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## Key indicators

Single-crystal X-ray study  
 $T = 293$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å  
 $R$  factor = 0.044  
 $wR$  factor = 0.107  
Data-to-parameter ratio = 17.2For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.6-Chloro-2,3,4,5-tetrahydro-7,8-dimethoxy-  
1-(4-methoxyphenyl)-1*H*-3-benzazepine

The title compound,  $\text{C}_{19}\text{H}_{22}\text{NClO}_3$ , was synthesized by an intramolecular condensation reaction of *N*-[2-hydroxy-2-(methoxyphenyl)ethyl]-2-(2-chloro-3,4-dimethoxyphenyl)-ethylamine in trifluoroacetic acid with 36 *N*  $\text{H}_2\text{SO}_4$  at room temperature and obtained in 69% yield. The crystal structure determined by X-ray diffraction shows normal bond lengths and angles. The seven-membered ring adopts a half-chair conformation.

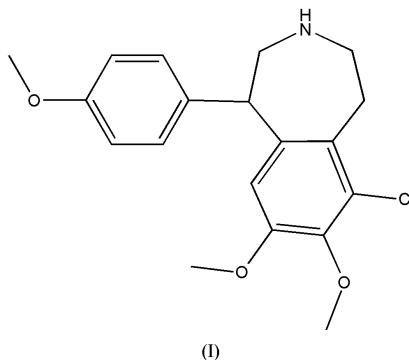
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## Comment

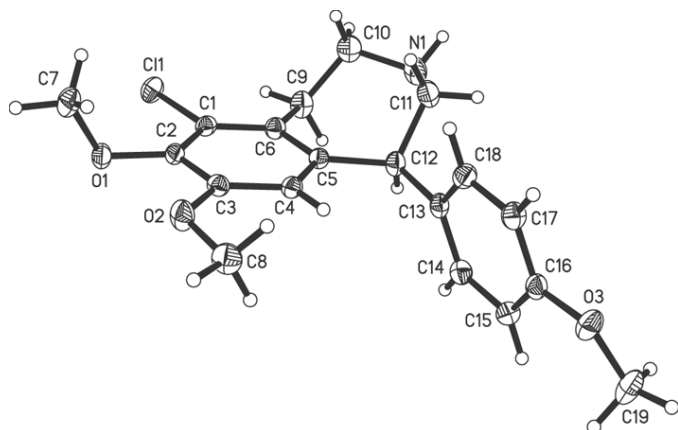
The title compound, (I) (Fig. 1), is an important pharmaceutical intermediate: removal of the methoxy groups with  $\text{BBr}_3$  gives fenoldopam. Fenoldopam is a renal vasodilator and useful in treating hypertension and in renal ischemia. Fenoldopam is also a good agent for studying D-1 receptors and the consequences of their stimulation in the periphery of the kidneys (McCarthy *et al.*, 1986; Weinstock *et al.*, 1980, 1986).



All bond lengths and angles in (I) are normal (Table 1). The C—C bond distances and C—C—C angles in the benzene rings are in the ranges 1.379 (2)–1.403 (2) Å and 117.04 (15)–123.32 (15)°, respectively. The two benzene rings make a dihedral angle of 103.8 (2)°. In the seven-membered ring, the interatomic distances of 1.456 (3) Å for N1—C10 and 1.464 (3) Å for N1—C11 reveal their single-bond character. The seven-membered ring adopts a half-chair conformation: atoms C5, C6, C9 and C12 are coplanar, while atoms C10, C11 and N1 deviate from this plane by 1.220 (3), 1.252 (3) and 1.133 (3) Å, respectively.

## Experimental

A solution of *N*-[2-hydroxy-2-(methoxyphenyl)ethyl]-2-(2-chloro-3,4-dimethoxyphenyl)ethylamine (78.7 g) in trifluoroacetic acid (590 ml) was treated at 298 K with 36 *N*  $\text{H}_2\text{SO}_4$  (17.9 ml) and then



**Figure 1**

View of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

stirred for 3.5 h at 298 K. Anhydrous NaOAc (79.3 g) was added, which raised the pot temperature to 333 K. The reaction mixture was concentrated at less than 328 K under vacuum, and the residue was diluted with water and made basic with 14 *N* aqueous ammonia with cooling. The mixture was extracted with  $\text{CH}_2\text{Cl}_2$  and the organic layer was dried over  $\text{MgSO}_4$  and concentrated under vacuum to give a yellow solid. Recrystallization from EtOAc and washing the product with diethyl ether gave 51.7 g (69%) of crystals. Suitable crystals were obtained by evaporation of an ethanol solution (m.p. 414–415 K). IR (KBr,  $\nu \text{ cm}^{-1}$ ): 3348, 2931, 2819, 1607, 1562, 1511, 1485, 1301, 1249, 1096, 1040, 833, 786;  $^1\text{H NMR}$  ( $\text{CDCl}_3$ ,  $\delta$ , p.p.m.): 7.05 (*d*, 2H,  $J = 8.4$  Hz), 6.89 (*d*, 2H,  $J = 8.4$  Hz), 6.38 (*s*, 1H), 4.22 (*d*, 1H,  $J = 5.7$  Hz), 3.84–3.69 (*m*, 3H), 3.43 (*dd*, 1H), 3.33 (*dd*, 1H,  $J = 2.2, 13.6$  Hz), 3.12 (*m*, 1H), 3.10 (*m*, 1H), 2.93 (*d*, 2H,  $J = 4.0$  Hz), 1.94 (*s*, 1H); analysis calculated for  $\text{C}_{19}\text{H}_{22}\text{ClNO}_3$ : C 65.61, H 6.38, N 4.03%; found: C 65.49, H 6.46, N 4.08%.

#### Crystal data

$\text{C}_{19}\text{H}_{22}\text{ClNO}_3$	$Z = 2$
$M_r = 347.83$	$D_x = 1.345 \text{ Mg m}^{-3}$
Triclinic, $P\bar{1}$	Mo $K\alpha$ radiation
$a = 9.1365$ (2) Å	Cell parameters from 2832 reflections
$b = 9.3759$ (2) Å	$\theta = 2.2\text{--}27.4^\circ$
$c = 10.9936$ (3) Å	$\mu = 0.24 \text{ mm}^{-1}$
$\alpha = 113.675$ (1) $^\circ$	$T = 293$ (2) K
$\beta = 92.350$ (1) $^\circ$	Prism, colorless
$\gamma = 93.164$ (2) $^\circ$	$0.22 \times 0.12 \times 0.11 \text{ mm}$
$V = 859.11$ (4) Å $^3$	

#### Data collection

Rigaku R-AXIS RAPID diffractometer	3806 independent reflections
$\omega$ scans	2804 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (ABSCOR; Higashi, 1995)	$R_{\text{int}} = 0.025$
$T_{\text{min}} = 0.966$ , $T_{\text{max}} = 0.974$	$\theta_{\text{max}} = 27.5^\circ$
6003 measured reflections	$h = -11 \rightarrow 11$
	$k = -12 \rightarrow 12$
	$l = -14 \rightarrow 14$

#### Refinement

Refinement on $F^2$	$w = 1/[\sigma^2(F_o^2) + (0.0475P)^2 + 0.2704P]$
$R[F^2 > 2\sigma(F^2)] = 0.044$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.107$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.01$	$\Delta\rho_{\text{max}} = 0.33 \text{ e \AA}^{-3}$
3806 reflections	$\Delta\rho_{\text{min}} = -0.21 \text{ e \AA}^{-3}$
221 parameters	
H atoms treated by a mixture of independent and constrained refinement	

**Table 1**

Selected geometric parameters (Å,  $^\circ$ ).

C11—C1	1.7473 (17)	C4—C5	1.391 (2)
O1—C2	1.3767 (19)	C5—C6	1.403 (2)
O1—C7	1.426 (2)	C5—C12	1.531 (2)
O2—C3	1.367 (2)	C6—C9	1.515 (2)
O2—C8	1.417 (2)	C9—C10	1.517 (3)
O3—C16	1.375 (2)	C11—C12	1.534 (3)
O3—C19	1.423 (3)	C16—C17	1.388 (3)
N1—C10	1.456 (3)	C17—C18	1.379 (2)
N1—C11	1.464 (3)	C12—C13	1.517 (2)
C1—C2	1.383 (2)	C13—C14	1.383 (3)
C1—C6	1.395 (2)	C13—C18	1.401 (3)
C2—C3	1.388 (2)	C14—C15	1.390 (3)
C3—C4	1.387 (2)	C15—C16	1.383 (3)
C2—O1—C7	114.19 (14)	C1—C6—C9	121.80 (15)
C3—O2—C8	117.56 (14)	C5—C6—C9	121.10 (15)
C16—O3—C19	117.74 (17)	C6—C9—C10	115.07 (17)
C10—N1—C11	114.27 (16)	N1—C10—C9	111.64 (17)
C2—C1—C6	123.32 (15)	N1—C11—C12	113.19 (17)
C2—C1—C11	115.82 (13)	C13—C12—C5	113.69 (14)
C6—C1—C11	120.85 (14)	C13—C12—C11	109.46 (15)
O1—C2—C1	120.85 (15)	C5—C12—C11	112.44 (15)
O1—C2—C3	120.42 (15)	C14—C13—C18	117.71 (16)
C1—C2—C3	118.65 (15)	C14—C13—C12	120.84 (17)
O2—C3—C4	124.73 (15)	C18—C13—C12	121.37 (17)
O2—C3—C2	115.79 (15)	C13—C14—C15	121.90 (18)
C4—C3—C2	119.48 (16)	O3—C16—C15	124.66 (17)
C3—C4—C5	121.41 (15)	O3—C16—C17	115.13 (17)
C4—C5—C6	120.05 (15)	C15—C16—C17	120.20 (16)
C4—C5—C12	120.12 (14)	C18—C17—C16	119.82 (18)
C6—C5—C12	119.79 (15)	C17—C18—C13	121.19 (17)
C1—C6—C5	117.04 (16)	C16—C15—C14	119.16 (17)

All H atoms were initially located in a difference Fourier map. The methyl H atoms were then constrained to an ideal geometry with C—H distances of 0.96 Å and  $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ , but each group was allowed to rotate freely about its C—C bond. The position of the amine H atom was refined freely along with an isotropic displacement parameter. All other H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H distances of 0.93 Å and  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ .

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSK, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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