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Ethyl 4-{5-[1-(hydroxymethyl)ethylamino-carbonyl]-1,3-benzodioxol-4-yl}-7-methoxy-3-benzodioxole-5-carboxylate

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Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.009 Å
R factor = 0.056
wR factor = 0.166
Data-to-parameter ratio = 6.6

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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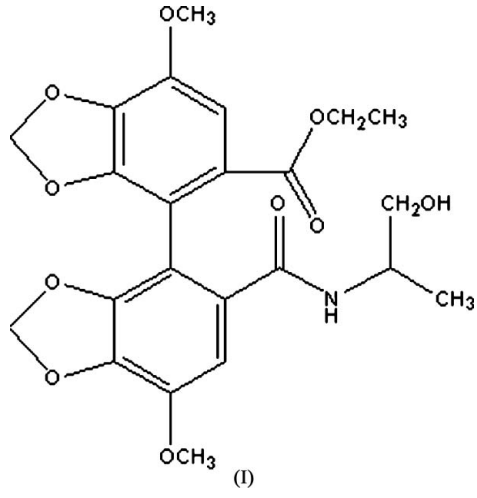
In the title compound, $C_{23}H_{25}NO_{10}$, the five-membered rings adopt envelope conformations and the dihedral angle between the two benzene rings is 67.1 (7)°. The crystal structure is stabilized by intramolecular N—H···O and intermolecular O—H···O hydrogen-bond interactions.

Comment

The title compound, (I), is a biphenyl derivative of dimethyl 4,4′-dimethoxy-5,6,5′,6′-dimethylenedioxybiphenyl-2,2′-dicarboxylate (DDB), which may act to moderate liver ailments, and is thus effective in the treatment of acute and chronic hepatitis and in lowering the level of serum glutamic pyruvic transaminase (SGPT) (Xie *et al.*, 1982, 1983).



(I)

The molecular structure of (I) is illustrated in Fig. 1. The bond lengths and angles are unremarkable. The five-membered rings adopt envelope conformations (in ring C14/O7/C15/C13/O6, C14 is the flap atom, and in ring C1/O2/C2/C7/O1, C1 is the flap atom). The angle between the two benzene rings is 67.1 (7)°. Intermolecular O—H···O hydrogen bonds help to stabilize the crystal structure (Fig. 2 and Table 1).

Experimental

The title compound, (I), was prepared according to a literature procedure (Xie *et al.*, 1982; Bunin, 1998). For the reaction, one molar equivalent of the polymeric active ester derivative was reacted with 2-propanol formamide in the presence of diisopropylethylamine (DIEA) at room temperature for 48 h to give compound (I) in 85% yield as a white powder (m.p. 412 K); single crystals suitable for X-ray analysis were obtained by slow evaporation of a dichloromethane

solution. ^{1}H NMR ($CDCl_3$, p.p.m.): 7.08 (*s*, 1H), 7.22 (*s*, 1H), 6.28 (*d*, 1H), 6.03 (*d*, 2H), 6.00 (*d*, 2H), 4.22 (*q*, 2H), 3.98 (*s*, 3H), 3.95 (*s*, 3H), 3.51 (*br*, 1H), 3.40 (*m*, 1H), 2.75 (*br s*,1H), 1.19 (*t*, 3H), 0.93 (*d*, 1H), 0.75 (*d*, 3H).

Crystal data

$C_{23}H_{25}NO_{10}$
M_r = 475.44
Monoclinic, $P2_1$
a = 9.0277 (15) Å
b = 14.185 (2) Å
c = 9.7695 (17) Å
β = 114.266 (3)°
V = 1140.5 (3) Å^3
Z = 2
D_x = 1.384 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2246 reflections
θ = 2.6–23.4°
μ = 0.11 mm^{-1}
T = 294 (2) K
Block, white
0.24 × 0.20 × 0.12 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.974, T_{max} = 0.987
5834 measured reflections
2099 independent reflections
1699 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
θ_{max} = 25.0°
$h = -10 \rightarrow 9$
$k = -16 \rightarrow 16$
$l = -11 \rightarrow 9$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.166
S = 1.06
2099 reflections
316 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.1001P)^2 + 0.5453P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.65 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O4	0.91 (7)	2.48 (7)	3.367 (8)	166 (5)
O10—H10···O9^{i}	0.82	1.99	2.811 (7)	174

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

The H atom bonded to the N atom was located in a difference map and allowed to refine freely. The other H atoms were positioned geometrically and constrained to ride on their parent atoms, with C—H = 0.93–0.97 Å and O—H = 0.82 Å, and with U_{iso}(H) = xU_{eq}(parent atom), where x = 1.5 for methyl C and O atoms or 1.2 for the others. In the absence of significant anomalous scattering, Friedel pairs were merged prior to the final refinement, and the absolute configuration was assigned according to the known absolute configuration of the dimethyl 4,4′-dimethoxy-5,6,5′,6′-dimethylenedioxybiphenyl-2-carboxyethylate-2′-*N*-(2-propanol)formamide employed in the synthesis.

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*.

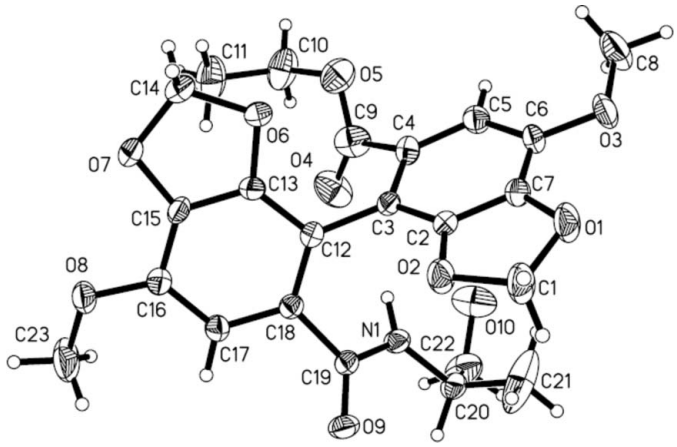


Figure 1
The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are represented by circles of arbitrary size.

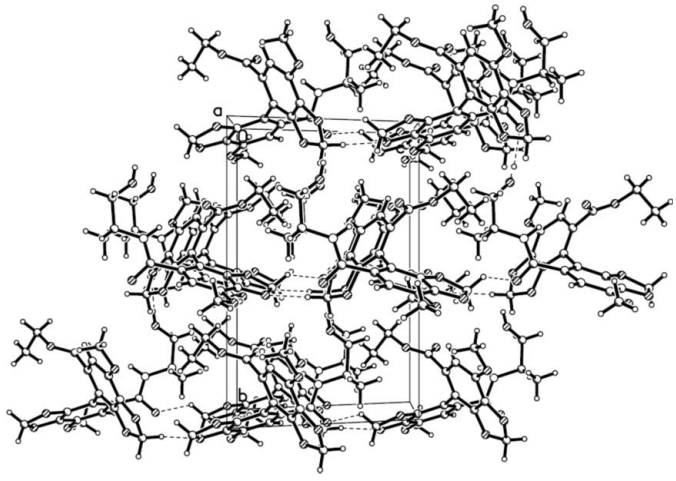


Figure 2
The crystal structure of (I), viewed along the *a* axis. Dashed lines indicate hydrogen bonds.

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References

Bruker (1997). *SMART* (Version 5.611), *SAINT* (Version 6.0) and *SHELXTL* (Version 5.10). Bruker AXS Inc., Madison, Wisconsin, USA.
Bunin, B. A. (1998). *The Combinatorial Index*, pp. 262–266. New York: Academic Press.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Xie, J. X., Zhou, J., Zhang, C. Z., Yang, J. H., Jin, H. Q. & Chen, J. X. (1982). *Acta Pharm. Sin.* **17**, 23–27.
Xie, J. X., Zhou, J., Zhang, C. Z., Yang, J. H., Jin, H. Q. & Chen, J. X. (1983). *Sci. Sin. B*, **26**, 1291–1303.