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Crystal structure of (3*E*)-3-{(2*E*)-3-[4-(dimethylamino)phenyl]prop-2-enylidene}-3,4-dihydro-2*H*-chromen-4-one

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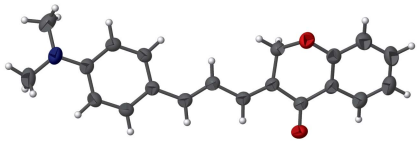
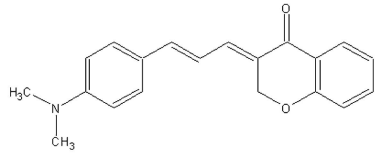
In the title compound, $C_{20}H_{19}NO_2$, the (dimethylamino)phenyl ring and the chromanone ring system are linked *via* an α-β unsaturated carbon bridge. The dihedral angle between the two terminal phenyl rings is 29.66 (6)°. The tetrahydro-4*H*-pyran-4 one ring in the chromanone moiety adopts a sofa conformation. The crystal packing is stabilized only by van der Waals forces.

3D view



Chemical scheme



Structure description

Chalcones are open chain flavonoids having a variety of biological activities, including antioxidant, anti-inflammation, antimicrobial, antiprotozoal and antiulcer properties (Dimmock *et al.*, 1999). More importantly, chalcones have also shown anticancer activity as inhibitors of cancer cell proliferation, carcinogenesis and metastasis (Zi & Simoneau, 2005). The Claisen–Schmidt condensation reaction between substituted acetophenones and aryl aldehydes under basic conditions has been widely used to synthesize chalcone derivatives (Robinson *et al.*, 2013; Tiwari *et al.*, 2010). As part of our studies in this area, the title compound (Fig. 1) was synthesized and its crystal structure determined.

The C—N distances in the (dimethylamino)phenyl moiety are in the range 1.366 (2) to 1.447 (2) Å and are comparable with the values reported for a similar structure (Adam *et al.*, 2015). The C—O and C=O distances in the chromanone moiety [1.365 (2)–1.441 (2) Å and 1.227 (2) Å, respectively] are typical of those in reported structures (Gopaul *et al.*, 2012). In the molecule, neither the α-β unsaturated carbon bridge nor the dimethylamino substituent are coplanar with their attached phenyl ring; the dihedral angle between the phenyl ring and the dimethylamino group is 10.3 (2)° while that

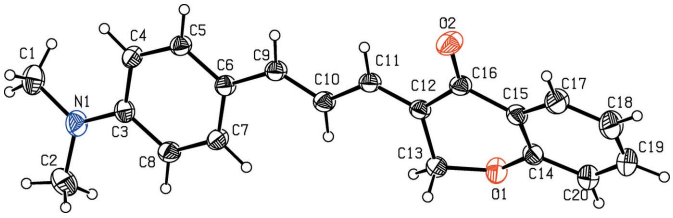


Figure 1
The molecular structure of the title compound, with displacement ellipsoids drawn at the 30% probability level. H atoms are shown as small circles of arbitrary radii.

between the α-β unsaturated carbon bridge and the phenyl ring is 8.58 (9)°. This is also true of the phenyl and tetrahydro-4*H*-pyran-4-one rings of the chromanone ring system, which make a dihedral angle of 6.2 (1)°. As a result of these dihedral angles which are all in the same direction, even though most of the title compound makes up a single conjugated system, there is a significant twist between the two end phenyl rings, which make a dihedral angle of 29.66 (6)°. The sum of the angles around the N atom is 359.61°, indicating sp^2 hybridization. The tetrahydro-4*H*-pyran-4-one ring in the chromanone moiety adopts a sofa conformation with atom C13 displaced from the other ring atoms by 0.5656 (15) Å and with puckering parameters of $q2$ = 0.3795 (15) Å, $\varphi2$ = 71.43 (2)°, $q3$ = 0.1835 (15) Å, Q_T = 0.4216 (14) Å and $\theta2$ = 64.20 (2)°.

It is well known that the ketone atom in chalcones is usually an active participant in hydrogen-bond formation or C—H···O interactions. However, in the present compound this is not the case and the crystal structure is stabilized only by van der Waals forces (Fig. 2).

Synthesis and crystallization

In a 250 ml round-bottomed flask, 4-chromanone (0.9 g, 0.006 mol) and 4-dimethylaminocinnamaldehyde (1 g, 0.006 mol) were added to absolute alcohol and stirred for 5 min. Then a solution of NaOH (0.3 g,10 ml) was added and stirred for 2 h. The mixture was kept overnight at room temperature and the precipitate was generated by adding a sufficient amount of crushed ice. The yield was filtered and washed with distilled water several times to remove any trace of NaOH remaining in the product. The crude chalcone derivative was recrystallized twice from ethyl methylketone to give red block-shaped diffraction-quality crystals of the title compound (yield 70%; m.p. 175°C).

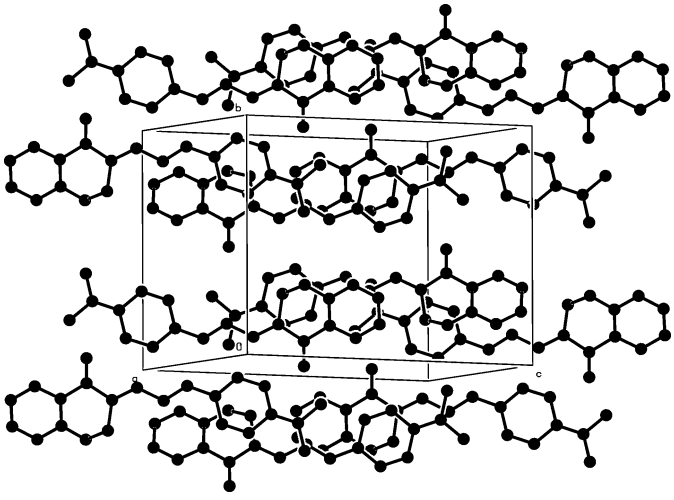


Figure 2
The packing of the molecules in the unit cell.

Table 1
Experimental details.

Crystal data	
Chemical formula	$C_{20}H_{19}NO_2$
M_r	305.36
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	296
a, b, c (Å)	9.3620 (4), 11.6073 (5), 15.0732 (7)
β (°)	98.859 (2)
V (Å^3)	1618.43 (12)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.08
Crystal size (mm)	0.20 × 0.20 × 0.15
Data collection	
Diffractometer	Bruker Kappa APEX3 CMOS
Absorption correction	Multi-scan (*SADABS*; Bruker, 2016)
T_{min}, T_{max}	0.984, 0.988
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	12086, 3331, 2487
R_{int}	0.037
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.625
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.044, 0.133, 1.05
No. of reflections	3275
No. of parameters	210
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.16, −0.14

Computer programs: *APEX3*, *SAINT* and *XPREP* (Bruker, 2016), *SHELXT2014/5* (Sheldrick, 2015*a*), *SHELXL2014/7* (Sheldrick, 2015*b*) and *ORTEP-3 for Windows* (Farrugia, 2012).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1.

Acknowledgements

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full crystallographic data

IUCrData (2018). **3**, x181273 [https://doi.org/10.1107/S2414314618012737]

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Crystal data

$C_{20}H_{19}NO_2$
M_r = 305.36
Monoclinic, $P2_1/c$
a = 9.3620 (4) Å
b = 11.6073 (5) Å
c = 15.0732 (7) Å
β = 98.859 (2)°
V = 1618.43 (12) Å^3
Z = 4
$F(000)$ = 648
D_x = 1.253 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 5210 reflections
θ = 3.2–26.4°
μ = 0.08 mm^{-1}
T = 296 K
Block, red
0.20 × 0.20 × 0.15 mm

Data collection

Bruker Kappa APEX3 CMOS diffractometer
ω and φ scan
Absorption correction: multi-scan (SADABS; Bruker, 2016)
T_{min} = 0.984, T_{max} = 0.988
12086 measured reflections
3331 independent reflections
2487 reflections with $I > 2\sigma(I)$
R_{int} = 0.037
θ_{max} = 26.4°, θ_{min} = 3.7°
h = −10→11
k = −14→14
l = −18→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.133
S = 1.05
3275 reflections
210 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0692P)^2 + 0.2609P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. H atoms were positioned geometrically and treated as riding on their parent atoms and refined with, C—H distances of 0.93–0.97 Å, with U_{iso}(H) = 1.5 U_{eq}(CH_3), and U_{iso}(H) = 1.2U_{eq}(C) for all other H atoms.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.0964 (2)	0.68888 (17)	0.79247 (11)	0.0640 (5)
H1A	0.0838	0.7306	0.8457	0.096*
H1B	0.1727	0.6335	0.8067	0.096*
H1C	0.0082	0.6497	0.7693	0.096*
C2	0.0885 (2)	0.88711 (17)	0.73183 (13)	0.0720 (5)
H2A	0.0361	0.8954	0.7814	0.108*
H2B	0.0275	0.9086	0.6772	0.108*
H2C	0.1723	0.9360	0.7409	0.108*
C3	0.19368 (15)	0.73066 (12)	0.65409 (9)	0.0419 (3)
C4	0.21389 (15)	0.61255 (12)	0.63836 (9)	0.0433 (3)
H4	0.1859	0.5583	0.6777	0.052*
C5	0.27444 (15)	0.57621 (12)	0.56564 (9)	0.0416 (3)
H5	0.2842	0.4975	0.5566	0.050*
C6	0.32198 (15)	0.65288 (11)	0.50480 (9)	0.0395 (3)
C7	0.30258 (16)	0.77064 (12)	0.52103 (9)	0.0438 (3)
H7	0.3331	0.8245	0.4823	0.053*
C8	0.24020 (16)	0.80844 (12)	0.59197 (9)	0.0459 (3)
H8	0.2280	0.8871	0.5996	0.055*
C9	0.38700 (15)	0.60951 (12)	0.43012 (9)	0.0430 (3)
H9	0.3857	0.5300	0.4227	0.052*
C10	0.44891 (16)	0.67015 (12)	0.37032 (9)	0.0437 (3)
H10	0.4570	0.7497	0.3767	0.052*
C11	0.50278 (16)	0.61550 (12)	0.29683 (9)	0.0427 (3)
H11	0.4934	0.5358	0.2935	0.051*
C12	0.56533 (15)	0.66574 (11)	0.23197 (8)	0.0394 (3)
C13	0.58723 (16)	0.79282 (12)	0.22502 (10)	0.0444 (3)
H13A	0.5089	0.8247	0.1825	0.053*
H13B	0.5830	0.8278	0.2830	0.053*
C14	0.74836 (15)	0.76685 (12)	0.12115 (9)	0.0425 (3)
C15	0.68871 (16)	0.65880 (12)	0.09684 (9)	0.0428 (3)
C16	0.60480 (16)	0.59667 (12)	0.15718 (9)	0.0432 (3)
C17	0.7188 (2)	0.60855 (15)	0.01730 (11)	0.0606 (4)
H17	0.6778	0.5378	−0.0010	0.073*
C18	0.8080 (2)	0.66229 (17)	−0.03405 (13)	0.0723 (5)
H18	0.8262	0.6284	−0.0871	0.087*
C19	0.8709 (2)	0.76745 (17)	−0.00662 (13)	0.0667 (5)
H19	0.9342	0.8024	−0.0403	0.080*
C20	0.84022 (17)	0.81996 (15)	0.06980 (11)	0.0553 (4)
H20	0.8810	0.8911	0.0871	0.066*
N1	0.13334 (15)	0.76841 (12)	0.72573 (9)	0.0556 (4)
O1	0.72288 (11)	0.82258 (9)	0.19689 (7)	0.0509 (3)
O2	0.57429 (15)	0.49431 (9)	0.14612 (8)	0.0648 (4)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0704 (11)	0.0793 (12)	0.0468 (9)	−0.0040 (9)	0.0230 (8)	−0.0043 (8)
C2	0.0828 (13)	0.0659 (11)	0.0727 (12)	0.0152 (9)	0.0286 (10)	−0.0171 (9)
C3	0.0403 (7)	0.0463 (8)	0.0395 (7)	−0.0012 (6)	0.0078 (6)	−0.0048 (6)
C4	0.0474 (8)	0.0423 (7)	0.0416 (7)	−0.0044 (6)	0.0115 (6)	0.0035 (6)
C5	0.0483 (8)	0.0347 (7)	0.0423 (7)	−0.0006 (5)	0.0088 (6)	0.0003 (5)
C6	0.0444 (7)	0.0382 (7)	0.0361 (7)	0.0009 (5)	0.0067 (6)	−0.0003 (5)
C7	0.0531 (8)	0.0384 (7)	0.0413 (7)	−0.0019 (6)	0.0114 (6)	0.0030 (6)
C8	0.0554 (9)	0.0352 (7)	0.0485 (8)	0.0007 (6)	0.0126 (7)	−0.0032 (6)
C9	0.0523 (8)	0.0385 (7)	0.0390 (7)	0.0036 (6)	0.0094 (6)	0.0009 (5)
C10	0.0532 (8)	0.0376 (7)	0.0416 (7)	0.0033 (6)	0.0109 (6)	−0.0006 (6)
C11	0.0545 (8)	0.0351 (7)	0.0397 (7)	0.0060 (6)	0.0107 (6)	0.0020 (5)
C12	0.0461 (8)	0.0350 (7)	0.0377 (7)	0.0058 (5)	0.0081 (6)	0.0000 (5)
C13	0.0515 (8)	0.0366 (7)	0.0477 (8)	0.0020 (6)	0.0162 (6)	−0.0028 (6)
C14	0.0393 (7)	0.0449 (7)	0.0438 (7)	0.0050 (6)	0.0079 (6)	0.0015 (6)
C15	0.0475 (8)	0.0417 (7)	0.0406 (7)	0.0078 (6)	0.0112 (6)	0.0004 (6)
C16	0.0548 (8)	0.0360 (7)	0.0402 (7)	0.0049 (6)	0.0112 (6)	−0.0002 (5)
C17	0.0826 (12)	0.0519 (9)	0.0526 (9)	0.0027 (8)	0.0271 (8)	−0.0065 (7)
C18	0.0941 (14)	0.0718 (12)	0.0598 (10)	0.0076 (10)	0.0401 (10)	−0.0050 (9)
C19	0.0667 (11)	0.0730 (12)	0.0680 (11)	0.0031 (9)	0.0346 (9)	0.0088 (9)
C20	0.0487 (9)	0.0566 (9)	0.0633 (10)	−0.0017 (7)	0.0171 (7)	0.0027 (7)
N1	0.0654 (8)	0.0558 (8)	0.0507 (7)	−0.0001 (6)	0.0251 (6)	−0.0083 (6)
O1	0.0528 (6)	0.0498 (6)	0.0527 (6)	−0.0102 (5)	0.0164 (5)	−0.0122 (5)
O2	0.1039 (10)	0.0350 (6)	0.0621 (7)	−0.0063 (5)	0.0341 (7)	−0.0080 (5)

Geometric parameters (Å, º)

C1—N1	1.446 (2)	C10—C11	1.4338 (19)
C1—H1A	0.9600	C10—H10	0.9300
C1—H1B	0.9600	C11—C12	1.3474 (19)
C1—H1C	0.9600	C11—H11	0.9300
C2—N1	1.447 (2)	C12—C16	1.4759 (18)
C2—H2A	0.9600	C12—C13	1.4951 (18)
C2—H2B	0.9600	C13—O1	1.4414 (18)
C2—H2C	0.9600	C13—H13A	0.9700
C3—N1	1.3658 (18)	C13—H13B	0.9700
C3—C4	1.409 (2)	C14—O1	1.3647 (17)
C3—C8	1.417 (2)	C14—C20	1.387 (2)
C4—C5	1.376 (2)	C14—C15	1.399 (2)
C4—H4	0.9300	C15—C17	1.400 (2)
C5—C6	1.3988 (19)	C15—C16	1.478 (2)
C5—H5	0.9300	C16—O2	1.2272 (17)
C6—C7	1.4054 (19)	C17—C18	1.373 (2)
C6—C9	1.4499 (19)	C17—H17	0.9300
C7—C8	1.367 (2)	C18—C19	1.390 (3)
C7—H7	0.9300	C18—H18	0.9300

C8—H8	0.9300	C19—C20	1.372 (2)
C9—C10	1.3434 (19)	C19—H19	0.9300
C9—H9	0.9300	C20—H20	0.9300
N1—C1—H1A	109.5	C12—C11—H11	116.1
N1—C1—H1B	109.5	C10—C11—H11	116.1
H1A—C1—H1B	109.5	C11—C12—C16	120.46 (12)
N1—C1—H1C	109.5	C11—C12—C13	123.88 (12)
H1A—C1—H1C	109.5	C16—C12—C13	115.42 (12)
H1B—C1—H1C	109.5	O1—C13—C12	113.18 (11)
N1—C2—H2A	109.5	O1—C13—H13A	108.9
N1—C2—H2B	109.5	C12—C13—H13A	108.9
H2A—C2—H2B	109.5	O1—C13—H13B	108.9
N1—C2—H2C	109.5	C12—C13—H13B	108.9
H2A—C2—H2C	109.5	H13A—C13—H13B	107.8
H2B—C2—H2C	109.5	O1—C14—C20	117.34 (13)
N1—C3—C4	121.86 (13)	O1—C14—C15	122.01 (13)
N1—C3—C8	121.65 (13)	C20—C14—C15	120.61 (14)
C4—C3—C8	116.49 (13)	C14—C15—C17	118.23 (14)
C5—C4—C3	120.99 (13)	C14—C15—C16	120.32 (12)
C5—C4—H4	119.5	C17—C15—C16	121.31 (13)
C3—C4—H4	119.5	O2—C16—C12	123.32 (13)
C4—C5—C6	122.62 (13)	O2—C16—C15	121.56 (13)
C4—C5—H5	118.7	C12—C16—C15	115.10 (12)
C6—C5—H5	118.7	C18—C17—C15	120.89 (16)
C5—C6—C7	116.23 (12)	C18—C17—H17	119.6
C5—C6—C9	120.14 (12)	C15—C17—H17	119.6
C7—C6—C9	123.63 (12)	C17—C18—C19	119.88 (16)
C8—C7—C6	122.04 (13)	C17—C18—H18	120.1
C8—C7—H7	119.0	C19—C18—H18	120.1
C6—C7—H7	119.0	C20—C19—C18	120.36 (16)
C7—C8—C3	121.62 (13)	C20—C19—H19	119.8
C7—C8—H8	119.2	C18—C19—H19	119.8
C3—C8—H8	119.2	C19—C20—C14	119.94 (16)
C10—C9—C6	127.97 (13)	C19—C20—H20	120.0
C10—C9—H9	116.0	C14—C20—H20	120.0
C6—C9—H9	116.0	C3—N1—C1	121.17 (14)
C9—C10—C11	121.74 (13)	C3—N1—C2	120.99 (14)
C9—C10—H10	119.1	C1—N1—C2	117.45 (14)
C11—C10—H10	119.1	C14—O1—C13	114.07 (11)
C12—C11—C10	127.81 (13)		
N1—C3—C4—C5	179.78 (13)	C11—C12—C16—O2	5.5 (2)
C8—C3—C4—C5	−0.6 (2)	C13—C12—C16—O2	−169.17 (14)
C3—C4—C5—C6	1.4 (2)	C11—C12—C16—C15	−172.74 (12)
C4—C5—C6—C7	−1.0 (2)	C13—C12—C16—C15	12.58 (18)
C4—C5—C6—C9	179.05 (12)	C14—C15—C16—O2	−166.30 (14)
C5—C6—C7—C8	−0.3 (2)	C17—C15—C16—O2	9.4 (2)

C9—C6—C7—C8	179.70 (13)	C14—C15—C16—C12	11.97 (19)
C6—C7—C8—C3	1.1 (2)	C17—C15—C16—C12	−172.32 (13)
N1—C3—C8—C7	179.00 (13)	C14—C15—C17—C18	1.9 (3)
C4—C3—C8—C7	−0.6 (2)	C16—C15—C17—C18	−173.87 (15)
C5—C6—C9—C10	−173.96 (14)	C15—C17—C18—C19	0.7 (3)
C7—C6—C9—C10	6.1 (2)	C17—C18—C19—C20	−2.4 (3)
C6—C9—C10—C11	−176.87 (13)	C18—C19—C20—C14	1.4 (3)
C9—C10—C11—C12	179.21 (14)	O1—C14—C20—C19	179.38 (14)
C10—C11—C12—C16	−175.39 (13)	C15—C14—C20—C19	1.3 (2)
C10—C11—C12—C13	−1.2 (2)	C4—C3—N1—C1	4.3 (2)
C11—C12—C13—O1	141.69 (13)	C8—C3—N1—C1	−175.22 (14)
C16—C12—C13—O1	−43.83 (17)	C4—C3—N1—C2	−168.28 (15)
O1—C14—C15—C17	179.08 (13)	C8—C3—N1—C2	12.2 (2)
C20—C14—C15—C17	−2.9 (2)	C20—C14—O1—C13	154.73 (13)
O1—C14—C15—C16	−5.1 (2)	C15—C14—O1—C13	−27.24 (18)
C20—C14—C15—C16	172.89 (13)	C12—C13—O1—C14	51.19 (16)