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Key indicators

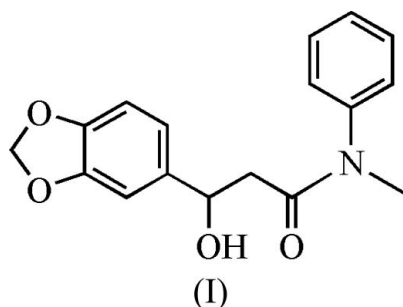
Single-crystal X-ray study
 $T = 294$ K
Mean $\sigma(\text{C}-\text{C}) = 0.003$ Å
 R factor = 0.043
 wR factor = 0.132
Data-to-parameter ratio = 15.2For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.3-(1,3-Benzodioxol-5-yl)-3-hydroxy-*N*-methyl-*N*-phenylpropionamide

The title compound, $\text{C}_{17}\text{H}_{17}\text{NO}_4$, was synthesized by the Reformatsky reaction of 1,3-benzodioxole-5-carbaldehyde and *N*-methyl-*N*-phenylcarbamic bromide. The two benzene rings are inclined at a dihedral angle of $50.6(3)^\circ$.

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Comment

A new compound, (I), was synthesized by an extension of the Reformatsky reaction (Bieber *et al.*, 1997). In our laboratory, we have recently designed a new system in the reaction of 1,3-benzodioxole-5-carbaldehyde with *N*-methyl-*N*-phenylcarbamic bromide. An X-ray crystal structure determination of (I) has been carried out and the results are presented here.



The molecular structure of (I) and the atom-numbering scheme are shown in Fig. 1. The dihedral angle between the two benzene rings is $50.6(3)^\circ$. The angle $\text{C}9-\text{C}10-\text{N}1$ is $117.8(19)^\circ$, indicating that C10 is sp^2 -hybridized. Atoms C5, C8, C9 and C10 are approximately coplanar [$\text{C}5-\text{C}8-\text{C}9-\text{C}10 = -166.92(18)^\circ$; mean deviation from plane is 0.0796 Å, maximum deviation from the plane is 0.0859 Å for C9]. The $\text{C}sp^2-\text{N}$ bonds [$\text{C}10-\text{N}1 = 1.346(3)$ Å and $\text{C}12-\text{N}1 = 1.438(3)$ Å] are significantly shorter than the $\text{C}sp^3-\text{N}$ bond [$\text{C}11-\text{N}1 = 1.466(3)$ Å] due to $\pi-\pi$ conjugation.

Experimental

The title compound was synthesized by adding *N*-methyl-*N*-phenylcarbamic bromide (3 mmol) to a solution of 1,3-benzodioxole-5-carbaldehyde (1 mmol) in dichloromethane (5 ml). Zinc powder (6 mmol) and a trace amount of iodine were added to the mixture. After the mixture had been refluxed for 11 h, the reaction was quenched with a saturated solution of ammonium chloride (8 ml). The mixture was extracted with dichloromethane and dried over magnesium sulfate. After removal of the solvent under reduced pressure, the residue was purified by flash chromatography (using ethyl acetate–petroleum ether). A colorless powder (yield 89%) was obtained. Slow evaporation of an ethyl acetate–petroleum ether solution (1:6) afforded the title compound as single crystals.

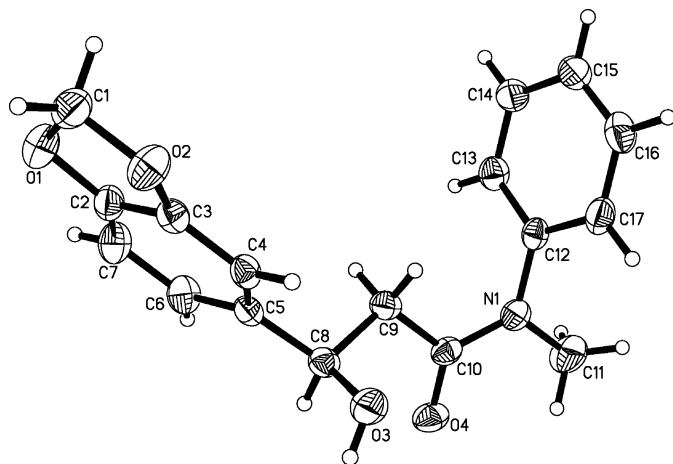


Figure 1
The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

Crystal data

$C_{17}H_{17}NO_4$	$Z = 4$
$M_r = 299.32$	$D_x = 1.341 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 15.864 (5) \text{ \AA}$	$\mu = 0.10 \text{ mm}^{-1}$
$b = 11.590 (4) \text{ \AA}$	$T = 294 (2) \text{ K}$
$c = 8.356 (3) \text{ \AA}$	Block, colorless
$\beta = 105.183 (6)^\circ$	$0.22 \times 0.18 \times 0.12 \text{ mm}$
$V = 1482.9 (8) \text{ \AA}^3$	

Data collection

Bruker SMART CCD area-detector diffractometer	8186 measured reflections
φ and ω scans	3038 independent reflections
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	1648 reflections with $I > 2\sigma(I)$
$T_{\min} = 0.963$, $T_{\max} = 0.989$	$R_{\text{int}} = 0.042$
	$\theta_{\max} = 26.4^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.043$
 $wR(F^2) = 0.132$
 $S = 1.00$
 3038 reflections
 200 parameters
 H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0596P)^2 + 0.1641P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.002$
 $\Delta\rho_{\max} = 0.30 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.19 \text{ e \AA}^{-3}$

Table 1

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

O1—C2	1.379 (3)	O2—C1	1.426 (3)
O1—C1	1.419 (3)	O3—C8	1.425 (2)
O2—C3	1.376 (3)	O4—C10	1.231 (2)
O1—C1—O2	107.76 (18)	O4—C10—N1	121.4 (2)
O3—C8—C9	107.33 (17)	C17—C12—N1	120.3 (2)
C1—O2—C3—C4	172.4 (2)	C11—N1—C12—C13	103.5 (3)
C4—C5—C8—O3	29.3 (3)		

All carbon-bound H atoms were positioned geometrically and refined as riding ($C-H = 0.93-0.98 \text{ \AA}$). For the CH and CH_2 groups, $U_{\text{iso}}(H)$ values were set equal to $1.2U_{\text{eq}}(C)$, and for methyl groups set equal to $1.5U_{\text{eq}}(C)$. The O3—H3 distance was constrained to 0.82 \AA , with $U_{\text{iso}}(H) = 1.5U_{\text{eq}}(C)$.

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

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