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2-{3-[2-(4-Hydroxyphenyl)vinyl]-5,5-dimethylcyclohex-2-en-1-ylidene}malononitrile

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Key indicators

Single-crystal X-ray study
T = 291 K
Mean σ(C–C) = 0.003 Å
R factor = 0.039
wR factor = 0.095
Data-to-parameter ratio = 9.5

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, $C_{19}H_{18}N_2O$, was studied in the course of our investigations on organic non-linear optical (NLO) materials. Its molecule with the exception of the $C(CH_3)_2$ group is nearly planar.

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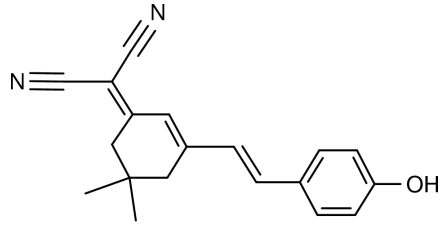
Comment

The structure of the title compound, (I) (Fig. 1), was studied in the course of our investigations on organic non-linear optical (NLO) materials. Its molecule with the exception of the $C(CH_3)_2$ group is nearly planar; the disubstituted C atom is displaced by 0.624 (2) Å from the mean plane of the remaining atoms of the cyclohexene ring. The hydrogen bond formed by the hydroxy group with the N atom of one of the cyano groups [O—H···N: O···N 2.958 (3) Å and O—H···N 173°] links the molecules in the crystal into infinite chains stretching along the [013] direction.



(I)

Experimental

The starting compound, 3,5,5-trimethyl(cyclohex-2-enylidene)-malonodinitrile, $C_{12}H_{14}N_2$, was prepared by means of Knoevenagel condensation of malonodinitrile (660 mg, 10 mmol, Fluka) and isophorone (1.382 g, 10 mmol, Fluka). Both compounds were dissolved in *N*,*N*-dimethylformamide (50 ml). The solution was stirred for eight hours at room temperature. Piperidinium acetate (600 mg) was used as a catalyst. A yellow precipitate was obtained from the resulting dark-yellow solution after evaporation of half of the solvent. The product was filtered and recrystallized from 95% ethanol. Yield 70%, m.p. 346–347 K. The title compound was prepared according to a published procedure (Lemke, 1970) from 3,5,5-trimethyl(cyclohex-2-enylidene)malonodinitrile (1.86 g, 10 mmol) and 4-hydroxybenzaldehyde (1.22 g, 10 mmol) in a 150 ml trichloromethane solution with continuous stirring for two days at room temperature. Piperidinium acetate was used as a catalyst. The orange–red solution was purified by column chromatography on silica gel. The orange precipitate (λ_{max} = 421 nm in MeOH) was recrystallized from glacial acetic acid [Yield 65% m.p. 478–479 K; literature m.p. 483–484 K (Lemke, 1970)]. Crystals were grown by slow (several days) evaporation from ethyl acetate.

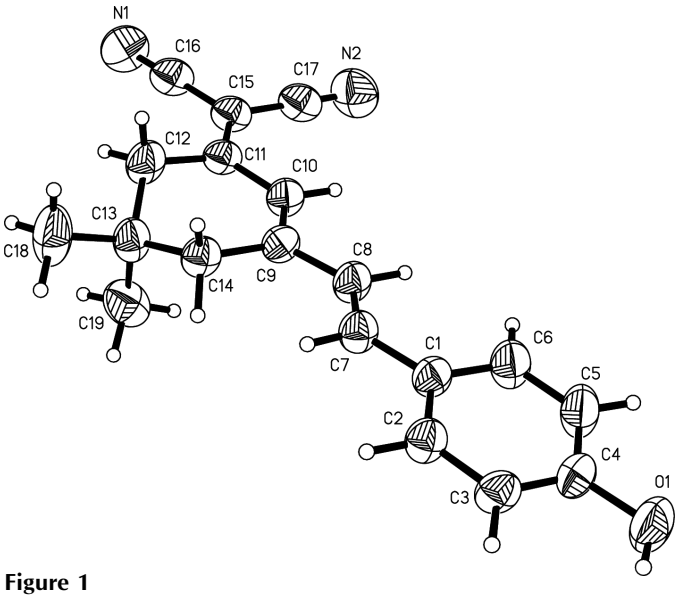


Figure 1
View of the title compound showing the labelling of all non-H atoms. Displacement ellipsoids are shown at 50% probability levels. H atoms are drawn as circles of arbitrary radii.

Crystal data

$C_{19}H_{18}N_2O$
M_r = 290.35
Orthorhombic, $Pna2_1$
a = 15.4413 (3) Å
b = 10.9988 (3) Å
c = 9.5699 (2) Å
V = 1625.31 (6) Å^3
Z = 4
D_x = 1.187 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 10060 reflections
θ = 3.1–27.4°
μ = 0.07 mm^{-1}
T = 291 (1) K
Block, red
0.25 × 0.23 × 0.23 mm

Data collection

Nonius KappaCCD diffractometer
201 frames *via* ω rotation ($\Delta\omega$ = 1%) and two times 20 s per frame (two sets at different κ angles)
1921 measured reflections
1921 independent reflections
1302 reflections with $I > 2\sigma(I)$
R_{int} = 0.032
θ_{max} = 27.4°
$h = -19 \rightarrow 19$
$k = -14 \rightarrow 14$
$l = -12 \rightarrow 12$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.095
S = 0.99
1921 reflections
203 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0555P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.12 e Å^{-3}
$\Delta\rho_{min}$ = −0.11 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.017 (3)

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N1^{i}	0.82	2.14	2.958 (3)	173.3

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

H atoms were placed in calculated positions with U_{iso} constrained to be 1.5 times U_{eq} of the carrier atom for the methyl H atoms and 1.2 times U_{eq} for the remaining H atoms.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL*97 and *PARST*95 (Nardelli, 1995).

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