F58	0.0835 (7)	0.0979 (3)	0.2279 (8)	0.089 (5)
F59	0.1992 (7)	0.0321 (2)	0.2988 (7)	0.085 (4)
F60	0.0996 (7)	0.0327 (3)	0.0763 (7)	0.102 (5)
C61	0.393 (1)	0.1494 (3)	0.302 (1)	0.038 (5)
O62	0.3176 (6)	0.1301 (2)	0.4174 (7)	0.048 (4)
C63	0.313 (1)	0.1549 (4)	0.555 (1)	0.051 (6)
O64	0.3790 (8)	0.1892 (3)	0.5978 (7)	0.071 (5)
C65	0.212 (1)	0.1364 (5)	0.653 (1)	0.058 (5)
C66	0.163 (1)	0.1661 (4)	0.771 (1)	0.072 (7)
C67	0.060 (2)	0.1519 (6)	0.860 (1)	0.09 (1)
C68	0.008 (1)	0.1081 (7)	0.826 (2)	0.09 (1)
C69	0.055 (1)	0.0743 (5)	0.711 (2)	0.083 (8)
C70	0.157 (1)	0.0920 (5)	0.622 (1)	0.069 (7)
C71	0.313 (1)	0.1964 (3)	0.239 (1)	0.035 (5)
C72	0.358 (1)	0.2158 (4)	0.113 (1)	0.052 (6)
C73	0.288 (1)	0.2576 (5)	0.053 (1)	0.064 (7)
C74	0.171 (1)	0.2787 (4)	0.118 (2)	0.070 (8)
C75	0.125 (1)	0.2604 (5)	0.243 (1)	0.066 (7)
C76	0.193 (1)	0.2188 (4)	0.303 (1)	0.048 (6)

Table 4. *Selected geometric parameters* (Å, °) *for* (3)

C3—C21	1.52 (1)	C45—C61	1.53 (1)
C15—C21	1.53 (1)	C53—C61	1.52 (1)
C21—O22	1.48 (1)	C61—O62	1.44 (1)
C21—C31	1.53 (1)	C61—C71	1.55 (1)
C3—C21—C15	109.5 (8)	C45—C61—C53	108.6 (8)
C3—C21—O22	102.6 (7)	C45—C61—O62	110.7 (7)
C3—C21—C31	106.2 (8)	C45—C61—C71	114.8 (8)
C15—C21—O22	109.3 (7)	C53—C61—O62	105.5 (8)
C15—C21—C31	116.4 (7)	C53—C61—C71	106.0 (7)
O22—C21—C31	112.0 (7)	O62—C61—C71	110.6 (8)

Data collection and cell refinement: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1989). Data reduction: *TEXSAN PROCESS* (Molecular Structure Corporation, 1985). Program(s) used to solve structure: *TEXSAN*; *MITHRIL* (Gilmore, 1984). Program(s) used to refine structure: *TEXSAN LS*. Molecular graphics: *TEXSAN*; *ORTEPII* (Johnson, 1976). Software used to prepare material for publication: *TEXSAN FINISH*. Literature survey: *CSSR* (1984).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: LI1106). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure Analysis of a Reversible Monoamine Oxidase A Inhibitor: 3-{4-[(*R*)-3-Hydroxybutoxy]phenyl}-(*R*)-5-methoxymethyl-1,3-oxazolidin-2-one

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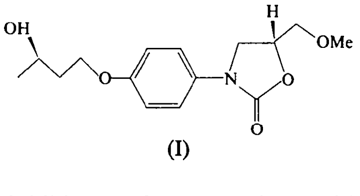
Abstract

The structure of a new reversible type-A MAO inhibitor and analogue of befloxatone is reported. Within experimental deviations, the oxazolidinone moiety of the title compound is planar and quasi coplanar with the adjacent phenyl ring [12.2 (5)°]. The sum of the angles at the N atom is 359.8°, indicating sp^2 hybridization of this atom. This is consistent with electron delocalization between the N atom and the carbonyl group [N—(C═O) 1.365 (4) Å] in the oxazolidinone ring. The lateral butoxy chain is all *trans*. As a result, the molecule is very flat with only the (*R*)-5-methoxymethyl chain and the terminal hydroxyl group out of the plane. The hydroxyl group is involved in the hydrogen bonding responsible for crystal cohesion.

Comment

In the course of our study of monoamine oxidase (MAO) inhibitors (Moureau *et al.*, 1992; Wouters, Perpete, Norberg, Evrard & Durant, 1994), we report here the X-ray structure of the title compound (I), a new reversible type-A MAO inhibitor and analogue of befloxatone (Koenig *et al.*, 1992).



(I)

Original inhibitors of monoamine oxidase (MAO) have been developed by Delalande Research (Groupe Synthelabo) in the aryloxazolidinone series. They belong to the (*R*)-5-methoxymethyl-3-aryloxazolidin-2-one family, are selective for the A form of the enzyme, are reversible and competitive against tyramine (Koenig *et al.*, 1992). Structure–activity results clearly revealed the crucial role that a hydroxybutoxy lateral chain has

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on MAO affinity and selectivity. Therefore, the crystal structure analysis of the title compound was undertaken to ascertain the conformation of the lateral butoxy chain and to examine the forces responsible for the crystal-packing cohesion in order to establish which functional groups of the aryloxazolidinone could interact with the MAO active site. The main structural features are identical to those observed for other aryloxazolidinones (Wouters *et al.*, 1993). A view of the molecule is presented in Fig. 1.

Within experimental deviations, the oxazolidinone moiety is planar and quasi coplanar with the adjacent phenyl ring (Table 2), leading to a distance between O1 and H10 of 2.24 Å, which is smaller than the van der Waals contact (2.55 Å). The angles at the N8 atom sum to 359.8°, indicating sp^2 character. This is consistent with electron delocalization between the N atom and the carbonyl functional group [N—(C=O) 1.365 (4) Å] in the oxazolidinone ring. The lateral butoxy chain adopts an all-*trans* conformation (Table 2) extending the planar phenyl-oxazolidinone entity. As a result, the molecule is very flat, with only the (*R*)-5-methoxymethyl chain and the terminal hydroxyl group out of the plane. The hydroxyl group is involved in intermolecular hydrogen bonding (Table 3) responsible for crystal cohesion (Fig. 2) and could lead to intermolecular hydrogen bonds with an appropriate residue of the active site of MAO (Wouters *et al.*, 1994). Relative positioning of this essential functional group is currently under investigation among several befloxatone analogues and would lead to a better understanding of the stereochemical requirements at the MAO active site.

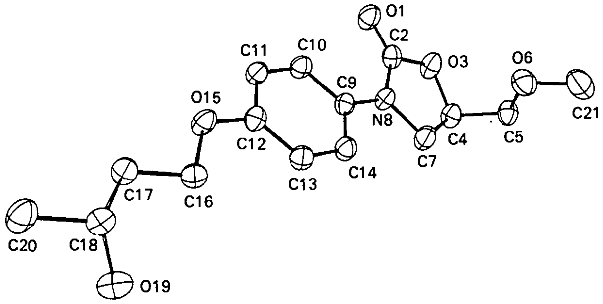

Fig. 1. View of $C_{15}H_{21}NO_5$ showing the labelling of the non-H atoms. Displacement ellipsoids are shown at 50% probability levels.

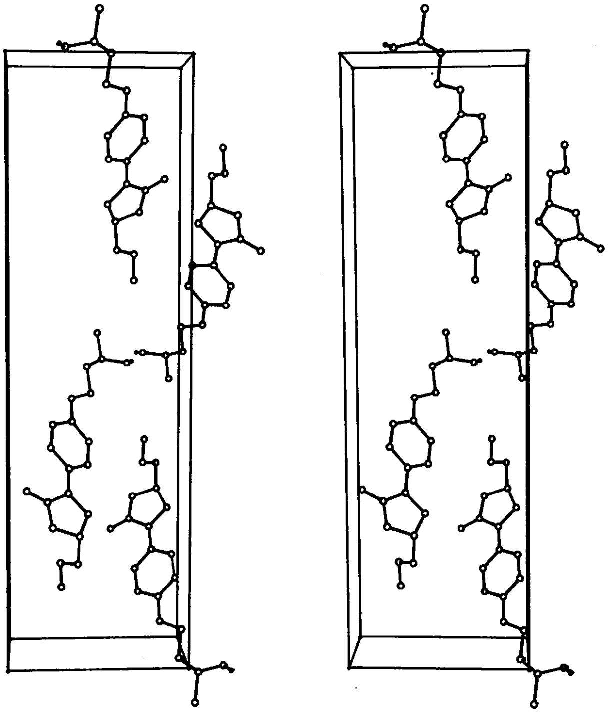

Fig. 2. Stereoview of the molecular conformation and crystal packing of the title compound.

Experimental

Crystal data

$C_{15}H_{21}NO_5$
$M_r = 295.34$
Orthorhombic
$P2_12_12_1$
$a = 9.387$ (2) Å
$b = 31.005$ (8) Å
$c = 5.1952$ (10) Å
$V = 1512.1$ (6) Å^3
$Z = 4$
$D_x = 1.297$ Mg m^{-3}

Cu $K\alpha$ radiation
$\lambda = 1.54178$ Å
Cell parameters from 25 reflections
$\theta = 11$–$30°$
$\mu = 0.77$ mm^{-1}
$T = 173$ K
Prism
0.38 × 0.11 × 0.09 mm
Colourless

Data collection

CAD-4 Enraf–Nonius diffractometer
$\theta/2\theta$ scans
2738 measured reflections
2229 independent reflections
2109 observed reflections [$I > 2.5\sigma(I)$]
$R_{\text{int}} = 0.0357$

$\theta_{\text{max}} = 72°$
$h = 0 \rightarrow 11$
$k = -32 \rightarrow 38$
$l = 0 \rightarrow 6$
3 standard reflections frequency: 60 min intensity variation: 1%

Refinement

Refinement on F
$R = 0.0528$
$wR = 0.0664$
$S = 1.95$
2109 reflections
253 parameters
Calculated weights $w = 1/[\sigma^2(F_o) + 0.002F^2]$
$(\Delta/\sigma)_{\text{max}} = 0.167$

$\Delta\rho_{\text{max}} = 0.288$ e Å^{-3}
$\Delta\rho_{\text{min}} = -0.344$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{\text{eq}} = (1/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
O1	0.8943 (2)	0.80950 (10)	−0.7816 (5)	0.0434 (6)
O3	0.7562 (2)	0.75410 (10)	−0.8952 (5)	0.0396 (6)
O6	0.7119 (2)	0.68370 (10)	−0.5640 (5)	0.0461 (7)

O15	0.6413 (2)	0.93930 (10)	0.0747 (4)	0.0331 (6)
O19	0.3091 (2)	0.99390 (10)	0.5199 (5)	0.0382 (6)
N8	0.6712 (2)	0.79920 (10)	−0.5981 (5)	0.0298 (6)
C2	0.7835 (3)	0.79050 (10)	−0.7568 (6)	0.0325 (7)
C4	0.6137 (3)	0.73810 (10)	−0.8353 (6)	0.0360 (8)
C5	0.6217 (4)	0.69050 (10)	−0.7765 (7)	0.0380 (8)
C7	0.5628 (3)	0.76570 (10)	−0.6090 (7)	0.0396 (9)
C9	0.6640 (3)	0.83420 (10)	−0.4253 (6)	0.0280 (7)
C10	0.7622 (3)	0.86850 (10)	−0.4344 (6)	0.0312 (7)
C11	0.7506 (3)	0.90240 (10)	−0.2632 (6)	0.0306 (7)
C12	0.6425 (3)	0.90340 (10)	−0.0802 (6)	0.0290 (7)
C13	0.5454 (3)	0.86980 (10)	−0.0710 (6)	0.0325 (8)
C14	0.5564 (3)	0.83560 (10)	−0.2424 (6)	0.0319 (8)
C16	0.5271 (3)	0.94480 (10)	0.2533 (6)	0.0293 (7)
C17	0.5473 (3)	0.98980 (10)	0.3631 (6)	0.0317 (8)
C18	0.4551 (3)	1.00020 (10)	0.5934 (6)	0.0304 (7)
C20	0.4781 (4)	1.04590 (10)	0.6872 (7)	0.0425 (10)
C21	0.7069 (5)	0.64030 (10)	−0.4822 (10)	0.0580 (14)

Table 2. *Selected geometric parameters* (Å, °)

O1—C2	1.202 (4)	C4—C5	1.509 (4)
O3—C2	1.362 (4)	C4—C7	1.531 (5)
O3—C4	1.460 (4)	C9—C10	1.408 (4)
O6—C5	1.407 (4)	C9—C14	1.387 (4)
O6—C21	1.412 (5)	C10—C11	1.381 (4)
O15—C12	1.374 (4)	C11—C12	1.391 (4)
O15—C16	1.428 (4)	C12—C13	1.385 (4)
O19—C18	1.436 (3)	C13—C14	1.389 (4)
N8—C2	1.365 (4)	C16—C17	1.519 (4)
N8—C7	1.455 (4)	C17—C18	1.511 (4)
N8—C9	1.410 (4)	C18—C20	1.514 (4)
C2—O3—C4	110.0 (2)	N8—C9—C14	119.7 (3)
C5—O6—C21	111.0 (3)	C10—C9—C14	118.4 (3)
C12—O15—C16	118.9 (2)	C9—C10—C11	120.1 (3)
C2—N8—C7	112.1 (3)	C10—C11—C12	121.0 (3)
C2—N8—C9	125.0 (2)	O15—C12—C11	115.1 (3)
C7—N8—C9	122.7 (2)	O15—C12—C13	125.7 (3)
O1—C2—O3	120.8 (3)	C11—C12—C13	119.1 (3)
O1—C2—N8	129.5 (3)	C12—C13—C14	120.2 (3)
O3—C2—N8	109.7 (2)	C9—C14—C13	121.1 (3)
O3—C4—C5	109.3 (3)	O15—C16—C17	105.1 (2)
O3—C4—C7	105.1 (2)	C16—C17—C18	114.9 (2)
C5—C4—C7	114.0 (3)	O19—C18—C17	107.9 (2)
O6—C5—C4	109.6 (3)	O19—C18—C20	110.4 (3)
N8—C7—C4	102.2 (2)	C17—C18—C20	111.9 (3)
N8—C9—C10	121.9 (3)		
C4—O3—C2—O1	−178.4 (3)	C5—C4—C7—N8	129.6 (3)
C4—O3—C2—N8	2.7 (3)	O3—C4—C5—O6	60.1 (3)
C2—O3—C4—C5	−130.9 (3)	C7—C4—C5—O6	−57.1 (3)
C2—O3—C4—C7	−8.2 (3)	O3—C4—C7—N8	10.1 (3)
C21—O6—C5—C4	172.1 (3)	N8—C9—C10—C11	179.4 (3)
C16—O15—C12—C11	174.8 (3)	C14—C9—C10—C11	0.3 (5)
C16—O15—C12—C13	−3.8 (4)	N8—C9—C14—C13	−179.4 (3)
C12—O15—C16—C17	−172.2 (2)	C10—C9—C14—C13	−0.3 (5)
C7—N8—C2—O1	−174.3 (3)	C9—C10—C11—C12	−0.1 (6)
C9—N8—C2—O1	0.4 (5)	C10—C11—C12—O15	−178.7 (3)
C7—N8—C2—O3	4.5 (4)	C10—C11—C12—C13	−0.1 (4)
C9—N8—C2—O3	179.2 (3)	O15—C12—C13—C14	178.6 (3)
C7—N8—C9—C10	−173.7 (3)	C11—C12—C13—C14	0.1 (5)
C2—N8—C9—C14	−168.7 (3)	C12—C13—C14—C9	0.1 (6)
C7—N8—C9—C14	5.4 (4)	O15—C16—C17—C18	−169.2 (2)
C2—N8—C9—C10	12.2 (5)	C16—C17—C18—O19	−56.7 (3)
C2—N8—C7—C4	−9.2 (3)	C16—C17—C18—C20	−178.4 (3)
C9—N8—C7—C4	176.0 (3)		

Table 3. *Hydrogen-bonding geometry* (Å, °)

D—H···*A*	H···*A*	*D*—H···*A*
O19—H19···O19^{i}	2.850 (4)	174.0 (50)

Symmetry code: (i) $\frac{1}{2} - x, 2 - y, \frac{1}{2} + z$.

Most calculations were performed with *SHELX*76 (Sheldrick, 1976), the structure was solved using *SHELXS*86 (Sheldrick, 1985) and *PLATON* (Spek, 1990) was used for the generation of the CIF.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: DU1076). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1994). C**50**, 1492–1494

(*Z*,*Z*)-5-Anilino-1,1,1,6,6,6-hexafluoro-3,4-diazahexa-2,4-diene-2-amine

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Abstract

The title molecule, $C_{10}H_8F_6N_4$, has been characterized crystallographically, thus establishing its isomeric form. This form has a C═N—N═C substructure with a torsion angle of 145 (1)° about the N—N bond. There is an extensive hydrogen-bonding system, such that each