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2-{3-[2-(1*H*-Indol-3-yl)vinyl]-5,5-dimethylcyclohex-2-enylidene}malononitrile

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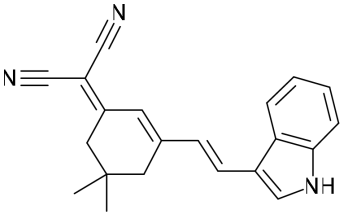
Key indicators

Single-crystal X-ray study
T = 291 K
Mean σ(C–C) = 0.003 Å
R factor = 0.052
wR factor = 0.130
Data-to-parameter ratio = 18.0

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

The title compound, $C_{21}H_{19}N_3$, first synthesized by Lemke [*Chem. Ber.* (1970), **103**, 1894–1898], due to its solvatochromic behaviour is supposed to be a good candidate for non-linear optical (NLO) and electrooptical applications. The molecule is nearly planar, with the exception of the $C(CH_3)_2$ group; the disubstituted C atom is displaced by 0.627 (2) Å from the mean plane of the remaining atoms of the cyclohexene ring. The hydrogen bond formed by the indolyl NH group with the N atom of one of the cyano groups [N···N 3.168 (3) Å and N—H···N 148°], links the molecules into infinite chains stretching along the $[10\bar{1}]$ direction of the crystal. The existence of this hydrogen bond was also confirmed by FT–IR spectral data [ν_{N-H} = 3388 cm^{-1} in the solid state (KBr pellet)].



(I)

Experimental

The title compound was synthesized according to Lemke (1970). The preparation of the starting compound, [3,5,5-trimethyl(cyclohex-2-enylidene)]malonodinitrile, is described in one of our previous papers (Kolev *et al.*, 2001). The starting compound (6 mmol, 20% excess) was dissolved in 80 ml of dry toluene under continuous stirring. Indole-3-aldehyde (5 mmol Across), dissolved in 50 ml of dry toluene, was added to the solution. Nearly 1 ml of triethylamine was used as a catalyst. The solution became dark-red after a few minutes and the resulting compound started precipitating. After 16 h reaction time, the solution was cooled and the resulting title compound was isolated and recrystallized twice from glacial acetic acid and from dry toluene [m.p. 519–521 K; literature m.p. 520–522 K (Lemke, 1970)]. The purity of the compound was confirmed by elemental analysis, IR, UV-vis and mass spectrometry. Crystals were grown from a glacial acetic acid solution by slow evaporation at room temperature over a period of several weeks.

Crystal data

$C_{21}H_{19}N_3$
M_r = 313.39
Monoclinic, $P2_1/n$
a = 15.5033 (3) Å
b = 7.5309 (2) Å
c = 15.9609 (4) Å
β = 110.774 (1)°
V = 1742.34 (7) Å^3
Z = 4

D_x = 1.195 Mg m^{-3}
Mo *K*α radiation
Cell parameters from 15212 reflections
θ = 3.0–27.5°
μ = 0.07 mm^{-1}
T = 291 (1) K
Plate, red
0.40 × 0.30 × 0.05 mm

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Data collection

Nonius KappaCCD diffractometer
292 frames *via* ω-rotation ($\Delta\omega = 1°$) with 3 sets at different κ-angles and two times 20 s per frame
15 212 measured reflections
3946 independent reflections
1785 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.027$
$\theta_{\rm max} = 27.5°$
$h = -20 \rightarrow 20$
$k = -9 \rightarrow 9$
$l = -20 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.052$
$wR(F^2) = 0.130$
$S = 0.92$
3946 reflections
219 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0595P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.13$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.18$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3···N2$^{\rm i}$	0.86	2.41	3.168 (3)	148
C21—H21···N1$^{\rm ii}$	0.93	2.55	3.301 (3)	138

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

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

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *DENZO* and *SCALEPACK*; data reduction: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); 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).

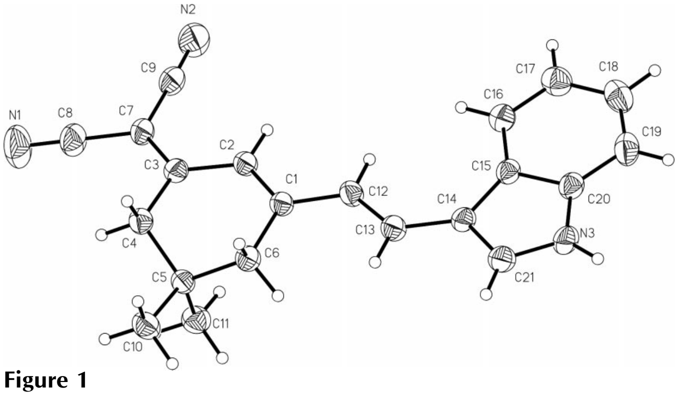

Figure 1
View of the title compound showing the labelling of all non-H atoms. Displacement ellipsoids are shown at the 50% probability levels. H atoms are drawn as circles of arbitrary radii.

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