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Methyl 3-*para*-anisyl-4-(2-hydroxybenzoyl)isoxazole-5-carboxylate

Bouchra Filali Baba,[a] Abdelali Kerbal,[a] Najib Bitit,[a] Brahim El-Bali,[b] Jean Escudie,[c] Henry Ranaivonjatovo[c] and Michael Bolte[d]*

[a]Laboratoire de Chimie Organique, Faculté des Sciences, Dhar El Mahraz, Université Sidi Mohammed Ben Abdelah, 1796 Atlas, Fès, Morocco, [b]Départment de Chimie, Faculté des Sciences Dhar El Mehraz, Fès, Morocco, [c]Laboratoire de l'Hétérochimie Fondamentale et Appliquée, UPRES Associé au CNRS No. 5069, Université P. Sabatier, 118 Route de Narbonne, 31062 Toulouse, France, and [d]Institut für Organische Chemie, J.-W.-Goethe-Universität Frankfurt, Marie-Curie-Strasse 11, 60439 Frankfurt/Main, Germany
Correspondence e-mail: bolte@chemie.uni-frankfurt.de

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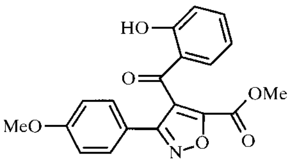
The title compound, $C_{19}H_{15}NO_6$, contains a planar isoxazole ring. An intramolecular hydrogen bond is formed between the OH group attached to a phenyl ring and a carbonyl O atom.

Comment

In one of our research fields, the dipole–dipolarophile approach has been investigated. In the case of dipolar 1,3-cycloadditions, we have studied the reaction of diarylnitrilimines with dipolarophile derivatives of lindanone (Kerbal *et al.*, 1988), titralone (Theobald *et al.*, 1990) or lisochromanone (Kerbal *et al.*, 1990). Recently, our research has been focused on the reaction of arylnitriloxides with isoquinoline arylidines (Badri *et al.*, 1999). We are trying to extend this research to the behaviour of new heterocyclic dipolarophiles, particularly that of esters of 4-oxo-4*H*-1-benzopyrancarboxylate with arylnitriloxides. However, the isoxazolinic cycloadducts obtained so far seem to transform to more stable isoxazole products due to the opening of the pyronic cycle. Since NMR spectroscopy did not provide sufficient information about the regiochemistry of the reaction product we have carried out the X-ray structure analysis of the title compound, (I). The isoxazole ring of the title compound is planar (r.m.s. deviation = 0.004 Å) with the methoxycarbonyl moiety coplanar to it. The phenyl ring at C3 encloses an angle of 35.55 (3)° with the isoxazole ring. The other phenyl ring is nearly perpendicular [77.44 (5)°] to the isoxazole ring. An intramolecular hydrogen bond is formed between the hydroxyl group attached to a phenyl ring and a carbonyl O atom, so that a new six-membered ring is formed. Furthermore, there is a short intermolecular contact between the ester carbonyl O atom (O51) and the hydroxyl group.



(I)

Experimental

4-Oxo-4*H*-1-benzopyran-2-carboxylate (9.8 mmol, 2 g) methyl ester and hydroxycarbamoyl chloride (12 mmol) were mixed in anhydrous benzene (30 ml). The mixture was stirred, with regular addition of triethylamine (4 ml), for 15 min. After 2 h, the triethylamine chlorohydrate was eliminated and the solvent evaporated. The residue was then recrystallized from methanol.

Crystal data

$C_{19}H_{15}NO_6$
$M_r = 353.32$
Monoclinic, $P2_1/n$
$a = 11.172$ (1) Å
$b = 9.337$ (1) Å
$c = 15.634$ (1) Å
$\beta = 94.10$ (1)°
$V = 1626.7$ (2) Å^3
$Z = 4$
$D_x = 1.443$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 8192 reflections
$\theta = 1$–25°
$\mu = 0.109$ mm^{-1}
$T = 173$ (2) K
Block, light yellow
0.60 × 0.50 × 0.30 mm

Data collection

Siemens CCD three-circle diffractometer
ω scans
Absorption correction: empirical (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.938$, $T_{\max} = 0.968$
32 334 measured reflections
4240 independent reflections
3444 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.030$
$\theta_{\max} = 29.57°$
$h = -15 \rightarrow 15$
$k = -12 \rightarrow 12$
$l = -21 \rightarrow 21$
400 standard reflections
frequency: 960 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.105$
$S = 1.024$
4240 reflections
239 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0507P)^2 + 0.6770P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.62$ e Å^{-3}
$\Delta\rho_{\min} = -0.24$ e Å^{-3}

Table 1
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O43—H43···O51$^{\rm i}$	0.91 (2)	2.61 (2)	3.0593 (14)	111.7 (16)
O43—H43···O41	0.91 (2)	1.80 (2)	2.6221 (13)	149 (2)

Symmetry code: (i) $\frac{1}{2}-x, y-\frac{1}{2}, \frac{3}{2}-z$.

All H atoms were initially located by difference Fourier synthesis. Subsequently, their positions were idealized and constrained to ride on their parent atoms with C—H(aromatic) = 0.95 Å or C—H(methyl) = 0.98 Å, and fixed individual displacement parameters [$U(\text{H}) = 1.5U_{\text{eq}}(\text{C}_{\text{methyl}})$ or $1.2U_{\text{eq}}(\text{C})$]. The hydroxyl H atom was refined freely.

Data collection: *SMART* (Siemens, 1995); cell refinement: *SMART*; data reduction: *SAINT* (Siemens, 1995); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997).

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