

(2*E*)-3-(6-Methoxynaphthalen-2-yl)-1-(pyridin-3-yl)prop-2-en-1-one and its cyclocondensation product with guanidine, (4*RS*)-2-amino-4-(6-methoxynaphthalen-2-yl)-6-(pyridin-3-yl)-3,4-dihydropyrimidine monohydrate: two types of hydrogen-bonded sheet

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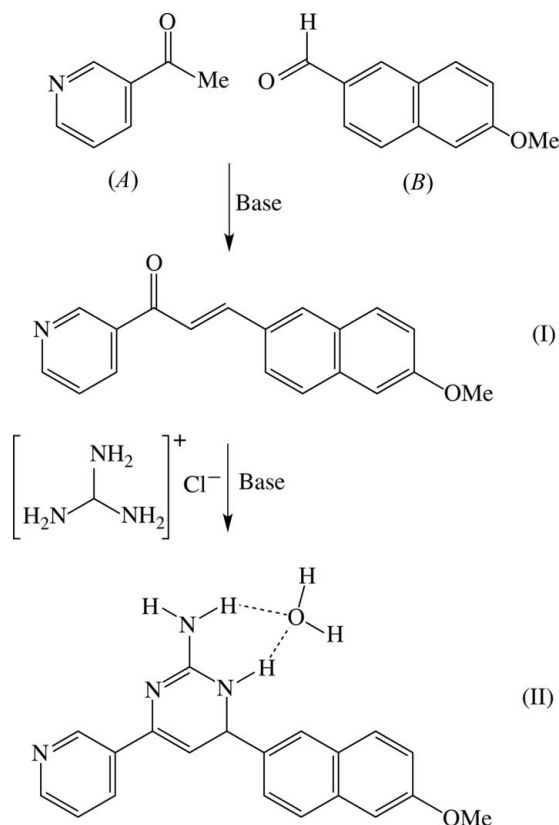
The structures of a chalcone and of its cyclocondensation product with guanidine are reported. In (2*E*)-3-(6-methoxynaphthalen-2-yl)-1-(pyridin-3-yl)prop-2-en-1-one, C₁₉H₁₅NO₂, (I), the planes of the pyridine and naphthalene units make dihedral angles with that of the central spacer unit of 23.61 (13) and 23.57 (15)°, respectively, and a dihedral angle of 47.24 (9)° with each other. The molecules of (I) are linked into sheets by a combination of C—H···O and C—H···π(arene) hydrogen bonds. In the cyclocondensation product (4*RS*)-2-amino-4-(6-methoxynaphthalen-2-yl)-6-(pyridin-3-yl)-3,4-dihydropyrimidine monohydrate, C₂₀H₁₈N₄O·H₂O, (II), the dihydropyrimidine ring adopts a conformation best described as a shallow boat. The molecular components are linked by two N—H···O hydrogen bonds, two O—H···N hydrogen bonds and one N—H···N hydrogen bond to form complex sheets, with the methoxynaphthalene interdigitated between inversion-related pairs of sheets.

Keywords: crystal structure; hydrogen bonding; chalcones; cyclocondensation; prop-2-en-1-one; 3,4-dihydropyrimidine; Claisen condensation.

1. Introduction

Chalcones (1,3-disubstituted prop-2-en-1-ones of the form R¹COCH=CHR²) which contain methoxy substituents exhibit potential as effective pharmaceutical agents in a

number of applications, such as anticancer agents (Lawrence *et al.*, 2006) and anti-infective and anti-inflammatory agents (Nowakowska, 2007). It has been observed that, when methoxy groups are present in chalcones, they can act as good acceptors of hydrogen bonds, while electron-rich naphthyl rings can participate in π–π stacking interactions, and both of these properties can play important roles in orientating inhibitors within the active sites of enzymes (Mascarello *et al.*, 2010). In addition, pyrimidine derivatives display a wide range of biological and pharmacological properties, such as anticancer (Petrie *et al.*, 1985), anti-inflammatory (Sondhi *et al.*, 2001) and antitumour (Baraldi *et al.*, 2002) activities. Prompted by these considerations, we have now prepared and structurally characterized the title chalcone (2*E*)-3-(6-methoxynaphthalen-2-yl)-1-(pyridin-3-yl)prop-2-en-1-one, (I) (Fig. 1), which incorporates a methoxy-substituted naphthyl group. Compound (I) was prepared by Claisen condensation between 3-acetyl pyridine (A) (see Scheme) and 6-methoxynaphthalene-2-carbaldehyde (B).



Chalcones can exhibit two distinct reactivity modes, namely Michael addition at the C=C double bond and condensation at the carbonyl group, and when these two modes are active in tandem new cyclic structures can result. Thus, for example, the cyclocondensation reactions of chalcones with simple hydrazines lead to the formation of dihydropyrazole derivatives (Fun *et al.*, 2010; Jasinski, Guild *et al.*, 2010; Jasinski, Pek *et al.*, 2010; Samshuddin *et al.*, 2010). Following these precedents, we have now prepared the title dihydropyrimidine derivative (4*RS*)-2-amino-4-(6-methoxynaphthalen-2-yl)-6-(pyridin-3-yl)-3,4-dihydropyrimidine monohydrate, (II) (Fig. 2), by

cyclocondensation of chalcone (I) with guanidinium chloride under basic conditions (see Scheme), and we report here the structure of (II) also. The aims of the present study are the determination of the conformations of the molecular constitutions of (I) and (II) and the exploration of their supra-molecular assembly, in particular, the influence of both the naphthyl unit and the methoxy substituent on this assembly.

2. Experimental

2.1. Synthesis and crystallization

For the synthesis of (I), a solution of aqueous potassium hydroxide (15 ml of a 10% *w/v* solution) was added to a mixture of 3-acetylpyridine (0.01 mol) and 6-methoxy-2-naphthaldehyde (0.01 mol) in ethanol (40 ml). This mixture was then stirred at 280 K for 3 h. The resulting solid product was collected by filtration and recrystallized from ethanol (yield 87%, m.p. 433–435 K). Colourless crystals of (I) suitable for single-crystal X-ray diffraction were grown by slow evaporation, at ambient temperature and in air, of a solution in methanol–toluene (1:1 *v/v*).

For the synthesis of (II), a mixture of (I) (0.01 mol) and guanidinium chloride (0.01 mol) in ethanol (25 ml) was heated under reflux for 24 h in the presence of sodium ethoxide (3.1 ml of a 21% *w/v* solution in ethanol). The mixture was allowed to cool to ambient temperature and was then refrigerated overnight. The resulting solid product was collected

by filtration and recrystallized from ethanol (yield 62%, m.p. 413–415 K). Yellow crystals of (II) suitable for single-crystal X-ray diffraction were grown by slow evaporation, at ambient temperature and in the presence of air, of a solution in *N,N*-dimethylformamide.

2.2. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. All H atoms were located in difference maps and then treated as riding atoms. C-bound H atoms were treated as riding in geometrically idealized positions, with C–H = 0.95 (alkenyl, aromatic and pyridyl), 0.98 (methyl) or 1.00 Å (aliphatic C–H), and with $U_{\text{iso}}(\text{H}) = kU_{\text{eq}}(\text{C})$, where $k = 1.5$ for the methyl groups, which were permitted to rotate but not to tilt, and 1.2 for all other C-bound H atoms. N- or O-bound H atoms were permitted to ride at the positions located in difference maps, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{N})$ or $1.5U_{\text{eq}}(\text{O})$, giving the N–H and O–H distances shown in Tables 3 and 5. An attempt was made to establish the correct orientation of the structure of (I) with respect to the polar-axis direction by use of the Flack x parameter (Flack, 1983), calculated using 1281 quotients of type $[(I^+) - (I^-)] / [(I^+) + (I^-)]$ (Parsons *et al.*, 2013), giving a value $x = 0.022$ (404), truncated in the CIF to 0.0 (4). Examination of the refined structures using *PLATON* (Spek, 2009) showed that neither of them contained any solvent-accessible voids.

Table 1
Experimental details.

	(I)	(II)
Crystal data		
Chemical formula	C ₁₉ H ₁₅ NO ₂	C ₂₀ H ₁₈ N ₄ O·H ₂ O
M_r	289.32	348.40
Crystal system, space group	Orthorhombic, <i>Pca</i> 2 ₁	Monoclinic, <i>P2</i> ₁ / <i>c</i>
Temperature (K)	200	200
a, b, c (Å)	14.2636 (6), 16.6789 (6), 6.0084 (2)	17.2244 (5), 5.9552 (2), 18.5173 (6)
α, β, γ (°)	90, 90, 90	90, 112.830 (1), 90
V (Å ³)	1429.41 (9)	1750.61 (10)
Z	4	4
Radiation type	Mo $K\alpha$	Mo $K\alpha$
μ (mm ⁻¹)	0.09	0.09
Crystal size (mm)	0.45 × 0.28 × 0.13	0.67 × 0.54 × 0.32
Data collection		
Diffractometer	Bruker APEXII CCD area-detector diffractometer	Bruker APEXII CCD area-detector diffractometer
Absorption correction	Multi-scan (<i>SADABS</i> ; Sheldrick, 2003)	Multi-scan (<i>SADABS</i> ; Sheldrick, 2003)
$T_{\text{min}}, T_{\text{max}}$	0.811, 0.989	0.895, 0.972
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	3474, 3440, 3106	15830, 4319, 3596
R_{int}	0.021	0.014
$(\sin \theta/\lambda)_{\text{max}}$ (Å ⁻¹)	0.668	0.667
Refinement		
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.035, 0.100, 1.08	0.041, 0.116, 1.04
No. of reflections	3440	4319
No. of parameters	200	236
No. of restraints	1	0
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained
$\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}$ (e Å ⁻³)	0.25, -0.18	0.29, -0.17
Absolute structure	Flack x parameter determined using 1281 quotients $[(I^+) - (I^-)] / [(I^+) + (I^-)]$ (Parsons <i>et al.</i> , 2013)	-
Absolute structure parameter	0.0 (4)	-

Computer programs: *APEX2* (Bruker, 2009), *SAINT* (Bruker, 2009), *SHELXS97* (Sheldrick, 2008), *PLATON* (Spek, 2009) and *SHELXL2014* (Sheldrick, 2014).

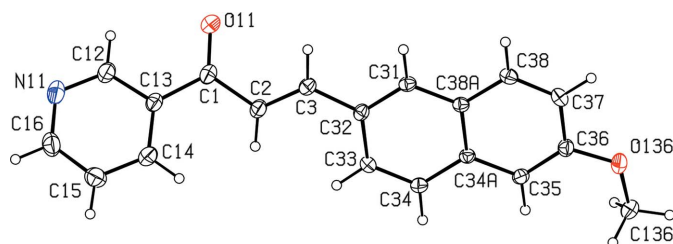


Figure 1
The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

Table 2
Selected geometric parameters (Å, °) for (I).

C31—C32	1.376 (3)	C36—C37	1.417 (3)
C32—C33	1.427 (3)	C37—C38	1.359 (3)
C33—C34	1.363 (3)	C38—C38a	1.419 (3)
C34—C34a	1.420 (3)	C38a—C31	1.412 (2)
C34a—C35	1.420 (3)	C34a—C38a	1.426 (2)
C35—C36	1.371 (3)		
O136—C36—C35	125.51 (19)	O136—C36—C37	113.91 (17)
C13—C1—C2—C3	−161.6 (2)	C2—C3—C32—C31	−174.8 (2)
C1—C2—C3—C32	−175.30 (19)	C35—C36—O136—C136	8.9 (3)
C2—C1—C13—C12	−160.4 (2)		

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

*Cg*1, *Cg*2 and *Cg*3 represent the centroids of the N11/C12—C16, C31—C34/C34a/C38a and C34a/C35—C38/C38a rings, respectively.

<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>
C16—H16···O136 ⁱ	0.95	2.45	3.364 (3)	162
C136—H36A···N11 ⁱⁱ	0.98	2.63	3.526 (3)	152
C3—H3··· <i>Cg</i> 3 ⁱⁱⁱ	0.95	2.94	3.518 (2)	121
C12—H12··· <i>Cg</i> 1 ^{iv}	0.95	2.93	3.555 (3)	125
C31—H31··· <i>Cg</i> 2 ⁱⁱⁱ	0.95	2.81	3.527 (2)	133
C34—H34··· <i>Cg</i> 2 ^v	0.95	2.67	3.391 (2)	133

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

3. Results and discussion

Compound (I) (Fig. 1) crystallizes in the space group $Pca2_1$, but in the absence of any atom heavier than O, the Flack x parameter (Flack, 1983), even as calculated by the Parsons method (Parsons *et al.*, 2013), was associated with a very large s.u. value. Hence, the assignment of the orientation of the structure of (I) relative to the polar-axis direction cannot be regarded as robust. Compound (II) crystallizes as a monohydrate and it is possible to select a compact asymmetric unit in which the two independent components are linked by two $N-H\cdots O$ hydrogen bonds (Table 5), so forming an $R_2^2(6)$ (Bernstein *et al.*, 1995) motif (Fig. 2). The organic component of (II) contains a stereogenic centre at atom C4 and the reference molecule was selected as one having the *R* configuration at atom C4. The centrosymmetric space group confirms that this compound crystallizes as a racemic mixture.

In compound (I), the central spacer unit between atoms C13 and C32 (Fig. 1) adopts a nearly planar all-*trans* conformation,

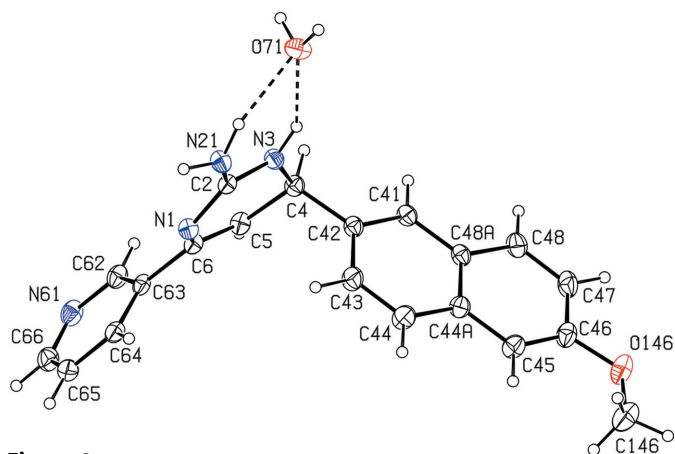


Figure 2
The independent molecular components of (II), showing the atom-labelling scheme and the $N-H\cdots O$ hydrogen bonds (dashed lines) linking the components within the selected asymmetric unit. Displacement ellipsoids are drawn at the 30% probability level.

Table 4
Selected geometric parameters (Å, °) for (II).

N1—C2	1.3189 (15)	C43—C44	1.3640 (19)
C2—N3	1.3447 (15)	C44—C44a	1.4172 (17)
N3—C4	1.4668 (15)	C44a—C45	1.4211 (18)
C4—C5	1.5005 (17)	C45—C46	1.3637 (19)
C5—C6	1.3389 (17)	C46—C47	1.407 (2)
C6—N1	1.4096 (14)	C47—C48	1.365 (2)
C2—N21	1.3563 (14)	C48—C48a	1.4202 (16)
C41—C42	1.3692 (16)	C48a—C41	1.4224 (17)
C42—C43	1.4141 (18)	C44a—C48a	1.4133 (18)
C45—C46—O146	125.24 (14)	C47—C46—O146	114.39 (12)
C45—C46—O146—C146	−6.6 (2)		

Table 5
Hydrogen-bond geometry (Å, °) for (II).

<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>
N3—H3···O71	0.92	2.05	2.8795 (14)	149
N21—H211···O71	0.90	2.10	2.9132 (15)	150
N21—H212···N1 ⁱ	0.89	2.14	3.0327 (15)	175
O71—H711···N61 ⁱⁱ	0.88	1.90	2.7751 (16)	171
O71—H712···N1 ⁱⁱⁱ	0.90	2.10	2.9940 (14)	169

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

as shown by the relevant torsion angles (Table 2). However, neither of the adjacent ring systems is coplanar with the central unit. The dihedral angle between the mean plane of the spacer unit (C1—C3/O11) and that of the pyridyl ring is $23.57 (15)^\circ$, and that between the spacer and the naphthalene unit is $23.61 (13)^\circ$. The dihedral angle between the two ring systems is $47.24 (9)^\circ$.

The dihydropyrimidine ring in (II) is nonplanar, with ring-puckering parameters (Cremer & Pople, 1975; calculated for the atom sequence N1/C2/N3/C4—C6) of $Q = 0.2251 (12) \text{ \AA}$, $\theta = 106.9 (3)^\circ$ and $\varphi = 352.3 (3)^\circ$. The atomic displacements from the mean plane of this ring are such that atoms N1 and C4 are displaced to one side of the mean plane and the other four ring atoms are displaced to the other side, albeit all by different amounts. The best single description of the ring conformation

is that of a shallow boat, with atoms N1 and C4 providing the bow and stern of the boat. The ring-puckering angle θ is, in fact, intermediate between the ideal values for boat and envelope conformations, *viz.* 90.0 and 115.3°, respectively (Boeyens, 1978). The amino group in (II) adopts a markedly pyramidal geometry, with a sum of the interbond angles at atom N21 of 348°.

In the naphthalene units of both compounds, the bonds Cx1–Cx2, Cx3–Cx4, Cx5–Cx6 and Cx7–Cx8, where $x = 3$ for (I) and $x = 4$ for (II) (Figs. 1 and 2), are characteristically (Glidewell & Lloyd, 1984) all shorter than the other C–C bonds in these ring systems (Tables 2 and 4). In each of (I) and (II), the methoxy C atom lies close to the plane of the adjacent aryl ring, with displacements from this plane of 0.223 (4) Å in (I) and 0.175 (2) Å in (II). Associated with this near-coplanarity, the two exocyclic C–C–O angles in each of (I) and (II) differ by *ca* 10°, as usually found in such circumstances (Seip & Seip, 1973; Ferguson *et al.*, 1996).

There are a number of short intermolecular contacts in the structure of (I) (Table 3), but only two of these can be regarded as structurally significant. Thus, the C–H···N contact involves a C–H bond from a methyl group which is of low acidity and almost certainly undergoing rapid rotation around the adjacent C–O bond (Riddell & Rogerson, 1996, 1997). Of the four C–H··· π contacts, all have quite narrow C–H···Cg angles (*cf.* Wood *et al.*, 2009) and three of them have quite long H···Cg distances. Accordingly, only the contact involving atom C34 is regarded as structurally significant. The C–H···O hydrogen bond links molecules related by translation to form a C(15) chain running parallel to the

[010] direction. The C–H··· π (arene) hydrogen bond involving atom C34 links molecules related by the *c*-glide plane at $x = \frac{1}{4}$ to form a chain running parallel to the [001] direction, and the combination of the [010] and [001] chains, each containing a single type of hydrogen bond, links the molecules into a sheet lying parallel to (100) (Fig. 3). These sheets lie in the domain $0 < x < \frac{1}{2}$, and a second such sheet, related to the first by the action of the 2_1 screw axes at $x = \frac{1}{2}$, lies in the domain $\frac{1}{2} < x < 1.0$, but there are no significant direction-specific interactions between adjacent sheets.

As noted above, the two molecular components in the asymmetric unit selected for (II) are linked by two N–H···O hydrogen bonds (Fig. 2). These bimolecular aggregates are linked by three further hydrogen bonds, one of N–H···N type and two of O–H···N type (Table 5). The combination of these three hydrogen bonds links the bimolecular aggregates into sheets, and each of them gives rise to a characteristic substructure (Ferguson *et al.*, 1998*a,b*; Gregson *et al.*, 2000). These substructures allow straightforward analysis of the complex structure of the sheet.

In the simplest of the substructures, of zero dimensionality, inversion-related pairs of dihydropyrimidine molecules are linked by inversion-related pairs of N–H···N hydrogen bonds to form an $R_2^2(8)$ motif, which is flanked by a pair of $R_2^1(6)$ rings built from the N–H···O hydrogen bonds (Fig. 4).

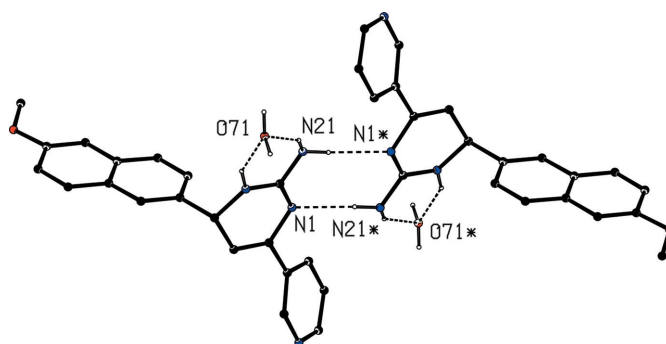


Figure 4
Part of the crystal structure of (II), showing the formation of a centrosymmetric aggregate built from N–H···O and N–H···N hydrogen bonds (dashed lines). For the sake of clarity, C-bound H atoms and the unit-cell outline have been omitted. Atoms marked with an asterisk (*) are at the symmetry position ($-x + 2, -y + 1, -z + 1$).

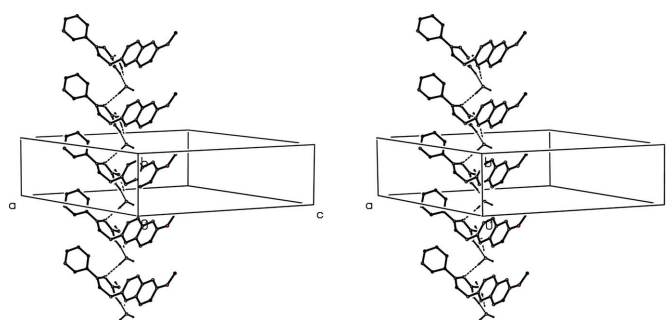


Figure 5
A stereoview of part of the crystal structure of (II), showing the formation of a $C_2^2(6)C_2^2(6)[R_2^1(6)]$ chain of rings parallel to [010]. The N–H···O and O–H···N hydrogen bonds are shown as dashed lines. For the sake of clarity, C-bound H atoms have been omitted.

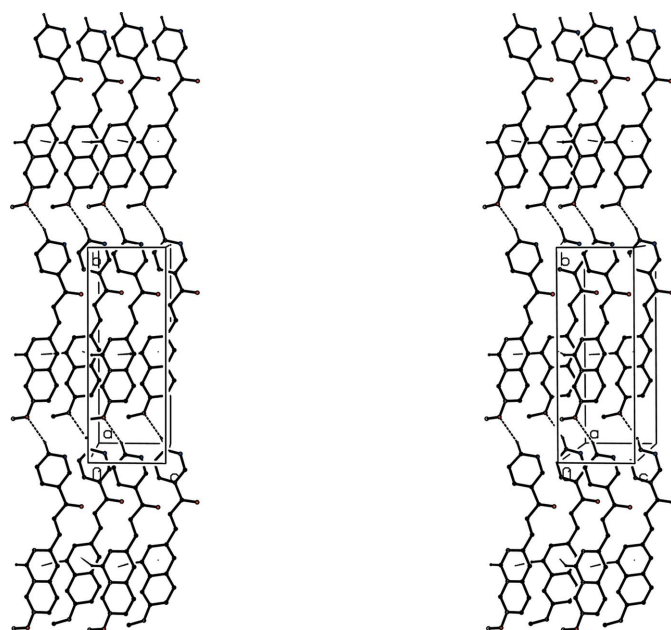


Figure 3
A stereoview of part of the crystal structure of (I), showing the formation of a sheet lying parallel to (100) and built from C–H···O and C–H··· π (arene) hydrogen bonds, shown as dashed and thin solid lines, respectively. For the sake of clarity, H atoms not involved in the motifs shown have been omitted.

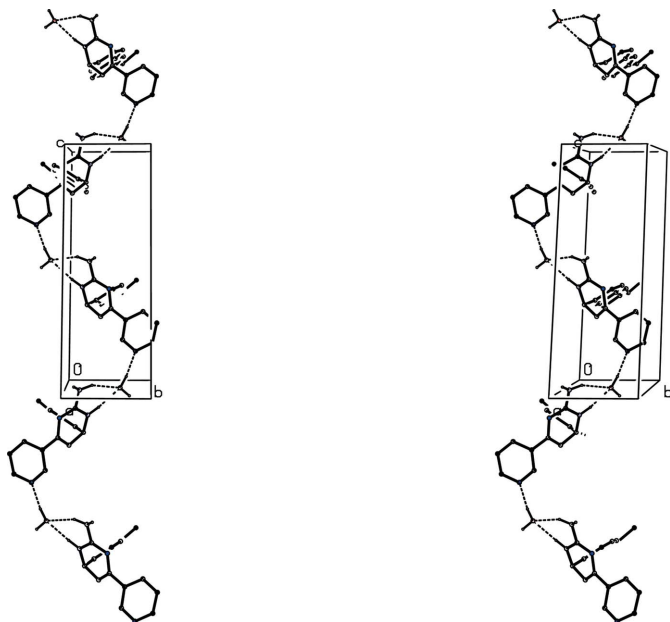


Figure 6

A stereoview of part of the crystal structure of (II), showing the formation of a $C_2^2(10)C_2^2(10)[R_2^1(6)]$ chain of rings parallel to [001]. The N—H...O and O—H...N hydrogen bonds are shown as dashed lines. For the sake of clarity, H atoms bonded to C atoms have been omitted.

The formation of the four-molecule aggregate utilizes fully the N—H...N and N—H...O hydrogen bonds, and thus leaves just the O—H...N hydrogen bonds available for the formation of further linkages.

Each of the two O—H...N hydrogen bonds gives rise to a chain-of-rings motif. The hydrogen bond having atom N1 as the acceptor links bimolecular aggregates (Fig. 2) which are related by translation to form a $C_2^2(6)C_2^2(6)[R_2^1(6)]$ chain of rings running parallel to the [010] direction (Fig. 5), and that having atom N61 as the acceptor links bimolecular aggregates which are related by the *c*-glide plane at $y = \frac{1}{4}$ to form a $C_2^2(10)C_2^2(10)[R_2^1(6)]$ chain of rings running parallel to the [001] direction (Fig. 6). The combination of these three substructures generates a sheet lying parallel to (100). The reference sheet lies in the domain $\frac{1}{2} < x < 1.0$ and a second sheet, related to the first by inversion, lies in the domain $0 < x < \frac{1}{2}$. The mutual arrangement of the sheets within the unit cell is such that the hydrogen bonds lie towards the outer margins of the domain of *x*, while the 6-methoxynaphthalene units are interdigitated in the central part of this domain (Fig. 7). Despite this, however, there are no direction-specific interactions between adjacent naphthalene units, as the distances between naphthalene ring centroids in adjacent molecules are all $>5 \text{ \AA}$.

Thus, while the methoxy O atom in (I) acts as a hydrogen-bond acceptor, that in (II) does not. The naphthalene substituent in (I) acts as an acceptor of a C—H... π (arene) hydrogen bond, although that in (II) does not, but π – π stacking interactions involving the naphthalene units are absent in both structures.

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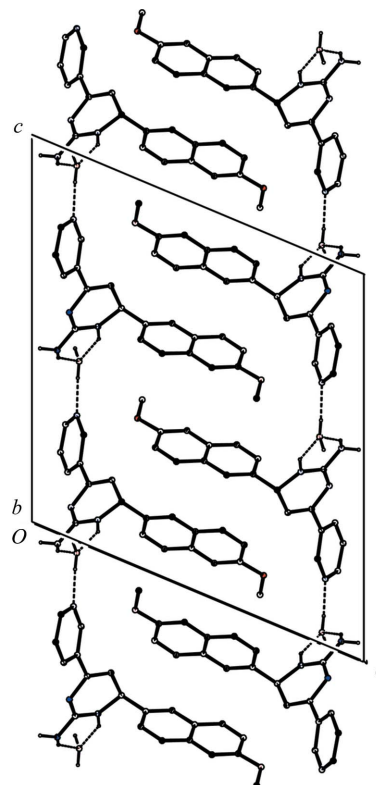


Figure 7

A projection along [010] of part of the crystal structure of (II), showing the interdigitation of the 6-methoxynaphthalene units in adjacent inversion-related sheets parallel to (100). The N—H...O and O—H...N hydrogen bonds are shown as dashed lines.

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(2E)-3-(6-Methoxynaphthalen-2-yl)-1-(pyridin-3-yl)prop-2-en-1-one and its cyclocondensation product with guanidine, (4RS)-2-amino-4-(6-methoxynaphthalen-2-yl)-6-(pyridin-3-yl)-3,4-dihydropyrimidine monohydrate: two types of hydrogen-bonded sheet

Prakash S. Nayak, Badiadka Narayana, Hemmige S. Yathirajan, Eric C. Hosten, Richard Betz and Christopher Glidewell

Computing details

For both compounds, data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT* (Bruker, 2009); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2014); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *PLATON* (Spek, 2009) and *SHELXL2014* (Sheldrick, 2014).

(I) (2E)-3-(6-Methoxynaphthalen-2-yl)-1-(pyridin-3-yl)prop-2-en-1-one

Crystal data

$C_{19}H_{15}NO_2$	$D_x = 1.344 \text{ Mg m}^{-3}$
$M_r = 289.32$	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
Orthorhombic, <i>Pca</i> 2 ₁	Cell parameters from 3441 reflections
$a = 14.2636 (6) \text{ \AA}$	$\theta = 1.2\text{--}28.4^\circ$
$b = 16.6789 (6) \text{ \AA}$	$\mu = 0.09 \text{ mm}^{-1}$
$c = 6.0084 (2) \text{ \AA}$	$T = 200 \text{ K}$
$V = 1429.41 (9) \text{ \AA}^3$	Plate, colourless
$Z = 4$	$0.45 \times 0.28 \times 0.13 \text{ mm}$
$F(000) = 608$	

Data collection

Bruker APEXII CCD area-detector diffractometer	3440 independent reflections
Radiation source: sealed tube	3106 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{\text{int}} = 0.021$
Absorption correction: multi-scan (<i>SADABS</i> ; Sheldrick, 2003)	$\theta_{\text{max}} = 28.4^\circ$, $\theta_{\text{min}} = 2.4^\circ$
$T_{\text{min}} = 0.811$, $T_{\text{max}} = 0.989$	$h = -19 \rightarrow 13$
3474 measured reflections	$k = -22 \rightarrow 22$
	$l = -8 \rightarrow 8$

Refinement

Refinement on F^2	3440 reflections
Least-squares matrix: full	200 parameters
$R[F^2 > 2\sigma(F^2)] = 0.035$	1 restraint
$wR(F^2) = 0.100$	Hydrogen site location: inferred from neighbouring sites
$S = 1.08$	

H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0517P)^2 + 0.2103P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.25 \text{ e } \text{\AA}^{-3}$

$\Delta\rho_{\min} = -0.18 \text{ e } \text{\AA}^{-3}$
 Absolute structure: Flack x parameter
 determined using 1281 quotients
 $[(I+)-(I-)]/[(I+)+(I-)]$ (Parsons *et al.*, 2013)
 Absolute structure parameter: 0.0 (4)

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.

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.13608 (15)	0.78897 (12)	0.6903 (4)	0.0346 (5)
O11	0.14016 (13)	0.77917 (10)	0.8914 (3)	0.0456 (5)
C2	0.13695 (16)	0.72033 (12)	0.5335 (4)	0.0344 (5)
H2	0.1525	0.7285	0.3814	0.041*
C3	0.11583 (14)	0.64709 (11)	0.6073 (3)	0.0296 (4)
H3	0.0963	0.6435	0.7581	0.036*
N11	0.15568 (17)	1.01214 (11)	0.6698 (4)	0.0512 (6)
C12	0.15867 (17)	0.93578 (13)	0.7316 (4)	0.0398 (5)
H12	0.1813	0.9239	0.8766	0.048*
C13	0.13077 (15)	0.87166 (12)	0.5973 (4)	0.0325 (4)
C14	0.09803 (17)	0.88972 (14)	0.3858 (4)	0.0413 (5)
H14	0.0793	0.8481	0.2874	0.050*
C15	0.0929 (2)	0.96912 (14)	0.3199 (4)	0.0475 (6)
H15	0.0695	0.9830	0.1770	0.057*
C16	0.1225 (2)	1.02770 (15)	0.4657 (5)	0.0500 (6)
H16	0.1192	1.0820	0.4190	0.060*
C31	0.08734 (13)	0.50274 (11)	0.5847 (3)	0.0260 (4)
H31	0.0596	0.5063	0.7281	0.031*
C32	0.11921 (13)	0.57162 (10)	0.4838 (3)	0.0260 (4)
C33	0.15895 (13)	0.56566 (11)	0.2665 (3)	0.0268 (4)
H33	0.1793	0.6129	0.1924	0.032*
C34	0.16819 (14)	0.49323 (11)	0.1636 (3)	0.0271 (4)
H34	0.1956	0.4908	0.0197	0.033*
C34a	0.13757 (13)	0.42139 (11)	0.2677 (3)	0.0241 (4)
C35	0.14779 (14)	0.34528 (11)	0.1646 (3)	0.0281 (4)
H35	0.1784	0.3409	0.0247	0.034*
C36	0.11333 (14)	0.27813 (11)	0.2677 (4)	0.0300 (4)
C37	0.06901 (14)	0.28379 (11)	0.4781 (4)	0.0316 (4)
H37	0.0449	0.2369	0.5470	0.038*
C38	0.06070 (13)	0.35573 (11)	0.5823 (3)	0.0296 (4)
H38	0.0318	0.3584	0.7246	0.035*
C38a	0.09457 (13)	0.42696 (10)	0.4814 (3)	0.0244 (4)
O136	0.11714 (12)	0.20216 (9)	0.1850 (3)	0.0408 (4)
C136	0.1722 (2)	0.18877 (14)	-0.0102 (4)	0.0470 (6)

H36A	0.1718	0.1315	-0.0468	0.070*
H36B	0.2368	0.2063	0.0169	0.070*
H36C	0.1458	0.2193	-0.1347	0.070*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0352 (11)	0.0314 (10)	0.0374 (11)	-0.0034 (8)	0.0022 (9)	-0.0078 (9)
O11	0.0639 (12)	0.0394 (8)	0.0334 (8)	-0.0061 (8)	-0.0013 (8)	-0.0058 (7)
C2	0.0408 (11)	0.0294 (10)	0.0332 (11)	-0.0022 (9)	0.0036 (9)	-0.0064 (8)
C3	0.0287 (9)	0.0305 (9)	0.0296 (9)	0.0022 (8)	0.0004 (8)	-0.0047 (8)
N11	0.0654 (14)	0.0318 (9)	0.0566 (13)	0.0001 (9)	-0.0037 (12)	-0.0083 (10)
C12	0.0446 (12)	0.0340 (10)	0.0408 (13)	-0.0004 (9)	-0.0025 (10)	-0.0078 (9)
C13	0.0339 (10)	0.0302 (9)	0.0335 (10)	-0.0017 (8)	0.0040 (9)	-0.0063 (8)
C14	0.0486 (14)	0.0392 (11)	0.0362 (11)	-0.0040 (10)	-0.0018 (11)	-0.0078 (10)
C15	0.0543 (15)	0.0472 (13)	0.0411 (14)	0.0050 (11)	0.0007 (11)	0.0057 (11)
C16	0.0596 (16)	0.0311 (11)	0.0593 (16)	0.0038 (11)	0.0056 (14)	0.0018 (11)
C31	0.0243 (8)	0.0309 (9)	0.0228 (8)	0.0036 (7)	0.0007 (7)	-0.0013 (7)
C32	0.0248 (9)	0.0250 (9)	0.0281 (10)	0.0024 (7)	-0.0016 (8)	-0.0037 (8)
C33	0.0277 (9)	0.0261 (9)	0.0268 (9)	-0.0007 (7)	0.0009 (8)	0.0035 (8)
C34	0.0277 (9)	0.0310 (9)	0.0227 (8)	0.0008 (7)	0.0017 (8)	0.0000 (8)
C34a	0.0245 (9)	0.0261 (9)	0.0217 (8)	0.0017 (7)	-0.0018 (8)	-0.0001 (7)
C35	0.0320 (10)	0.0291 (9)	0.0232 (9)	0.0020 (7)	-0.0011 (8)	-0.0022 (8)
C36	0.0327 (10)	0.0252 (9)	0.0321 (10)	0.0022 (8)	-0.0041 (9)	-0.0017 (8)
C37	0.0322 (10)	0.0279 (9)	0.0347 (10)	-0.0010 (8)	0.0006 (9)	0.0069 (8)
C38	0.0290 (9)	0.0333 (10)	0.0264 (9)	0.0023 (7)	0.0026 (8)	0.0041 (8)
C38a	0.0227 (9)	0.0275 (9)	0.0231 (8)	0.0023 (7)	-0.0017 (7)	0.0009 (7)
O136	0.0542 (10)	0.0258 (6)	0.0425 (9)	-0.0012 (6)	0.0039 (8)	-0.0043 (6)
C136	0.0720 (17)	0.0332 (11)	0.0357 (11)	0.0053 (11)	0.0025 (12)	-0.0077 (10)

Geometric parameters (Å, °)

C1—O11	1.221 (3)	C33—C34	1.363 (3)
C1—C2	1.482 (3)	C33—H33	0.9500
C1—C13	1.490 (3)	C34—C34a	1.420 (3)
C2—C3	1.334 (3)	C34—H34	0.9500
C2—H2	0.9500	C34a—C35	1.420 (3)
C3—C32	1.462 (3)	C35—C36	1.371 (3)
C3—H3	0.9500	C35—H35	0.9500
N11—C12	1.327 (3)	C36—O136	1.362 (2)
N11—C16	1.340 (4)	C36—C37	1.417 (3)
C12—C13	1.398 (3)	C37—C38	1.359 (3)
C12—H12	0.9500	C37—H37	0.9500
C13—C14	1.387 (3)	C38—C38a	1.419 (3)
C14—C15	1.384 (3)	C38a—C31	1.412 (2)
C14—H14	0.9500	C38—H38	0.9500
C15—C16	1.378 (4)	C34a—C38a	1.426 (2)
C15—H15	0.9500	O136—C136	1.429 (3)

C16—H16	0.9500	C136—H36A	0.9800
C31—C32	1.376 (3)	C136—H36B	0.9800
C31—H31	0.9500	C136—H36C	0.9800
C32—C33	1.427 (3)		
O11—C1—C2	121.7 (2)	C34—C33—C32	121.01 (17)
O11—C1—C13	119.84 (19)	C34—C33—H33	119.5
C2—C1—C13	118.48 (19)	C32—C33—H33	119.5
C3—C2—C1	119.61 (19)	C33—C34—C34a	121.21 (17)
C3—C2—H2	120.2	C33—C34—H34	119.4
C1—C2—H2	120.2	C34a—C34—H34	119.4
C2—C3—C32	127.75 (19)	C35—C34a—C34	122.05 (17)
C2—C3—H3	116.1	C35—C34a—C38a	119.68 (17)
C32—C3—H3	116.1	C34—C34a—C38a	118.27 (17)
C12—N11—C16	117.0 (2)	C36—C35—C34a	119.77 (17)
N11—C12—C13	124.3 (2)	C36—C35—H35	120.1
N11—C12—H12	117.9	C34a—C35—H35	120.1
C13—C12—H12	117.9	O136—C36—C35	125.51 (19)
C14—C13—C12	117.3 (2)	O136—C36—C37	113.91 (17)
C14—C13—C1	124.18 (19)	C35—C36—C37	120.58 (18)
C12—C13—C1	118.5 (2)	C38—C37—C36	120.60 (18)
C15—C14—C13	119.2 (2)	C38—C37—H37	119.7
C15—C14—H14	120.4	C36—C37—H37	119.7
C13—C14—H14	120.4	C37—C38—C38a	120.86 (18)
C16—C15—C14	118.7 (2)	C37—C38—H38	119.6
C16—C15—H15	120.7	C38a—C38—H38	119.6
C14—C15—H15	120.7	C31—C38a—C38	122.48 (17)
N11—C16—C15	123.5 (2)	C31—C38a—C34a	119.04 (16)
N11—C16—H16	118.2	C38—C38a—C34a	118.48 (17)
C15—C16—H16	118.2	C36—O136—C136	117.82 (17)
C32—C31—C38a	122.00 (16)	O136—C136—H36A	109.5
C32—C31—H31	119.0	O136—C136—H36B	109.5
C38a—C31—H31	119.0	H36A—C136—H36B	109.5
C31—C32—C33	118.41 (16)	O136—C136—H36C	109.5
C31—C32—C3	118.98 (18)	H36A—C136—H36C	109.5
C33—C32—C3	122.52 (17)	H36B—C136—H36C	109.5
O11—C1—C2—C3	18.8 (3)	C32—C33—C34—C34a	0.8 (3)
C13—C1—C2—C3	-161.6 (2)	C33—C34—C34a—C35	-179.09 (18)
C1—C2—C3—C32	-175.30 (19)	C33—C34—C34a—C38a	1.5 (3)
C16—N11—C12—C13	0.8 (4)	C34—C34a—C35—C36	-177.12 (18)
N11—C12—C13—C14	0.1 (4)	C38a—C34a—C35—C36	2.3 (3)
N11—C12—C13—C1	-178.7 (2)	C34a—C35—C36—O136	178.79 (19)
O11—C1—C13—C14	-159.6 (2)	C34a—C35—C36—C37	-1.1 (3)
C2—C1—C13—C14	20.9 (3)	O136—C36—C37—C38	179.47 (19)
O11—C1—C13—C12	19.1 (3)	C35—C36—C37—C38	-0.6 (3)
C2—C1—C13—C12	-160.4 (2)	C36—C37—C38—C38a	1.2 (3)
C12—C13—C14—C15	-1.2 (3)	C32—C31—C38a—C38	-179.03 (18)

C1—C13—C14—C15	177.6 (2)	C32—C31—C38a—C34a	1.0 (3)
C13—C14—C15—C16	1.3 (4)	C37—C38—C38a—C31	-179.88 (19)
C12—N11—C16—C15	-0.6 (4)	C37—C38—C38a—C34a	0.0 (3)
C14—C15—C16—N11	-0.4 (4)	C35—C34a—C38a—C31	178.18 (18)
C38a—C31—C32—C33	1.2 (3)	C34—C34a—C38a—C31	-2.4 (2)
C38a—C31—C32—C3	-175.47 (17)	C35—C34a—C38a—C38	-1.8 (3)
C2—C3—C32—C31	-174.8 (2)	C34—C34a—C38a—C38	177.67 (18)
C2—C3—C32—C33	8.6 (3)	C35—C36—O136—C136	8.9 (3)
C31—C32—C33—C34	-2.2 (3)	C37—C36—O136—C136	-171.2 (2)
C3—C32—C33—C34	174.39 (18)		

Hydrogen-bond geometry (Å, °)

Cg1, Cg2 and Cg3 represent the centroids of the rings N11/C12–C16, C31–C34/C34a/C38a and C34a/C35–C38/C38a, respectively.

<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>
C16—H16...O136 ⁱ	0.95	2.45	3.364 (3)	162
C136—H36A...N11 ⁱⁱ	0.98	2.63	3.526 (3)	152
C3—H3...Cg3 ⁱⁱⁱ	0.95	2.94	3.518 (2)	121
C12—H12...Cg1 ^{iv}	0.95	2.93	3.555 (3)	125
C31—H31...Cg2 ⁱⁱⁱ	0.95	2.81	3.527 (2)	133
C34—H34...Cg2 ^v	0.95	2.67	3.391 (2)	133

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

(II) (RS)-2-Amino-4-(6-methoxynaphthalen-2-yl)-6-(pyridin-3-yl)-3,4-dihydropyrimidine monohydrate*Crystal data*

$C_{20}H_{18}N_4O \cdot H_2O$

$M_r = 348.40$

Monoclinic, $P2_1/c$

$a = 17.2244$ (5) Å

$b = 5.9552$ (2) Å

$c = 18.5173$ (6) Å

$\beta = 112.830$ (1)°

$V = 1750.61$ (10) Å³

$Z = 4$

$F(000) = 736$

$D_x = 1.322$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 4319 reflections

$\theta = 2.2$ – 28.3 °

$\mu = 0.09$ mm⁻¹

$T = 200$ K

Block, yellow

$0.67 \times 0.54 \times 0.32$ mm

Data collection

Bruker APEXII CCD area-detector
diffractometer

Radiation source: sealed tube

φ and ω scans

Absorption correction: multi-scan
(*SADABS*; Sheldrick, 2003)

$T_{\min} = 0.895$, $T_{\max} = 0.972$

15830 measured reflections

4319 independent reflections

3596 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.014$

$\theta_{\max} = 28.3$ °, $\theta_{\min} = 2.2$ °

$h = -22 \rightarrow 22$

$k = -7 \rightarrow 7$

$l = -24 \rightarrow 22$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.041$

$wR(F^2) = 0.116$

$S = 1.04$

4319 reflections

236 parameters

0 restraints

Hydrogen site location: mixed

H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0554P)^2 + 0.561P]$$

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

$$\Delta\rho_{\max} = 0.29 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.17 \text{ e } \text{\AA}^{-3}$$

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.

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
N1	0.89139 (6)	0.52266 (17)	0.41737 (5)	0.0298 (2)
C2	0.87565 (7)	0.3476 (2)	0.45317 (6)	0.0277 (2)
N3	0.80838 (6)	0.21208 (18)	0.42197 (6)	0.0316 (2)
H3	0.8063	0.0906	0.4520	0.038*
C4	0.73670 (7)	0.2640 (2)	0.34898 (7)	0.0314 (3)
H4	0.7175	0.1223	0.3182	0.038*
C5	0.76718 (7)	0.4235 (2)	0.30296 (7)	0.0326 (3)
H5	0.7360	0.4394	0.2482	0.039*
C6	0.83758 (7)	0.5437 (2)	0.33777 (6)	0.0286 (2)
N21	0.92997 (6)	0.29906 (19)	0.52711 (6)	0.0340 (2)
H211	0.9280	0.1599	0.5450	0.041*
H212	0.9812	0.3593	0.5417	0.041*
C41	0.58418 (7)	0.2716 (2)	0.33298 (7)	0.0313 (3)
H41	0.5752	0.1391	0.3023	0.038*
C42	0.66312 (7)	0.3639 (2)	0.36432 (7)	0.0308 (3)
C43	0.67574 (8)	0.5617 (2)	0.40969 (9)	0.0421 (3)
H43	0.7302	0.6276	0.4311	0.051*
C44	0.61107 (8)	0.6597 (3)	0.42323 (9)	0.0455 (3)
H44	0.6213	0.7918	0.4543	0.055*
C44a	0.52881 (7)	0.5675 (2)	0.39167 (7)	0.0344 (3)
C45	0.46096 (8)	0.6671 (2)	0.40564 (8)	0.0405 (3)
H45	0.4701	0.7986	0.4369	0.049*
C46	0.38268 (8)	0.5734 (3)	0.37404 (8)	0.0404 (3)
C47	0.36847 (8)	0.3804 (3)	0.32683 (9)	0.0446 (3)
H47	0.3134	0.3188	0.3042	0.054*
C48	0.43269 (8)	0.2804 (3)	0.31298 (8)	0.0406 (3)
H48	0.4220	0.1494	0.2813	0.049*
C48a	0.51539 (7)	0.3709 (2)	0.34562 (7)	0.0311 (3)
O146	0.31249 (6)	0.6528 (2)	0.38374 (7)	0.0538 (3)
C146	0.32473 (11)	0.8329 (3)	0.43708 (10)	0.0573 (4)
H46A	0.2715	0.8665	0.4425	0.086*
H46B	0.3438	0.9659	0.4174	0.086*
H46C	0.3674	0.7908	0.4883	0.086*
N61	0.87654 (7)	0.8262 (2)	0.17333 (7)	0.0429 (3)
C62	0.85457 (8)	0.6778 (2)	0.21618 (7)	0.0356 (3)
H62	0.8297	0.5405	0.1921	0.043*

C63	0.86594 (7)	0.7125 (2)	0.29423 (6)	0.0293 (2)
C64	0.90497 (7)	0.9109 (2)	0.32890 (7)	0.0331 (3)
H64	0.9159	0.9400	0.3824	0.040*
C65	0.92794 (9)	1.0666 (2)	0.28517 (8)	0.0402 (3)
H65	0.9539	1.2042	0.3079	0.048*
C66	0.91230 (9)	1.0179 (3)	0.20805 (9)	0.0437 (3)
H66	0.9276	1.1256	0.1781	0.052*
O71	0.86814 (7)	-0.15413 (16)	0.53121 (6)	0.0436 (2)
H711	0.8726	-0.2222	0.5751	0.065*
H712	0.8813	-0.2572	0.5020	0.065*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0284 (5)	0.0340 (5)	0.0245 (4)	-0.0007 (4)	0.0077 (4)	0.0021 (4)
C2	0.0261 (5)	0.0307 (6)	0.0271 (5)	0.0031 (4)	0.0112 (4)	-0.0001 (4)
N3	0.0279 (5)	0.0325 (5)	0.0327 (5)	-0.0003 (4)	0.0099 (4)	0.0045 (4)
C4	0.0260 (5)	0.0345 (6)	0.0305 (6)	-0.0021 (5)	0.0076 (4)	-0.0026 (5)
C5	0.0286 (5)	0.0416 (7)	0.0257 (5)	0.0015 (5)	0.0085 (4)	0.0012 (5)
C6	0.0267 (5)	0.0340 (6)	0.0257 (5)	0.0043 (4)	0.0107 (4)	0.0011 (4)
N21	0.0312 (5)	0.0375 (6)	0.0292 (5)	-0.0024 (4)	0.0072 (4)	0.0065 (4)
C41	0.0291 (5)	0.0325 (6)	0.0292 (5)	-0.0044 (5)	0.0080 (4)	-0.0022 (5)
C42	0.0267 (5)	0.0334 (6)	0.0302 (5)	-0.0018 (5)	0.0086 (4)	0.0014 (5)
C43	0.0277 (6)	0.0417 (7)	0.0531 (8)	-0.0082 (5)	0.0115 (5)	-0.0130 (6)
C44	0.0333 (6)	0.0423 (8)	0.0566 (8)	-0.0063 (6)	0.0128 (6)	-0.0191 (6)
C44a	0.0287 (6)	0.0372 (7)	0.0341 (6)	-0.0007 (5)	0.0086 (5)	-0.0003 (5)
C45	0.0358 (6)	0.0440 (8)	0.0398 (7)	0.0025 (6)	0.0127 (5)	-0.0042 (6)
C46	0.0305 (6)	0.0530 (8)	0.0380 (6)	0.0062 (6)	0.0137 (5)	0.0092 (6)
C47	0.0266 (6)	0.0543 (9)	0.0496 (8)	-0.0074 (6)	0.0110 (5)	0.0026 (7)
C48	0.0313 (6)	0.0439 (8)	0.0428 (7)	-0.0095 (5)	0.0103 (5)	-0.0048 (6)
C48a	0.0263 (5)	0.0344 (6)	0.0291 (5)	-0.0042 (5)	0.0070 (4)	0.0024 (5)
O146	0.0342 (5)	0.0753 (8)	0.0545 (6)	0.0089 (5)	0.0201 (5)	0.0028 (6)
C146	0.0525 (9)	0.0758 (12)	0.0480 (8)	0.0214 (8)	0.0243 (7)	0.0085 (8)
N61	0.0432 (6)	0.0574 (8)	0.0340 (5)	0.0101 (5)	0.0215 (5)	0.0100 (5)
C62	0.0326 (6)	0.0459 (7)	0.0298 (6)	0.0054 (5)	0.0137 (5)	0.0026 (5)
C63	0.0227 (5)	0.0387 (6)	0.0272 (5)	0.0066 (4)	0.0105 (4)	0.0050 (5)
C64	0.0314 (6)	0.0377 (7)	0.0328 (6)	0.0060 (5)	0.0154 (5)	0.0034 (5)
C65	0.0393 (7)	0.0381 (7)	0.0480 (7)	0.0037 (5)	0.0222 (6)	0.0058 (6)
C66	0.0452 (7)	0.0478 (8)	0.0466 (7)	0.0075 (6)	0.0272 (6)	0.0141 (6)
O71	0.0618 (6)	0.0328 (5)	0.0380 (5)	0.0013 (4)	0.0215 (4)	0.0009 (4)

Geometric parameters (Å, °)

N1—C2	1.3189 (15)	C46—O146	1.3734 (16)
C2—N3	1.3447 (15)	C46—C47	1.407 (2)
N3—C4	1.4668 (15)	C47—C48	1.365 (2)
N3—H3	0.9212	C47—H47	0.9500
C4—C5	1.5005 (17)	C48—C48a	1.4202 (16)

C4—C42	1.5232 (16)	C48a—C41	1.4224 (17)
C4—H4	1.0000	C44a—C48a	1.4133 (18)
C5—C6	1.3389 (17)	C48—H48	0.9500
C5—H5	0.9500	O146—C146	1.417 (2)
C6—N1	1.4096 (14)	C146—H46A	0.9800
C2—N21	1.3563 (14)	C146—H46B	0.9800
N21—H211	0.8981	C146—H46C	0.9800
N21—H212	0.8912	N61—C62	1.3368 (17)
C6—C63	1.4848 (16)	N61—C66	1.338 (2)
C41—C42	1.3692 (16)	C62—C63	1.3966 (16)
C41—H41	0.9500	C62—H62	0.9500
C42—C43	1.4141 (18)	C63—C64	1.3877 (18)
C43—C44	1.3640 (19)	C64—C65	1.3857 (18)
C43—H43	0.9500	C64—H64	0.9500
C44—C44a	1.4172 (17)	C65—C66	1.378 (2)
C44—H44	0.9500	C65—H65	0.9500
C44a—C45	1.4211 (18)	C66—H66	0.9500
C45—C46	1.3637 (19)	O71—H711	0.8845
C45—H45	0.9500	O71—H712	0.9034
C2—N1—C6	114.59 (10)	C46—C45—H45	120.1
N1—C2—N3	124.70 (10)	C44a—C45—H45	120.1
N1—C2—N21	118.69 (10)	C45—C46—O146	125.24 (14)
N3—C2—N21	116.61 (10)	C45—C46—C47	120.38 (12)
C2—N3—C4	123.13 (10)	C47—C46—O146	114.39 (12)
C2—N3—H3	116.1	C48—C47—C46	120.99 (12)
C4—N3—H3	120.3	C48—C47—H47	119.5
N3—C4—C5	107.56 (9)	C46—C47—H47	119.5
N3—C4—C42	111.98 (10)	C47—C48—C48a	120.42 (13)
C5—C4—C42	111.00 (10)	C47—C48—H48	119.8
N3—C4—H4	108.7	C48a—C48—H48	119.8
C5—C4—H4	108.7	C44a—C48a—C48	118.25 (12)
C42—C4—H4	108.7	C44a—C48a—C41	119.46 (11)
C6—C5—C4	121.08 (10)	C48—C48a—C41	122.29 (12)
C6—C5—H5	119.5	C46—O146—C146	116.82 (12)
C4—C5—H5	119.5	O146—C146—H46A	109.5
C5—C6—N1	124.13 (11)	O146—C146—H46B	109.5
C5—C6—C63	121.87 (10)	H46A—C146—H46B	109.5
N1—C6—C63	114.00 (10)	O146—C146—H46C	109.5
C2—N21—H211	117.3	H46A—C146—H46C	109.5
C2—N21—H212	115.8	H46B—C146—H46C	109.5
H211—N21—H212	115.8	C62—N61—C66	117.56 (12)
C42—C41—C48a	121.14 (11)	N61—C62—C63	123.91 (13)
C42—C41—H41	119.4	N61—C62—H62	118.0
C48a—C41—H41	119.4	C63—C62—H62	118.0
C41—C42—C43	118.98 (11)	C64—C63—C62	116.95 (11)
C41—C42—C4	121.70 (11)	C64—C63—C6	121.84 (10)
C43—C42—C4	119.28 (10)	C62—C63—C6	121.20 (11)

C44—C43—C42	121.15 (12)	C65—C64—C63	119.75 (12)
C44—C43—H43	119.4	C65—C64—H64	120.1
C42—C43—H43	119.4	C63—C64—H64	120.1
C43—C44—C44a	121.04 (13)	C66—C65—C64	118.66 (14)
C43—C44—H44	119.5	C66—C65—H65	120.7
C44a—C44—H44	119.5	C64—C65—H65	120.7
C48a—C44a—C44	118.23 (11)	N61—C66—C65	123.12 (13)
C48a—C44a—C45	120.15 (11)	N61—C66—H66	118.4
C44—C44a—C45	121.63 (12)	C65—C66—H66	118.4
C46—C45—C44a	119.80 (13)	H711—O71—H712	106.6
C6—N1—C2—N3	-8.17 (16)	C44a—C45—C46—C47	-0.9 (2)
C6—N1—C2—N21	172.54 (10)	C45—C46—C47—C48	1.4 (2)
N1—C2—N3—C4	-10.46 (18)	O146—C46—C47—C48	-178.96 (13)
N21—C2—N3—C4	168.84 (10)	C46—C47—C48—C48a	-0.5 (2)
C2—N3—C4—C5	23.21 (15)	C44—C44a—C48a—C48	-179.05 (13)
C2—N3—C4—C42	-99.00 (13)	C45—C44a—C48a—C48	1.22 (18)
N3—C4—C5—C6	-19.82 (16)	C44—C44a—C48a—C41	0.49 (18)
C42—C4—C5—C6	103.00 (13)	C45—C44a—C48a—C41	-179.23 (12)
C4—C5—C6—N1	4.27 (19)	C47—C48—C48a—C44a	-0.7 (2)
C4—C5—C6—C63	-176.20 (11)	C47—C48—C48a—C41	179.73 (12)
C2—N1—C6—C5	11.10 (17)	C42—C41—C48a—C44a	-0.35 (18)
C2—N1—C6—C63	-168.46 (10)	C42—C41—C48a—C48	179.17 (12)
C48a—C41—C42—C43	-0.24 (18)	C45—C46—O146—C146	-6.6 (2)
C48a—C41—C42—C4	-177.88 (11)	C47—C46—O146—C146	173.76 (13)
N3—C4—C42—C41	-124.93 (12)	C66—N61—C62—C63	0.70 (19)
C5—C4—C42—C41	114.84 (13)	N61—C62—C63—C64	-2.14 (18)
N3—C4—C42—C43	57.43 (15)	N61—C62—C63—C6	178.00 (11)
C5—C4—C42—C43	-62.80 (14)	C5—C6—C63—C64	144.25 (12)
C41—C42—C43—C44	0.7 (2)	N1—C6—C63—C64	-36.18 (15)
C4—C42—C43—C44	178.40 (13)	C5—C6—C63—C62	-35.90 (17)
C42—C43—C44—C44a	-0.6 (2)	N1—C6—C63—C62	143.67 (11)
C43—C44—C44a—C48a	-0.1 (2)	C62—C63—C64—C65	2.19 (17)
C43—C44—C44a—C45	179.67 (14)	C6—C63—C64—C65	-177.96 (11)
C48a—C44a—C45—C46	-0.4 (2)	C63—C64—C65—C66	-0.94 (18)
C44—C44a—C45—C46	179.85 (13)	C62—N61—C66—C65	0.7 (2)
C44a—C45—C46—O146	179.50 (12)	C64—C65—C66—N61	-0.6 (2)

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>
N3—H3...O71	0.92	2.05	2.8795 (14)	149
N21—H211...O71	0.90	2.10	2.9132 (15)	150
N21—H212...N1 ⁱ	0.89	2.14	3.0327 (15)	175
O71—H711...N61 ⁱⁱ	0.88	1.90	2.7751 (16)	171
O71—H712...N1 ⁱⁱⁱ	0.90	2.10	2.9940 (14)	169

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