



Hall, C. L., Hamilton, V., Potticary, J., Cremeens, M. E., Pridmore, N. E., Sparkes, H. A., D'ambruoso, G. D., Warren, S. D., Matsumoto, M., & Hall, S. R. (2020). Crystal structures of three functionalized chalcones: 4'-di-methyl-amino-3-nitro-chalcone, 3-di-methyl-amino-3'-nitrochalcone and 3'-nitro-chalcone. *Acta Crystallographica Section E: Crystallographic Communications*, E76, 1599-1604.
<https://doi.org/10.1107/s2056989020011858>

Publisher's PDF, also known as Version of record

License (if available):
CC BY

Link to published version (if available):
[10.1107/s2056989020011858](https://doi.org/10.1107/s2056989020011858)

[Link to publication record in Explore Bristol Research](#)
PDF-document

This is the final published version of the article (version of record). It first appeared online via International Union of Crystallography at <https://doi.org/10.1107/S2056989020011858> . Please refer to any applicable terms of use of the publisher.

University of Bristol - Explore Bristol Research

General rights

This document is made available in accordance with publisher policies. Please cite only the published version using the reference above. Full terms of use are available:
<http://www.bristol.ac.uk/red/research-policy/pure/user-guides/ebr-terms/>

Received 9 July 2020

Accepted 27 August 2020

Edited by D. Chopra, Indian Institute of Science
Education and Research Bhopal, India**Keywords:** chalcone; dimethylamino; nitro;
crystal structure.**CCDC references:** 2025821; 2025820;
2025819**Supporting information:** this article has
supporting information at journals.iucr.org/e

Crystal structures of three functionalized chalcones: 4'-dimethylamino-3-nitrochalcone, 3-dimethylamino-3'-nitrochalcone and 3'-nitrochalcone

Charlie L. Hall,^a Victoria Hamilton,^a Jason Potticary,^a Matthew E. Cremeens,^b
Natalie E. Pridmore,^a Hazel A. Sparkes,^a Gemma D. D'ambrosio,^b Stephen D.
Warren,^b Masaomi Matsumoto^b and Simon R. Hall^{a*}

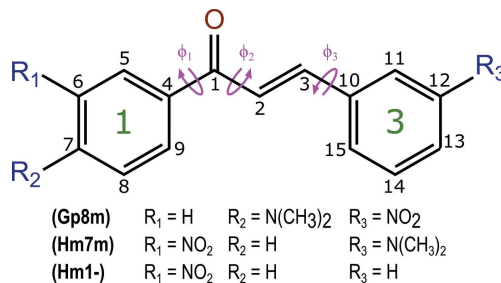
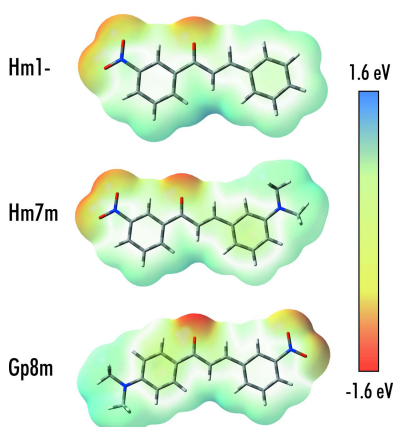
^aSchool of Chemistry, University of Bristol, Cantock's Close, Bristol, BS8 1TS, England, and ^bDepartment of Chemistry & Biochemistry, Gonzaga University, 502 E Boone Ave, Spokane, WA 99258, USA. *Correspondence e-mail: simon.hall@bristol.ac.uk

The structure of three functionalized chalcones (1,3-diarylprop-2-en-1-ones), containing combinations of nitro and dimethylamino functional groups, are presented, namely, 1-[4-(dimethylamino)phenyl]-3-(3-nitrophenyl)prop-2-en-1-one, C₁₇H₁₆N₂O₃, Gp8m, 3-[3-(dimethylamino)phenyl]-1-(3-nitrophenyl)prop-2-en-1-one, C₁₇H₁₆N₂O₃, Hm7m and 1-(3-nitrophenyl)-3-phenylprop-2-en-1-one, C₁₅H₁₁NO₃, Hm1-. Each of the molecules contains bonding motifs seen in previously solved crystal structures of functionalized chalcones, adding to the large dataset available for these small organic molecules. The structures of all three of the title compounds contain similar bonding motifs, resulting in two-dimensional planes of molecules formed *via* C—H···O hydrogen-bonding interactions involving the nitro- and ketone groups. The structure of Hm1- is very similar to the crystal structure of a previously solved isomer [Jing (2009). *Acta Cryst. E* **65**, o2510].

1. Chemical context

Chalcones, 1,3-diarylprop-2-en-1-ones, are a group of organic molecules containing two aromatic rings joined by an enone backbone. Chalcones are studied for a range of medicinal purposes, with many reviews published on their biological applicability (see, for example, Rammohan *et al.*, 2020; Zhuang *et al.*, 2017; Singh *et al.*, 2014).

A range of chalcones, functionalized on either aromatic ring, can be readily synthesized *via* an aldol condensation reaction (Mandge *et al.*, 2007). Altering the functional groups on the chalcone structure has been shown to yield a variety of useful properties, including changes in colour and fluorescent properties (Ibnaouf *et al.*, 2018).



In this work, the structures of three chalcones: 4'-dimethylamino-3-nitrochalcone [Gp8m, R₁ = N(CH₃)₂, R₂ = H,

Table 1

Torsion and ring angles ($^{\circ}$) describing the planarity of molecules in each crystal structure.

Torsion angles calculated using the definitions: $\Phi_1 = \text{C5}-\text{C4}-\text{C1}-\text{C2}$, $\Phi_2 = \text{C4}-\text{C1}-\text{C2}-\text{C3}$ and $\Phi_3 = \text{C2}-\text{C3}-\text{C10}-\text{C11}$. Ring twist and fold angles were calculated using the mean planes of the 1- and 3-rings. All values were calculated using OLEX2 (Dolomanov *et al.*, 2009).

	Gm8p	Hm7m	Hm1- (1)	Hm1- (2)
Φ_1	-173.15 (11)	172.9 (2)	177.4 (2)	-164.6 (2)
Φ_2	158.84 (12)	168.8 (2)	174.4 (2)	175.9 (2)
Φ_3	-169.23 (13)	-175.0 (2)	-170.3 (3)	-172.9 (9)
Ring twist angle	3.61 (4)	13.8 (8)	1.88 (8)	12.58 (8)
Ring fold angle	11.46 (4)	0.59 (8)	2.58 (8)	6.66 (8)

$R_3 = \text{NO}_2$], 3'-nitro-3-dimethylaminochalcone [Hm7m, $R_1 = \text{NO}_2$, $R_2 = \text{N}(\text{CH}_3)_2$, $R_3 = \text{H}$] and 3'-nitrochalcone [Hm1-, $R_1 = \text{NO}_2$, $R_2 = \text{H}$, $R_3 = \text{H}$] are presented. The crystal structures of these molecules add to the large dataset available for molecules based on the chalcone backbone. In particular, these structures add to the small amount of data available for chalcones substituted with a nitro group on the 3-ring.

2. Structural commentary

The planarity of the chalcone molecules is defined by the torsion angles $\Phi_1 = \text{C5}-\text{C4}-\text{C1}-\text{C2}$, $\Phi_2 = \text{C4}-\text{C1}-\text{C2}-\text{C3}$ and $\Phi_3 = \text{C2}-\text{C3}-\text{C10}-\text{C11}$. The torsion angle $\text{C1}-\text{C2}-\text{C3}-\text{C10}$ is planar to within 1° of 180° in all three structures. The torsion angles, along with the numbering of the molecules, are highlighted in the scheme. The 1-ring of the molecule is defined as the aromatic ring attached to C1 and the 3-ring is that attached to C3. The long axis of each molecule is defined to be along the $\text{C2}-\text{C12}$ axis, and the short axis is defined to be along $\text{H2}-\text{C2}$. Table 1 presents a summary of the torsion and ring angles in the title structures.

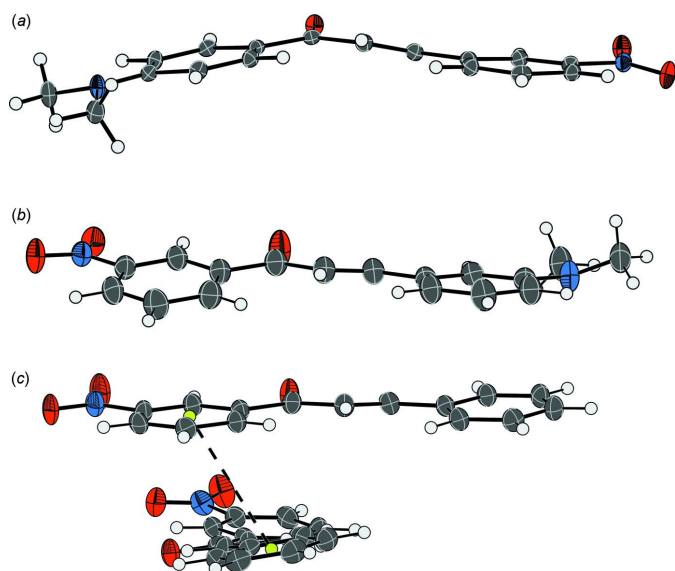


Figure 1

Displacement ellipsoid plots showing the asymmetric units of the solved crystal structures (a) Gp8m, (b) Hm7m and (c) Hm1-. Displacement ellipsoids are shown at the 50% probability level. The stacking interaction between the 1- and 3-rings of the molecules in the asymmetric unit of Hm1- is highlighted.

Gp8m (Fig. 1a) crystallizes in space group $P2_1/c$ with a single molecule in the asymmetric unit. The molecule deviates from planarity, with $\Phi_2 = 158.84 (12)^{\circ}$, meaning that there is a fold angle of $11.46 (4)^{\circ}$ between the planes of the 1- and 3-rings of the molecule. The nitro group on the 3-ring is twisted out of the plane of the ring [$\text{C11}-\text{C12}-\text{N2}-\text{O2} = 10.09 (18)^{\circ}$].

Hm7m (Fig. 1b) crystallizes in space group $P2_1/n$ with a single molecule in the asymmetric unit. The combination of torsion angles along the long-axis of the molecule means that although the backbone remains relatively planar [$\text{C4}-\text{C10}-\text{C2} = 2.86 (6)^{\circ}$], the 1- and 3-rings are twisted with respect to each other, with a twist angle of $13.80 (8)^{\circ}$ between the planes of the rings.

Hm1- crystallizes in space group $P2_1/c$ and contains two molecules in the asymmetric unit. One of the molecules (1) is almost planar, with a twist angle of only $1.88 (8)^{\circ}$ between the planes of the 1- and 3-rings of the molecule. The second molecule (2) is less planar with $\Phi_1 = -164.6 (2)$ and $\Phi_2 = -172 (9)^{\circ}$, leading to a twist angle of $12.85 (8)^{\circ}$ between the planes of the 1- and 3-rings. There is a stacking interaction between the aryl rings of the two molecules in the asymmetric unit, with a centroid-to-centroid distance of $3.82782 (17) \text{ \AA}$ (Fig. 1c).

In each of the molecular structures, the functionalized group in the *meta*-position sits on the same side of the molecule as the carbonyl oxygen group (1-ring: C6, 3-ring: C12). This is likely due to the optimization of hydrogen-bonding motifs in the crystal structures.

3. Supramolecular features

Although being in a different space group, the crystal structure of Gp8m is very similar to that of a previously reported chalcone 3'-nitro,4-dimethylaminochalcone (Rosli *et al.*, 2007). This may be expected, as the only difference between these molecules is that the functional groups are on opposite rings. Within the crystal structure, chains of molecules form down the long axis of the molecule *via* short contacts between the dimethylamino and nitro groups (Fig. 2a; $\text{C17}-\text{H17B} \cdots \text{O3}^{\text{iii}}$). These molecules form stacks parallel to the *b* axis, with alternate molecules the opposite way around such that the nitro group sits above the 1-ring of the adjacent molecule. The final 3D structure is completed by a linking of the stacks *via* $\text{C}-\text{H} \cdots \text{O}$ cyclic hydrogen bonding ($\text{C3}-\text{H3} \cdots \text{O1}^{\text{i}}$, $\text{C5}-$

Table 2
Hydrogen-bond geometry (Å, °) for Gp8m.

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$C3-H3\cdots O1^i$	0.95	2.47	3.3124 (16)	148
$C5-H5\cdots O2^i$	0.95	2.67	3.3067 (17)	125
$C11-H11\cdots O1^i$	0.95	2.88	3.5773 (16)	131
$C15-H15\cdots O1^{ii}$	0.95	2.68	3.5435 (16)	152
$C17-H17B\cdots O3^{iii}$	0.98	2.68	3.5755 (17)	152

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

Table 3
Hydrogen-bond geometry (Å, °) for Hm7m.

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$C15-H15\cdots O3^i$	0.95	2.50	3.442 (2)	174
$C16-H16B\cdots O2^{ii}$	0.98	2.58	3.520 (2)	160

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

$H5\cdots O2^i$, $C11-H11\cdots O1^i$) and hydrogen bonds involving the carbonyl group (Fig. 2*b*; $C15-H15\cdots O1^{ii}$). Numerical details of the hydrogen-bond geometry and symmetry codes are given in Table 2.

Within the crystal structure of Hm7m, sheets are formed in the plane of the aromatic rings of the molecule. Within the plane, pairs of inverted molecules form *via* cyclic hydrogen bonding between the dimethylamino and nitro groups, offset in the short axis of the molecule ($C16-H16B\cdots O2^{ii}$). The pairs of molecules then form sheets *via* a trifurcated hydrogen-bonding interaction involving the nitro group ($C15-H15\cdots O3^i$, $C15-H2\cdots O3^i$, $C15-H9\cdots O3^i$). These sheets

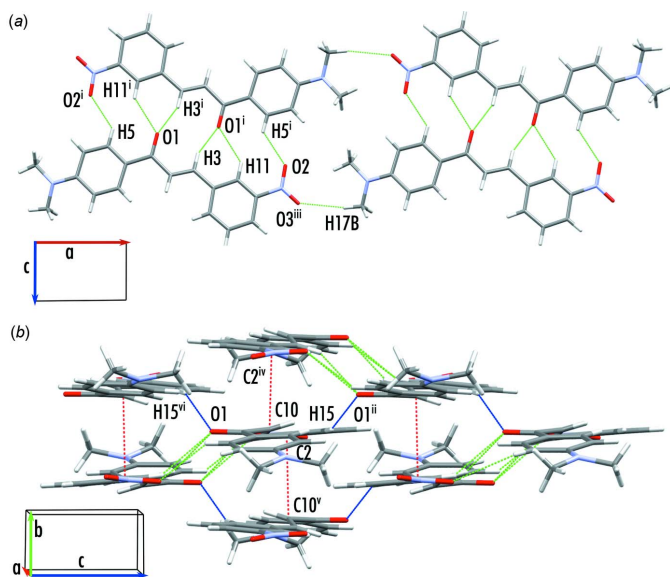


Figure 2
Bonding motifs present in the crystal structure of Gp8m. (a) Interactions in the plane of the rings of the molecules. (b) Hydrogen bonds offset along the short axis of the molecule. Cyclic hydrogen bonds highlighted in (a) are shown in green and an additional hydrogen-bonding motif is shown in blue. Stacking interactions are highlighted in red. [Symmetry codes: (i) $-x + 1, -y + 1, -z$; (ii) $x, -y + \frac{3}{2}, z + \frac{1}{2}$; (iii) $x - 1, y, z$; (iv) $-x + 1, y + \frac{1}{2}, -z - \frac{1}{2}$; (v) $-x + 1, y + \frac{1}{2}, -z + \frac{1}{2}$; (vi) $x, -y + \frac{3}{2}, z - \frac{1}{2}$.]

Table 4
Hydrogen-bond geometry (Å, °) for Hm1-.

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$C2-H2\cdots O3^i$	0.95	2.51	3.456 (3)	174
$C3-H3\cdots O4^{ii}$	0.95	2.50	3.328 (3)	146
$C9-H9\cdots O3^i$	0.95	2.58	3.527 (3)	175
$C15-H15\cdots O3^i$	0.95	2.47	3.399 (3)	166
$C17-H17\cdots O6^{iii}$	0.95	2.57	3.491 (3)	164
$C18-H18\cdots O1^{iv}$	0.95	2.46	3.300 (3)	147
$C30-H30\cdots O6^{iii}$	0.95	2.58	3.456 (3)	154
$C26-H26\cdots O1^{iv}$	0.95	2.54	3.328 (3)	140

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

make up the 3D structure *via* a stacking interaction, where the nitro group of one molecule sits over the 1- ring of another (Fig. 3). Numerical details of the hydrogen-bond geometry and symmetry codes are given in Table 3.

The crystal structure of Hm1- contains two molecules in the asymmetric unit cell, which differ slightly in their planarity. Sheets of molecules form *via* the same interactions as in Hm7m; however, the pairs of molecules form between different independent molecules, meaning they are not directly related by an inversion centre. Furthermore, the absence of the dimethylamino group means that the molecules are shifted relatively along the long axis of the molecule, forming hydrogen bonds that utilize the carbonyl oxygen (Fig. 4*a*). The stacking interactions that make up the 3D structure of Hm1- are more complex than those in Hm7m, and are highlighted in Fig. 4*b*. Molecule 1 forms a direct stack with a symmetrically equivalent molecule, with an inversion centre relating the molecules. There is a half stack that forms between the 1-ring of molecule 1 and the 3-ring of molecule 2, which sit at approximately 90° to each other. Finally, molecule 2 forms a half stack with a symmetrically equivalent molecule, where the 1-ring of each molecule sits on top of the other.

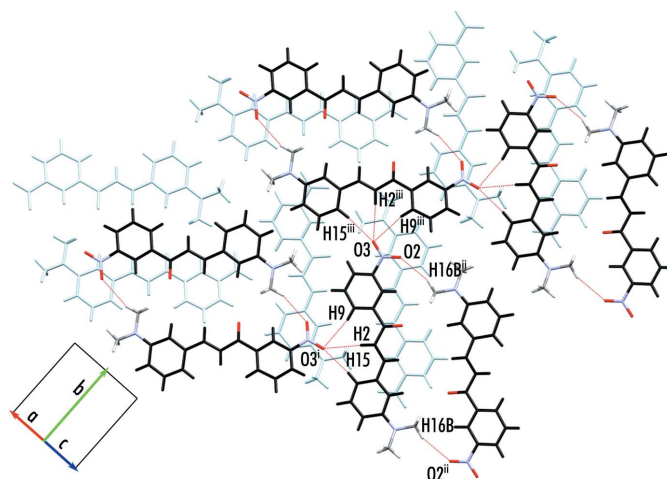


Figure 3
Bonding motifs present in the crystal structure of Hm7m. Two sheets of molecules are highlighted, coloured in black and light blue for contrast. [Symmetry codes: (i) $-x + \frac{3}{2}, y - \frac{1}{2}, -z + \frac{1}{2}$; (ii) $-x, -y + 1, -z + 1$; (iii) $-x + 1, -y + 1, -z + 1$.]

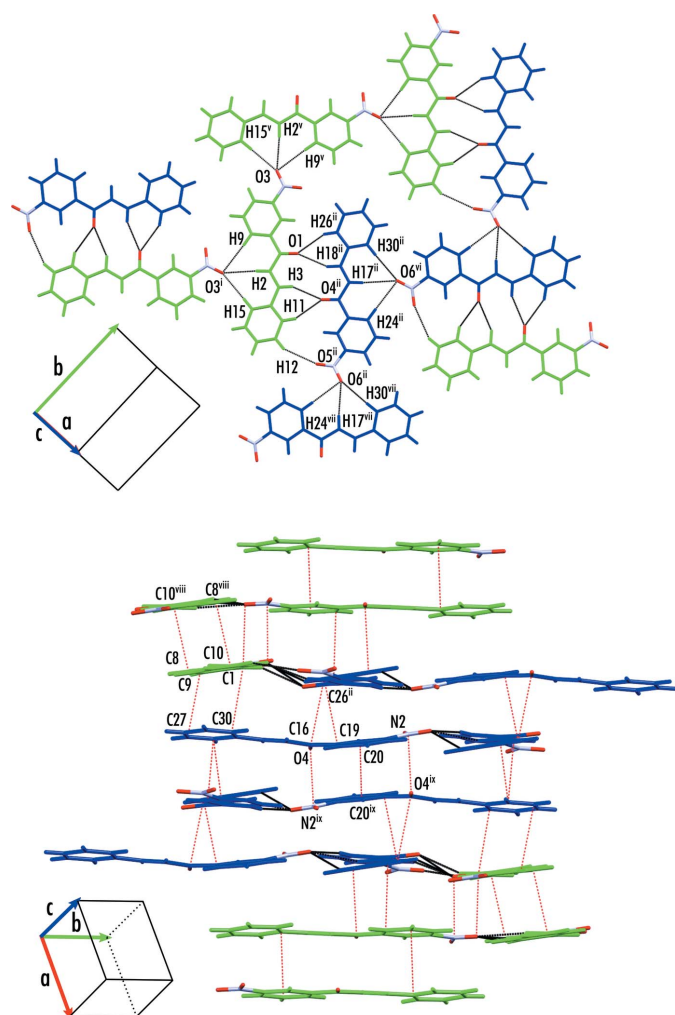


Figure 4
Bonding motifs present in the crystal structure of Hm1-. (a) Hydrogen-bonding interactions forming two-dimensional planes of molecules. (b) Stacking interactions present in the crystal structure. Molecules are coloured according to their symmetry equivalence as green (1) and blue (2). [Symmetry codes: (i) $-x, y - \frac{1}{2}, -z + \frac{1}{2}$; (ii) $x, -y + \frac{1}{2}, z + \frac{1}{2}$; (v) $-x, y + \frac{1}{2}, -z + \frac{1}{2}$; (vi) $-x + 1, -y + 1, -z + 2$; (vii) $-x + 1, -y, -z + 2$; (viii) $-x, -y, -z + 1$; (ix) $-x + 1, -y + 1, -z + 1$.]

Numerical details of the hydrogen-bond geometry are given in Table 4.

4. Database survey

A survey of the Cambridge Structural Database (CSD, version 5.41, last update March 2020; Groom *et al.*, 2016) revealed 38 structures of chalcones functionalized with either nitro or dimethylamino-groups in either the *meta* or *para* positions of the 1- or 3-ring. None of the structures contain chalcones with a dimethylamino group on the 1- ring, as in Gp8m. However, there are 14 structures of chalcones substituted with a dimethylamino group on the 3-ring, likely owing to their fluorescent properties (Jiang *et al.*, 1994; Tomasch *et al.*, 2012).

17 of the 29 structures that contain nitro ring substitutions contain the bonding motif between the nitro group and the

region between H15, H2 and H9, as observed in Hm7m and Hm1-. This is likely caused by the optimization of electrostatic interactions, as highlighted by the electrostatic potentials in Fig. 5. The layered motif in Hm7m is the same as that present in the structure of 3'-nitro-3,5-dimethoxychalcone (Qiu & Yang, 2006). The planes of molecules seen in Hm1- are similar to those seen in the structure of 4'-nitrochalcone (BUDXOO; Jing, 2009).

5. Synthesis and crystallization

Each of the functionalized chalcones was synthesized *via* an aldol condensation reaction between a suitably functionalized benzaldehyde and acetophenone. While syntheses were not specifically reported for Gp8m and Hm7m, the first reports for Hm1- appeared in 1929 and 1935 (Dilthey *et al.*, 1929; Weygand *et al.*, 1935).

Ethanol (1.5 mL, 95%) and a stir bar were added to two separate vessels; one contained the benzaldehyde (3 mmol) and the other contained the acetophenone (3 mmol). Each vessel was gently heated over a hot plate until complete dissolution and then cooled to room temperature; depending on the solubility of the starting materials, solids precipitated upon cooling. Once cooled, NaOH (aq) (0.4 mL, 50% by wgt) was added to the vessel containing the acetophenone and vigorously stirred. The benzaldehyde mixture was added to the acetophenone and NaOH mixture. The resulting reaction mixture was vigorously mixed until a slurry or paste formed. Water was added to the vessel and the contents were agitated

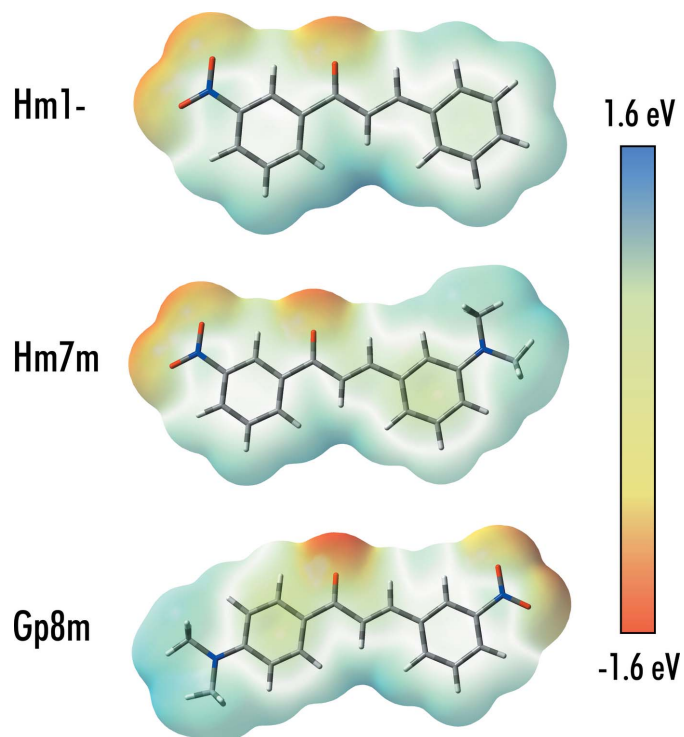


Figure 5
Electrostatic potential maps for optimized configurations of Gp8m, Hm7m and Hm1- molecules. Optimizations were carried out using Gaussian09 [B3LYP/6-31 G(d); Frisch *et al.*, 2009]. The electrostatic potential is mapped onto the 0.0004 SCF electron density surface.

Table 5
Experimental details.

	Hm1-	Gp8m	Hm7m
Crystal data			
Chemical formula	C ₁₅ H ₁₁ NO ₃	C ₁₇ H ₁₆ N ₂ O ₃	C ₁₇ H ₁₆ N ₂ O ₃
<i>M_r</i>	253.25	296.32	296.32
Crystal system, space group	Monoclinic, <i>P</i> ₂ / <i>c</i>	Monoclinic, <i>P</i> ₂ / <i>c</i>	Monoclinic, <i>P</i> ₂ / <i>n</i>
Temperature (K)	100	100	200
<i>a</i> , <i>b</i> , <i>c</i> (Å)	14.7856 (8), 15.9841 (9), 10.3188 (6)	17.3171 (7), 7.0708 (3), 11.3487 (4)	7.7552 (4), 15.6998 (7), 12.0525 (7)
β (°)	99.210 (4)	90.761 (3)	100.668 (3)
<i>V</i> (Å ³)	2407.3 (2)	1389.48 (10)	1442.09 (13)
<i>Z</i>	8	4	4
Radiation type	Mo <i>K</i> α	Mo <i>K</i> α	Mo <i>K</i> α
μ (mm ⁻¹)	0.10	0.10	0.10
Crystal size (mm)	0.61 × 0.35 × 0.25	0.39 × 0.35 × 0.19	0.39 × 0.33 × 0.25
Data collection			
Diffractometer	Bruker APEXII Kappa CCD area detector	Bruker APEXII Kappa CCD area detector	Bruker APEXII Kappa CCD area detector
Absorption correction	Multi-scan (<i>SADABS</i> ; Bruker, 2016)	Multi-scan (<i>SADABS</i> ; Bruker, 2016)	Multi-scan (<i>SADABS</i> ; Bruker, 2016)
<i>T</i> _{min} , <i>T</i> _{max}	0.610, 0.746	0.666, 0.746	0.629, 0.746
No. of measured, independent and observed [<i>I</i> > 2 σ (<i>I</i>)] reflections	25080, 4408, 2657	12270, 3320, 2561	11497, 3056, 1882
<i>R</i> _{int}	0.085	0.034	0.058
(<i>sin</i> θ / λ) _{max} (Å ⁻¹)	0.602	0.659	0.633
Refinement			
<i>R</i> [<i>F</i> ² > 2 σ (<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.049, 0.141, 0.99	0.043, 0.123, 1.04	0.049, 0.133, 1.02
No. of reflections	4408	3320	3056
No. of parameters	343	212	201
H-atom treatment	H-atom parameters constrained	H atoms treated by a mixture of independent and constrained refinement	H-atom parameters constrained
$\Delta\rho_{\max}$, $\Delta\rho_{\min}$ (e Å ⁻³)	0.28, -0.25	0.31, -0.28	0.19, -0.24

Computer programs: *APEX2* (Bruker, 2016), *S SAINT* (Bruker, 2016), *SUPERFLIP* (Palatinus & Chapuis, 2007; Palatinus & van der Lee, 2008; Palatinus *et al.*, 2012), *SHELXL* (Sheldrick, 2015), *OLEX2* (Dolomanov *et al.*, 2009) and *Mercury* (Macrae *et al.*, 2020), *OLEX2* (Dolomanov *et al.*, 2009).

with a micro spatula. The solids were collected by vacuum filtration and purified by recrystallization with ethanol. ¹H NMR (400 MHz, CDCl₃, referenced to TMS): δ (ppm) for Gp8m are 8.51 (1H, *s*), 8.22 (1H, *d*, *J* = 8.0 Hz), 8.02 (2H, *d*, *J* = 8.9 Hz), 7.90 (1H, *d*, *J* = 7.5 Hz), 7.79 (1H, *d*, *J* = 15.6 Hz), 7.70 (1H, *d*, *J* = 15.6 Hz), 7.59 (1H, *t*, *J* = 8.0 Hz), 6.72 (2H, *d*, *J* = 8.9 Hz), 3.11 (6H, *s*); for Hm1- are 8.83 (1H, *t*, *J* = 1.9 Hz), 8.44 (1H, *ddd*, *J* = 8.2, 2.2, 1.0 Hz), 8.35 (1H, *ddd*, *J* = 7.8, 1.4, 1.4 Hz), 7.89 (1H, *d*, *J* = 15.6 Hz), 7.72 (1H, *t*, *J* = 8.0 Hz), 7.67 (2H, *m*), 7.54 (1H, *d*, *J* = 15.6 Hz), 7.45 (3H, *m*); and for Hm7m are 8.82 (1H, *t*, *J* = 1.9 Hz), 8.42 (1H, *ddd*, *J* = 8.2, 2.2, 1.0 Hz), 8.34 (1H, *ddd*, *J* = 7.7, 1.2, 1.2 Hz), 7.85 (1H, *d*, *J* = 15.6 Hz), 7.71 (1H, *t*, *J* = 8.0 Hz), 7.48 (1H, *d*, *J* = 15.6 Hz), 7.30 (1H, *t*, *J* = 7.9 Hz), 7.06 (1H, *d*, *J* = 7.6 Hz), 6.92 (1H, *dd*, *J* = 2.1, 1.6 Hz), 6.82 (1H, *dd*, *J* = 8.2, 2.5 Hz). ¹³C NMR (100 MHz, CDCl₃, referenced to solvent, 77.16 ppm): δ (ppm) for Gp8m are 186.86, 153.82, 148.83, 139.52, 137.49, 134.45, 131.16, 130.03, 125.51, 125.05, 124.21, 122.14, 111.02, 40.21; for Hm7m are 188.41, 150.97, 148.49, 148.27, 139.79, 135.06, 134.23, 129.96, 129.79, 127.02, 123.39, 120.42, 116.63, 115.42, 112.82, 40.60; and for Hm1- are 188.09, 148.50, 146.87, 139.58, 134.40, 134.22, 131.33, 130.04, 129.21, 128.86, 127.18, 123.37, 120.72.

Crystals of Hm7m suitable for structural solution *via* single crystal X-ray diffraction were produced *via* evaporation of a 10 mg mL⁻¹ acetone solution. Crystals of three separate

colours were observed (yellow needles, orange needles and red block-like crystals); however, only crystals of a red block-like morphology were suitable for structure solution. Hm7m appeared to go through a phase transition between 100 K and 200 K which caused the crystal to crack. For this reason, single crystal X-ray diffraction was carried out at 200 K.

Crystals of Hm1- and Gp8m suitable for structural solution *via* single crystal X-ray diffraction were produced *via* evaporation of an ethanol solution of concentration 10 mg mL⁻¹. Crystals of Gp8m appeared as fine yellow needles and Hm1- as colourless block-like crystals. Each single crystal was mounted onto a glass capillary using paraffin oil.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 5. All hydrogen atoms were located geometrically (aromatic C–H = 0.95 Å, methyl C–H = 0.99 Å) and refined using a riding model [*U*_{iso}(H) = 1.2*U*_{eq}(C-aromatic) or 1.5*U*_{eq}(C-methyl)].

Acknowledgements

The GU co-authors thank J. Hazen, S. Economu and B. Hendricks for their assistance, as well as the Howard Hughes

Medical Institute for supporting equipment acquisition through its Undergraduate Science Education Program.

Funding information

Funding for this research was provided by: EPSRC (grant No. EP/L015544/1 to C. L. Hall; grant No. EP/L016648/1 to V. Hamilton); European Union's Horizon 2020 Research and Innovation Programme (grant No. 736899).

References

- Bruker. (2016). *APEX2, SAINT and SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Dilthey, W., Neuhaus, L. & Schommer, W. (1929). *J. Prakt. Chem.* **123**, 235–240.
- Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). *J. Appl. Cryst.* **42**, 339–341.
- Frisch, M. J., Trucks, G. W., Schlegel, H. B., Scuseria, G. E., Robb, M. A., Cheeseman, J. R., Scalmani, G., Barone, V., Mennucci, B., Petersson, G. A., Nakatsuji, H., Caricato, M., Li, X., Hratchian, H. P., Izmaylov, A. F., Bloino, J., Zheng, G., Sonnenberg, J. L., Hada, M., Ehara, M., Toyota, K., Fukuda, R., Hasegawa, J., Ishida, M., Nakajima, T., Honda, Y., Kitao, O., Nakai, H., Vreven, T., Montgomery, J. A. Jr, Peralta, J. E., Ogliaro, F., Bearpark, M., Heyd, J. J., Brothers, E., Kudin, K. N., Staroverov, V. N., Kobayashi, R., Normand, J., Raghavachari, K., Rendell, A., Burant, J. C., Iyengar, S. S., Tomasi, J., Cossi, M., Rega, N., Millam, J. M., Klene, M., Knox, J. E., Cross, J. B., Bakken, V., Adamo, C., Jaramillo, J., Gomperts, R., Stratmann, R. E., Yazyev, O., Austin, A. J., Cammi, R., Pomelli, C., Ochterski, J. W., Martin, R. L., Morokuma, K., Zakrzewski, V. G., Voth, G. A., Salvador, P., Dannenberg, J. J., Dapprich, S., Daniels, A. D., Farkas, Ö., Foresman, J. B., Ortiz, J. V., Cioslowski, J. & Fox, D. J. (2009). *GAUSSIAN09*. Gaussian Inc., Wallingford, CT, USA.
- Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). *Acta Cryst.* **B72**, 171–179.
- Ibnaouf, H. K., Elzupir, A. O., AlSalhi, M. S. & Alaamer, A. S. (2018). *Opt. Mater.* **76**, 216–221.
- Jiang, Y., Wang, X. & Lin, L. (1994). *J. Phys. Chem.* **98**, 12367–12372.
- Jing, L.-H. (2009). *Acta Cryst.* **E65**, o2510.
- Macrae, C. F., Sovago, I., Cottrell, S. J., Galek, P. T. A., McCabe, P., Pidcock, E., Platings, M., Shields, G. P., Stevens, J. S., Towler, M. & Wood, P. A. (2020). *J. Appl. Cryst.* **53**, 226–235.
- Mandge, S., Singh, H. P., Gupta, S. D. & Hari Narayana Moorthy, N. S. (2007). *Trends Appl. Sci. Res.* **2**, 52–56.
- Palatinus, L. & Chapuis, G. (2007). *J. Appl. Cryst.* **40**, 786–790.
- Palatinus, L. & van der Lee, A. (2008). *J. Appl. Cryst.* **41**, 975–984.
- Palatinus, L., Prathapa, S. J. & van Smaalen, S. (2012). *J. Appl. Cryst.* **45**, 575–580.
- Qiu, X.-Y. & Yang, S.-L. (2006). *Acta Cryst.* **E62**, o5939–o5940.
- Rammohan, A., Reddy, J. S., Sravya, G., Rao, C. N. & Zyryanov, G. V. (2020). *Environ. Chem. Lett.* **18**, 433–458.
- Rosli, M. M., Patil, P. S., Fun, H.-K., Razak, I. A. & Dharmaparakash, S. M. (2007). *Acta Cryst.* **E63**, o2692.
- Sheldrick, G. M. (2015). *Acta Cryst.* **C71**, 3–8.
- Singh, P., Anand, A. & Kumar, V. (2014). *Eur. J. Med. Chem.* **85**, 758–777.
- Tomasch, M., Schwed, J. S., Weizel, L. & Stark, H. (2012). *Frontiers in Systems Neuroscience*, **6**, 14.
- Weygand, C. & Schächter, F. (1935). *Ber. Dtsch. Chem. Ges. A/B*, **68**, 227–234.
- Zhuang, C., Zhang, W., Sheng, C., Zhang, W., Xing, C. & Miao, Z. (2017). *Chem. Rev.* **117**, 7762–7810.

supporting information

Acta Cryst. (2020). E76, 1599-1604 [https://doi.org/10.1107/S2056989020011858]

Crystal structures of three functionalized chalcones: 4'-dimethylamino-3-nitrochalcone, 3-dimethylamino-3'-nitrochalcone and 3'-nitrochalcone

Charlie L. Hall, Victoria Hamilton, Jason Potticary, Matthew E. Cremeens, Natalie E. Pridmore, Hazel A. Sparkes, Gemma D. D'ambruoso, Stephen D. Warren, Masaomi Matsumoto and Simon R. Hall

Computing details

For all structures, data collection: *APEX2* (Bruker, 2016); cell refinement: *SAINTE* (Bruker, 2016); data reduction: *SAINTE* (Bruker, 2016); program(s) used to solve structure: Superflip (Palatinus & Chapuis, 2007; Palatinus & van der Lee, 2008; Palatinus *et al.*, 2012); program(s) used to refine structure: *SHELXL* (Sheldrick, 2015); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009) and *Mercury* (Macrae *et al.*, 2020); software used to prepare material for publication: *OLEX2* (Dolomanov *et al.*, 2009).

1-[4-(Dimethylamino)phenyl]-3-(3-nitrophenyl)prop-2-en-1-one (Gp8m)

Crystal data

$C_{17}H_{16}N_2O_3$	$F(000) = 624$
$M_r = 296.32$	$D_x = 1.416 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
$a = 17.3171 (7) \text{ \AA}$	Cell parameters from 2510 reflections
$b = 7.0708 (3) \text{ \AA}$	$\theta = 2.4\text{--}27.7^\circ$
$c = 11.3487 (4) \text{ \AA}$	$\mu = 0.10 \text{ mm}^{-1}$
$\beta = 90.761 (3)^\circ$	$T = 100 \text{ K}$
$V = 1389.48 (10) \text{ \AA}^3$	Plate, clear yellow
$Z = 4$	$0.39 \times 0.35 \times 0.19 \text{ mm}$

Data collection

Bruker APEXII Kappa CCD area detector diffractometer	12270 measured reflections
Radiation source: fine-focus sealed tube	3320 independent reflections
Graphite monochromator	2561 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{\text{int}} = 0.034$
Absorption correction: multi-scan (SADABS; Bruker, 2016)	$\theta_{\text{max}} = 27.9^\circ$, $\theta_{\text{min}} = 2.4^\circ$
$T_{\text{min}} = 0.666$, $T_{\text{max}} = 0.746$	$h = -22 \rightarrow 22$
	$k = -9 \rightarrow 9$
	$l = -14 \rightarrow 14$

Refinement

Refinement on F^2	3320 reflections
Least-squares matrix: full	212 parameters
$R[F^2 > 2\sigma(F^2)] = 0.043$	0 restraints
$wR(F^2) = 0.123$	Primary atom site location: iterative
$S = 1.04$	

Secondary atom site location: difference Fourier map
 Hydrogen site location: mixed
 H atoms treated by a mixture of independent and constrained refinement

$$w = 1/[\sigma^2(F_o^2) + (0.0678P)^2 + 0.1379P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.31 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.28 \text{ e } \text{\AA}^{-3}$

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.40268 (5)	0.64764 (14)	0.01669 (8)	0.0193 (2)
N1	0.07275 (6)	0.53937 (17)	0.25056 (10)	0.0205 (3)
C1	0.38550 (7)	0.64612 (17)	0.12161 (11)	0.0145 (3)
O2	0.81017 (6)	0.60742 (15)	0.17555 (9)	0.0254 (3)
N2	0.79887 (6)	0.59843 (16)	0.28197 (10)	0.0189 (3)
C2	0.44638 (7)	0.65714 (18)	0.21487 (12)	0.0162 (3)
H2	0.432687	0.695454	0.292062	0.019*
O3	0.85104 (6)	0.57987 (16)	0.35600 (9)	0.0292 (3)
C3	0.51981 (7)	0.61451 (17)	0.19275 (12)	0.0156 (3)
H3	0.532011	0.578168	0.114587	0.019*
C4	0.30435 (7)	0.62866 (17)	0.15869 (11)	0.0141 (3)
C5	0.24711 (7)	0.59749 (18)	0.07252 (11)	0.0154 (3)
H5	0.261307	0.595641	-0.008048	0.019*
C6	0.17102 (8)	0.56948 (18)	0.10089 (12)	0.0168 (3)
H6	0.133772	0.548627	0.040059	0.020*
C7	0.14770 (7)	0.57146 (18)	0.22001 (11)	0.0155 (3)
C8	0.20495 (7)	0.60892 (18)	0.30661 (12)	0.0172 (3)
H8	0.190843	0.615961	0.387096	0.021*
C9	0.28085 (7)	0.63541 (18)	0.27630 (12)	0.0163 (3)
H9	0.318238	0.658831	0.336557	0.020*
C10	0.58321 (7)	0.61938 (17)	0.27977 (11)	0.0149 (3)
C11	0.65932 (7)	0.60289 (18)	0.24156 (11)	0.0154 (3)
H11	0.669921	0.586105	0.160316	0.018*
C12	0.71904 (7)	0.61135 (17)	0.32371 (12)	0.0159 (3)
C13	0.70704 (8)	0.62847 (18)	0.44304 (12)	0.0176 (3)
H13	0.749151	0.632712	0.497489	0.021*
C14	0.63139 (8)	0.63927 (18)	0.48083 (12)	0.0187 (3)
H14	0.621331	0.647757	0.562745	0.022*
C15	0.57042 (8)	0.63789 (18)	0.40118 (12)	0.0174 (3)
H15	0.519110	0.649624	0.428883	0.021*
C16	0.01685 (9)	0.4836 (2)	0.16067 (14)	0.0246 (3)
C17	0.05477 (8)	0.5002 (2)	0.37305 (12)	0.0235 (3)
H17A	0.083637	0.388553	0.399692	0.035*
H17B	-0.000728	0.476410	0.380111	0.035*

H17C	0.069292	0.609223	0.421831	0.035*
H16A	0.0117 (10)	0.580 (2)	0.0989 (16)	0.033 (5)*
H16B	0.0326 (10)	0.362 (2)	0.1247 (15)	0.035 (5)*
H16C	-0.0330 (12)	0.467 (3)	0.1955 (17)	0.049 (5)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0175 (5)	0.0265 (5)	0.0139 (5)	0.0000 (4)	0.0017 (4)	0.0010 (4)
N1	0.0131 (6)	0.0338 (7)	0.0144 (6)	-0.0006 (5)	0.0011 (4)	-0.0003 (5)
C1	0.0165 (7)	0.0124 (6)	0.0147 (6)	0.0013 (5)	0.0006 (5)	-0.0002 (5)
O2	0.0188 (5)	0.0376 (6)	0.0199 (5)	-0.0004 (4)	0.0024 (4)	-0.0025 (4)
N2	0.0158 (6)	0.0198 (6)	0.0210 (6)	0.0003 (4)	-0.0025 (5)	-0.0024 (5)
C2	0.0175 (7)	0.0163 (6)	0.0148 (6)	-0.0001 (5)	-0.0003 (5)	-0.0010 (5)
O3	0.0167 (5)	0.0425 (7)	0.0281 (6)	0.0048 (4)	-0.0078 (4)	-0.0017 (5)
C3	0.0174 (7)	0.0157 (6)	0.0136 (6)	-0.0008 (5)	-0.0001 (5)	0.0003 (5)
C4	0.0154 (6)	0.0121 (6)	0.0147 (7)	0.0008 (5)	-0.0005 (5)	0.0002 (5)
C5	0.0182 (6)	0.0164 (6)	0.0117 (6)	0.0021 (5)	0.0010 (5)	-0.0005 (5)
C6	0.0156 (6)	0.0202 (7)	0.0143 (6)	0.0005 (5)	-0.0037 (5)	-0.0008 (5)
C7	0.0151 (6)	0.0160 (6)	0.0154 (6)	0.0016 (5)	0.0002 (5)	0.0006 (5)
C8	0.0187 (7)	0.0220 (7)	0.0111 (6)	0.0019 (5)	0.0010 (5)	0.0001 (5)
C9	0.0168 (7)	0.0181 (7)	0.0140 (6)	0.0007 (5)	-0.0027 (5)	-0.0009 (5)
C10	0.0163 (6)	0.0127 (6)	0.0157 (7)	0.0001 (5)	-0.0006 (5)	0.0005 (5)
C11	0.0169 (7)	0.0149 (6)	0.0143 (6)	0.0006 (5)	-0.0005 (5)	-0.0003 (5)
C12	0.0152 (6)	0.0145 (6)	0.0181 (7)	0.0018 (5)	-0.0006 (5)	0.0000 (5)
C13	0.0188 (7)	0.0163 (7)	0.0176 (7)	0.0005 (5)	-0.0054 (5)	-0.0004 (5)
C14	0.0254 (7)	0.0185 (7)	0.0122 (6)	-0.0001 (5)	0.0003 (5)	-0.0007 (5)
C15	0.0164 (7)	0.0175 (7)	0.0183 (7)	0.0003 (5)	0.0017 (5)	-0.0009 (5)
C16	0.0148 (7)	0.0382 (9)	0.0207 (8)	-0.0023 (6)	-0.0002 (6)	-0.0016 (7)
C17	0.0168 (7)	0.0359 (8)	0.0181 (7)	0.0008 (6)	0.0046 (5)	0.0051 (6)

Geometric parameters (Å, °)

O1—C1	1.2312 (15)	C8—H8	0.9500
N1—C7	1.3668 (16)	C8—C9	1.3759 (18)
N1—C16	1.4516 (18)	C9—H9	0.9500
N1—C17	1.4552 (17)	C10—C11	1.3979 (18)
C1—C2	1.4860 (18)	C10—C15	1.4045 (18)
C1—C4	1.4775 (17)	C11—H11	0.9500
O2—N2	1.2277 (15)	C11—C12	1.3845 (18)
N2—O3	1.2326 (14)	C12—C13	1.3781 (18)
N2—C12	1.4700 (17)	C13—H13	0.9500
C2—H2	0.9500	C13—C14	1.3861 (19)
C2—C3	1.3339 (18)	C14—H14	0.9500
C3—H3	0.9500	C14—C15	1.3808 (19)
C3—C10	1.4672 (18)	C15—H15	0.9500
C4—C5	1.4005 (17)	C16—H16A	0.983 (18)
C4—C9	1.4013 (18)	C16—H16B	0.989 (17)

C5—H5	0.9500	C16—H16C	0.96 (2)
C5—C6	1.3749 (18)	C17—H17A	0.9800
C6—H6	0.9500	C17—H17B	0.9800
C6—C7	1.4161 (18)	C17—H17C	0.9800
C7—C8	1.4117 (18)		
C7—N1—C16	119.57 (11)	C8—C9—H9	119.2
C7—N1—C17	119.33 (11)	C11—C10—C3	119.30 (12)
C16—N1—C17	118.06 (12)	C11—C10—C15	118.27 (12)
O1—C1—C2	120.67 (11)	C15—C10—C3	122.42 (12)
O1—C1—C4	121.28 (12)	C10—C11—H11	120.4
C4—C1—C2	118.04 (11)	C12—C11—C10	119.11 (12)
O2—N2—O3	123.44 (11)	C12—C11—H11	120.4
O2—N2—C12	118.47 (11)	C11—C12—N2	118.52 (12)
O3—N2—C12	118.09 (11)	C13—C12—N2	118.48 (12)
C1—C2—H2	119.2	C13—C12—C11	122.99 (12)
C3—C2—C1	121.55 (12)	C12—C13—H13	121.2
C3—C2—H2	119.2	C12—C13—C14	117.67 (12)
C2—C3—H3	117.5	C14—C13—H13	121.2
C2—C3—C10	125.07 (12)	C13—C14—H14	119.5
C10—C3—H3	117.5	C15—C14—C13	120.98 (13)
C5—C4—C1	118.77 (11)	C15—C14—H14	119.5
C5—C4—C9	117.30 (12)	C10—C15—H15	119.5
C9—C4—C1	123.90 (12)	C14—C15—C10	120.91 (12)
C4—C5—H5	119.0	C14—C15—H15	119.5
C6—C5—C4	122.05 (12)	N1—C16—H16A	111.4 (10)
C6—C5—H5	119.0	N1—C16—H16B	109.9 (10)
C5—C6—H6	119.7	N1—C16—H16C	109.8 (11)
C5—C6—C7	120.56 (12)	H16A—C16—H16B	109.4 (14)
C7—C6—H6	119.7	H16A—C16—H16C	107.8 (15)
N1—C7—C6	121.62 (12)	H16B—C16—H16C	108.4 (14)
N1—C7—C8	121.00 (12)	N1—C17—H17A	109.5
C8—C7—C6	117.38 (12)	N1—C17—H17B	109.5
C7—C8—H8	119.5	N1—C17—H17C	109.5
C9—C8—C7	121.06 (12)	H17A—C17—H17B	109.5
C9—C8—H8	119.5	H17A—C17—H17C	109.5
C4—C9—H9	119.2	H17B—C17—H17C	109.5
C8—C9—C4	121.59 (12)		
O1—C1—C2—C3	-19.7 (2)	C4—C5—C6—C7	0.1 (2)
O1—C1—C4—C5	5.38 (18)	C5—C4—C9—C8	1.23 (19)
O1—C1—C4—C9	-176.74 (12)	C5—C6—C7—N1	-178.37 (12)
N1—C7—C8—C9	177.95 (12)	C5—C6—C7—C8	1.93 (19)
C1—C2—C3—C10	-179.22 (11)	C6—C7—C8—C9	-2.35 (19)
C1—C4—C5—C6	176.37 (12)	C7—C8—C9—C4	0.8 (2)
C1—C4—C9—C8	-176.68 (12)	C9—C4—C5—C6	-1.66 (19)
O2—N2—C12—C11	10.09 (18)	C10—C11—C12—N2	-178.54 (11)
O2—N2—C12—C13	-170.87 (11)	C10—C11—C12—C13	2.5 (2)

N2—C12—C13—C14	-179.61 (11)	C11—C10—C15—C14	-0.17 (19)
C2—C1—C4—C5	-173.15 (11)	C11—C12—C13—C14	-0.61 (19)
C2—C1—C4—C9	4.74 (18)	C12—C13—C14—C15	-1.66 (19)
C2—C3—C10—C11	-169.23 (13)	C13—C14—C15—C10	2.1 (2)
C2—C3—C10—C15	11.5 (2)	C15—C10—C11—C12	-2.01 (18)
O3—N2—C12—C11	-170.20 (12)	C16—N1—C7—C6	6.14 (19)
O3—N2—C12—C13	8.84 (18)	C16—N1—C7—C8	-174.17 (13)
C3—C10—C11—C12	178.72 (12)	C17—N1—C7—C6	166.08 (12)
C3—C10—C15—C14	179.07 (12)	C17—N1—C7—C8	-14.22 (19)
C4—C1—C2—C3	158.84 (12)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C3—H3...O1 ⁱ	0.95	2.47	3.3124 (16)	148
C5—H5...O2 ⁱ	0.95	2.67	3.3067 (17)	125
C11—H11...O1 ⁱ	0.95	2.88	3.5773 (16)	131
C15—H15...O1 ⁱⁱ	0.95	2.68	3.5435 (16)	152
C17—H17B...O3 ⁱⁱⁱ	0.98	2.68	3.5755 (17)	152

Symmetry codes: (i) $-x+1, -y+1, -z$; (ii) $x, -y+3/2, z+1/2$; (iii) $x-1, y, z$.

3-[3-(Dimethylamino)phenyl]-1-(3-nitrophenyl)prop-2-en-1-one (Hm7m)*Crystal data*C₁₇H₁₆N₂O₃*M_r* = 296.32Monoclinic, *P*2₁/*n**a* = 7.7552 (4) Å*b* = 15.6998 (7) Å*c* = 12.0525 (7) Å β = 100.668 (3)°*V* = 1442.09 (13) Å³*Z* = 4*F*(000) = 624*D_x* = 1.365 Mg m⁻³Mo *K*α radiation, λ = 0.71073 Å

Cell parameters from 1681 reflections

 θ = 2.6–24.0° μ = 0.10 mm⁻¹*T* = 200 K

Block, clear orange

0.39 × 0.33 × 0.25 mm

*Data collection*Bruker APEXII Kappa CCD area detector
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

 φ and ω scans

Absorption correction: multi-scan

(SADABS; Bruker, 2016)

T_{min} = 0.629, *T_{max}* = 0.746

11497 measured reflections

3056 independent reflections

1882 reflections with *I* > 2σ(*I*)*R_{int}* = 0.058 θ_{\max} = 26.7°, θ_{\min} = 2.2°*h* = -7→9*k* = -19→19*l* = -15→13*Refinement*Refinement on *F*²

Least-squares matrix: full

R[*F*² > 2σ(*F*²)] = 0.049*wR*(*F*²) = 0.133*S* = 1.02

3056 reflections

201 parameters

0 restraints

Primary atom site location: iterative

Secondary atom site location: difference Fourier
mapHydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0622P)^2 + 0.0748P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$

$$\Delta\rho_{\max} = 0.19 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.24 \text{ e } \text{\AA}^{-3}$$

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.3894 (3)	0.47781 (12)	0.36738 (19)	0.0361 (5)
N1	0.7377 (2)	0.69758 (11)	0.21902 (15)	0.0364 (4)
O1	0.2877 (2)	0.53451 (9)	0.38190 (16)	0.0554 (5)
C2	0.3564 (3)	0.38878 (12)	0.39197 (18)	0.0356 (5)
H2	0.427006	0.345535	0.367975	0.043*
N2	-0.1631 (2)	0.18000 (10)	0.60898 (17)	0.0411 (5)
O2	0.62970 (19)	0.74937 (9)	0.23772 (14)	0.0445 (4)
C3	0.2296 (3)	0.36699 (12)	0.44729 (18)	0.0343 (5)
H3	0.162525	0.412295	0.469835	0.041*
O3	0.8619 (2)	0.71586 (10)	0.17312 (14)	0.0518 (5)
C4	0.5534 (3)	0.50134 (12)	0.32408 (18)	0.0316 (5)
C5	0.5701 (3)	0.58465 (12)	0.28981 (17)	0.0315 (5)
H5	0.478821	0.624712	0.291478	0.038*
C6	0.7212 (3)	0.60857 (12)	0.25325 (18)	0.0320 (5)
C7	0.8569 (3)	0.55285 (13)	0.24965 (19)	0.0388 (6)
H7	0.960216	0.571074	0.224810	0.047*
C8	0.8390 (3)	0.47026 (13)	0.2829 (2)	0.0431 (6)
H8	0.930866	0.430648	0.280479	0.052*
C9	0.6885 (3)	0.44375 (13)	0.32009 (19)	0.0386 (5)
H9	0.677876	0.386332	0.342771	0.046*
C10	0.1823 (3)	0.28126 (12)	0.47704 (18)	0.0311 (5)
C11	0.0358 (2)	0.27097 (12)	0.52753 (17)	0.0315 (5)
H11	-0.028145	0.319845	0.542776	0.038*
C12	-0.0194 (3)	0.19012 (12)	0.55642 (18)	0.0326 (5)
C13	0.0783 (3)	0.12007 (13)	0.5326 (2)	0.0406 (6)
H13	0.044151	0.064351	0.550403	0.049*
C14	0.2239 (3)	0.13081 (13)	0.4835 (2)	0.0441 (6)
H14	0.289136	0.082183	0.468976	0.053*
C15	0.2770 (3)	0.21003 (13)	0.4552 (2)	0.0395 (6)
H15	0.377263	0.216077	0.421012	0.047*
C16	-0.2649 (3)	0.25448 (14)	0.6266 (2)	0.0482 (6)
H16A	-0.187783	0.297063	0.669840	0.072*
H16B	-0.356654	0.238239	0.668615	0.072*
H16C	-0.319124	0.278526	0.553447	0.072*
C17	-0.2612 (3)	0.10108 (14)	0.5915 (2)	0.0464 (6)
H17A	-0.304660	0.092786	0.510518	0.070*

H17B	-0.360675	0.103638	0.631031	0.070*
H17C	-0.184628	0.053444	0.620960	0.070*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0362 (12)	0.0342 (12)	0.0402 (14)	-0.0013 (9)	0.0131 (11)	-0.0013 (9)
N1	0.0346 (10)	0.0413 (10)	0.0350 (12)	-0.0075 (8)	0.0110 (9)	0.0033 (8)
O1	0.0486 (10)	0.0365 (9)	0.0918 (15)	0.0039 (7)	0.0405 (10)	0.0111 (8)
C2	0.0374 (12)	0.0329 (11)	0.0389 (14)	-0.0031 (9)	0.0133 (11)	-0.0015 (9)
N2	0.0430 (11)	0.0332 (10)	0.0531 (13)	-0.0082 (8)	0.0248 (10)	-0.0013 (9)
O2	0.0434 (9)	0.0373 (9)	0.0564 (12)	0.0024 (7)	0.0188 (8)	0.0063 (7)
C3	0.0348 (11)	0.0319 (11)	0.0382 (14)	-0.0029 (8)	0.0115 (10)	-0.0020 (9)
O3	0.0476 (10)	0.0526 (10)	0.0637 (13)	-0.0116 (7)	0.0324 (9)	0.0073 (8)
C4	0.0325 (11)	0.0301 (11)	0.0340 (13)	-0.0026 (8)	0.0111 (10)	-0.0020 (9)
C5	0.0298 (11)	0.0327 (11)	0.0338 (13)	-0.0015 (8)	0.0108 (10)	-0.0014 (9)
C6	0.0330 (11)	0.0335 (11)	0.0310 (12)	-0.0053 (9)	0.0096 (10)	-0.0002 (9)
C7	0.0328 (11)	0.0441 (13)	0.0434 (15)	-0.0037 (9)	0.0175 (11)	-0.0006 (10)
C8	0.0353 (12)	0.0400 (13)	0.0578 (17)	0.0046 (9)	0.0189 (12)	-0.0017 (11)
C9	0.0411 (12)	0.0322 (12)	0.0455 (15)	0.0002 (9)	0.0159 (11)	0.0003 (10)
C10	0.0306 (10)	0.0314 (11)	0.0322 (13)	-0.0052 (8)	0.0077 (10)	-0.0016 (9)
C11	0.0319 (11)	0.0306 (11)	0.0330 (13)	0.0008 (8)	0.0090 (10)	-0.0029 (9)
C12	0.0351 (11)	0.0329 (11)	0.0308 (13)	-0.0049 (9)	0.0084 (10)	-0.0017 (9)
C13	0.0452 (13)	0.0274 (11)	0.0513 (16)	-0.0060 (9)	0.0145 (12)	-0.0011 (10)
C14	0.0441 (13)	0.0310 (11)	0.0615 (17)	0.0015 (9)	0.0211 (13)	-0.0060 (11)
C15	0.0371 (12)	0.0376 (12)	0.0481 (15)	-0.0031 (9)	0.0191 (11)	-0.0037 (10)
C16	0.0449 (13)	0.0449 (14)	0.0622 (18)	-0.0052 (10)	0.0292 (13)	-0.0033 (11)
C17	0.0441 (13)	0.0441 (13)	0.0537 (17)	-0.0120 (10)	0.0160 (12)	0.0068 (11)

Geometric parameters (Å, °)

C1—O1	1.223 (2)	C8—H8	0.9500
C1—C2	1.461 (3)	C8—C9	1.389 (3)
C1—C4	1.507 (3)	C9—H9	0.9500
N1—O2	1.218 (2)	C10—C11	1.394 (2)
N1—O3	1.2303 (19)	C10—C15	1.389 (3)
N1—C6	1.469 (3)	C11—H11	0.9500
C2—H2	0.9500	C11—C12	1.404 (3)
C2—C3	1.331 (3)	C12—C13	1.395 (3)
N2—C12	1.389 (2)	C13—H13	0.9500
N2—C16	1.448 (3)	C13—C14	1.378 (3)
N2—C17	1.449 (3)	C14—H14	0.9500
C3—H3	0.9500	C14—C15	1.373 (3)
C3—C10	1.457 (3)	C15—H15	0.9500
C4—C5	1.385 (3)	C16—H16A	0.9800
C4—C9	1.392 (3)	C16—H16B	0.9800
C5—H5	0.9500	C16—H16C	0.9800
C5—C6	1.378 (2)	C17—H17A	0.9800

C6—C7	1.376 (3)	C17—H17B	0.9800
C7—H7	0.9500	C17—H17C	0.9800
C7—C8	1.372 (3)		
O1—C1—C2	121.68 (18)	C8—C9—H9	120.0
O1—C1—C4	118.63 (18)	C11—C10—C3	118.49 (16)
C2—C1—C4	119.69 (17)	C15—C10—C3	122.07 (17)
O2—N1—O3	123.37 (17)	C15—C10—C11	119.43 (17)
O2—N1—C6	118.95 (15)	C10—C11—H11	119.2
O3—N1—C6	117.68 (16)	C10—C11—C12	121.59 (17)
C1—C2—H2	119.3	C12—C11—H11	119.2
C3—C2—C1	121.38 (18)	N2—C12—C11	121.59 (17)
C3—C2—H2	119.3	N2—C12—C13	121.05 (17)
C12—N2—C16	118.67 (16)	C13—C12—C11	117.34 (18)
C12—N2—C17	118.37 (16)	C12—C13—H13	119.6
C16—N2—C17	115.23 (17)	C14—C13—C12	120.73 (18)
C2—C3—H3	116.4	C14—C13—H13	119.6
C2—C3—C10	127.12 (18)	C13—C14—H14	119.2
C10—C3—H3	116.4	C15—C14—C13	121.64 (18)
C5—C4—C1	117.81 (16)	C15—C14—H14	119.2
C5—C4—C9	119.36 (17)	C10—C15—H15	120.4
C9—C4—C1	122.81 (18)	C14—C15—C10	119.26 (18)
C4—C5—H5	120.5	C14—C15—H15	120.4
C6—C5—C4	119.01 (17)	N2—C16—H16A	109.5
C6—C5—H5	120.5	N2—C16—H16B	109.5
C5—C6—N1	118.21 (17)	N2—C16—H16C	109.5
C7—C6—N1	119.26 (17)	H16A—C16—H16B	109.5
C7—C6—C5	122.52 (19)	H16A—C16—H16C	109.5
C6—C7—H7	120.9	H16B—C16—H16C	109.5
C8—C7—C6	118.21 (17)	N2—C17—H17A	109.5
C8—C7—H7	120.9	N2—C17—H17B	109.5
C7—C8—H8	119.5	N2—C17—H17C	109.5
C7—C8—C9	120.91 (18)	H17A—C17—H17B	109.5
C9—C8—H8	119.5	H17A—C17—H17C	109.5
C4—C9—H9	120.0	H17B—C17—H17C	109.5
C8—C9—C4	119.98 (19)		
C1—C2—C3—C10	179.8 (2)	C4—C5—C6—N1	-179.00 (19)
C1—C4—C5—C6	177.95 (19)	C4—C5—C6—C7	-0.2 (3)
C1—C4—C9—C8	-177.7 (2)	C5—C4—C9—C8	0.6 (3)
N1—C6—C7—C8	179.5 (2)	C5—C6—C7—C8	0.6 (3)
O1—C1—C2—C3	-10.6 (4)	C6—C7—C8—C9	-0.5 (4)
O1—C1—C4—C5	-7.7 (3)	C7—C8—C9—C4	-0.1 (4)
O1—C1—C4—C9	170.6 (2)	C9—C4—C5—C6	-0.4 (3)
C2—C1—C4—C5	172.9 (2)	C10—C11—C12—N2	178.6 (2)
C2—C1—C4—C9	-8.8 (3)	C10—C11—C12—C13	0.0 (3)
C2—C3—C10—C11	-175.0 (2)	C11—C10—C15—C14	0.2 (3)
C2—C3—C10—C15	4.1 (4)	C11—C12—C13—C14	0.5 (3)

N2—C12—C13—C14	-178.0 (2)	C12—C13—C14—C15	-0.8 (4)
O2—N1—C6—C5	9.5 (3)	C13—C14—C15—C10	0.4 (4)
O2—N1—C6—C7	-169.4 (2)	C15—C10—C11—C12	-0.4 (3)
C3—C10—C11—C12	178.7 (2)	C16—N2—C12—C11	4.5 (3)
C3—C10—C15—C14	-178.9 (2)	C16—N2—C12—C13	-177.0 (2)
O3—N1—C6—C5	-170.91 (19)	C17—N2—C12—C11	152.4 (2)
O3—N1—C6—C7	10.2 (3)	C17—N2—C12—C13	-29.1 (3)
C4—C1—C2—C3	168.8 (2)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
C15—H15 \cdots O3 ⁱ	0.95	2.50	3.442 (2)	174
C16—H16B \cdots O2 ⁱⁱ	0.98	2.58	3.520 (2)	160

Symmetry codes: (i) $-x+3/2, y-1/2, -z+1/2$; (ii) $-x, -y+1, -z+1$.**1-(3-Nitrophenyl)-3-phenylprop-2-en-1-one (Hm1-)***Crystal data*C₁₅H₁₁NO₃*M_r* = 253.25Monoclinic, *P*2₁/*c**a* = 14.7856 (8) Å*b* = 15.9841 (9) Å*c* = 10.3188 (6) Å β = 99.210 (4)°*V* = 2407.3 (2) Å³*Z* = 8*F*(000) = 1056*D_x* = 1.398 Mg m⁻³Mo *K* α radiation, λ = 0.71073 Å

Cell parameters from 2603 reflections

 θ = 2.4–24.0° μ = 0.10 mm⁻¹*T* = 100 K

Block, clear colourless

0.61 × 0.35 × 0.25 mm

*Data collection*Bruker APEXII Kappa CCD area detector
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

 φ and ω scans

Absorption correction: multi-scan

(SADABS; Bruker, 2016)

T_{min} = 0.610, *T_{max}* = 0.746

25080 measured reflections

4408 independent reflections

2657 reflections with *I* > 2 σ (*I*)*R_{int}* = 0.085 θ_{\max} = 25.4°, θ_{\min} = 1.4°*h* = -17→17*k* = -18→19*l* = -11→12*Refinement*Refinement on *F*²

Least-squares matrix: full

R[*F*² > 2 σ (*F*²)] = 0.049*wR*(*F*²) = 0.141*S* = 0.99

4408 reflections

343 parameters

0 restraints

Primary atom site location: iterative

Secondary atom site location: difference Fourier
mapHydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + (0.0721P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} < 0.001$ $\Delta\rho_{\max} = 0.28 \text{ e \AA}^{-3}$ $\Delta\rho_{\min} = -0.24 \text{ e \AA}^{-3}$

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.15404 (12)	0.16690 (10)	0.61388 (17)	0.0359 (5)
O2	0.01454 (14)	0.38743 (11)	0.34410 (18)	0.0473 (6)
O3	-0.05654 (12)	0.36438 (11)	0.14763 (17)	0.0383 (5)
N1	-0.01292 (15)	0.34002 (13)	0.2521 (2)	0.0323 (5)
C1	0.12448 (16)	0.11250 (15)	0.5350 (2)	0.0254 (6)
C2	0.14090 (16)	0.02299 (14)	0.5655 (2)	0.0255 (6)
H2	0.112881	-0.018632	0.506696	0.031*
C3	0.19525 (16)	0.00085 (14)	0.6761 (2)	0.0253 (6)
H3	0.220584	0.045244	0.731502	0.030*
C4	0.07058 (15)	0.13797 (14)	0.4054 (2)	0.0231 (6)
C5	0.05461 (16)	0.22346 (14)	0.3858 (2)	0.0248 (6)
H5	0.077179	0.262547	0.452566	0.030*
C6	0.00593 (16)	0.25020 (14)	0.2690 (2)	0.0252 (6)
C7	-0.02844 (17)	0.19594 (15)	0.1684 (2)	0.0279 (6)
H7	-0.062116	0.216229	0.088457	0.033*
C8	-0.01236 (16)	0.11190 (15)	0.1879 (2)	0.0271 (6)
H8	-0.034651	0.073409	0.120088	0.032*
C9	0.03611 (16)	0.08256 (15)	0.3055 (2)	0.0258 (6)
H9	0.045864	0.024162	0.317841	0.031*
C10	0.22033 (16)	-0.08377 (14)	0.7218 (2)	0.0238 (6)
C11	0.26673 (16)	-0.09467 (15)	0.8493 (2)	0.0266 (6)
H11	0.282266	-0.047021	0.903293	0.032*
C12	0.29063 (17)	-0.17356 (15)	0.8988 (2)	0.0301 (6)
H12	0.321996	-0.179789	0.986089	0.036*
C13	0.26862 (17)	-0.24322 (16)	0.8206 (3)	0.0325 (6)
H13	0.284275	-0.297540	0.854305	0.039*
C14	0.22364 (17)	-0.23354 (15)	0.6929 (3)	0.0302 (6)
H14	0.209076	-0.281420	0.639024	0.036*
C15	0.19979 (17)	-0.15487 (15)	0.6432 (2)	0.0282 (6)
H15	0.169329	-0.148993	0.555349	0.034*
O4	0.35937 (12)	0.39992 (10)	0.37652 (16)	0.0314 (4)
O5	0.44124 (12)	0.62138 (10)	0.68540 (17)	0.0357 (5)
O6	0.52132 (14)	0.59755 (11)	0.87609 (17)	0.0447 (5)
N2	0.48438 (15)	0.57425 (13)	0.7669 (2)	0.0317 (5)
C16	0.38593 (16)	0.34642 (15)	0.4587 (2)	0.0261 (6)
C17	0.36217 (17)	0.25751 (15)	0.4363 (2)	0.0273 (6)
H17	0.385793	0.216860	0.499959	0.033*
C18	0.30757 (16)	0.23402 (15)	0.3268 (2)	0.0254 (6)
H18	0.285045	0.277608	0.267756	0.031*

C19	0.44235 (15)	0.37308 (14)	0.5864 (2)	0.0231 (5)
C20	0.44373 (15)	0.45800 (14)	0.6154 (2)	0.0236 (6)
H20	0.413123	0.496873	0.553799	0.028*
C21	0.48994 (17)	0.48510 (15)	0.7343 (2)	0.0262 (6)
C22	0.53737 (16)	0.43117 (16)	0.8256 (2)	0.0294 (6)
H22	0.568880	0.451388	0.907075	0.035*
C23	0.53761 (16)	0.34715 (16)	0.7950 (2)	0.0283 (6)
H23	0.570586	0.309004	0.855604	0.034*
C24	0.49037 (16)	0.31779 (15)	0.6772 (2)	0.0266 (6)
H24	0.490620	0.259682	0.657880	0.032*
C25	0.27816 (15)	0.14936 (15)	0.2866 (2)	0.0243 (6)
C30	0.29847 (16)	0.07941 (15)	0.3671 (3)	0.0286 (6)
H30	0.331611	0.086022	0.453284	0.034*
C29	0.27051 (17)	0.00044 (15)	0.3220 (3)	0.0323 (6)
H29	0.284069	-0.046713	0.377719	0.039*
C28	0.22299 (18)	-0.01024 (16)	0.1962 (3)	0.0357 (7)
H28	0.204939	-0.064705	0.165510	0.043*
C27	0.20172 (17)	0.05826 (16)	0.1152 (3)	0.0329 (6)
H27	0.168703	0.051101	0.029069	0.039*
C26	0.22886 (16)	0.13736 (15)	0.1605 (2)	0.0270 (6)
H26	0.213718	0.184348	0.104843	0.032*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0504 (12)	0.0234 (10)	0.0272 (10)	0.0002 (8)	-0.0146 (9)	-0.0021 (8)
O2	0.0765 (15)	0.0291 (11)	0.0296 (11)	0.0078 (10)	-0.0120 (10)	-0.0019 (9)
O3	0.0471 (12)	0.0345 (11)	0.0276 (11)	0.0066 (9)	-0.0113 (9)	0.0096 (8)
N1	0.0386 (13)	0.0309 (12)	0.0249 (13)	0.0054 (10)	-0.0025 (11)	0.0036 (10)
C1	0.0241 (13)	0.0262 (13)	0.0239 (14)	-0.0004 (10)	-0.0020 (11)	0.0002 (11)
C2	0.0295 (14)	0.0218 (13)	0.0237 (14)	-0.0008 (10)	-0.0008 (11)	-0.0023 (10)
C3	0.0298 (14)	0.0219 (13)	0.0230 (14)	-0.0028 (10)	0.0008 (11)	-0.0005 (10)
C4	0.0228 (13)	0.0245 (13)	0.0213 (13)	-0.0006 (10)	0.0011 (11)	0.0032 (11)
C5	0.0289 (14)	0.0236 (14)	0.0207 (13)	-0.0008 (10)	0.0005 (11)	0.0001 (10)
C6	0.0301 (14)	0.0241 (13)	0.0205 (13)	0.0053 (11)	0.0015 (11)	0.0022 (11)
C7	0.0290 (14)	0.0349 (15)	0.0185 (13)	0.0012 (11)	0.0001 (11)	0.0041 (11)
C8	0.0290 (14)	0.0294 (14)	0.0215 (14)	-0.0017 (11)	0.0003 (11)	-0.0023 (11)
C9	0.0275 (13)	0.0259 (14)	0.0230 (14)	0.0001 (10)	0.0010 (11)	0.0013 (11)
C10	0.0245 (13)	0.0245 (13)	0.0213 (13)	-0.0012 (10)	0.0003 (11)	0.0017 (10)
C11	0.0291 (14)	0.0261 (13)	0.0233 (14)	-0.0003 (11)	0.0007 (11)	0.0007 (11)
C12	0.0287 (14)	0.0348 (15)	0.0248 (14)	0.0021 (11)	-0.0024 (12)	0.0061 (12)
C13	0.0365 (15)	0.0248 (14)	0.0358 (16)	0.0039 (11)	0.0047 (13)	0.0062 (12)
C14	0.0348 (15)	0.0255 (14)	0.0299 (15)	-0.0010 (11)	0.0042 (12)	-0.0016 (11)
C15	0.0324 (14)	0.0275 (14)	0.0236 (14)	-0.0006 (11)	0.0015 (12)	0.0021 (11)
O4	0.0389 (10)	0.0259 (9)	0.0255 (10)	-0.0009 (8)	-0.0074 (8)	0.0028 (8)
O5	0.0439 (11)	0.0262 (10)	0.0331 (11)	0.0037 (8)	-0.0057 (9)	-0.0007 (8)
O6	0.0665 (14)	0.0374 (11)	0.0248 (11)	-0.0098 (10)	-0.0090 (10)	-0.0076 (9)
N2	0.0390 (13)	0.0318 (12)	0.0227 (13)	-0.0049 (10)	0.0002 (11)	-0.0043 (10)

C16	0.0252 (13)	0.0274 (14)	0.0256 (14)	0.0022 (11)	0.0034 (11)	-0.0016 (11)
C17	0.0335 (14)	0.0224 (13)	0.0243 (14)	0.0002 (11)	-0.0003 (12)	0.0021 (11)
C18	0.0275 (14)	0.0247 (13)	0.0234 (14)	0.0020 (10)	0.0016 (11)	0.0009 (10)
C19	0.0245 (13)	0.0246 (14)	0.0199 (13)	-0.0026 (10)	0.0027 (11)	0.0005 (10)
C20	0.0258 (13)	0.0236 (13)	0.0207 (13)	-0.0022 (10)	0.0014 (11)	0.0014 (10)
C21	0.0294 (14)	0.0252 (13)	0.0241 (14)	-0.0010 (11)	0.0044 (11)	-0.0006 (11)
C22	0.0296 (14)	0.0366 (15)	0.0206 (14)	-0.0043 (11)	-0.0002 (11)	-0.0002 (11)
C23	0.0249 (13)	0.0316 (15)	0.0269 (15)	0.0014 (11)	-0.0009 (11)	0.0070 (11)
C24	0.0291 (14)	0.0258 (14)	0.0238 (14)	-0.0005 (10)	0.0007 (11)	0.0008 (11)
C25	0.0195 (12)	0.0272 (14)	0.0262 (14)	0.0006 (10)	0.0036 (11)	-0.0035 (11)
C30	0.0272 (13)	0.0274 (14)	0.0300 (15)	0.0014 (11)	0.0004 (11)	-0.0005 (11)
C29	0.0334 (15)	0.0239 (14)	0.0399 (17)	-0.0009 (11)	0.0071 (13)	-0.0014 (12)
C28	0.0363 (16)	0.0263 (15)	0.0450 (18)	-0.0023 (12)	0.0079 (14)	-0.0093 (13)
C27	0.0278 (14)	0.0366 (16)	0.0333 (16)	-0.0029 (11)	0.0022 (12)	-0.0095 (12)
C26	0.0270 (13)	0.0271 (14)	0.0266 (14)	0.0006 (11)	0.0034 (11)	-0.0018 (11)

Geometric parameters (Å, °)

O1—C1	1.223 (3)	O4—C16	1.224 (3)
O2—N1	1.232 (3)	O5—N2	1.229 (3)
O3—N1	1.227 (2)	O6—N2	1.229 (3)
N1—C6	1.468 (3)	N2—C21	1.469 (3)
C1—C2	1.477 (3)	C16—C17	1.473 (3)
C1—C4	1.499 (3)	C16—C19	1.504 (3)
C2—H2	0.9500	C17—H17	0.9500
C2—C3	1.334 (3)	C17—C18	1.333 (3)
C3—H3	0.9500	C18—H18	0.9500
C3—C10	1.460 (3)	C18—C25	1.461 (3)
C4—C5	1.396 (3)	C19—C20	1.389 (3)
C4—C9	1.393 (3)	C19—C24	1.396 (3)
C5—H5	0.9500	C20—H20	0.9500
C5—C6	1.370 (3)	C20—C21	1.375 (3)
C6—C7	1.385 (3)	C21—C22	1.383 (3)
C7—H7	0.9500	C22—H22	0.9500
C7—C8	1.373 (3)	C22—C23	1.380 (3)
C8—H8	0.9500	C23—H23	0.9500
C8—C9	1.388 (3)	C23—C24	1.384 (3)
C9—H9	0.9500	C24—H24	0.9500
C10—C11	1.394 (3)	C25—C30	1.397 (3)
C10—C15	1.401 (3)	C25—C26	1.399 (3)
C11—H11	0.9500	C30—H30	0.9500
C11—C12	1.385 (3)	C30—C29	1.386 (3)
C12—H12	0.9500	C29—H29	0.9500
C12—C13	1.383 (4)	C29—C28	1.384 (4)
C13—H13	0.9500	C28—H28	0.9500
C13—C14	1.387 (4)	C28—C27	1.383 (4)
C14—H14	0.9500	C27—H27	0.9500
C14—C15	1.382 (3)	C27—C26	1.385 (3)

C15—H15	0.9500	C26—H26	0.9500
O2—N1—C6	118.5 (2)	O5—N2—O6	123.2 (2)
O3—N1—O2	122.9 (2)	O5—N2—C21	118.7 (2)
O3—N1—C6	118.5 (2)	O6—N2—C21	118.1 (2)
O1—C1—C2	121.2 (2)	O4—C16—C17	121.5 (2)
O1—C1—C4	118.9 (2)	O4—C16—C19	118.7 (2)
C2—C1—C4	119.9 (2)	C17—C16—C19	119.7 (2)
C1—C2—H2	120.1	C16—C17—H17	119.9
C3—C2—C1	119.7 (2)	C18—C17—C16	120.1 (2)
C3—C2—H2	120.1	C18—C17—H17	119.9
C2—C3—H3	116.2	C17—C18—H18	116.1
C2—C3—C10	127.5 (2)	C17—C18—C25	127.9 (2)
C10—C3—H3	116.2	C25—C18—H18	116.1
C5—C4—C1	116.7 (2)	C20—C19—C16	116.9 (2)
C9—C4—C1	124.5 (2)	C20—C19—C24	119.1 (2)
C9—C4—C5	118.8 (2)	C24—C19—C16	124.0 (2)
C4—C5—H5	120.5	C19—C20—H20	120.4
C6—C5—C4	119.1 (2)	C21—C20—C19	119.2 (2)
C6—C5—H5	120.5	C21—C20—H20	120.4
C5—C6—N1	118.2 (2)	C20—C21—N2	118.1 (2)
C5—C6—C7	122.8 (2)	C20—C21—C22	122.5 (2)
C7—C6—N1	118.9 (2)	C22—C21—N2	119.3 (2)
C6—C7—H7	121.0	C21—C22—H22	121.0
C8—C7—C6	118.0 (2)	C23—C22—C21	118.1 (2)
C8—C7—H7	121.0	C23—C22—H22	121.0
C7—C8—H8	119.6	C22—C23—H23	119.6
C7—C8—C9	120.7 (2)	C22—C23—C24	120.8 (2)
C9—C8—H8	119.6	C24—C23—H23	119.6
C4—C9—H9	119.7	C19—C24—H24	119.8
C8—C9—C4	120.6 (2)	C23—C24—C19	120.4 (2)
C8—C9—H9	119.7	C23—C24—H24	119.8
C11—C10—C3	118.8 (2)	C30—C25—C18	123.1 (2)
C11—C10—C15	118.2 (2)	C30—C25—C26	118.3 (2)
C15—C10—C3	123.0 (2)	C26—C25—C18	118.6 (2)
C10—C11—H11	119.3	C25—C30—H30	119.8
C12—C11—C10	121.3 (2)	C29—C30—C25	120.3 (2)
C12—C11—H11	119.3	C29—C30—H30	119.8
C11—C12—H12	120.2	C30—C29—H29	119.8
C13—C12—C11	119.7 (2)	C28—C29—C30	120.5 (2)
C13—C12—H12	120.2	C28—C29—H29	119.8
C12—C13—H13	120.1	C29—C28—H28	120.0
C12—C13—C14	119.8 (2)	C27—C28—C29	120.1 (2)
C14—C13—H13	120.1	C27—C28—H28	120.0
C13—C14—H14	119.7	C28—C27—H27	120.2
C15—C14—C13	120.6 (2)	C28—C27—C26	119.6 (2)
C15—C14—H14	119.7	C26—C27—H27	120.2
C10—C15—H15	119.8	C25—C26—H26	119.4

C14—C15—C10	120.3 (2)	C27—C26—C25	121.2 (2)
C14—C15—H15	119.8	C27—C26—H26	119.4
O1—C1—C2—C3	-5.7 (4)	O4—C16—C17—C18	-2.7 (4)
O1—C1—C4—C5	-2.5 (3)	O4—C16—C19—C20	14.0 (3)
O1—C1—C4—C9	177.5 (2)	O4—C16—C19—C24	-167.8 (2)
O2—N1—C6—C5	-1.1 (3)	O5—N2—C21—C20	-2.2 (3)
O2—N1—C6—C7	176.9 (2)	O5—N2—C21—C22	-179.3 (2)
O3—N1—C6—C5	-180.0 (2)	O6—N2—C21—C20	175.9 (2)
O3—N1—C6—C7	-2.0 (3)	O6—N2—C21—C22	-1.1 (3)
N1—C6—C7—C8	-178.0 (2)	N2—C21—C22—C23	176.8 (2)
C1—C2—C3—C10	-179.1 (2)	C16—C17—C18—C25	178.7 (2)
C1—C4—C5—C6	179.8 (2)	C16—C19—C20—C21	176.3 (2)
C1—C4—C9—C8	-179.3 (2)	C16—C19—C24—C23	-177.3 (2)
C2—C1—C4—C5	177.4 (2)	C17—C16—C19—C20	-164.6 (2)
C2—C1—C4—C9	-2.6 (4)	C17—C16—C19—C24	13.6 (4)
C2—C3—C10—C11	-170.3 (2)	C17—C18—C25—C30	5.8 (4)
C2—C3—C10—C15	9.6 (4)	C17—C18—C25—C26	-172.9 (2)
C3—C10—C11—C12	178.7 (2)	C18—C25—C30—C29	-178.4 (2)
C3—C10—C15—C14	-178.6 (2)	C18—C25—C26—C27	177.9 (2)
C4—C1—C2—C3	174.4 (2)	C19—C16—C17—C18	175.9 (2)
C4—C5—C6—N1	177.9 (2)	C19—C20—C21—N2	-175.3 (2)
C4—C5—C6—C7	0.0 (4)	C19—C20—C21—C22	1.7 (4)
C5—C4—C9—C8	0.7 (4)	C20—C19—C24—C23	0.9 (4)
C5—C6—C7—C8	-0.2 (4)	C20—C21—C22—C23	-0.1 (4)
C6—C7—C8—C9	0.7 (4)	C21—C22—C23—C24	-1.1 (4)
C7—C8—C9—C4	-0.9 (4)	C22—C23—C24—C19	0.7 (4)
C9—C4—C5—C6	-0.2 (4)	C24—C19—C20—C21	-2.0 (3)
C10—C11—C12—C13	0.3 (4)	C25—C30—C29—C28	0.6 (4)
C11—C10—C15—C14	1.3 (4)	C30—C25—C26—C27	-0.9 (4)
C11—C12—C13—C14	0.6 (4)	C30—C29—C28—C27	-1.0 (4)
C12—C13—C14—C15	-0.6 (4)	C29—C28—C27—C26	0.5 (4)
C13—C14—C15—C10	-0.4 (4)	C28—C27—C26—C25	0.5 (4)
C15—C10—C11—C12	-1.2 (4)	C26—C25—C30—C29	0.3 (4)

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C2—H2 \cdots O3 ⁱ	0.95	2.51	3.456 (3)	174
C3—H3 \cdots O4 ⁱⁱ	0.95	2.50	3.328 (3)	146
C9—H9 \cdots O3 ⁱ	0.95	2.58	3.527 (3)	175
C15—H15 \cdots O3 ⁱ	0.95	2.47	3.399 (3)	166
C17—H17 \cdots O6 ⁱⁱⁱ	0.95	2.57	3.491 (3)	164
C18—H18 \cdots O1 ^{iv}	0.95	2.46	3.300 (3)	147
C30—H30 \cdots O6 ⁱⁱⁱ	0.95	2.58	3.456 (3)	154
C26—H26 \cdots O1 ^{iv}	0.95	2.54	3.328 (3)	140

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