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Dimethyl 5,5-dicyano-2-hydroxy-4,6-diphenylcyclohex-1-ene-1,3-dicarboxylate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.037
wR factor = 0.096
Data-to-parameter ratio = 14.2

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

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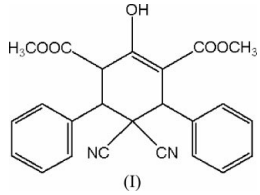
In the title molecule, $C_{24}H_{20}N_2O_5$, the cyclohexene ring adopts a half-chair conformation. The molecular structure shows some intra- and intermolecular hydrogen bonds.

Comment

A series of 4*H*-pyran and cyclohexanone derivatives were prepared *via* a three-component reaction of dimethyl acetonedicarboxylate, aromatic aldehydes and malononitrile.

In the molecule of the title compound, (I), the dihedral angle between the two phenyl rings is 88.8 (1)° (*PARST*; Nardelli, 1995). The cyclohexene ring adopts a half-chair conformation. The total puckering amplitude (Cremer & Pople, 1975) for this ring is Q_T = 0.527 (1) Å. According to Duax *et al.* (1976), the ring conformation is half-chair, with a local pseudo-twofold axis passing through the mid-points of the C8—C9 and C11—C12 bonds; it is deformed towards a sofa, with a local pseudo-twofold axis along C9···C12.



(I)

The bond lengths and angles in (I) are comparable to the corresponding values in methyl 6-amino-5-cyano-2-methoxycarbonylmethyl-4-phenyl-4*H*-pyran-3-carboxylate (Öztürk *et al.*, 2004). All bond distances and angles are as expected.

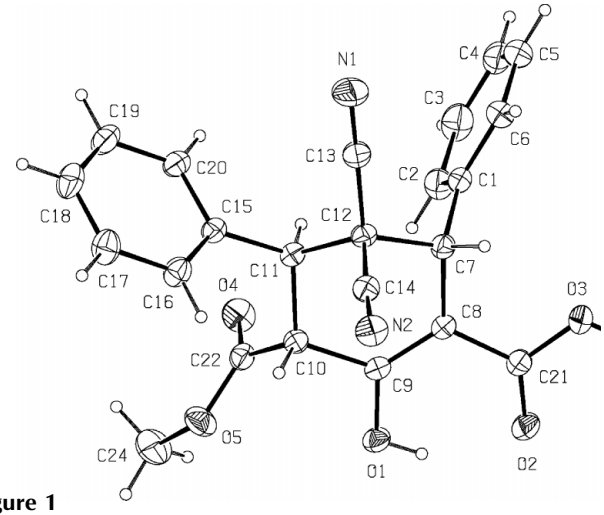


Figure 1
An *ORTEP* plot of (I), with the atom-numbering scheme and 20% probability displacement ellipsoids.

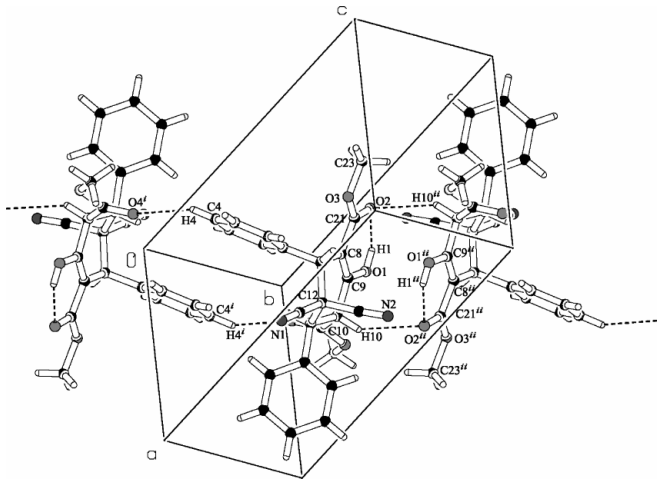


Figure 2
View of the hydrogen bonding (dashed lines) in (I). [Symmetry codes: (i) $-x, 1-y, -z$; (ii) $1-x, 2-y, 1-z$.]

The crystal structure of (I) is stabilized by intra- and intermolecular hydrogen bonds. The hydrogen-bonding geometry is given in Table 2 and can be seen in Fig. 2.

Experimental

The synthesis of (I) has been reported previously (Heber & Stoyanov, 2003). IR (cm^{-1}, KBr): 3029, 2963, 2840,1738, 1661, 1640,1492, 1436, 1400,1367, 1308. EIMS *m/z* (%): 416 (M^+, 31), 384 (6), 357 (6), 325 (59), 298 (6), 262 (36), 230 (32), 202 (100), 171 (80), 154 (20), 140 (8), 121 (30), 103 (28), 91 (9), 77 (16), 59 (12), 43 (2). ^{1}H NMR (300 MHz, DMSO-d_6): δ 3.62 (*s*, 3H, OCH_3), 3.67 (*s*, 3H, OCH_3), 3.69 (*d*, 1H, J = 11.8 Hz), 4.30 (*d*, 1H, J = 11.8 Hz), 5.07 (*s*, 1H), 7.29–7.62 (*m*, 10H aromatic), 12.30 (*s*, 1H, OH). Analysis calculated for $C_{24}H_{20}N_2O_5$: C 69.22, H 4.84, N 6.73%; found: C 69.11, H 4.87, N 6.64%.

Crystal data

$C_{24}H_{20}N_2O_5$
M_r = 416.42
Triclinic, $P\overline{1}$
a = 8.8185 (10) Å
b = 10.9462 (13) Å
c = 12.8297 (14) Å
α = 97.514 (9)°
β = 106.713 (9)°
γ = 109.678 (9)°
V = 1080.7 (2) Å^3
Z = 2
D_x = 1.280 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 6497 reflections
θ = 1.7–28.4°
μ = 0.09 mm^{-1}
T = 293 K
Irregular, colorless
0.46 × 0.37 × 0.25 mm

Data collection

Stoe IPDS-II diffractometer
ω scans
Absorption correction: by integration (*X-RED*32; Stoe & Cie, 2002)
T_{min} = 0.967, T_{max} = 0.985
9110 measured reflections
4138 independent reflections
2631 reflections with $I > 2\sigma(I)$
R_{int} = 0.033
θ_{max} = 26.0°
$h = -10 \rightarrow 10$
$k = -13 \rightarrow 13$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.096
S = 0.89
4138 reflections
292 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0552P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C9	1.339 (2)	O5—C22	1.321 (2)
O2—C21	1.223 (2)	O5—C24	1.453 (3)
O3—C21	1.326 (2)	N1—C13	1.134 (3)
O3—C23	1.450 (3)	N2—C14	1.137 (2)
O4—C22	1.194 (2)		
C21—O3—C23	116.06 (14)	O2—C21—C8	123.73 (15)
C22—O5—C24	116.39 (15)	O3—C21—C8	113.46 (14)
O1—C9—C10	111.15 (13)	O2—C21—O3	122.81 (16)
O1—C9—C8	124.40 (15)	O4—C22—C10	123.71 (16)
N1—C13—C12	178.89 (19)	O5—C22—C10	111.56 (14)
N2—C14—C12	176.5 (2)	O4—C22—O5	124.72 (16)
C23—O3—C21—C8	178.54 (14)	C9—C8—C21—O2	3.4 (2)
C23—O3—C21—O2	−0.8 (2)	C7—C8—C21—O3	3.0 (2)
C24—O5—C22—C10	−173.06 (19)	C21—C8—C9—O1	−1.3 (2)
C24—O5—C22—O4	6.3 (3)	O1—C9—C10—C11	−168.56 (13)
C8—C7—C12—C14	69.31 (16)	C9—C10—C22—O5	99.93 (17)
C8—C7—C12—C13	−173.62 (13)	C11—C10—C22—O4	44.4 (2)
C7—C8—C9—O1	179.81 (14)	C11—C10—C22—O5	−136.20 (16)
C7—C8—C21—O2	−177.67 (15)	C10—C11—C12—C14	−54.71 (19)
C7—C8—C9—C10	−1.9 (2)	C10—C11—C12—C13	−173.43 (14)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O2	0.82	1.83	2.548 (2)	145
C4—H4···O4^{i}	0.93	2.54	3.317 (3)	141
C7—H7···O3	0.96 (2)	2.42 (2)	2.720 (2)	97 (1)
C10—H10···O2^{ii}	0.96 (2)	2.50 (1)	3.323 (2)	144 (1)
C11—H11···O4	0.99 (2)	2.53 (2)	2.878 (2)	101 (1)

Symmetry codes: (i) $-x, 1-y, -z$; (ii) $1-x, 2-y, 1-z$.

The H atoms attached to atoms C7, C10 and C11 were clearly visible in a difference electron-density map and were refined freely. A rotating group model was used for the methyl and hydroxy groups, with their H atoms in idealized positions. These and other H atoms positioned geometrically were allowed to ride on the parent atoms, with aromatic C—H = 0.93 Å, methyl C—H = 0.96 Å and hydroxy O—H = 0.82 Å. The U_{iso}(H) values were set at $1.5U_{eq}$(C) for the methyl and hydroxy H atoms, and at $1.2U_{eq}$(C) for other C-bound H atoms.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED*32 (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*III (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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