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2-[5,5-Dimethyl-3-(2-*p*-tolyl-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 = 0.043
wR factor = 0.082
Data-to-parameter ratio = 19.8

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

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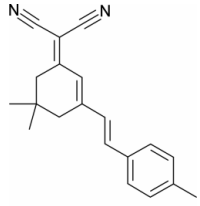
The crystal structure of the title compound, $C_{20}H_{20}N_2$, contains two independent molecules in the asymmetric unit. These molecules differ significantly only in the torsion angles of the dimethylcyclohexene moieties, which have twisted conformations. The molecules are nearly planar except for the $C(CH_3)_2$ groups.

Comment

In the course of our investigations on organic electro-optical, non-linear optical and photorefractive materials we determined the crystal structure of the title compound, (I). As a result of its solvatochromism, it is expected to be a good candidate for photorefractive and electro-optical applications.



(I)

The conversion of basic 2-(3,5,5-trimethyl-cyclohex-2-enylidene)-malononitrile into the corresponding 2-[5,5-dimethyl-3-(2-*p*-tolyl-vinyl)-cyclohex-2-enylidene]-malononitrile affords a way to enhance the charge-transfer transition at the molecular level – a requisite for the design of efficient second- and third-order non-linear optical materials.

The crystal structure contains no classical H-bonds, but C11—H11···N21 [H···*A* = 2.62, *D*···*A* = 3.391 (2) Å, *D*—H···*A* = 141°] is a possible non-classical H-bond.

The dihedral angle between the least-squares planes through atoms C1, C2, C3, C11, C12 and through C13, C14, C15, C16, C17, C18 is 8.24 (9)°, whereas the corresponding angle in the other molecule in the asymmetric unit is only 3.74 (9)°

Experimental

The title compound was synthesized for the first time, according to a general procedure described by Lemke (1970): 3.72 g (20 mmol) 2-(3,5,5-trimethyl-cyclohex-2-enylidene)-malononitrile and 3.71 ml (20 mmol) 4-methyl-benzaldehyde were dissolved in 80 ml dry toluene with continuous stirring and heating under reflux for 60 min. Piperidine (1 ml) was used as a catalyst. After a few minutes, the solution became dark red. A bright yellow precipitate was obtained after evaporation of the solvent. The product was filtered and washed with ethanol and recrystallized from ethyl acetate. Yield of the recrystallized product: 33%; melting point: 428–429 K. UV–vis(EtOH): λ_{max} (log ε) = 270.5 nm (3.702), 398.5 nm (4.329).

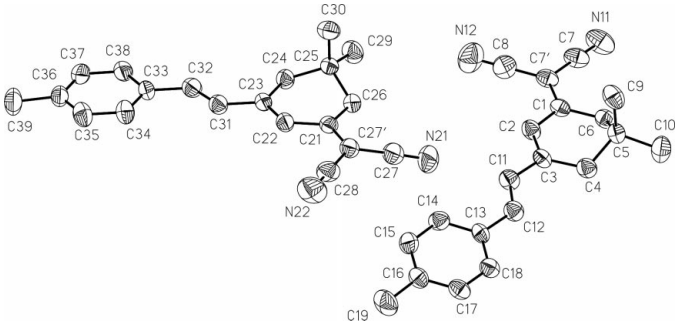


Figure 1
View of the title compound, showing the labelling of all non-H atoms. Displacement ellipsoids are shown at the 50% probability level. H atoms are omitted for clarity.

1094 The purity of the compound was confirmed by elemental analysis, IR, UV–vis and mass spectrometry. Crystals were grown from ethyl acetate by slow evaporation at room temperature over a period of a week.

Crystal data

$C_{20}H_{20}N_2$	D_x = 1.095 Mg m^{-3}
M_r = 288.38	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 62924 reflections
a = 13.5270 (3) Å	θ = 3.0–27.5°
b = 16.1781 (3) Å	μ = 0.06 mm^{-1}
c = 16.3616 (4) Å	T = 291 (1) K
β = 102.3807 (9)°	Block, orange
V = 3497.32 (13) Å^3	0.50 × 0.40 × 0.32 mm
Z = 8	

Data collection

Nonius KappaCCD diffractometer	R_{int} = 0.021
ω scans with κ offsets	θ_{max} = 27.5°
Absorption correction: none	$h = -17 \rightarrow 17$
62924 measured reflections	$k = -20 \rightarrow 20$
7998 independent reflections	$l = -21 \rightarrow 20$
2602 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	H-atoms parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.043	$w = 1/[\sigma^2(F_o^2) + (0.02P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.082	$(\Delta/\sigma)_{max}$ = 0.004
S = 0.96	$\Delta\rho_{max}$ = 0.11 e Å^{-3}
7998 reflections	$\Delta\rho_{min}$ = −0.16 e Å^{-3}
403 parameters	

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 and 1.2 times U_{eq} for the remaining H atoms. The methyl groups were allowed to rotate but not to tip.

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), *PLATON* (Spek, 2001).

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