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2-(4-Methoxyphenyl)-5-(4-methylpent-3-enyl)-2,3,3a,4,7,7a-hexahydro-1*H*-isoindole-1,3-dione

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Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.008 Å
Disorder in main residue
R factor = 0.074
wR factor = 0.223
Data-to-parameter ratio = 11.9

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

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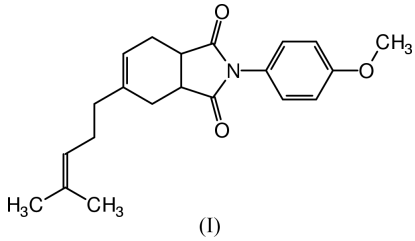
The structure of the title compound, $C_{21}H_{25}NO_3$, comprises a U-shaped molecule with two disordered atoms in the pentene chain. The two components of the two disordered alkane C atoms exist with unequal partial occupancies of 0.70 and 0.30. The dihedral angle between the planar imide ring [r.m.s. deviation 0.038 (3) Å] and the phenyl ring is 62.56 (14)°. C—H···O close contacts are observed from two different aromatic C atoms to the two oxo O atoms.

Comment

The title compound, (I), was prepared by a Diels–Alder reaction between 1-(4-methoxyphenyl)-3-pyrroline-2,5-dione and 7-methyl-3-methyleneocta-1,6-diene. The naming of (I) has proved difficult, with several variants for the main ring system being previously given. The basic unsubstituted structure (Kirfel, 1976) was named as a tetrahydrophthalimide and several other substituted analogues have been similarly named, although numbering schemes differ significantly as to which four atoms are saturated. Other names include hydroisoindoline-1,3-dione (with differing numbers of saturated C atoms), cyclohex-4-ene-1,2-dicarboximide and azabicyclo[4.3.0]non-3-ene-7,9-dione, with the first being the most popular.



(I)

The structure of (I) comprises a U-shaped molecule with two disordered atoms in the pentene chain (Fig. 1). The shape is dependent on the boat conformation displayed by the hydrophthalimide group. Of 136 structures (Cambridge Structural Database, September 2002 release; Allen, 2002) containing a phthalimide ring similar to (I), only 48 do not have any bridging across the cyclohexene ring. From the 48, 23 compounds contain an *N*-phenyl moiety, of which 11 share a similar boat conformation to (I). This conformation is also displayed by 5-(2-methyl-1,3-dioxo-2,3,3a,4,7,7a-hexahydro-1*H*-isoindol-5-yl)pent-2-enoic acid (Yliniemela *et al.*, 1995), the nearest structural analogue to (I). In the latter structure, the pent-2-enoic acid chain is not disordered. In (I), the two components (*A* and *B*) of the two disordered alkane C atoms (C51 and C52) exist with unequal partial occupancies of 0.70 and 0.30, respectively, for *A* and *B*. The dihedral angle between the planar imide ring [r.m.s. deviation 0.038 (3) Å]

and the phenyl ring is 62.56 (14)°. C—H···O close contacts are observed from two different aromatic C atoms to the two oxo O atoms (Table 1).

Experimental

The title compound was obtained from Key Organics Ltd and crystals were grown from an ethanol solution.

Crystal data

$C_{21}H_{25}NO_3$
M_r = 339.42
Monoclinic, $P2_1/c$
a = 17.001 (2) Å
b = 6.5492 (10) Å
c = 17.145 (3) Å
β = 110.584 (5)°
V = 1787.1 (5) Å^3
Z = 4
D_x = 1.262 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 20434 reflections
θ = 2.9–27.5°
μ = 0.08 mm^{-1}
T = 150 (2) K
Prism, colourless
0.20 × 0.15 × 0.03 mm

Data collection

Bruker–Nonius KappaCCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995)
T_{min} = 0.983, T_{max} = 0.998
8778 measured reflections
2934 independent reflections
1368 reflections with $I > 2\sigma(I)$
R_{int} = 0.129
θ_{max} = 25.0°
$h = -18 \rightarrow 20$
$k = -7 \rightarrow 7$
$l = -19 \rightarrow 20$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.074
$wR(F^2)$ = 0.223
S = 0.94
2934 reflections
247 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1159P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C22—H22···O3^{i}	0.95	2.55	3.134 (5)	120
C25—H25···O1^{ii}	0.95	2.49	3.340 (5)	149

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

All H atoms were included in the refinement, at calculated positions, in riding model approximation, with C—H distances of 0.95 (Ar—H), 1.00 (CH), 0.99 (CH_2) and 0.98 Å (CH_3), while the isotropic displacement parameters were set at $1.25U_{eq}$(C). The R_{int} value of 0.129 is the result of weak high-angle data.

Data collection: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT* (Hooft, 1998); cell refinement: *DENZO* and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON*97 (Spek, 1997); software used to prepare material for publication: *SHELXL*97.

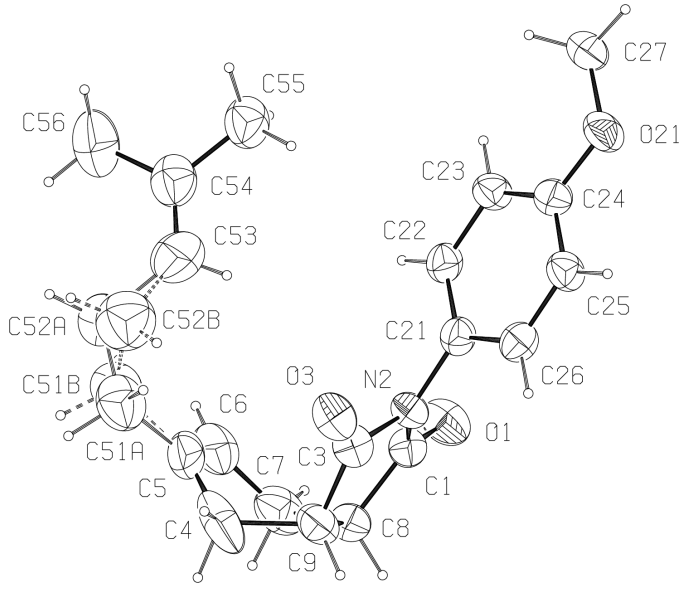


Figure 1
The molecular configuration and atom-numbering scheme for the title compound, with displacement ellipsoids drawn at the 50% probability level.

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