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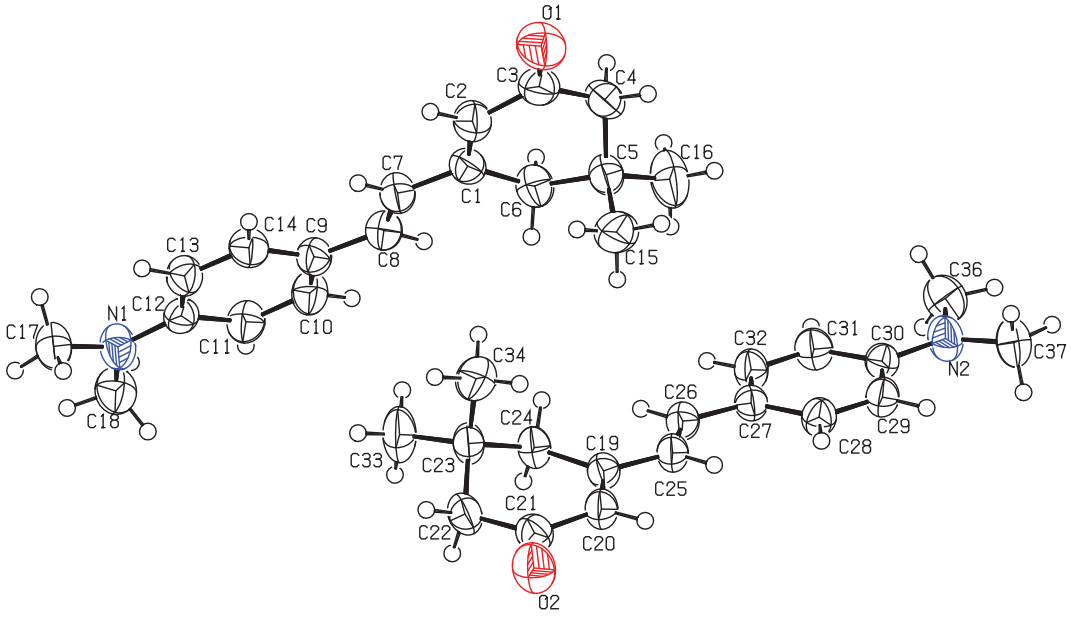
The crystal structure of (*E*)-3-(4-(dimethylamino) styryl)-5,5-dimethylcyclohex-2-en-1-one, $C_{18}H_{23}NO$



https://doi.org/10.1515/ncrs-2019-0167
Received March 5, 2019; accepted April 29, 2019; available online May 27, 2019

Abstract

$C_{18}H_{23}NO$, triclinic, $P\bar{1}$ (no. 2), $a = 6.0033(3)$ Å, $b = 11.5667(7)$ Å, $c = 23.5556(12)$ Å, $\alpha = 77.121(5)°$, $\beta = 89.635(4)°$, $\gamma = 78.852(5)°$, $V = 1563.27(15)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0630$, $wR_{ref}(F^2) = 0.1733$, $T = 296(2)$ K.

CCDC no.: 1882603

Table 1: Data collection and handling.

Crystal:	Yellow needle
Size:	0.41 × 0.18 × 0.11 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.07 mm^{-1}
Diffractometer, scan mode:	SuperNova, ω
θ_{max}, completeness:	29.3°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	13184, 7367, 0.025
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3826
$N(param)_{refined}$:	361
Programs:	CrysAlis^{PRO} [1], SHELX [2, 3], WinGX/ORTEP [4]

The asymmetric unit containing two title molecules is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	x	y	z	U_{iso}*/U_{eq}
C1	0.2744(4)	0.34186(19)	0.65408(8)	0.0493(5)
C2	0.1171(4)	0.2772(2)	0.64710(9)	0.0595(6)
H2	0.018947	0.305060	0.614439	0.071*
C3	0.0918(4)	0.1667(2)	0.68751(9)	0.0579(6)
C4	0.2603(4)	0.1205(2)	0.73797(9)	0.0594(6)
H4A	0.390623	0.068031	0.726498	0.071*
H4B	0.191609	0.072401	0.769805	0.071*
C5	0.3416(4)	0.2209(2)	0.75957(8)	0.0535(6)
C6	0.4278(4)	0.3039(2)	0.70776(8)	0.0568(6)
H6A	0.450686	0.375950	0.719659	0.068*
H6B	0.574582	0.262738	0.697914	0.068*
C7	0.3026(4)	0.4450(2)	0.60875(8)	0.0554(6)
H7	0.194826	0.469767	0.578065	0.067*
C8	0.4672(4)	0.5077(2)	0.60672(8)	0.0555(6)
H8	0.571195	0.483510	0.638224	0.067*
C9	0.5051(4)	0.60955(19)	0.56131(8)	0.0497(5)
C10	0.6912(4)	0.6619(2)	0.56556(9)	0.0581(6)
H10	0.794451	0.628249	0.596904	0.070*
C11	0.7301(4)	0.7615(2)	0.52539(8)	0.0552(6)
H11	0.858003	0.793216	0.530164	0.066*
C12	0.5804(3)	0.81583(18)	0.47754(8)	0.0453(5)
C13	0.3969(4)	0.7599(2)	0.47133(8)	0.0533(5)
H13	0.297461	0.790704	0.439030	0.064*
C14	0.3617(4)	0.6605(2)	0.51207(8)	0.0544(6)
H14	0.238031	0.626080	0.506638	0.065*
C15	0.1459(5)	0.2910(2)	0.78725(10)	0.0736(7)
H15A	0.196655	0.354167	0.800858	0.110*
H15B	0.023400	0.325817	0.758861	0.110*
H15C	0.094160	0.237127	0.819473	0.110*
C16	0.5359(5)	0.1654(3)	0.80525(10)	0.0882(9)
H16A	0.659879	0.121094	0.788046	0.132*
H16B	0.586273	0.228594	0.818937	0.132*
H16C	0.483361	0.111658	0.837376	0.132*
C17	0.4440(4)	0.9799(2)	0.39285(9)	0.0667(7)
H17A	0.494278	1.049248	0.370081	0.100*
H17B	0.424846	0.926179	0.368330	0.100*
H17C	0.301838	1.005549	0.409562	0.100*
C18	0.7921(4)	0.9789(2)	0.44672(9)	0.0674(7)
H18A	0.786315	1.048500	0.415293	0.101*
H18B	0.775782	1.003931	0.482991	0.101*
H18C	0.935223	0.924481	0.447381	0.101*
C19	0.0877(4)	0.62862(18)	0.84842(7)	0.0440(5)
C20	−0.1187(4)	0.68426(19)	0.86141(8)	0.0505(5)
H20	−0.170642	0.658919	0.898525	0.061*
C21	−0.2640(4)	0.78153(19)	0.82052(9)	0.0519(5)
C22	−0.1729(4)	0.8214(2)	0.76112(8)	0.0583(6)
H22A	−0.093121	0.886449	0.761820	0.070*
H22B	−0.299764	0.853375	0.733088	0.070*
C23	−0.0124(4)	0.72067(19)	0.74071(8)	0.0521(5)
C24	0.1740(4)	0.6623(2)	0.78802(8)	0.0510(5)
H24A	0.261262	0.590010	0.778278	0.061*
H24B	0.275974	0.717886	0.788046	0.061*
C25	0.2293(4)	0.53755(18)	0.89265(8)	0.0472(5)
H25	0.164915	0.514840	0.928424	0.057*
C26	0.4425(4)	0.48296(18)	0.88733(8)	0.0469(5)
H26	0.504602	0.505591	0.851236	0.056*
C27	0.5901(3)	0.39242(18)	0.93094(8)	0.0435(5)
C28	0.5254(4)	0.34733(19)	0.98718(8)	0.0485(5)
H28	0.381357	0.378316	0.998367	0.058*
C29	0.6669(4)	0.25874(19)	1.02665(8)	0.0498(5)
H29	0.616797	0.231639	1.063794	0.060*
C30	0.8843(4)	0.20855(18)	1.01217(8)	0.0452(5)
C31	0.9543(4)	0.2566(2)	0.95626(8)	0.0546(5)
H31	1.100072	0.228037	0.945315	0.065*
C32	0.8093(4)	0.34526(19)	0.91768(8)	0.0524(5)
H32	0.860557	0.375025	0.880990	0.063*
C33	0.0969(5)	0.7734(3)	0.68429(9)	0.0819(8)
H33A	0.197655	0.709625	0.671686	0.123*
H33B	−0.019577	0.811790	0.654641	0.123*
H33C	0.180988	0.831917	0.691225	0.123*
C34	−0.1439(5)	0.6262(2)	0.72955(9)	0.0727(7)
H34A	−0.041809	0.563171	0.716728	0.109*
H34B	−0.211182	0.592288	0.764891	0.109*
H34C	−0.261129	0.664044	0.700026	0.109*
C36	1.2431(4)	0.0605(2)	1.03544(10)	0.0687(7)
H36A	1.313324	−0.001625	1.067941	0.103*
H36B	1.227208	0.025473	1.002734	0.103*
H36C	1.335978	0.120376	1.025354	0.103*
C37	0.9356(4)	0.0577(2)	1.10488(9)	0.0697(7)
H37A	1.054137	−0.004141	1.126425	0.105*
H37B	0.883344	0.116330	1.127566	0.105*
H37C	0.811587	0.021907	1.096468	0.105*
N1	0.6099(3)	0.91835(17)	0.43847(7)	0.0598(5)
N2	1.0223(3)	0.11629(17)	1.05089(7)	0.0597(5)
O1	−0.0552(3)	0.11073(16)	0.67974(7)	0.0827(6)
O2	−0.4503(3)	0.83117(16)	0.83340(6)	0.0750(5)

Source of material

The title compound was synthesized following the literature method [5] where *p*-dimethylaminobenzaldehyde (1 g, 6.702 mmol) and isophorone (1 g, 7.23 mmol) were mixed in 15 mL of [EtOH + 50% NaOH] and stirred at room temperature for 48 h. Then the reaction mixture was quenched with cold water to produce precipitate and the pH was adjusted to 7.5 before filtration. The filtered product was washed with cold water and cold ethanol and recrystalized from chloroform under slow evaporation.

Experimental details

The data collection was accomplished using CrysAlis^{PRO} software [1] at 296 K under the Mo Kα radiation. The structure solution was performed using SHELXS-2016/6 [2] and refined SHELXL-2016/6 [3], in-built with WinGX [4]. The figure was generated through ORTEP [4]. All the aromatic C—H hydrogen atoms were positioned geometrically with C—H = 0.93 Å and treated as riding atoms with U_{iso}(H) = 1.2 U_{eq}(C). Methyl and methylene hydrogen atoms were also

positioned geometrically with C—H = 0.96 Å for methyl and C—H = 0.97 Å for methylene and treated as riding atoms where U_{iso}(H) was set to 1.5 U_{eq}(C) for methyl and 1.2 U_{eq}(C) for methylene carbon atoms.

Comment

Nonlinear optic compounds have extensively been studied due to their use in optoelectronic devices [6, 7]. These compounds are composed with two types of chromophores where one acts as electron donor while other acts as electron acceptor (D–π–A). The sign π represents the electrons which are in conjugation between the D/A moieties [8]. Polyenes are among the best carriers which transfer the electrons between two chromophores. In this report we are reporting the crystal structure of a molecule which belongs to this family.

In the crystal structure of the title compound (*cf.* the figure), two independent molecules [A = (C1—C18) & B = (C19—C36)] have been observed with the same conformation. The structures of some related compounds were found in literature [9–11]. The geometry around the central double bond lying between the two rings is *trans* in both molecules. The cycloenone ring adopted half-chair conformation with the root mean square deviation of 0.1862(2) Å and 0.1864(2) Å for molecule A and B, respectively [12, 13]. The cyclohexenone rings adopted the half chair conformation with the root mean square deviations of the plane produced from the fitted atoms these rings are 0.1862(2) Å and 0.1864(1) Å for molecule A and B, respectively. The puckering parameters for these nonplanar rings are measured [where Q = 0.4566 Å, Θ = 54.63° & φ = 233.19° for molecule A and Q = 0.4566 Å, Θ = 54.63° & φ = 233.19° and molecule B]. The dihedral angle between the aromatic ring (C1—C6) and the mean plane cyclohexenone ring (C9—C14) is 15.0° in molecule A. While in molecule these two rings are twisted at dihedral angle of 12.1°. The plane produced from the fitted atoms of dimethylamino group is twisted by 10.3° and 11.567° with its parent ring in molecule A and B respectively.

Acknowledgements: This paper was funded by the Deanship of Scientific Research (DSR) at King Abdulaziz University, Jeddah, under grant no. RG-03-102-428. The authors, therefore, acknowledge with thanks DSR for technical and financial support.

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