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Crystal structures, syntheses, and spectroscopic and electrochemical measurements of two push–pull chromophores: 2-[4-(dimethylamino)benzylidene]-1*H*-indene-1,3(2*H*)-dione and (*E*)-2-{3-[4-(dimethylamino)phenyl]allylidene}-1*H*-indene-1,3(2*H*)-dione

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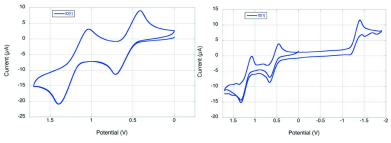
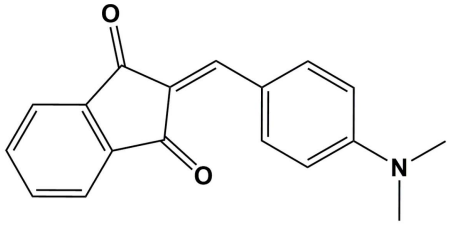
The title pull–push chromophores, 2-[4-(dimethylamino)benzylidene]-1*H*-indene-1,3(2*H*)-dione, $C_{18}H_{15}NO_2$ (ID[1]) and (*E*)-2-{3-[4-(dimethylamino)-phenyl]allylidene}-1*H*-indene-1,3(2*H*)-dione, $C_{20}H_{17}NO_2$ (ID[2]), have donor–π-bridge–acceptor structures. The molecule with the short π-bridge, ID[1], is almost planar while for the molecule with a longer bridge, ID[2], is less planar. The benzene ring is inclined to the mean plane of the 2,3-dihydro-1*H*-indene unit by 3.19 (4)° in ID[1] and 13.06 (8)° in ID[2]. The structures of three polymorphs of compound ID[1] have been reported: the α-polymorph [space group $P2_1/c$; Magomedova & Zvonkova (1978). *Kristallografiya*, **23**, 281–288], the β-polymorph [space group $P2_1/c$; Magomedova & Zvonkova (1980). *Kristallografiya*, **25** 1183–1187] and the γ-polymorph [space group $Pna2_1$; Magomedova, Neigauz, Zvonkova & Novakovskaya (1980). *Kristallografiya*, **25**, 400–402]. The molecular packing in ID[1] studied here is centrosymmetric (space group $P2_1/c$) and corresponds to the β-polymorph structure. The molecular packing in ID[2] is non-centrosymmetric (space group $P2_1$), which suggests potential NLO properties for this crystalline material. In both compounds, there is short intramolecular C—H···O contact present, enclosing an *S*(7) ring motif. In the crystal of ID[1], molecules are linked by C—H···O hydrogen bonds and C—H···π interactions, forming layers parallel to the *bc* plane. In the crystal of ID[2], molecules are liked by C—H···O hydrogen bonds to form 2_1 helices propagating along the *b*-axis direction. The molecules in the helix are linked by offset π–π interactions with, for example, a centroid–centroid distance of 3.9664 (13) Å (= *b* axis) separating the indene rings, and an offset of 1.869 Å. Spectroscopic and electrochemical measurements show the ability of these compounds to easily transfer electrons through the π-conjugated chain.

1. Chemical context

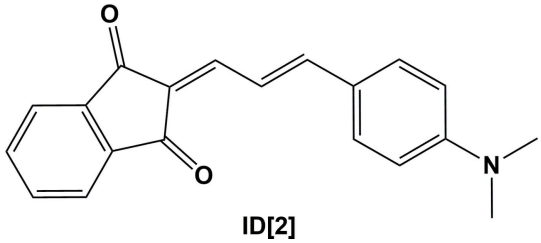
Organic molecules containing donor and acceptor groups connected by a conjugated π-bridge (push–pull chromophores) are important in many areas of materials chemistry, especially organic electronics and optoelectronics. Applications of pull–push molecules can be related to their properties such as intramolecular charge transfer and specific molecular arrangements in the solid state. Intramolecular charge transfer from donor to acceptor *via* a π-bridge defines their colour, light absorption and emission, hyperpolarizability and other optoelectronic effects. Applications of pull–push chromophores include non-linear optics (NLO; Ortiz *et al.*,



1994), as luminescent sensors (Duarte *et al.*, 2011; Qin *et al.*, 2015), solid-state lasers (Samuel & Turnbull, 2007), organic light-emitting diodes (Muller *et al.*, 2003), organic field-effect transistors (Suponitsky *et al.*, 2006; Oliveira *et al.*, 2018) and many more. The spectroscopic properties of pull–push molecules are related to the donor and acceptor strength in these molecules and to the length of the π-bridge. Many such compounds have been studied, but not all of their crystal structures have been reported. Such compounds are important for their NLO properties (Andreu *et al.*, 2003; Raimundo *et al.*, 2002). Herein, we report on the crystal structures, syntheses and spectroscopic and electrochemical properties of the title donor–π-bridge–acceptor structures, ID[1] and ID[2]. The structures of three polymorphs of ID[1] have been reported previously; the α-polymorph (Magomedova & Zvonkova, 1978), the β-polymorph (Magomedova & Zvonkova, 1980) and the γ-polymorph (Magomedova, Neigauz *et al.*, 1980). We have repeated the structural study of ID[1] in order to establish exactly which polymorph we obtained. It was then characterized by spectroscopic and electrochemical measurements.



ID[1]



ID[2]

2. Structural commentary

The molecular structures of ID[1] and ID[2] are illustrated in Fig. 1. The structural analysis of ID[1] synthesized by us established that it is the β-polymorph (Magomedova & Zvonkova, 1980), and it was then characterized with spectroscopic (§4) and electrochemical (§5) measurements.

Both molecules have acceptor–π-bridge–donor structures. It was found, as in our previous studies (Tillotson *et al.*, 2019), that with an increase of the length of the π-conjugated bridge the molecule becomes less planar, and the angles between the different planar fragments (acceptor–bridge, bridge–donor) become larger (Table 1).

Compound ID[1] has an almost planar structure, with the benzene ring (C10–C15) being inclined to the mean plane of the indene ring system (C1–C9) by 3.19 (4)°. In ID[2] the deviation from planarity is somewhat larger with the benzene ring (C10–C15) being inclined to the mean plane of the indene ring system (C1–C9) by 13.06 (8)°; see further details in Table 1.

Table 1
Dihedral angles between molecular fragments (°) and mean deviations (Å) of atoms from these fragments.

	ID[1]	ID[2]
Acceptor–bridge	1.31 (11)	11.6 (2)
Bridge–donor	2.63 (11)	4.9 (3)
Deviation in acceptor	0.0325	0.0159
Deviation in bridge	0.0000	0.0473
Deviation in donor	0.0035	0.0073

Table 2
Hydrogen-bond geometry (Å, °) for ID[1].

*Cg*2 and *Cg*3 are the centroids of the C4–C9 and C10–C15 rings, respectively.

D—H···A	D—H	H···A	D···A	D—H···A
C14—H14···O1	0.95	2.17	3.0143 (12)	147
C7—H7···O3^{i}	0.95	2.58	3.4813 (14)	159
C11—H11···O1^{ii}	0.95	2.37	3.2778 (11)	160
C16—H16*A*···*Cg*3^{iii}	0.98	2.91	3.7948 (12)	151
C17—H17*A*···*Cg*2^{iv}	0.98	2.83	3.6591 (13)	143

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

3. Supramolecular features

Molecules of ID[1] and ID[2] have significant dipole moments, which is very common for NLO chromophores. Because of this, molecules have a trend to antiparallel packing, which is observed in the crystal structures of both ID[1] and ID[2].

In the crystal of ID[1], molecules form two almost perpendicular stacks with anangle of *ca* 84.47° between them. The molecules, which stack in an antiparallel or head-to-tail

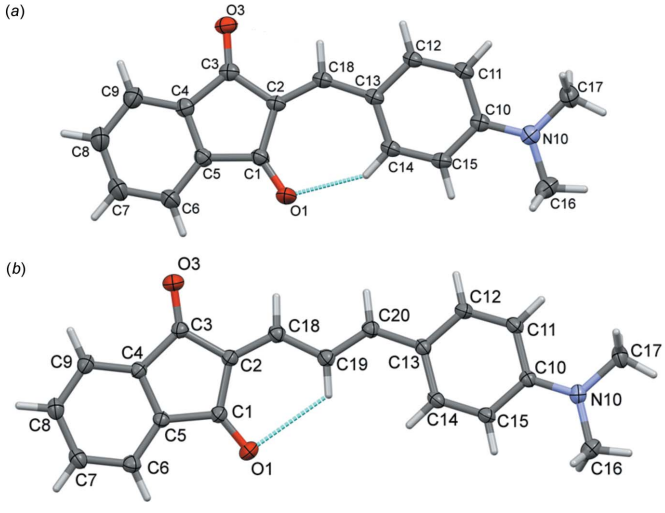


Figure 1
A view of the molecular structure of the title compounds, (*a*) ID[1] and (*b*) ID[2], with atom labelling. Displacement ellipsoids are drawn at the 50% probability level. The intramolecular C—H···O hydrogen bonds (Tables 2 and 3) are shown as dashed lines.

Table 3
Hydrogen-bond geometry (Å, °) for ID[2].

D—H···A	D—H	H···A	D···A	D—H···A
C19—H19···O1	0.95	2.51	3.118 (3)	122
C17—H17C···O3^{i}	0.98	2.50	3.463 (3)	169

Symmetry code: (i) $-x+1, y+\frac{1}{2}, -z+1$.

fashion, are linked by C—H···O hydrogen bonds (Fig. 2, Table 2), forming layers lying parallel to the *bc* plane. Within the layers there are C—H···π interactions present (Table 2).

In the crystal of ID[2], molecules form stacks with parallel molecular positions, and shifted positions of stacks extended along the *b*-axis direction within the acentric space group $P2_1$. The ID[2] molecules are packed in a herringbone fashion (Fig. 3). Here, the angle between two molecules from different stacks is *ca* 60.8°. The molecules are linked by C—H···O hydrogen bonds (Table 3), forming a 2_1 helix that propagates along the *b*-axis direction. The molecules in the helix are linked by offset π–π interactions with, for example, a centroid–centroid distance $Cg1\cdots Cg1^{i}$ of 3.9664 (13) Å [symmetry code: (i) $x, y-1, z$] separating the indene ring systems (C1–C9), with an offset of 1.869 Å.

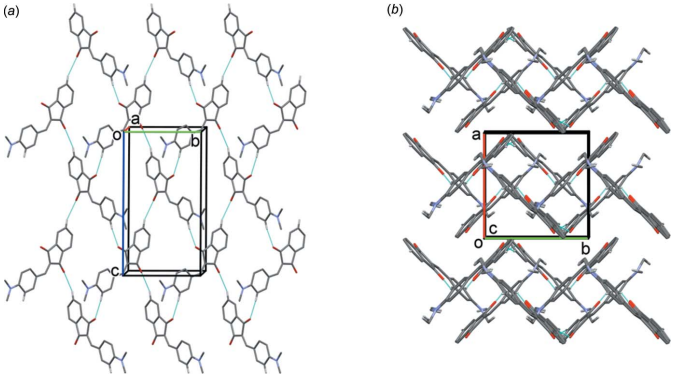


Figure 2
Views along (*a*) the *a* axis and (*b*) the *c* axis of the crystal packing of ID[1]. The hydrogen bonds (Table 2) are shown as dashed lines. For clarity, only the H atoms involved in the intermolecular interactions have been included.

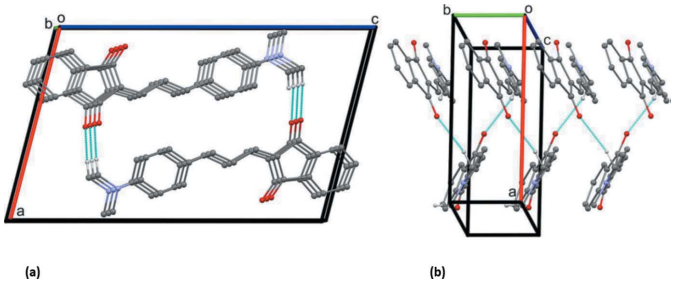


Figure 3
Views along (*a*) the *b* axis and (*b*) the *c* axis of the crystal packing of ID[2]. The hydrogen bonds (Table 3) are shown as dashed lines. For clarity, only the H atoms involved in the intermolecular interactions have been included.

Table 4
Absorption maxima (nm) for ID[1] and ID[2] in chloroform and acetonitrile.

Solvent	ID[1]	ID[2]
Chloroform	483	539
Acetonitrile	480	526

4. Spectroscopic studies

Absorbance spectra were obtained for both ID[1] and ID[2] in chloroform and acetonitrile. For donor–acceptor polyenes, the dominating feature of the absorbance spectrum is the π–π^* transition that results from charge transfer from donor to acceptor. According to recent studies (Bogdanov *et al.*, 2019), it proves that dimethylaminophenyl polyenals have reversed solvatochromism, which is proved by the maxima of the absorption values (Table 4), showing that both ID[1] and ID[2] have their peaks higher in chloroform than in acetonitrile; see the absorption spectra of ID[1] and ID[2] in acetonitrile given in Fig. 4.

5. Electrochemical measurements

Donor–acceptor polyenes can be characterized by electrochemical measurements to show their ability to transfer electrons. The voltammagrams (Fig. 5) demonstrate a completely reversible oxidation process and a partially reversible reduction process. When only swept between 0 V and 1.7 V the oxidation process is reversible (Fig. 5*a*),

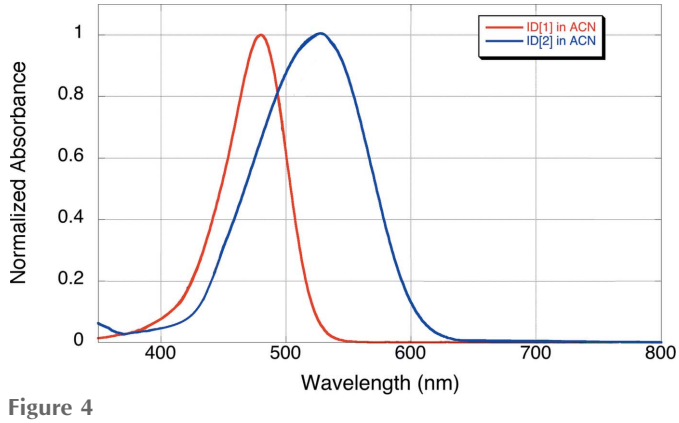


Figure 4
Normalized absorbance spectra (nm) in acetonitrile for ID[1] and ID[2].

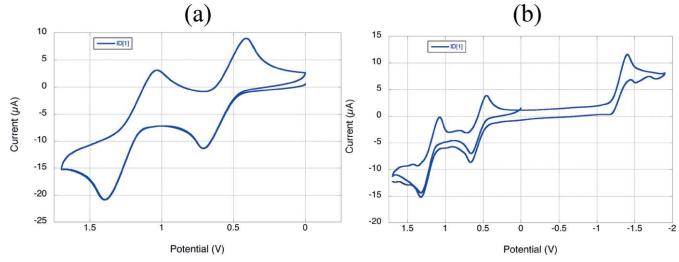


Figure 5
Cyclic voltammagrams of ID[1]: (*a*) sweep from 0 to 1.7 V and (*b*) sweep from −1.9 to 1.7 V.

Table 5
Experimental details.

	ID[1]	ID[2]
Crystal data		
Chemical formula	$C_{18}H_{15}NO_2$	$C_{20}H_{17}NO_2$
M_r	277.31	303.35
Crystal system, space group	Monoclinic, $P2_1/c$	Monoclinic, $P2_1$
Temperature (K)	150	150
a, b, c (Å)	9.2298 (9), 9.0302 (9), 16.7375 (17)	11.072 (2), 3.9664 (8), 17.557 (4)
β (°)	97.863 (1)	104.500 (2)
V (Å^3)	1381.9 (2)	746.5 (3)
Z	4	2
Radiation type	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	0.09	0.09
Crystal size (mm)	0.40 × 0.20 × 0.15	0.40 × 0.12 × 0.10
Data collection		
Diffractometer	Bruker APEXII CCD	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Krause *et al.*, 2015)	Multi-scan (*SADABS*; Krause *et al.*, 2015)
T_{min}, T_{max}	0.673, 0.746	0.584, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	15794, 4391, 3812	8685, 4531, 4358
R_{int}	0.041	0.044
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.741	0.739
Refinement		
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.047, 0.139, 1.07	0.044, 0.121, 1.12
No. of reflections	4391	4531
No. of parameters	192	210
No. of restraints	0	1
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.27, −0.40	0.22, −0.33
Absolute structure	–	Flack x determined using 1732 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter	–	−0.1 (5)

Computer programs: *APEX3* and *SAINT* (Bruker, 2015), *SHELXT2017/1* (Sheldrick, 2015*a*), *SHELXL2018/3* (Sheldrick, 2015*b*), *Mercury* (Macrae *et al.*, 2008), *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2010).

however, when swept to −1.9 V the reduction is only partly reversible (Fig. 5*b*). This represents the ability of the compound to 'easily' transfer electrons through the chain from donor towards acceptor.

Note: cyclic voltammagrams of ID[1] were made against $FeCp_2^{+/0}$ (internal reference $E_{1/2}^{+/0}$ = 0.55 V *vs* Ag/AgCl) in dichloromethane with 0.1 *M* ^{n}Bu$_4$NPF$_6$). Measurements were recorded at 50 mV s^{-1} using a BAS Potentiostat using a glassy carbon working electrode, Pt wire auxilliary electrode and a Ag/AgCl reference electrode.

6. Database survey

A search of the Cambridge Structural Database (CSD Version 5.40, update May 2019; Groom *et al.*, 2016) for the substructure of ID[1] yielded 27 hits. Three of them, 2-(*p*-diethylaminobenzylidene)-1,3-indandione (CSD refcode TELWEM; Khodorkovsky *et al.*, 1996), which has ethyl groups instead of methyl groups in the donor, 2-{[4-(diphenylamino)phenyl]methylidene}-1*H*-indene-1,3(2*H*)-dione (QENYEQ at 223 K: Hariharan *et al.*, 2018; QENYEQ01 at 150 K: Redon *et al.*, 2018), which has methyl groups in the donor, and 2-{[4-(dibutylamino)phenyl]methylidene}-1*H*-indene-1,3(2*H*)-dione (BIQYUY; Situ *et al.*, 2019), which has butyl groups in the donor. In these three compounds, the benzene ring is inclined to the mean plane of the 2,3-dihydro-1*H*-indene ring system by 7.6 (2), 1.66 (6)/1.49 (9) and 5.71 (9)°, for TELWEN, QENYEQ/QENYEQ01 and BIQYUY, respectively, compared to 3.19 (4)° in ID[1].

Also, out of all 27 hits there are three hits, (MBYINO: Magomedova *et al.*, 1978; MBYINO01: Magomedova, Neigauz *et al.*, 1980; MBYINO02: Magomedova & Zvonkova, 1980), which are the α, γ and β ID[1] polymorphs, respectively, published over 40 years ago. It should be mentioned that the crystal packing in the α and β polymorphs is centric (space group $P2_1/c$), while in the γ polymorph it is acentric (space group $Pna2_1$). The crystal structure of ID[1] we obtained corresponds to the β polymorph, *i.e.* the centrosymmetric modification MBYINO02. The dihedral angles between the benzene and indene rings for two independent molecules in MBYINO are *ca* 4.35 and 7.79°, compared to *ca* 7.36° in MBYINO01, and 3.54° in MBYINO02 (*cf.* 3.19 (4)° in the present crystal structure analysis of ID[1]).

A search of the CSD for the substructure of ID[2] yielded nine hits. Only two structures are similar to that of ID[2]. The first, 2-{3-[4-(dimethylamino)phenyl]prop-2-yn-1-ylidene}-1*H*-indene-1,3(2*H*)-dione (ZIGPIR; Solanke *et al.*, 2018), has a triple bond between atoms C18 and C19. The second, 2-[4-(dimethylamino)cinnamoyl]indan-1,3-dione (CNINDO; Magomedova, Zvonkova *et al.*, 1980) has a hydroxyl group attached to atom C18. Like ID[2], it crystallizes in the chiral monoclinic space group $P2_1$. The benzene ring is inclined to

the mean plane of the indene ring system by *ca* 11.42° in CNINDO compared to 13.06 (8)° in ID[2]. In ZIGPIR this dihedral angle is smaller at 8.6 (3)°.

7. Synthesis and crystallization

For the synthesis of the title compounds, two aldehydes were used: 4-(dimethylamino)benzaldehyde (A1; purchased from Aldrich) and 4-(dimethylamino)cinnamaldehyde (A2), which was synthesized as described previously (Tillotson, *et al.*, 2019). 2,3-Indanedione was purchased from Aldrich and used without further purification.

Synthesis of 2-[4-(dimethylamino)benzylidene]indane-1,3-dione (ID[1]): Aldehyde A1 (2.00 g, 13.4 mmol) and 1,3-indanedione (2.01 g, 13.4 mmol) were suspended in 100 ml of absolute ethanol. The mixture was gently heated until the solids had dissolved. After about 10 min of stirring the dissolution was complete, and a red crystalline precipitate began forming on the walls of the flask. The reaction mixture was stirred vigorously overnight, and the resulting product was collected by filtration then washed with cold ethanol and hexanes to give shiny dark-red crystals (yield 3.65 g, 98%; m.p. 477–478 K). ID[1] can be purified by recrystallization using numerous solvent systems (acetone, ethanol, ethyl acetate/hexane, dichloromethane/hexane and toluene (to name a few), many of which afforded single crystals. ^{1}H NMR (400 MHz, CD_2Cl_2) δ 8.52 (*d*, *J* = 9.16 Hz, 2H), 7.91–9.73 (*m*, 4H), 7.71 (*s*, 1H), 6.77 (*d*, *J* = 9.16 HZ, 2H), 3.14 (*s*, 6H) ppm. ^{13}C NMR (100 MHz, CD_2Cl_2) δ 191.6, 190.1, 154.5, 147.3, 142.7, 140.3, 138.2, 134.8, 134.5, 123.3, 122.7, 122.6, 122.2, 111.7, 40.3 ppm.

Synthesis of (*E*)-2-{3-[4-(dimethylamino)phenyl]allylidene}indane-1,3-dione (ID[2]): Aldehyde A2 (1.00 g, 5.71 mmol), 1,3-indanedione (0.85 g, 5.7 mmol) and piperidine (0.15 ml, 1.4 mmol) in ethanol (50 ml) were mixed and treated as for the synthesis of ID[1]. The crude product obtained was collected by filtration and washed with cold ethanol before being recrystallized from ethanol to give incredibly shiny and thin purple actinic crystals (1.55 g, 90%; m.p. 535–537 K). They were washed with hexane and dried under vacuum. ^{1}H NMR (400 MHz, CD_2Cl_2) δ 8.25 (*dd*, *J* = 15.0, 12.2 Hz, 1H), 7.90–7.86 (*m*, 2H), 7.76–7.73 (*m*, 2H), 7.60 (*d*, *J* = 12.2 Hz, 1H), 7.60 (*d*, *J* = 9.0 Hz, 2H), 7.34 (*d*, *J* = 15.0 Hz, 1H), 6.72 (*d*, *J* = 9.0 Hz, 2H), 3.08 (*s*, 6H) ppm.

8. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 5. For both structures, the C-bound hydrogen atoms were positioned geometrically and refined using a riding model: C—H = 0.95–0.98 Å with U_{iso}(H) = 1.5U_{eq}(C-methyl) and 1.2U_{iso}(C) for other H atoms.

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Crystal structures, syntheses, and spectroscopic and electrochemical measurements of two push–pull chromophores: 2-[4-(dimethylamino)benzylidene]-1*H*-indene-1,3(2*H*)-dione and (*E*)-2-{3-[4-(dimethylamino)phenyl]allylidene}-1*H*-indene-1,3(2*H*)-dione

Georgii Bogdanov, John P. Tillotson and Tatiana Timofeeva

Computing details

For both structures, data collection: *APEX3* (Bruker, 2015); cell refinement: *SAINT* (Bruker, 2015); data reduction: *SAINT* (Bruker, 2015); program(s) used to solve structure: *SHELXT2017/1* (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2018/3* (Sheldrick, 2015b); molecular graphics: *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXL2018/3* (Sheldrick, 2015b), *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2010).

2-[4-(Dimethylamino)benzylidene]-1*H*-indene-1,3(2*H*)-dione (ID1)

Crystal data

$C_{18}H_{15}NO_2$
M_r = 277.31
Monoclinic, $P2_1/c$
a = 9.2298 (9) Å
b = 9.0302 (9) Å
c = 16.7375 (17) Å
β = 97.863 (1)°
V = 1381.9 (2) Å^3
Z = 4
$F(000)$ = 584
D_x = 1.333 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 8518 reflections
θ = 2.2–31.5°
μ = 0.09 mm^{-1}
T = 150 K
Block, purple
0.40 × 0.20 × 0.15 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
φ and ω scans
Absorption correction: multi-scan (SADABS; Krause *et al.*, 2015)
T_{min} = 0.673, T_{max} = 0.746
15794 measured reflections
4391 independent reflections
3812 reflections with $I > 2\sigma(I)$
R_{int} = 0.041
θ_{max} = 31.8°, θ_{min} = 2.2°
h = −13→12
k = −12→12
l = −23→23

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.139
S = 1.07
4391 reflections
192 parameters
0 restraints
Primary atom site location: dual

Secondary atom site location: difference Fourier map	$w = 1/[\sigma^2(F_o^2) + (0.0835P)^2 + 0.2073P]$ where $P = (F_o^2 + 2F_c^2)/3$
Hydrogen site location: inferred from neighbouring sites	$(\Delta/\sigma)_{max} < 0.001$
H-atom parameters constrained	$\Delta\rho_{max} = 0.27$ e Å^{-3}
	$\Delta\rho_{min} = -0.40$ 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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O3	0.93689 (8)	0.32542 (8)	0.50819 (4)	0.03149 (17)
O1	0.60774 (9)	0.60246 (9)	0.32291 (4)	0.0379 (2)
N10	0.31173 (9)	0.97003 (9)	0.60039 (5)	0.02873 (18)
C13	0.60837 (9)	0.65851 (10)	0.52818 (5)	0.02205 (17)
C2	0.75196 (9)	0.49196 (9)	0.44122 (5)	0.02184 (17)
C18	0.71343 (9)	0.55144 (10)	0.51060 (5)	0.02284 (17)
H18	0.769381	0.512603	0.557817	0.027*
C4	0.89040 (10)	0.33908 (10)	0.36262 (5)	0.02526 (18)
C12	0.60627 (10)	0.69512 (10)	0.61024 (5)	0.02429 (18)
H12	0.673418	0.647010	0.649979	0.029*
C3	0.86960 (9)	0.37921 (10)	0.44692 (5)	0.02369 (18)
C11	0.51125 (10)	0.79732 (10)	0.63494 (5)	0.02506 (18)
H11	0.514728	0.819276	0.690677	0.030*
C15	0.40888 (10)	0.83354 (10)	0.49501 (5)	0.02435 (18)
H15	0.341038	0.880551	0.455185	0.029*
C14	0.50520 (10)	0.73202 (10)	0.47141 (5)	0.02373 (18)
H14	0.502565	0.710598	0.415658	0.028*
C10	0.40801 (9)	0.87012 (9)	0.57750 (5)	0.02255 (17)
C1	0.70060 (10)	0.51650 (10)	0.35504 (5)	0.02429 (18)
C5	0.78862 (10)	0.41613 (10)	0.30928 (5)	0.02495 (18)
C6	0.78078 (12)	0.39789 (12)	0.22630 (6)	0.0329 (2)
H6	0.710143	0.449691	0.190125	0.039*
C9	0.98955 (12)	0.24303 (13)	0.33465 (7)	0.0347 (2)
H9	1.059397	0.190603	0.370999	0.042*
C7	0.87982 (14)	0.30121 (14)	0.19785 (7)	0.0392 (3)
H7	0.876810	0.286448	0.141453	0.047*
C17	0.31815 (13)	1.01545 (13)	0.68433 (7)	0.0369 (2)
H17A	0.297002	0.930205	0.717083	0.055*
H17B	0.245649	1.093342	0.688545	0.055*
H17C	0.416109	1.053349	0.703830	0.055*
C16	0.19759 (12)	1.03450 (12)	0.54226 (7)	0.0367 (2)
H16A	0.242318	1.089217	0.501365	0.055*
H16B	0.138187	1.102198	0.570043	0.055*
H16C	0.135358	0.955511	0.516197	0.055*

C8	0.98325 (14)	0.22601 (14)	0.25148 (7)	0.0409 (3)
H8	1.050850	0.161833	0.230903	0.049*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O3	0.0289 (3)	0.0367 (4)	0.0275 (3)	0.0027 (3)	−0.0011 (3)	0.0068 (3)
O1	0.0502 (5)	0.0424 (4)	0.0201 (3)	0.0190 (3)	0.0011 (3)	0.0033 (3)
N10	0.0287 (4)	0.0296 (4)	0.0275 (4)	0.0028 (3)	0.0026 (3)	−0.0008 (3)
C13	0.0230 (4)	0.0253 (4)	0.0174 (3)	−0.0038 (3)	0.0009 (3)	0.0006 (3)
C2	0.0224 (4)	0.0237 (4)	0.0190 (4)	−0.0023 (3)	0.0015 (3)	0.0023 (3)
C18	0.0233 (4)	0.0263 (4)	0.0183 (3)	−0.0032 (3)	0.0003 (3)	0.0023 (3)
C4	0.0244 (4)	0.0261 (4)	0.0258 (4)	−0.0015 (3)	0.0052 (3)	0.0014 (3)
C12	0.0254 (4)	0.0291 (4)	0.0174 (3)	−0.0012 (3)	−0.0007 (3)	0.0010 (3)
C3	0.0214 (4)	0.0257 (4)	0.0236 (4)	−0.0035 (3)	0.0017 (3)	0.0027 (3)
C11	0.0277 (4)	0.0288 (4)	0.0178 (3)	−0.0024 (3)	0.0003 (3)	−0.0015 (3)
C15	0.0246 (4)	0.0280 (4)	0.0194 (4)	−0.0021 (3)	−0.0008 (3)	0.0034 (3)
C14	0.0253 (4)	0.0281 (4)	0.0172 (3)	−0.0032 (3)	0.0008 (3)	0.0012 (3)
C10	0.0224 (4)	0.0221 (4)	0.0228 (4)	−0.0046 (3)	0.0017 (3)	0.0006 (3)
C1	0.0291 (4)	0.0251 (4)	0.0187 (4)	0.0003 (3)	0.0031 (3)	0.0013 (3)
C5	0.0277 (4)	0.0256 (4)	0.0218 (4)	−0.0005 (3)	0.0047 (3)	0.0006 (3)
C6	0.0401 (5)	0.0365 (5)	0.0224 (4)	0.0059 (4)	0.0056 (4)	0.0000 (4)
C9	0.0308 (5)	0.0378 (5)	0.0363 (5)	0.0076 (4)	0.0072 (4)	0.0026 (4)
C7	0.0470 (6)	0.0444 (6)	0.0283 (5)	0.0076 (5)	0.0126 (4)	−0.0028 (4)
C17	0.0393 (5)	0.0389 (5)	0.0333 (5)	0.0040 (4)	0.0079 (4)	−0.0076 (4)
C16	0.0306 (5)	0.0324 (5)	0.0449 (6)	0.0041 (4)	−0.0026 (4)	0.0035 (4)
C8	0.0427 (6)	0.0441 (6)	0.0385 (5)	0.0122 (5)	0.0145 (5)	−0.0021 (5)

Geometric parameters (Å, º)

O3—C3	1.2243 (11)	C15—C14	1.3722 (13)
O1—C1	1.2249 (11)	C15—C10	1.4207 (12)
N10—C10	1.3578 (12)	C15—H15	0.9500
N10—C16	1.4543 (13)	C14—H14	0.9500
N10—C17	1.4570 (13)	C1—C5	1.4961 (13)
C13—C14	1.4152 (12)	C5—C6	1.3907 (13)
C13—C12	1.4155 (12)	C6—C7	1.3941 (15)
C13—C18	1.4281 (12)	C6—H6	0.9500
C2—C18	1.3700 (12)	C9—C8	1.3939 (16)
C2—C1	1.4721 (11)	C9—H9	0.9500
C2—C3	1.4820 (12)	C7—C8	1.3941 (17)
C18—H18	0.9500	C7—H7	0.9500
C4—C9	1.3879 (13)	C17—H17A	0.9800
C4—C5	1.3907 (13)	C17—H17B	0.9800
C4—C3	1.4943 (13)	C17—H17C	0.9800
C12—C11	1.3751 (13)	C16—H16A	0.9800
C12—H12	0.9500	C16—H16B	0.9800
C11—C10	1.4190 (12)	C16—H16C	0.9800

C11—H11	0.9500	C8—H8	0.9500
C10—N10—C16	121.29 (9)	C11—C10—C15	117.27 (8)
C10—N10—C17	121.15 (8)	O1—C1—C2	129.63 (8)
C16—N10—C17	117.56 (9)	O1—C1—C5	123.74 (8)
C14—C13—C12	116.40 (8)	C2—C1—C5	106.61 (7)
C14—C13—C18	126.35 (8)	C6—C5—C4	121.34 (9)
C12—C13—C18	117.25 (8)	C6—C5—C1	128.71 (8)
C18—C2—C1	133.27 (8)	C4—C5—C1	109.93 (8)
C18—C2—C3	119.19 (8)	C5—C6—C7	117.92 (9)
C1—C2—C3	107.54 (7)	C5—C6—H6	121.0
C2—C18—C13	134.62 (8)	C7—C6—H6	121.0
C2—C18—H18	112.7	C4—C9—C8	117.80 (10)
C13—C18—H18	112.7	C4—C9—H9	121.1
C9—C4—C5	120.99 (9)	C8—C9—H9	121.1
C9—C4—C3	130.22 (9)	C6—C7—C8	120.56 (10)
C5—C4—C3	108.79 (8)	C6—C7—H7	119.7
C11—C12—C13	122.85 (8)	C8—C7—H7	119.7
C11—C12—H12	118.6	N10—C17—H17A	109.5
C13—C12—H12	118.6	N10—C17—H17B	109.5
O3—C3—C2	127.57 (8)	H17A—C17—H17B	109.5
O3—C3—C4	125.36 (9)	N10—C17—H17C	109.5
C2—C3—C4	107.06 (7)	H17A—C17—H17C	109.5
C12—C11—C10	120.27 (8)	H17B—C17—H17C	109.5
C12—C11—H11	119.9	N10—C16—H16A	109.5
C10—C11—H11	119.9	N10—C16—H16B	109.5
C14—C15—C10	121.68 (8)	H16A—C16—H16B	109.5
C14—C15—H15	119.2	N10—C16—H16C	109.5
C10—C15—H15	119.2	H16A—C16—H16C	109.5
C15—C14—C13	121.53 (8)	H16B—C16—H16C	109.5
C15—C14—H14	119.2	C9—C8—C7	121.37 (10)
C13—C14—H14	119.2	C9—C8—H8	119.3
N10—C10—C11	121.43 (8)	C7—C8—H8	119.3
N10—C10—C15	121.31 (8)		
C1—C2—C18—C13	0.93 (17)	C12—C11—C10—C15	−0.50 (13)
C3—C2—C18—C13	−179.71 (9)	C14—C15—C10—N10	−179.59 (8)
C14—C13—C18—C2	2.59 (16)	C14—C15—C10—C11	0.04 (13)
C12—C13—C18—C2	−177.54 (9)	C18—C2—C1—O1	1.45 (18)
C14—C13—C12—C11	−0.66 (13)	C3—C2—C1—O1	−177.97 (10)
C18—C13—C12—C11	179.46 (8)	C18—C2—C1—C5	179.75 (9)
C18—C2—C3—O3	2.96 (14)	C3—C2—C1—C5	0.33 (9)
C1—C2—C3—O3	−177.53 (9)	C9—C4—C5—C6	1.13 (15)
C18—C2—C3—C4	−178.21 (8)	C3—C4—C5—C6	−178.70 (9)
C1—C2—C3—C4	1.30 (9)	C9—C4—C5—C1	−177.34 (9)
C9—C4—C3—O3	−3.52 (16)	C3—C4—C5—C1	2.83 (10)
C5—C4—C3—O3	176.28 (9)	O1—C1—C5—C6	−1.89 (16)
C9—C4—C3—C2	177.62 (10)	C2—C1—C5—C6	179.68 (10)

C5—C4—C3—C2	−2.58 (10)	O1—C1—C5—C4	176.43 (9)
C13—C12—C11—C10	0.83 (14)	C2—C1—C5—C4	−2.00 (10)
C10—C15—C14—C13	0.11 (13)	C4—C5—C6—C7	−0.91 (16)
C12—C13—C14—C15	0.18 (13)	C1—C5—C6—C7	177.25 (10)
C18—C13—C14—C15	−179.95 (8)	C5—C4—C9—C8	−0.25 (16)
C16—N10—C10—C11	−174.40 (9)	C3—C4—C9—C8	179.53 (10)
C17—N10—C10—C11	4.97 (14)	C5—C6—C7—C8	−0.15 (18)
C16—N10—C10—C15	5.21 (14)	C4—C9—C8—C7	−0.81 (18)
C17—N10—C10—C15	−175.41 (9)	C6—C7—C8—C9	1.0 (2)
C12—C11—C10—N10	179.13 (8)		

Hydrogen-bond geometry (Å, º)

*Cg*2 and *Cg*3 are the centroids of the C4–C9 and C10–C15 rings, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C14—H14···O1	0.95	2.17	3.0143 (12)	147
C7—H7···O3^{i}	0.95	2.58	3.4813 (14)	159
C11—H11···O1^{ii}	0.95	2.37	3.2778 (11)	160
C16—H16*A*···*Cg*3^{iii}	0.98	2.91	3.7948 (12)	151
C17—H17*A*···*Cg*2^{iv}	0.98	2.83	3.6591 (13)	143

Symmetry codes: (i) *x*, −*y*+1/2, *z*−1/2; (ii) *x*, −*y*+3/2, *z*+1/2; (iii) −*x*+1, −*y*+2, −*z*+1; (iv) −*x*+1, −*y*+1, −*z*+1.

(*E*)-2-{3-[4-(Dimethylamino)phenyl]allylidene}-1*H*-indene-1,3(2*H*)-dione (ID2)

Crystal data

$C_{20}H_{17}NO_2$
M_r = 303.35
Monoclinic, $P2_1$
a = 11.072 (2) Å
b = 3.9664 (8) Å
c = 17.557 (4) Å
β = 104.500 (2)°
V = 746.5 (3) Å^3
Z = 2
F(000) = 320
D_x = 1.350 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 6074 reflections
θ = 2.4–31.6°
μ = 0.09 mm^{-1}
T = 150 K
Needle, blue
0.40 × 0.12 × 0.10 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
φ and *ω* scans
Absorption correction: multi-scan (SADABS; Krause *et al.*, 2015)
T_{min} = 0.584, T_{max} = 0.746
8685 measured reflections
4531 independent reflections
4358 reflections with $I > 2\sigma(I)$
R_{int} = 0.044
θ_{max} = 31.7°, θ_{min} = 1.2°
h = −15→16
k = −5→5
l = −24→25

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.121
S = 1.12
4531 reflections
210 parameters
1 restraint
Primary atom site location: dual
Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0488P)^2 + 0.2055P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.22$ e Å^{-3}
$\Delta\rho_{min} = -0.33$ e Å^{-3}
Absolute structure: Flack *x* determined using 1732 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter: −0.1 (5)

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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O3	0.50946 (14)	0.2510 (5)	0.17850 (9)	0.0331 (4)
O1	0.11093 (14)	0.5986 (5)	0.20367 (9)	0.0328 (4)
N10	0.15603 (16)	0.3906 (5)	0.69990 (9)	0.0253 (4)
C13	0.29025 (17)	0.2553 (5)	0.49784 (10)	0.0203 (3)
C10	0.20054 (16)	0.3470 (5)	0.63500 (10)	0.0202 (3)
C12	0.35740 (17)	0.1388 (5)	0.57237 (11)	0.0232 (4)
H12	0.434333	0.025205	0.576557	0.028*
C9	0.37083 (18)	0.5984 (6)	0.02319 (11)	0.0250 (4)
H9	0.450310	0.528451	0.017745	0.030*
C11	0.31564 (18)	0.1834 (5)	0.63920 (11)	0.0236 (4)
H11	0.364293	0.103946	0.688397	0.028*
C7	0.16966 (19)	0.8643 (6)	−0.03035 (11)	0.0276 (4)
H7	0.113386	0.972479	−0.073153	0.033*
C14	0.17745 (18)	0.4244 (5)	0.49465 (11)	0.0224 (4)
H14	0.130688	0.510783	0.445647	0.027*
C19	0.29104 (17)	0.3097 (5)	0.35537 (11)	0.0230 (4)
H19	0.211433	0.414940	0.342126	0.028*
C18	0.35755 (17)	0.2675 (5)	0.29653 (10)	0.0222 (3)
H18	0.433577	0.144955	0.311130	0.027*
C20	0.33964 (17)	0.2017 (5)	0.43054 (11)	0.0222 (4)
H20	0.414971	0.076184	0.439446	0.027*
C17	0.2246 (2)	0.2557 (6)	0.77578 (11)	0.0294 (4)
H17A	0.236951	0.012715	0.770933	0.044*
H17B	0.176965	0.296073	0.815026	0.044*
H17C	0.305856	0.367609	0.792316	0.044*
C8	0.2871 (2)	0.7601 (6)	−0.03803 (11)	0.0273 (4)
H8	0.309669	0.800923	−0.085963	0.033*
C6	0.13502 (17)	0.8102 (5)	0.03978 (11)	0.0247 (4)
H6	0.056140	0.882629	0.045770	0.030*
C16	0.03720 (19)	0.5566 (6)	0.69463 (13)	0.0283 (4)
H16A	0.040550	0.787428	0.675361	0.043*
H16B	0.020234	0.562374	0.746783	0.043*
H16C	−0.029316	0.431904	0.658184	0.043*

C4	0.33549 (16)	0.5416 (5)	0.09239 (10)	0.0200 (3)
C15	0.13295 (17)	0.4683 (5)	0.56017 (11)	0.0224 (4)
H15	0.055820	0.581423	0.555547	0.027*
C3	0.40481 (17)	0.3712 (5)	0.16627 (10)	0.0216 (3)
C5	0.21913 (16)	0.6474 (5)	0.10052 (10)	0.0195 (3)
C2	0.32319 (17)	0.3854 (5)	0.22113 (10)	0.0212 (3)
C1	0.20501 (17)	0.5508 (5)	0.18022 (11)	0.0219 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O3	0.0219 (6)	0.0463 (10)	0.0311 (7)	0.0089 (7)	0.0068 (5)	0.0039 (7)
O1	0.0235 (6)	0.0485 (10)	0.0297 (7)	0.0070 (7)	0.0128 (5)	0.0051 (7)
N10	0.0264 (8)	0.0304 (9)	0.0201 (7)	0.0016 (7)	0.0078 (6)	0.0028 (7)
C13	0.0197 (7)	0.0221 (8)	0.0182 (7)	−0.0011 (7)	0.0029 (6)	0.0026 (6)
C10	0.0196 (7)	0.0211 (8)	0.0190 (7)	−0.0023 (7)	0.0035 (6)	0.0013 (6)
C12	0.0187 (7)	0.0278 (9)	0.0219 (8)	0.0035 (7)	0.0028 (6)	0.0038 (7)
C9	0.0241 (8)	0.0292 (10)	0.0240 (8)	−0.0017 (8)	0.0103 (7)	−0.0015 (7)
C11	0.0214 (8)	0.0286 (10)	0.0189 (7)	0.0020 (7)	0.0011 (6)	0.0041 (7)
C7	0.0290 (9)	0.0305 (10)	0.0208 (8)	−0.0019 (8)	0.0017 (7)	0.0038 (8)
C14	0.0203 (8)	0.0245 (9)	0.0198 (7)	−0.0002 (7)	0.0003 (6)	0.0041 (7)
C19	0.0212 (8)	0.0267 (9)	0.0206 (7)	−0.0009 (7)	0.0046 (6)	0.0010 (7)
C18	0.0217 (8)	0.0237 (9)	0.0206 (7)	−0.0007 (7)	0.0038 (6)	−0.0001 (7)
C20	0.0208 (8)	0.0242 (9)	0.0213 (8)	−0.0009 (7)	0.0046 (6)	0.0008 (7)
C17	0.0351 (10)	0.0348 (11)	0.0176 (8)	−0.0032 (9)	0.0051 (7)	0.0012 (8)
C8	0.0315 (9)	0.0313 (10)	0.0198 (8)	−0.0033 (8)	0.0076 (7)	0.0005 (7)
C6	0.0198 (8)	0.0288 (10)	0.0243 (8)	−0.0002 (7)	0.0035 (6)	0.0026 (7)
C16	0.0257 (9)	0.0299 (10)	0.0320 (10)	−0.0005 (8)	0.0121 (7)	−0.0022 (8)
C4	0.0186 (7)	0.0222 (8)	0.0191 (7)	−0.0014 (7)	0.0047 (6)	−0.0026 (7)
C15	0.0185 (8)	0.0261 (9)	0.0206 (8)	0.0021 (7)	0.0012 (6)	0.0032 (7)
C3	0.0187 (7)	0.0255 (9)	0.0206 (7)	0.0003 (7)	0.0045 (6)	−0.0011 (7)
C5	0.0173 (7)	0.0221 (8)	0.0186 (7)	−0.0013 (6)	0.0037 (6)	−0.0010 (6)
C2	0.0189 (7)	0.0254 (9)	0.0190 (7)	0.0001 (7)	0.0042 (6)	−0.0007 (7)
C1	0.0195 (7)	0.0266 (9)	0.0201 (7)	−0.0001 (7)	0.0059 (6)	0.0002 (7)

Geometric parameters (Å, º)

O3—C3	1.221 (2)	C19—C20	1.364 (3)
O1—C1	1.227 (2)	C19—C18	1.421 (2)
N10—C10	1.361 (2)	C19—H19	0.9500
N10—C16	1.453 (3)	C18—C2	1.365 (3)
N10—C17	1.460 (3)	C18—H18	0.9500
C13—C14	1.406 (3)	C20—H20	0.9500
C13—C12	1.411 (2)	C17—H17A	0.9800
C13—C20	1.437 (2)	C17—H17B	0.9800
C10—C11	1.416 (3)	C17—H17C	0.9800
C10—C15	1.423 (2)	C8—H8	0.9500
C12—C11	1.376 (3)	C6—C5	1.387 (3)

C12—H12	0.9500	C6—H6	0.9500
C9—C4	1.385 (2)	C16—H16A	0.9800
C9—C8	1.389 (3)	C16—H16B	0.9800
C9—H9	0.9500	C16—H16C	0.9800
C11—H11	0.9500	C4—C5	1.396 (2)
C7—C6	1.395 (3)	C4—C3	1.493 (3)
C7—C8	1.402 (3)	C15—H15	0.9500
C7—H7	0.9500	C3—C2	1.478 (2)
C14—C15	1.371 (3)	C5—C1	1.496 (2)
C14—H14	0.9500	C2—C1	1.479 (3)

C10—N10—C16	121.07 (16)	H17A—C17—H17B	109.5
C10—N10—C17	120.19 (17)	N10—C17—H17C	109.5
C16—N10—C17	118.67 (16)	H17A—C17—H17C	109.5
C14—C13—C12	116.72 (16)	H17B—C17—H17C	109.5
C14—C13—C20	123.71 (16)	C9—C8—C7	121.16 (18)
C12—C13—C20	119.57 (17)	C9—C8—H8	119.4
N10—C10—C11	121.65 (16)	C7—C8—H8	119.4
N10—C10—C15	120.86 (17)	C5—C6—C7	118.06 (18)
C11—C10—C15	117.49 (16)	C5—C6—H6	121.0
C11—C12—C13	122.48 (17)	C7—C6—H6	121.0
C11—C12—H12	118.8	N10—C16—H16A	109.5
C13—C12—H12	118.8	N10—C16—H16B	109.5
C4—C9—C8	118.26 (18)	H16A—C16—H16B	109.5
C4—C9—H9	120.9	N10—C16—H16C	109.5
C8—C9—H9	120.9	H16A—C16—H16C	109.5
C12—C11—C10	120.33 (16)	H16B—C16—H16C	109.5
C12—C11—H11	119.8	C9—C4—C5	120.77 (17)
C10—C11—H11	119.8	C9—C4—C3	129.72 (17)
C6—C7—C8	120.39 (18)	C5—C4—C3	109.52 (15)
C6—C7—H7	119.8	C14—C15—C10	121.01 (17)
C8—C7—H7	119.8	C14—C15—H15	119.5
C15—C14—C13	121.95 (17)	C10—C15—H15	119.5
C15—C14—H14	119.0	O3—C3—C2	127.48 (18)
C13—C14—H14	119.0	O3—C3—C4	126.02 (17)
C20—C19—C18	121.06 (18)	C2—C3—C4	106.49 (15)
C20—C19—H19	119.5	C6—C5—C4	121.36 (16)
C18—C19—H19	119.5	C6—C5—C1	129.12 (16)
C2—C18—C19	126.54 (18)	C4—C5—C1	109.51 (15)
C2—C18—H18	116.7	C18—C2—C3	123.48 (17)
C19—C18—H18	116.7	C18—C2—C1	128.36 (17)
C19—C20—C13	127.60 (18)	C3—C2—C1	108.14 (15)
C19—C20—H20	116.2	O1—C1—C2	128.88 (18)
C13—C20—H20	116.2	O1—C1—C5	124.79 (17)
N10—C17—H17A	109.5	C2—C1—C5	106.31 (15)
N10—C17—H17B	109.5		

C16—N10—C10—C11	−179.48 (19)	C5—C4—C3—O3	−179.7 (2)

C17—N10—C10—C11	−2.5 (3)	C9—C4—C3—C2	179.4 (2)
C16—N10—C10—C15	1.1 (3)	C5—C4—C3—C2	−1.0 (2)
C17—N10—C10—C15	178.13 (19)	C7—C6—C5—C4	0.3 (3)
C14—C13—C12—C11	0.5 (3)	C7—C6—C5—C1	−178.4 (2)
C20—C13—C12—C11	179.7 (2)	C9—C4—C5—C6	0.4 (3)
C13—C12—C11—C10	0.9 (3)	C3—C4—C5—C6	−179.24 (18)
N10—C10—C11—C12	179.1 (2)	C9—C4—C5—C1	179.36 (18)
C15—C10—C11—C12	−1.5 (3)	C3—C4—C5—C1	−0.3 (2)
C12—C13—C14—C15	−1.4 (3)	C19—C18—C2—C3	171.6 (2)
C20—C13—C14—C15	179.5 (2)	C19—C18—C2—C1	−6.9 (4)
C20—C19—C18—C2	−175.0 (2)	O3—C3—C2—C18	1.7 (3)
C18—C19—C20—C13	173.3 (2)	C4—C3—C2—C18	−176.93 (19)
C14—C13—C20—C19	2.4 (3)	O3—C3—C2—C1	−179.5 (2)
C12—C13—C20—C19	−176.7 (2)	C4—C3—C2—C1	1.9 (2)
C4—C9—C8—C7	0.1 (3)	C18—C2—C1—O1	−4.8 (4)
C6—C7—C8—C9	0.7 (3)	C3—C2—C1—O1	176.5 (2)
C8—C7—C6—C5	−0.9 (3)	C18—C2—C1—C5	176.7 (2)
C8—C9—C4—C5	−0.6 (3)	C3—C2—C1—C5	−2.0 (2)
C8—C9—C4—C3	179.0 (2)	C6—C5—C1—O1	1.7 (3)
C13—C14—C15—C10	0.8 (3)	C4—C5—C1—O1	−177.2 (2)
N10—C10—C15—C14	−179.92 (19)	C6—C5—C1—C2	−179.7 (2)
C11—C10—C15—C14	0.7 (3)	C4—C5—C1—C2	1.4 (2)
C9—C4—C3—O3	0.7 (4)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C19—H19···O1	0.95	2.51	3.118 (3)	122
C17—H17*C*···O3^{i}	0.98	2.50	3.463 (3)	169

Symmetry code: (i) −*x*+1, *y*+1/2, −*z*+1.