organic papers

2-[1-(2-Hydroxypropyliminio)ethyl]-5-methoxy-phenolate

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The title Schiff base compound, $C_{12}H_{17}NO_3$, synthesized by the reaction of paeonol and isopropanolamine in absolute methanol, crystallizes in a zwitterionic form. All non-H atoms, except for the 2-hydroxypropyl group, are approximately coplanar. The molecules are linked to each other by intermolecular hydrogen bonds.

Comment

Paeonol, 2-hydroxyl-4-methoxyacetophenone, is one of the main components of Moutan cortex. It has been reported to possess analgesic, sedative, anti-allergic, anti-oxidative, anti-inflammatory and antimicrobial properties (Chung, 1999; Papandreou *et al.*, 2002; Kim *et al.*, 2004). However, its insolubility in water and volatility impair its efficiency. Modifications to paeonol have been an active area of research. Here, we present the structure of the title compound, (I).
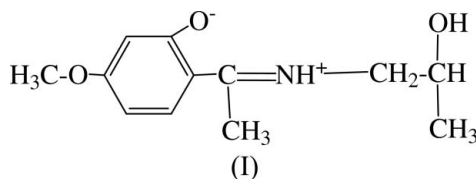
The molecular structure of (I) is shown in Fig. 1 and selected geometrical parameters are given in Table 1. The molecule is in a zwitterionic form with a strong intramolecular N—H⋯O hydrogen bond (Table 2). The C7=N bond distance is typical for a C=N double bond. The C1—O1 and C3—O2 bonds are shorter than the C12—O2 and C10—O3 bonds. This may be due to the effect of hyperconjugation. Atoms C10, C11 and O3 deviate from the mean plane formed by the rest of the non-H atoms in the molecule by −0.3880, −0.3727 and 0.5028 Å, respectively. The molecules are linked
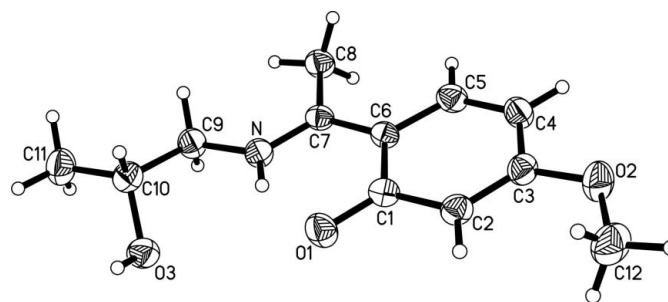

Figure 1
The molecular structure of (I), with 30% probability displacement ellipsoids. The disordered atoms H8D/H8E/H8F have been omitted for clarity.
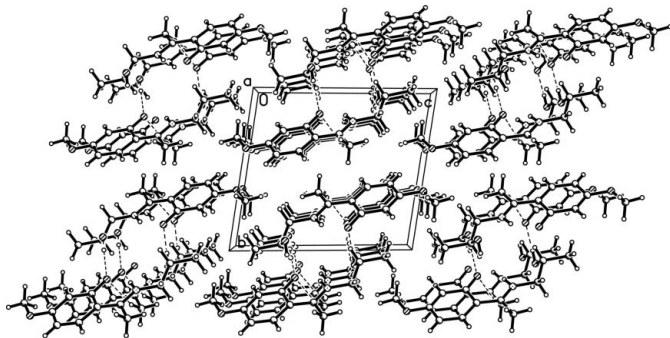
Figure 2
The crystal packing of (I). Dashed lines indicate hydrogen bonds.

to each other by O—H···O hydrogen bonds (Fig. 2 and Table 2).

Experimental

To a stirred absolute methanol solution (25 ml) of paeonol (0.83 g, 5 mmol) was added dropwise an absolute methanol solution (10 ml) of ethanolamine (0.38 g, 5.0 mmol) at room temperature. After stirring for 2 h at 320 K, the precipitate was filtered off, washed with methanol and dried *in vacuo*. Single crystals of (I) were obtained by slow evaporation of the resulting filtrate after 6 d.

Crystal data

$C_{12}H_{17}NO_3$	$V = 588.8$ (3) Å^3
$M_r = 223.27$	$Z = 2$
Triclinic, $P\bar{1}$	$D_x = 1.259$ Mg m^{-3}
$a = 5.714$ (2) Å	Mo $K\alpha$ radiation
$b = 9.672$ (4) Å	$\mu = 0.09$ mm^{-1}
$c = 10.869$ (3) Å	$T = 296$ (2) K
$\alpha = 98.19$ (2)°	Block, yellow
$\beta = 96.71$ (2)°	$0.54 \times 0.46 \times 0.12$ mm
$\gamma = 93.35$ (3)°	

Data collection

Siemens P4 diffractometer	$R_{int} = 0.017$
ω scans	$\theta_{max} = 25.5°$
Absorption correction: none	3 standard reflections
2506 measured reflections	every 97 reflections
2187 independent reflections	intensity decay: 3.9%
1070 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0268P)^2]$
$R[F^2 > 2\sigma(F^2)] = 0.041$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.081$	$(\Delta/\sigma)_{max} < 0.001$
$S = 0.99$	$\Delta\rho_{max} = 0.15$ e Å^{-3}
2187 reflections	$\Delta\rho_{min} = -0.15$ e Å^{-3}
155 parameters	Extinction correction: *SHELXL97*
H atoms treated by a mixture of independent and constrained refinement	Extinction coefficient: 0.013 (2)

Table 1
Selected bond lengths (Å).

O1—C1	1.290 (2)	C2—C3	1.354 (3)
O2—C3	1.365 (2)	C3—C4	1.400 (3)
O2—C12	1.434 (2)	C4—C5	1.358 (2)
O3—C10	1.417 (2)	C5—C6	1.406 (3)
N—C7	1.307 (2)	C6—C7	1.432 (2)
N—C9	1.463 (2)	C7—C8	1.494 (3)
C1—C2	1.428 (2)	C9—C10	1.503 (3)
C1—C6	1.440 (2)	C10—C11	1.517 (3)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3O···O1[i]	0.83 (1)	1.87 (2)	2.698 (2)	178 (3)
N—H0N···O1	0.87 (1)	1.75 (2)	2.525 (2)	147 (3)

Symmetry code: (i) $-x, -y, -z + 1$.

H3O and H0N were located in difference Fourier maps and were refined. All other H atoms were positioned geometrically and treated as riding on their parent atoms, with aromatic C—H distances of 0.93 Å, methyl C—H distances of 0.96 Å and methylene C—H distances of 0.97 Å. The U_{iso}(H) values were set at $1.2U_{eq}$(C). Atoms H8*A*/H8*B*/H8*C*/H8*D*/H8*E*/H8*F* were found to be disordered, and the site-occupancy factors of these atoms were refined to 0.5.

Data collection: *XSCANS* (Siemens, 1994); cell refinement: *XSCANS*; data reduction: *SHELXTL* (Siemens, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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