Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

1-(4-Aminophenyl)-3-(3,4,5-trimethoxyphenyl)prop-2-en-1-one–1-(4-aminophenyl)-3-(3-bromo-4,5-trimethoxyphenyl)prop-2-en-1-one (0.972/0.028)

William T. A. Harrison,[a]* H. J. Ravindra,[b] M. R. Suresh Kumar[b] and S. M. Dharmaprakash[b]

[a]Department of Chemistry, University of Aberdeen, Meston Walk, Aberdeen AB24 3UE, Scotland, and [b]Department of Physics, Mangalore University, Mangalagangotri 574 199, India
Correspondence e-mail: w.harrison@abdn.ac.uk

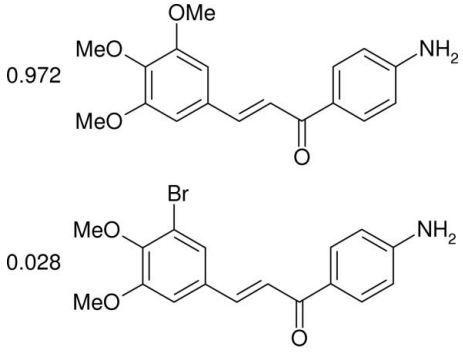
Received 23 August 2007; accepted 25 August 2007

Key indicators: single-crystal X-ray study; T = 291 K; mean σ(C–C) = 0.004 Å; disorder in main residue; R factor = 0.036; wR factor = 0.098; data-to-parameter ratio = 7.2.

In the title cocrystal, $0.972(C_{18}H_{19}NO_4).0.028(C_{17}H_{16}BrNO_3)$, which arose from an impure starting material, all the atoms are overlapped except for one OMe group and the Br atom. The dihedral angle between the benzene ring mean planes is 18.20 (13)°. A weak N—H···O hydrogen bond helps to establish the non-centrosymmetric crystal packing.

Related literature

For background, see: Uchida *et al.* (1998); Harrison *et al.* (2007); Ravindra *et al.* (2007). For reference structural data, see: Allen *et al.* (1995).



Experimental

Crystal data

$0.972C_{18}H_{19}NO_4 \cdot 0.028C_{17}H_{16}BrNO_3$
M_r = 314.81
Monoclinic, *Pn*
a = 4.2227 (4) Å
b = 12.2001 (12) Å
c = 15.4865 (16) Å
β = 97.506 (2)°
V = 790.99 (14) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.17 mm^{-1}
T = 291 (2) K
0.55 × 0.45 × 0.40 mm

Data collection

Bruker SMART1000 CCD diffractometer
Absorption correction: none
5129 measured reflections
1540 independent reflections
1380 reflections with $I > 2\sigma(I)$
R_{int} = 0.024

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.098
S = 1.04
1540 reflections
215 parameters
2 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H5B···O1^{i}	0.86	2.21	2.948 (3)	143

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

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

HJR and SMD thank DAE–BRNS for financial assistance.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2192).

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  doi:10.1107/S1600536807041840

supplementary materials

Acta Cryst. (2007). E**63**, o3970 [**doi:10.1107/S1600536807041840**]

1-(4-Aminophenyl)-3-(3,4,5-trimethoxyphenyl)prop-2-en-1-one-1-(4-aminophenyl)-3-(3-bromo-4,5-trimethoxyphenyl)prop-2-en-1-one (0.972/0.028)

W. T. A. Harrison, H. J. Ravindra, M. R. S. Kumar and S. M. Dharmaprakash

Comment

As part of our on-going studies of chalcone derivatives as possible non-linear optical materials (Uchida *et al.*, 1998; Harrison *et al.*, 2007), we now report the synthesis and structure of the non-centrosymmetric title co-crystal, (I) + (II), (Fig. 1). All the atoms are superimposed in the crystal, except for O4/C18/H18A/H18B/H18C in (I) and Br1 in (II). A similar co-crystal was recently described (Ravindra *et al.*, 2007).

The bond lengths and angles for (I) + (II) are normal (Allen *et al.*, 1995). The C16 and C18 atoms are almost co-planar with the C1–C6 benzene ring, whereas C17 is significantly displaced, by 1.026 (6) Å. The dihedral angle between two benzene rings (C1–C6 and C10–C15) is 18.20 (13)°. The central enone fragment (C7/C8/C9/O1) makes dihedral angles of 7.8 (3)° and 11.6 (3)° with C1–C6 and C10–C15, respectively.

The non-centrosymmetric packing for (I) + (II) is consolidated by a weak N—H···O hydrogen bond (Table 1), resulting in C(8) chains propagating in [10$\bar{1}$].

Experimental

A Claisen–Schmidt condensation reaction was used: a solution of ethanol (20 ml) and 10% aqueous sodium hydroxide (5 ml) solution were taken in a conical flask. A previously prepared portion of 3,4,5-trimethoxy benzaldehyde (0.001 mol) and 1-(4-aminophenyl)ethanone (0.001 mol) dissolved in ethanol (25 ml) was added to the conical flask with stirring and the temperature of the solution was maintained between 293 and 298 K. A precipitate was obtained after stirring the solution for about five minutes. The remaining portion of the aldehyde and ketone mixture was added and the solution was stirred for about 60 minutes. The solid product was filtered and washed with excess water to remove the alkali and dried. Yellow blocks of (I) + (II) were grown by slow evaporation of an acetone solution. The 3,4,5-trimethoxybenzaldehyde starting material was contaminated with some 3-bromo-4,5-dimethoxybenzaldehyde, resulting also in the formation of (II). The presence of the Br atom in the starting material as well as the product was confirmed by performing the halogen test.

Refinement

Due to negligible amonalous scattering, Friedel pairs were merged before refinement.

After initial modelling as the expected compound (I), high residuals (wR2 > 0.15) and a significant difference peak in the vicinity of O4 and C18 remained. The separation of the peak and C2 suggested the presence of a C—Br bond (Ravindra *et al.*, 2007). Refinement as a co-crystal of (I) + (II) (occupancies of the –O4—C18 and –Br1 groups/atoms attached to C2 refined with their sum constrained to unity) converged to a physically plausible answer with lower residuals.

The hydrogen atoms were placed in calculated positions (N—H = 0.86 Å, C—H = 0.95–0.99 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(C, N) or 1.5U_{eq}(methyl C). The methyl groups were allowed to rotate but not to tip to best fit the electron density.

Figures

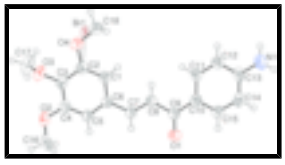

Fig. 1. View of the molecular structure of (I) + (II) showing 50% displacement ellipsoids (H atoms are drawn as spheres of arbitrary radius). The Br atom of (II) is connected to the benzene ring with a dashed line.

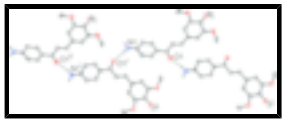

Fig. 2. Part of a hydrogen-bonded chain in (I) + (II) with hydrogen bonds shown as dashed lines. All the C-bound hydrogen atoms and Br1 omitted for clarity. Symmetry code as in Table 1; additionally (ii) $x + 1, y, z - 1$.

1-(4-Aminophenyl)-3-(3,4,5-trimethoxyphenyl)prop-2-en-1-one–1-(4- aminophenyl)-3-(3-bromo-4,5-trimethoxyphenyl)prop-2-en-1-one (0.972/0.028)

Crystal data

$0.972C_{18}H_{19}NO_4 \cdot 0.028C_{17}H_{16}BrNO_3$	$F_{000} = 333$
$M_r = 314.81$	$D_x = 1.322$ Mg m^{-3}
Monoclinic, *Pn*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: P -2yac	Cell parameters from 3137 reflections
a = 4.2227 (4) Å	θ = 5.0–26.0º
b = 12.2001 (12) Å	μ = 0.17 mm^{-1}
c = 15.4865 (16) Å	*T* = 291 (2) K
β = 97.506 (2)º	Block, yellow
V = 790.99 (14) Å^3	0.55 × 0.45 × 0.40 mm
Z = 2	

Data collection

Bruker SMART1000 CCD diffractometer	1380 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{int} = 0.024$
Monochromator: graphite	$\theta_{max} = 26.0$º
T = 291(2) K	$\theta_{min} = 5.0$º
ω scans	*h* = −5→5
Absorption correction: none	*k* = −13→15
5129 measured reflections	*l* = −19→19
1540 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map

Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.036$	H-atom parameters constrained
$wR(F^2) = 0.098$	$w = 1/[\sigma^2(F_o^2) + (0.0633P)^2 + 0.0717P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.04$	$(\Delta/\sigma)_{max} < 0.001$
1540 reflections	$\Delta\rho_{max} = 0.15$ e Å^{-3}
215 parameters	$\Delta\rho_{min} = -0.16$ e Å^{-3}
2 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

Special details

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

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
C1	0.6830 (7)	0.6305 (2)	0.59663 (17)	0.0456 (6)	
H1A	0.7560	0.6177	0.5434	0.055*	
C2	0.6978 (6)	0.5477 (2)	0.65815 (18)	0.0474 (6)	
C3	0.5932 (6)	0.5659 (2)	0.73839 (17)	0.0448 (6)	
C4	0.4792 (6)	0.6690 (2)	0.75739 (16)	0.0459 (6)	
C5	0.4595 (7)	0.7520 (2)	0.69540 (17)	0.0442 (6)	
H5	0.3800	0.8204	0.7080	0.053*	
C6	0.5592 (6)	0.7328 (2)	0.61415 (16)	0.0421 (6)	
C7	0.5229 (7)	0.8205 (2)	0.54870 (17)	0.0444 (6)	
H7	0.4230	0.8843	0.5639	0.053*	
C8	0.6162 (7)	0.8188 (2)	0.47081 (17)	0.0484 (6)	
H8	0.7331	0.7587	0.4559	0.058*	
C9	0.5449 (7)	0.9074 (2)	0.40564 (16)	0.0461 (6)	
C10	0.6270 (6)	0.8898 (2)	0.31701 (15)	0.0424 (6)	
C11	0.8102 (7)	0.8023 (2)	0.29464 (16)	0.0460 (6)	
H11	0.8890	0.7521	0.3373	0.055*	
C12	0.8783 (7)	0.7880 (2)	0.21047 (18)	0.0515 (6)	
H12	1.0016	0.7286	0.1974	0.062*	
C13	0.7639 (7)	0.8615 (2)	0.14522 (17)	0.0484 (6)	
C14	0.5748 (7)	0.9488 (2)	0.16634 (18)	0.0513 (7)	
H14	0.4933	0.9982	0.1234	0.062*	

C15	0.5078 (7)	0.9624 (2)	0.25052 (17)	0.0485 (6)	
H15	0.3810	1.0210	0.2634	0.058*	
C16	0.2691 (10)	0.7785 (3)	0.8611 (2)	0.0663 (9)	
H16A	0.2156	0.7752	0.9194	0.099*	
H16B	0.0805	0.7943	0.8214	0.099*	
H16C	0.4245	0.8352	0.8574	0.099*	
C17	0.8445 (10)	0.4671 (4)	0.8583 (3)	0.0888 (13)	
H17A	0.8033	0.4099	0.8978	0.133*	
H17B	0.8861	0.5344	0.8899	0.133*	
H17C	1.0269	0.4480	0.8304	0.133*	
C18	0.9253 (9)	0.4213 (3)	0.5689 (5)	0.0620 (9)	0.972 (3)
H18A	1.0191	0.3495	0.5729	0.093*	0.972 (3)
H18B	1.0854	0.4744	0.5600	0.093*	0.972 (3)
H18C	0.7570	0.4235	0.5208	0.093*	0.972 (3)
O1	0.4088 (7)	0.99177 (17)	0.42458 (14)	0.0702 (7)	
O2	0.3921 (5)	0.68052 (19)	0.83999 (14)	0.0622 (6)	
O3	0.5817 (5)	0.48021 (17)	0.79608 (14)	0.0593 (6)	
O4	0.8057 (7)	0.44333 (19)	0.64595 (19)	0.0619 (6)	0.972 (3)
N1	0.8291 (9)	0.8461 (2)	0.06121 (17)	0.0708 (8)	
H5A	0.9411	0.7907	0.0489	0.085*	
H5B	0.7579	0.8918	0.0211	0.085*	
Br1	0.912 (3)	0.4202 (10)	0.6089 (11)	0.040 (5)*	0.028 (3)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0555 (15)	0.0412 (14)	0.0408 (13)	−0.0016 (11)	0.0094 (11)	−0.0023 (10)
C2	0.0514 (15)	0.0376 (13)	0.0524 (15)	−0.0011 (11)	0.0035 (12)	0.0005 (12)
C3	0.0492 (15)	0.0405 (13)	0.0443 (13)	−0.0038 (11)	0.0048 (11)	0.0074 (10)
C4	0.0550 (15)	0.0467 (14)	0.0362 (13)	−0.0077 (12)	0.0071 (11)	0.0019 (10)
C5	0.0555 (15)	0.0382 (12)	0.0395 (13)	0.0008 (11)	0.0081 (11)	−0.0002 (10)
C6	0.0508 (14)	0.0384 (12)	0.0366 (12)	−0.0032 (11)	0.0041 (10)	0.0007 (10)
C7	0.0578 (15)	0.0344 (12)	0.0420 (13)	−0.0012 (11)	0.0102 (11)	−0.0010 (10)
C8	0.0663 (17)	0.0377 (13)	0.0421 (14)	0.0027 (11)	0.0112 (12)	−0.0009 (10)
C9	0.0643 (17)	0.0353 (13)	0.0402 (13)	−0.0011 (11)	0.0123 (12)	−0.0005 (10)
C10	0.0557 (15)	0.0337 (12)	0.0383 (13)	−0.0071 (11)	0.0085 (11)	0.0007 (10)
C11	0.0611 (16)	0.0390 (13)	0.0387 (13)	0.0027 (11)	0.0090 (11)	0.0058 (10)
C12	0.0695 (18)	0.0412 (14)	0.0467 (14)	0.0016 (13)	0.0178 (12)	−0.0011 (11)
C13	0.0653 (17)	0.0422 (14)	0.0384 (14)	−0.0162 (12)	0.0097 (12)	−0.0012 (11)
C14	0.0645 (18)	0.0468 (14)	0.0416 (13)	−0.0102 (12)	0.0035 (12)	0.0123 (11)
C15	0.0622 (16)	0.0364 (12)	0.0477 (14)	0.0030 (11)	0.0099 (12)	0.0050 (11)
C16	0.085 (2)	0.082 (2)	0.0358 (13)	−0.0068 (18)	0.0221 (13)	−0.0015 (15)
C17	0.081 (2)	0.090 (3)	0.090 (3)	−0.011 (2)	−0.010 (2)	0.050 (2)
C18	0.077 (3)	0.0448 (19)	0.065 (3)	0.0146 (16)	0.0096 (19)	−0.0057 (17)
O1	0.1155 (19)	0.0448 (11)	0.0544 (12)	0.0213 (12)	0.0265 (12)	0.0006 (9)
O2	0.0847 (15)	0.0613 (12)	0.0435 (11)	0.0035 (11)	0.0191 (10)	0.0172 (9)
O3	0.0662 (13)	0.0501 (12)	0.0604 (12)	−0.0052 (10)	0.0039 (9)	0.0197 (9)
O4	0.0846 (16)	0.0398 (11)	0.0636 (15)	0.0104 (11)	0.0186 (15)	0.0063 (10)

N1	0.117 (2)	0.0554 (15)	0.0423 (14)	−0.0101 (16)	0.0203 (14)	0.0006 (12)

Geometric parameters (Å, °)

C1—C2	1.384 (4)	C11—H11	0.9300
C1—C6	1.394 (4)	C12—C13	1.390 (4)
C1—H1A	0.9300	C12—H12	0.9300
C2—O4	1.374 (4)	C13—N1	1.377 (4)
C2—C3	1.390 (4)	C13—C14	1.395 (4)
C2—Br1	2.000 (17)	C14—C15	1.380 (4)
C3—O3	1.380 (3)	C14—H14	0.9300
C3—C4	1.392 (4)	C15—H15	0.9300
C4—O2	1.384 (3)	C16—O2	1.361 (4)
C4—C5	1.391 (4)	C16—H16A	0.9600
C5—C6	1.398 (3)	C16—H16B	0.9600
C5—H5	0.9300	C16—H16C	0.9600
C6—C7	1.468 (4)	C17—O3	1.380 (4)
C7—C8	1.317 (4)	C17—H17A	0.9600
C7—H7	0.9300	C17—H17B	0.9600
C8—C9	1.483 (4)	C17—H17C	0.9600
C8—H8	0.9300	C18—O4	1.382 (8)
C9—O1	1.233 (3)	C18—H18A	0.9600
C9—C10	1.475 (3)	C18—H18B	0.9600
C10—C11	1.389 (4)	C18—H18C	0.9600
C10—C15	1.401 (3)	N1—H5A	0.8600
C11—C12	1.382 (4)	N1—H5B	0.8600
C2—C1—C6	120.2 (2)	C11—C12—H12	119.7
C2—C1—H1A	119.9	C13—C12—H12	119.7
C6—C1—H1A	119.9	N1—C13—C12	120.3 (3)
O4—C2—C1	124.6 (3)	N1—C13—C14	121.1 (3)
O4—C2—C3	114.9 (2)	C12—C13—C14	118.5 (2)
C1—C2—C3	120.6 (2)	C15—C14—C13	120.5 (2)
C1—C2—Br1	106.7 (5)	C15—C14—H14	119.8
C3—C2—Br1	132.6 (5)	C13—C14—H14	119.8
O3—C3—C2	120.0 (3)	C14—C15—C10	121.3 (3)
O3—C3—C4	120.3 (2)	C14—C15—H15	119.3
C2—C3—C4	119.5 (2)	C10—C15—H15	119.3
O2—C4—C5	124.4 (2)	O2—C16—H16A	109.5
O2—C4—C3	115.4 (2)	O2—C16—H16B	109.5
C5—C4—C3	120.2 (2)	H16A—C16—H16B	109.5
C4—C5—C6	120.1 (2)	O2—C16—H16C	109.5
C4—C5—H5	120.0	H16A—C16—H16C	109.5
C6—C5—H5	120.0	H16B—C16—H16C	109.5
C1—C6—C5	119.4 (2)	O3—C17—H17A	109.5
C1—C6—C7	121.8 (2)	O3—C17—H17B	109.5
C5—C6—C7	118.9 (2)	H17A—C17—H17B	109.5
C8—C7—C6	127.2 (2)	O3—C17—H17C	109.5
C8—C7—H7	116.4	H17A—C17—H17C	109.5
C6—C7—H7	116.4	H17B—C17—H17C	109.5

C7—C8—C9	123.6 (2)	O4—C18—H18A	108.4
C7—C8—H8	118.2	O4—C18—H18B	109.7
C9—C8—H8	118.2	H18A—C18—H18B	109.5
O1—C9—C10	121.1 (2)	O4—C18—H18C	110.3
O1—C9—C8	120.4 (2)	H18A—C18—H18C	109.5
C10—C9—C8	118.4 (2)	H18B—C18—H18C	109.5
C11—C10—C15	117.5 (2)	C16—O2—C4	117.9 (2)
C11—C10—C9	123.4 (2)	C3—O3—C17	116.5 (3)
C15—C10—C9	119.1 (2)	C2—O4—C18	117.7 (3)
C12—C11—C10	121.6 (2)	C13—N1—H5A	120.0
C12—C11—H11	119.2	C13—N1—H5B	120.0
C10—C11—H11	119.2	H5A—N1—H5B	120.0
C11—C12—C13	120.6 (3)		
C6—C1—C2—O4	177.8 (3)	C7—C8—C9—C10	−172.5 (3)
C6—C1—C2—C3	−0.8 (4)	O1—C9—C10—C11	172.7 (3)
C6—C1—C2—Br1	−176.8 (4)	C8—C9—C10—C11	−10.1 (4)
O4—C2—C3—O3	−4.6 (4)	O1—C9—C10—C15	−9.3 (4)
C1—C2—C3—O3	174.1 (3)	C8—C9—C10—C15	167.8 (3)
Br1—C2—C3—O3	−11.1 (6)	C15—C10—C11—C12	1.2 (4)
O4—C2—C3—C4	180.0 (2)	C9—C10—C11—C12	179.1 (3)
C1—C2—C3—C4	−1.4 (4)	C10—C11—C12—C13	0.0 (4)
Br1—C2—C3—C4	173.5 (5)	C11—C12—C13—N1	−179.2 (3)
O3—C3—C4—O2	7.0 (4)	C11—C12—C13—C14	−1.1 (4)
C2—C3—C4—O2	−177.6 (2)	N1—C13—C14—C15	179.1 (3)
O3—C3—C4—C5	−173.2 (3)	C12—C13—C14—C15	1.0 (4)
C2—C3—C4—C5	2.3 (4)	C13—C14—C15—C10	0.1 (4)
O2—C4—C5—C6	178.8 (2)	C11—C10—C15—C14	−1.2 (4)
C3—C4—C5—C6	−1.0 (4)	C9—C10—C15—C14	−179.3 (3)
C2—C1—C6—C5	2.0 (4)	C5—C4—O2—C16	2.3 (4)
C2—C1—C6—C7	−176.5 (3)	C3—C4—O2—C16	−177.9 (3)
C4—C5—C6—C1	−1.1 (4)	C2—C3—O3—C17	94.6 (4)
C4—C5—C6—C7	177.4 (3)	C4—C3—O3—C17	−90.0 (4)
C1—C6—C7—C8	−4.9 (4)	C1—C2—O4—Br1	16.5 (12)
C5—C6—C7—C8	176.6 (3)	C3—C2—O4—Br1	−164.8 (12)
C6—C7—C8—C9	174.5 (3)	C1—C2—O4—C18	4.0 (4)
C7—C8—C9—O1	4.7 (5)	C3—C2—O4—C18	−177.4 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H5B···O1^{i}	0.86	2.21	2.948 (3)	143

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

Fig. 1

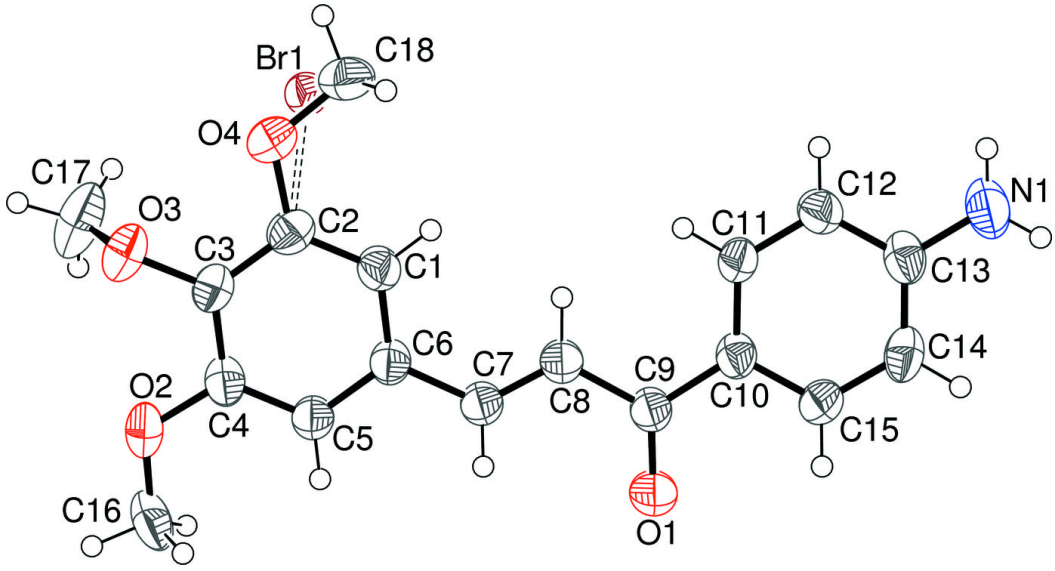
Br1
C18
O4
C17
O3
C3
C2
C1
C6
C7
C8
C9
C10
C11
C12
N1
C13
C14
C15
O2
C4
C5
C16
O1

Fig. 2