1.218 (3)	C4—C10	1.504 (3)
C9—O1—C2	118.48 (17)	O1—C2—C3	109.97 (18)
C13—O2—C3	116.12 (19)	C4—C3—C2	112.23 (19)
C17—O5—C7	118.13 (17)	C10—C4—C3	110.88 (19)
O1—C2—C12	104.53 (19)	O1—C9—C10	123.29 (19)
O1—C2—C11	108.16 (19)	C9—C10—C4	120.6 (2)
C9—O1—C2—C3	−40.6 (3)	O1—C9—C10—C4	0.2 (3)
O1—C2—C3—C4	57.2 (2)	C3—C4—C10—C9	16.7 (3)
C2—C3—C4—C10	−44.8 (3)		

The temperature of the crystal was controlled using an Oxford Cryosystems Cryostream Cooler (Cosier & Glazer, 1986). Data were collected over a hemisphere of reciprocal space, by a combination of three sets of exposures. Each set had a different φ angle for the crystal and each exposure of 10 s covered 0.3° in ω. The crystal-to-detector distance was 5.01 cm. Coverage of the unique set was over 88% complete to at least 25° in θ. The absence of crystal decay was established by repeating the initial frames at the end of the data collection and analysing the duplicate reflections. H atoms were added at calculated positions and refined using a riding model. Anisotropic displacement parameters were used for all non-H atoms; H atoms were given isotropic displacement parameters equal to 1.2 (1.5 for methyl H atoms) times the equivalent isotropic displacement parameter of the atom to which they are attached. S.u.'s on C—C distances do not exceed 0.004 Å. The bond-length distribution confirms the bond orders shown in the Scheme.

Data collection: *SMART* (Siemens, 1994*a*). Cell refinement: *SAINT* (Siemens, 1995). Data reduction: *SAINT*. Program(s) used to solve structure: *SHELXTL/PC* (Siemens, 1994*b*). Program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997). Molecular graphics: *SHELXTL/PC*. Software used to prepare material for publication: *SHELXTL/PC*.

We wish to acknowledge the use of the EPSRC's Chemical Database Service at Daresbury Laboratory (Fletcher *et al.*, 1996) for access to the Cambridge Structural Database (Allen & Kennard, 1993).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: BM1179). Services for accessing these data are described at the back of the journal.

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1-(3,4-Dimethoxy-α,β-dihydrocinnamoyl)pyrrole,† a Novel Amide from *Piper brachystachyum*

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Abstract

The isolation and structure of the title compound, $C_{15}H_{17}NO_3$, are described. The molecule is twisted so that the two ring systems are oriented at an angle of

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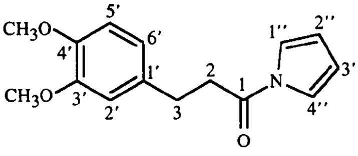
† Systematic name: 1-[3-(3,4-dimethoxyphenyl)propanoyl]pyrrole.

64.2 (1)° with respect to one another, and the *ortho*-methoxy groups, while being almost coplanar with the phenyl ring, point away from each other.

Comment

Piper species, widely distributed in the tropical and subtropical regions of the world, are used medicinally in various ways (Parmar *et al.*, 1997). This paper reports the isolation and structure of a novel amide, (I), extracted from *Piper brachystachyum*.



(I)

The molecular structure of the title compound, (I), is represented in Fig. 1. The bond lengths and angles are unexceptional. The two planar ring systems are oriented with a dihedral angle of 64.2 (1)°; this angle results from a succession of non-zero (or 180°) torsion angles involving the C1—C2—C3 backbone and the ring systems. The two methoxy groups are almost coplanar with the phenyl ring to which they are attached; the deviations from planarity are indicated by the values of the torsion angles C8′—O3—C4′—C3′ and C7′—O2—C3′—C4′ of 174.7 (2) and 176.0 (2)°, respectively. The methyl groups in these methoxy units point away from each other, presumably to reduce steric interactions. This conformation of the methoxy groups has been noted previously (Sharma *et al.*, 1997). No obvious short-range intermolecular contacts were observed. The phenyl rings pack one above the other, but as they are separated at a distance equal to the length of b [5.7516 (10) Å], π interactions can be discounted.

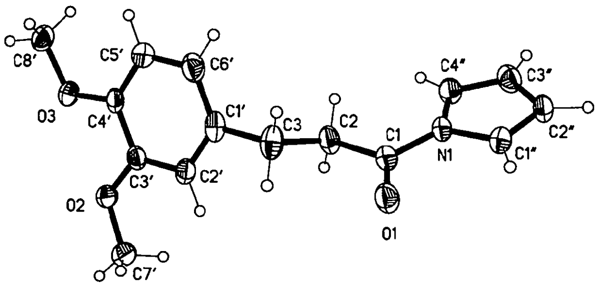


Fig. 1. View of the title molecule showing the atomic numbering. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small circles of arbitrary radii.

Experimental

The plant material was collected from the forests around Shillong (Meghalaya, India). Stems and leaves (2 kg) were dried and extracted with cold dichloromethane–methanol (1:1). The chloroform–petrol (3:2) eluates of the column chromatogram of the dried extract gave 200 mg of (I), which crystallized from petrol–chloroform as white needles; m.p. 381 K. Elemental analysis: found C 68.80, H 6.60, N 5.32%; $C_{15}H_{17}NO_3$ requires C 69.48, H 6.61, N 5.40%. IR ν_{max} (Nujol): 3150, 3100, 2860, 1700, 1610, 1600, 1520, 1325, 1300, 1260, 1220, 1155, 1020, 920, 850, 810, 795, 750 and 710 cm^{-1}. UV λ_{max} (MeOH): 285 nm. ^1H NMR ($CDCl_3$, 250 MHz): δ 3.03–3.10 (4H, *m*, H-2 and H-3), 3.83 (3H, *s*, $-OCH_3$), 3.84 (3H, *s*, $-OCH_3$), 6.27 (2H, *d*, J = 2.4 Hz, H-2″ and H-3″), 6.76–6.78 (3H, *m*, H-2′, H-5′ and H-6′), 7.29 p.p.m. (broad *s*, H-1″ and H-4″). ^{13}C NMR ($CDCl_3$, 62.9 MHz): δ 169.59 (>C=O), 148.90 (C-3′), 147.56 (C-4′), 132.70 (C-1′), 120.09 (C-6′), 118.79 (C-1″ and C-4″), 112.97 (C-2″ and C-3″), 111.74 (C-5′), 111.37 (C-2′), 55.78 ($-OCH_3$), 55.71 ($-OCH_3$), 36.47 (C-2), 29.91 (C-3). EIMS *m/z* (relative intensity): 259 (M^+) (70), 192 (32), 177 (7), 164 (30), 151 (100), 135 (4), 121 (5), 107 (10), 91 (8), 77 (9) and 67 (13).

Crystal data

$C_{15}H_{17}NO_3$
M_r = 259.30
Monoclinic
$P2_1/n$
a = 10.3731 (18) Å
b = 5.7516 (10) Å
c = 22.460 (4) Å
β = 101.992 (4)°
V = 1310.8 (4) $Å^3$
Z = 4
D_x = 1.314 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 3752 reflections
θ = 1.85–25.50°
μ = 0.092 mm^{-1}
T = 180 (2) K
Needle
0.48 × 0.35 × 0.20 mm
Colourless

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
Absorption correction: none
6472 measured reflections
2422 independent reflections

1858 reflections with $I > 2\sigma(I)$
R_{int} = 0.031
θ_{max} = 25.5°
$h = -12 \rightarrow 11$
$k = -6 \rightarrow 6$
$l = -27 \rightarrow 19$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.119
S = 1.023
2422 reflections
174 parameters
H atoms constrained
$w = 1/[\sigma^2(F_o^2) + (0.0606P)^2 + 0.3805P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max}$ = 0.005
$\Delta\rho_{max}$ = 0.22 e $Å^{-3}$
$\Delta\rho_{min}$ = −0.21 e $Å^{-3}$
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

O1—C1	1.211 (2)	N1—C4″	1.391 (2)
O2—C3′	1.372 (2)	N1—C1″	1.393 (2)
O2—C7′	1.426 (2)	C1″—C2″	1.350 (3)
O3—C4′	1.369 (2)	C2″—C3″	1.425 (3)
O3—C8′	1.428 (2)	C3″—C4″	1.352 (3)

C3′—O2—C7′	117.09 (14)	C4″—N1—C1″	108.00 (14)
C4′—O3—C8′	117.71 (14)		
C1″—N1—C1—O1	−1.9 (3)	C2—C3—C1′—C6′	−93.5 (2)
O1—C1—C2—C3	33.5 (3)	C7′—O2—C3′—C4′	−175.98 (15)
N1—C1—C2—C3	−146.33 (17)	C8′—O3—C4′—C3′	174.70 (15)
C1—C2—C3—C1′	−178.59 (17)		

The temperature of the crystal was controlled using an Oxford Cryosystems Cryostream Cooler (Cosier & Glazer, 1986). Data were collected over a hemisphere of reciprocal space, by a combination of three sets of exposures. Each set had a different φ angle for the crystal and each exposure of 10 s covered 0.3° in ω. The crystal to detector distance was 5.01 cm. Coverage of the unique set was over 99% complete to at least 25° in θ. The absence of crystal decay was monitored by repeating the initial frames at the end of the data collection and analysing the duplicate reflections. H atoms were added at calculated positions and refined using a riding model. Anisotropic displacement parameters were used for all non-H atoms; each H atom was given an isotropic displacement parameter equal to 1.2 (or 1.5 for methyl H atoms) times the equivalent isotropic displacement parameter of the atom to which it is attached.

Data collection: *SMART* (Siemens, 1994*a*). Cell refinement: *SAINT* (Siemens, 1995). Data reduction: *SAINT*. Program(s) used to solve structure: *SHELXTL/PC* (Siemens, 1994*b*). Program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997). Molecular graphics: *SHELXTL/PC*. Software used to prepare material for publication: *SHELXTL/PC*.

We wish to acknowledge the use of the EPSRC's Chemical Database Service at Daresbury Laboratory (Fletcher *et al.*, 1996) for access to the Cambridge Structural Database (Allen & Kennard, 1993). We also thank the Danish International Development Agency (DANIDA) and the Council for Scientific and Industrial Research (CSIR, New Delhi, India) for financial assistance.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: BM1181). Services for accessing these data are described at the back of the journal.

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1,4-Bis[3-(*N*-cyclohexyliminomethyl)-2-hydroxy-5-methylbenzyl]piperazine

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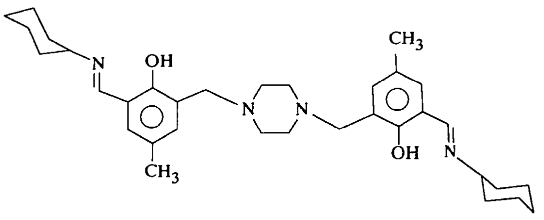
(*Received 20 October 1997; accepted 17 November 1997*)

Abstract

Molecules of the title molecule, $C_{34}H_{48}N_4O_2$, lie across crystallographic inversion centres. The piperazine and cyclohexyl rings adopt chair conformations and the hydroxyl group participates in an O—H···N intramolecular hydrogen bond.

Comment

The impetus for the study of binucleating ligands and their copper complexes has come mainly from three areas, *i.e.* homogeneous catalysis, as model systems for the study of mechanisms of magnetic exchange, and as speculative models for the copper active site in oxyhaemocyanin (Fenton *et al.*, 1982) and several metalloproteins (Kurtz, 1990). Many compounds of this type have more than one metal present. X-ray crystal structure analysis is useful for investigating the coordination-site change and transmetallation reactions (Casellato *et al.*, 1986).



(I)

Molecules of the title compound, (I), lie across crystallographic inversion centres and the asymmetric unit therefore contains one-half of a molecule. The N2 atom is in a pyramidal configuration. The bond lengths and

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