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2-{5,5-Dimethyl-3-[2-(2-methoxyphenyl)vinyl]cyclohex-2-enylidene}malononitrile

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Key indicators

Single-crystal X-ray study
T = 291 K
Mean σ(C–C) = 0.002 Å
R factor = 0.049
wR factor = 0.125
Data-to-parameter ratio = 18.3

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

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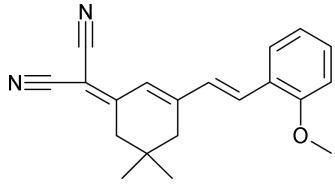
The title molecule, $C_{20}H_{20}N_2O$, is nearly planar except for the $C(CH_3)_2$ group. The disubstituted C atom is displaced by 0.635 (2) Å from the mean plane of the remaining atoms of the cyclohexene ring.

Comment

As part of our investigations on organic non-linear optical (NLO) materials, we determined the crystal structure of the title compound, (I) (Fig. 1), which was first synthesized by Desai & Shah (1997), according to the general procedure described by Lemke (1970). It was suggested that this compound could be a good candidate for NLO and electro-optical applications.



(I)

Experimental

The title compound was synthesized in a way similar to the general procedure of Lemke (1970). The preparation of the starting compound, 3,5,5-trimethyl(cyclohex-2-enylidene)malonodinitrile, $C_{12}H_{14}N_2$, is described in one of our previous papers (Kolev *et al.*, 2001). The starting compound (3.72 g, 20 mmol) was dissolved in 60 ml of dry methanol by continuous stirring at room temperature. 2-Methoxybenzaldehyde (2.72 g, 20 mmol, Acros) was added to the solution. Nearly 3 ml of triethylamine was used as a catalyst. The solution became yellow after a few minutes and the resulting compound precipitated. After 24 h reaction time, the solution was cooled and the resulting title compound was isolated. Purification was achieved *via* column chromatography on silica gel using chloroform as eluent. Yield 5.12 g (86%) (m.p. 466–468 K). The purity of the compound was confirmed by elemental analysis, IR, UV–vis and mass spectrometry. The crystals were grown from an ethyl acetate solution by slow evaporation at room temperature over a period of several days.

Crystal data

$C_{20}H_{20}N_2O$
M_r = 304.38
Orthorhombic, *Pbca*
a = 13.1374 (3) Å
b = 13.2089 (3) Å
c = 19.7506 (4) Å
V = 3427.33 (13) Å^3
Z = 8
D_x = 1.180 Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 20 401 reflections
θ = 3.1–27.5°
μ = 0.07 mm^{-1}
T = 291 (1) K
Plate, yellow
0.5 × 0.4 × 0.1 mm

Data collection

Nonius KappaCCD diffractometer	1907 reflections with $I > 2\sigma(I)$
198 frames *via* ω rotation ($\Delta\omega = 1^\circ$) with two sets at different κ angles and two times 10 s per frame	$R_{int} = 0.034$
	$\theta_{max} = 27.5^\circ$
	$h = -17 \rightarrow 17$
20 402 measured reflections	$k = -17 \rightarrow 17$
3890 independent reflections	$l = -25 \rightarrow 25$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.059P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.049$	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2) = 0.125$	$\Delta\rho_{max} = 0.14$ e Å^{-3}
$S = 0.98$	$\Delta\rho_{min} = -0.14$ e Å^{-3}
3890 reflections	Extinction correction: *SHELXL*97
212 parameters	Extinction coefficient: 0.0077 (9)
H-atom parameters constrained	

H atoms were placed in calculated positions with U_{iso} constrained to be $1.5U_{eq}$ of the carrier atom for the methyl–H and $1.2U_{eq}$ 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, *PARST*95 (Nardelli, 1995) and *PLATON* (Spek, 2001).

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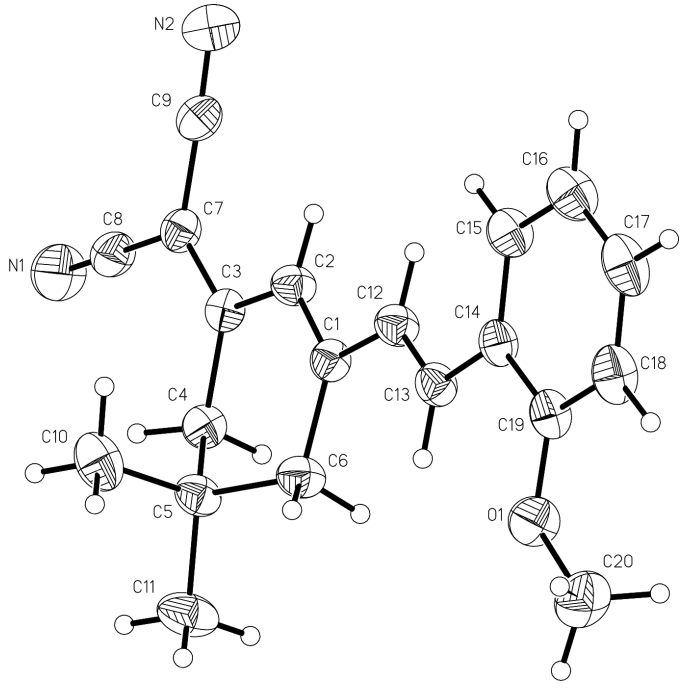

Figure 1
View of the title compound with the atom labelling for non-H atoms. Displacement ellipsoids are shown at the 50% probability level. H atoms are drawn as circles of arbitrary radii.

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