Acta Crystallographica Section C
Crystal Structure Communications
ISSN 0108-2701

Two seven-membered heterocycles with 1,2-diaza ring N atoms: 3,5,7-triphenyl-1,2-diazacyclohepta-1(7),2-diene and 3,7-bis(2-hydroxyphenyl)-5-phenyl-1,2-diazacyclohepta-1(7),2-diene

Yao-Cheng Shi* and Hong-Yang Duan

College of Chemistry and Chemical Engineering, Yangzhou University, 180 SiWangTing Road, Yangzhou 225002, People's Republic of China
Correspondence e-mail: ycshi@yzu.edu.cn

Received 10 August 2013
Accepted 23 August 2013

The title compounds, 3,5,7-triphenyl-1,2-diazacyclohepta-1(7),2-diene, $C_{23}H_{20}N_2$, (I), and 3,7-bis(2-hydroxyphenyl)-5-phenyl-1,2-diazacyclohepta-1(7),2-diene, $C_{23}H_{20}N_2O_2$, (II), constitute the first structurally characterized examples of seven-membered heterocycles with 1,2-diaza ring N atoms. Compound (I) crystallizes in the space group $P\overline{1}$, with two independent molecules in the asymmetric unit that differ in the conformation of one of the phenyl rings, while (II) crystallizes in the space group $C2/c$. The C_5N_2 ring in each of (I) and (II) adopts a twist-boat conformation. Compound (I) exhibits neither C—H$\cdots\pi$ interactions nor π–π stacking interactions, whereas (II) shows both intramolecular O—H$\cdots$N hydrogen bonds and a C—H$\cdots\pi$ interaction that joins the molecules into an infinite chain in the [010] direction.

Keywords: Seven-membered ring; nitrogen-containing heterocycle; conformational analysis; 1,5-diketone.

1. Introduction

Nitrogen-containing heterocycles (N-heterocycles) are ubiquitous in natural products, pharmaceuticals and materials science. Their synthesis represents a substantial chemical research effort that includes the total synthesis of natural products and medicinal chemistry (Behenna *et al.*, 2012). To the best of our knowledge, there are no reported examples of 1,2-diaza heterocycles with seven-membered rings (Pettersson, 2011). As part of an on-going project on the syntheses of N-heterocycles (Shi *et al.*, 2006*a*,*b*,*c*; Shi, Sui & Zhu, 2006; Shi, Zhu & Ng, 2007; Shi, Sui, Cheng *et al.*, 2007; Shi, Cheng, Sui *et al.*, 2007; Zhu, Shi, Shen *et al.*, 2011), in this article we describe the syntheses and crystal structures of two seven-membered N-heterocycles. Reactions of 1,5-diketones $ArCOCH_2CHPhCH_2COAr$ with hydrazine hydrate in the presence of HOAc afford the title compounds, *viz.* (I) (Ar = C_6H_5) and (II) (Ar = 2-HOC_6H_4).

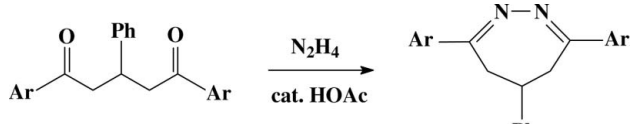
2. Experimental

2.1. Synthesis and crystallization

A mixture of $ArCOCH_2CHPhCH_2COAr$ (4 mmol) and 80% $N_2H_4{\cdot}H_2O$ (4 mmol) in the presence of HOAc (2 drops) in EtOH (25 ml) was refluxed for 12 h. For (I), the solvent was removed *in vacuo* and the resulting residue was isolated by chromatography on silica gel. Elution with petroleum ether–tetrahydrofuran (2:1 *v*/*v*) gave one colourless band which was recrystallized from petroleum ether (333–363 K) and CH_2Cl_2 to afford a white solid of (I) (yield 0.701 g, 54%). For (II), the resulting solid was collected, washed with EtOH, air-dried and recrystallized from petroleum ether (333–363 K) and CH_2Cl_2 to give a yellow solid (yield 0.713 g, 50%).

Analysis calculated for $C_{23}H_{20}N_2$, (I): C 85.15, H 6.21, N 8.63%; found: C 85.11, H 6.34, N 8.57%; m.p. 433–435 K. Spectroscopic analysis: IR (KBr disk, ν, cm^{-1}): 3029 (*m*), 2921 (*m*), 1667 (*m*), 1590 (*m*), 1547 (*m*), 1492 (*s*), 1446 (*s*), 1344 (*m*), 1254 (*m*), 1187 (*m*), 1065 (*m*), 1020 (*m*), 919 (*m*), 761 (*vs*), 697 (*vs*); UV ($\varepsilon_\lambda \times 10^4$, in DMF): 290.00 (1.02), 375.00 (0.86) nm; ^{1}H NMR (600 MHz, $CDCl_3$, TMS): δ 6.797–7.804 (*m*, 15H, $3C_6H_5$), 4.045–4.087 (*m*, 1H, CH), 3.002–3.018 (*m*, 4H, $2CH_2$).

Analysis calculated for $C_{23}H_{20}N_2O_2$, (II): C 77.51, H 5.66, N 7.86%; found: C 77.75, H 5.69, N 7.67%; m.p. 510–512 K. Spectroscopic analysis: IR (KBr disk, ν, cm^{-1}): 3434 (*m*), 3030 (*m*), 2921 (*m*), 1668 (*m*), 1590 (*m*), 1548 (*m*), 1493 (*s*), 1447 (*s*), 1344 (*s*), 1300 (*m*), 1255 (*m*), 1187 (*m*), 1020 (*m*), 919 (*m*), 762 (*vs*), 698 (*vs*); UV ($\varepsilon_\lambda \times 10^4$, in DMF): 290.00 (1.16), 377.00 (0.76) nm; ^{1}H NMR (600 MHz, $CDCl_3$, TMS): δ 12.956 (*s*, 2H, 2OH), 6.765–7.364 (*m*, 14H, $2C_6H_5$, C_6H_4), 4.083–4.124 (*m*, 1H, CH), 3.053–3.105 (*m*, 4H, $2CH_2$).



(I) Ar = C_6H_5
(II) Ar = 2-HOC_6H_4

2.2. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. H atoms attached to C atoms were placed at calculated positions and treated as riding atoms, with C—H = 0.93 (CH, aromatic), 0.97 (CH_2) or 0.98 Å (CH), and with U_{iso}(H) = 1.2U_{eq}(C). For (II), H atoms attached to O atoms were located in a difference Fourier map and refined freely with individual isotropic displacement parameters.

3. Results and discussion

Compound (I) crystallizes in the space group $P\overline{1}$, with two independent molecules in the asymmetric unit, labelled (I*a*)

 doi:10.1107/S0108270113023792

Table 1
Experimental details.

	(I)	(II)
Crystal data		
Chemical formula	$C_{23}H_{20}N_2$	$C_{23}H_{20}N_2O_2$
M_r	324.41	356.41
Crystal system, space group	Triclinic, $P\overline{1}$	Monoclinic, $C2/c$
Temperature (K)	296	296
a, b, c (Å)	9.4245 (14), 10.6605 (16), 18.405 (3)	23.9715 (14), 9.5669 (16), 18.2303 (12)
α, β, γ (°)	80.2989 (19), 85.094 (2), 78.1155 (19)	90, 119.256 (3), 90
V (Å^3)	1781.1 (5)	3647.5 (7)
Z	4	8
Radiation type	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	0.07	0.08
Crystal size (mm)	0.24 × 0.20 × 0.15	0.30 × 0.28 × 0.26
Data collection		
Diffractometer	Bruker SMART APEX CCD area-detector diffractometer	Bruker SMART APEX CCD area-detector diffractometer
Absorption correction	Multi-scan (*SADABS*; Sheldrick, 2004)	Multi-scan (*SADABS*; Sheldrick, 2004)
T_{min}, T_{max}	0.967, 0.982	0.967, 0.972
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	15860, 8141, 5062	12998, 3224, 1647
R_{int}	0.030	0.071
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.654	0.595
Refinement		
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.049, 0.149, 1.02	0.045, 0.109, 0.96
No. of reflections	8141	3224
No. of parameters	451	253
H-atom treatment	H-atom parameters constrained	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.18, −0.21	0.13, −0.14

Computer programs: *APEX2* (Bruker, 2009), *SAINT* (Bruker, 2009), *SIR2004* (Burla *et al.*, 2005), *SHELXTL* (Sheldrick, 2008), *PLATON* (Spek, 2009), *WinGX* (Farrugia, 2012) and *publCIF* (Westrip, 2010).

and (I*b*) (Fig. 1 and Table 2). A quaternion fit plot of (I*a*) on inverted (I*b*) (Spek, 2009) (Fig. 2) shows that molecules (I*a*) and (I*b*) are conformers, differing mainly in the rotation of one of the phenyl groups with respect to the rest of the molecule. The C1–C6 and C10–C15 planes form a dihedral angle of 59.15 (9)° for (I*a*). The corresponding angle in (I*b*) for the C24–C29 and C33–C38 planes is 51.68 (10)°. Atoms C7, C17, C30 and C40 each adopt sp^2-hybridization (Table 2). However, the C=N bonds are not conjugated with the respective phenyl groups, the dihedral angles between the C1–C6 and N1/C7/C8 planes, and the C18–C23 and N2/C16/C17 planes, being 26.9 (2) and 28.2 (2)°, respectively, for (I*a*), and the corresponding dihedral angles being 33.0 (2) and 17.9 (2)° for (I*b*). Furthermore, the two C=N bonds are not involved in conjugation with each other, with C—N—N—C torsion angles of −60.5 (2)° for (I*a*) and 61.8 (2)° for (I*b*). The seven-membered ring in (I*a*) displays a twist-boat conformation, with puckering parameters (Cremer & Pople, 1975; Zotov *et al.*, 1997) q_2 = 1.1007 (17) Å, q_3 = 0.0974 (17) Å, φ_2 = 39.77 (9)°, φ_3 = 196.9 (10)° and Q = 1.1049 (17) Å, and this is also indicated by the dihedral angles of 62.4 (2) and 63.0 (2)° formed by the C8/C9/C16 plane with the N1/C7/C8 and N2/C16/C17 planes, respectively. The seven-membered ring in (I*b*) also exhibits a similar twist-boat conformation, with puckering parameters q_2 = 1.1194 (17) Å, q_3 = 0.0983 (17) Å, φ_2 = 219.87 (9)°, φ_3 = 18.2 (10)° and Q = 1.1238 (18) Å. The C31/C32/C39 plane forms dihedral angles with the N3/C30/

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

C6—C7	1.488 (2)	C30—N3	1.284 (2)
C7—N1	1.286 (2)	C40—N4	1.291 (2)
C17—N2	1.289 (2)	C40—C41	1.488 (2)
C17—C18	1.487 (2)	N1—N2	1.4056 (19)
C29—C30	1.486 (2)	N3—N4	1.4083 (19)
N1—C7—C6	117.17 (14)	N3—C30—C29	117.17 (15)
N1—C7—C8	121.65 (15)	N3—C30—C31	121.53 (15)
C6—C7—C8	121.11 (14)	C29—C30—C31	121.22 (15)
N2—C17—C18	117.66 (15)	N4—C40—C41	116.91 (14)
N2—C17—C16	122.19 (15)	N4—C40—C39	121.36 (15)
C18—C17—C16	120.14 (14)	C41—C40—C39	121.68 (14)
C7—N1—N2—C17	−60.5 (2)	C30—N3—N4—C40	61.8 (2)

Table 3
Selected geometric parameters (Å, °) for (II).

C6—C7	1.471 (3)	C17—C18	1.468 (3)
C7—N1	1.297 (3)	N1—N2	1.394 (2)
C17—N2	1.300 (3)		
N1—C7—C6	117.0 (2)	N2—C17—C18	116.4 (2)
N1—C7—C8	120.1 (2)	N2—C17—C16	119.8 (2)
C6—C7—C8	122.8 (2)	C18—C17—C16	123.8 (2)
C7—N1—N2—C17	−59.8 (3)		

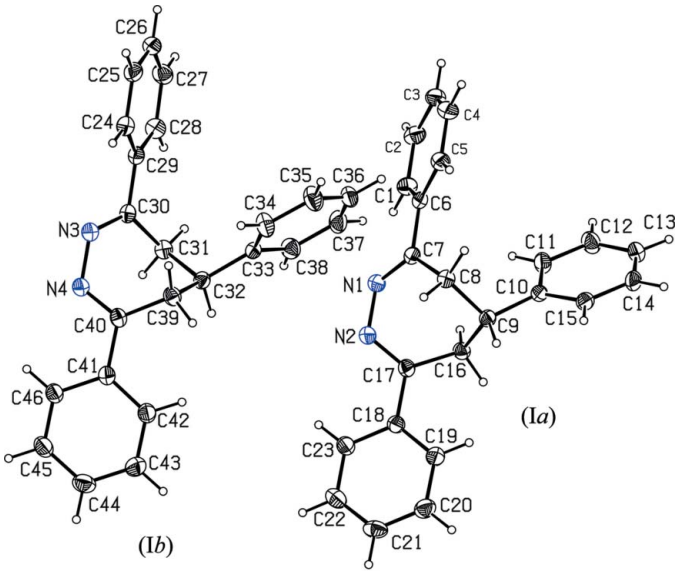


Figure 1
The two independent molecules [labelled (I*a*) and (I*b*)] of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 20% probability level.

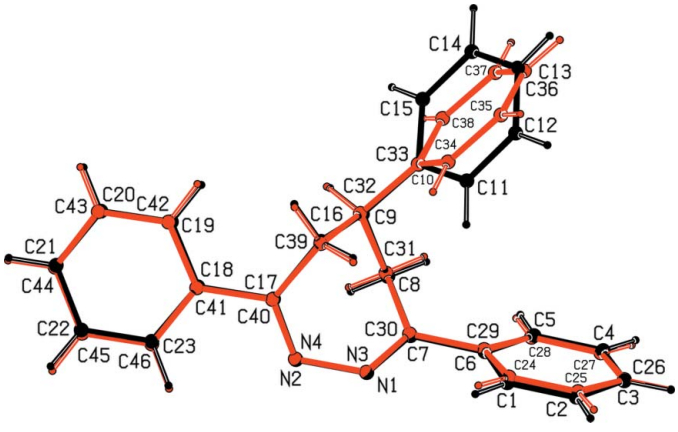


Figure 2
A quaternion *MOLFIT* plot (*PLATON*; Spek, 2009) of the two independent molecules, *viz.* (I*a*) and inverted (I*b*).

C31 and N4/C39/C40 planes of 63.0 (2) and 64.0 (2)°, respectively.

Compound (II) crystallizes in the space group *C*2/*c* (Fig. 3). Atoms C7 and C17 are both sp^2-hybridized (Table 3). Although the two C=N bonds are not conjugated with each other, with a C—N—N—C torsion angle of −59.8 (3)°, they participate in partial conjugation with their attached phenyl rings, as shown by the small dihedral angles of 3.6 (2)° between the C1–C6 and N1/C7/C8 planes and 4.8 (3)° between the C18–C23 and N2/C16/C17 planes. Thus, the C6—C7 and C17—C18 bonds in (II) are shorter than those in (I), the latter being typical single bonds (Csp^2—Csp^2). The C8/C9/C16 plane forms dihedral angles of 64.2 (3) and 60.3 (3)° with the N1/C7/C8 and N2/C16/C17 planes, respectively. This indicates that the seven-membered ring has a twist-boat conformation similar to that

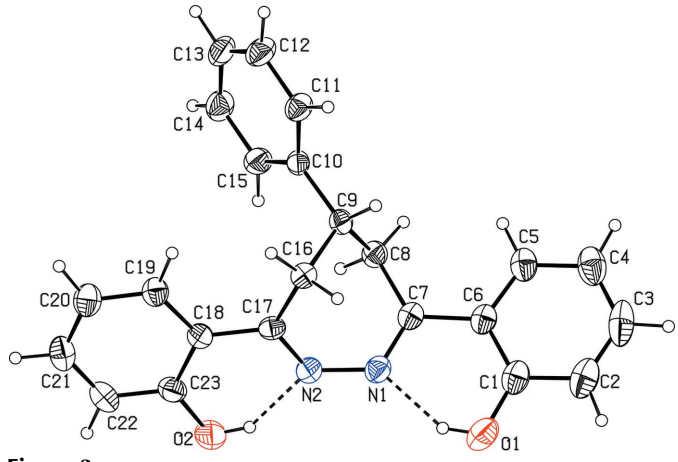


Figure 3
A view of (II), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 20% probability level. Dashed lines indicate intramolecular hydrogen bonds.

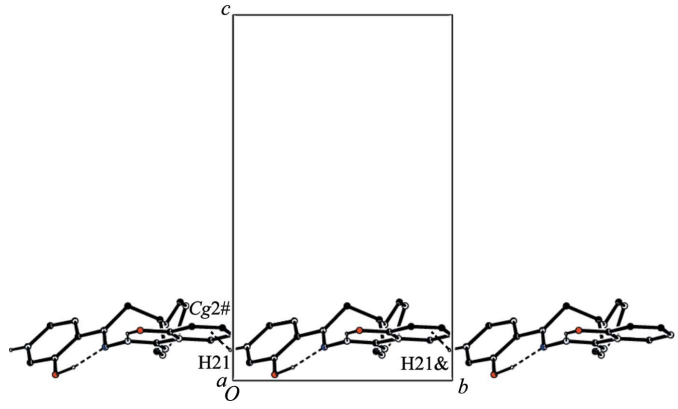


Figure 4
Part of the crystal structure of (II), showing the formation of the [010] chain linked by C—H···π interactions (dashed lines). For clarity, H atoms not involved in the motif shown have been omitted. Atoms labelled with a hash symbol (#) or ampersand (&) are at the symmetry positions (x, $y-1$, z) and (x, $y+1$, z), respectively.

of (I*a*), with puckering parameters q_2 = 1.087 (2) Å, q_3 = 0.116 (2) Å, φ_2 = 36.36 (13)°, φ_3 = 179.3 (12)° and Q = 1.093 (2) Å. Additionally, the C1–C6 and C10–C15 planes make a dihedral angle of 63.11 (13)°.

No π–π stacking interactions or C—H···π interactions are present in (I). The only supramolecular interaction in (II) is C21—H21···*Cg*2^{i} (π), where *Cg*2 is the centroid of the C10–C15 phenyl ring [symmetry code: (i) x, $y-1$, z]. Molecules of (II) join into an infinite chain in the *b*-axis direction (Table 4 and Fig. 4).

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

*Cg*2 is the centroid of the C10–C15 benzene ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1O···N1	0.95 (3)	1.71 (3)	2.539 (3)	144 (3)
O2—H2O···N2	0.97 (3)	1.65 (3)	2.532 (3)	150 (2)
C21—H21···*Cg*2^{i}	0.93	2.74	3.495 (3)	138

Symmetry code: (i) x, $y-1$, z.

As in the crystalline state of (II), a low-field signal of the OH group at 12.956 p.p.m. confirms the presence of intramolecular O—H···N hydrogen bonds in a $CDCl_3$ solution of (II) (Shi, Zhang, Cheng *et al.*, 2006; Shi & Zhang, 2007; Shi *et al.*, 2012).

The authors thank the Mao Zedong Foundation of China, Project Funded by the Priority Academic Program Development of Jiangsu Higher Education Institutions (PAPD), the Natural Science Foundation of China (grant No. 20572091) and the Natural Science Foundation of Jiangsu Province (grant No. 05KJB150151) for financial support of this work.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK3507). Services for accessing these data are described at the back of the journal.

References

Behenna, D. C., Liu, Y.-Y., Yurino, T., Kim, J., White, D. E., Virgil, S. C. & Stoltz, B. M. (2012). *Nat. Chem.* **4**, 130–133.
Bruker (2009). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Burla, M. C., Caliandro, R., Camalli, M., Carrozzini, B., Cascarano, G. L., De Caro, L., Giacovazzo, C., Polidori, G. & Spagna, R. (2005). *J. Appl. Cryst.* **38**, 381–388.
Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
Pettersson, B. (2011). PhD thesis, Karolinska Institutet, Stockholm, Sweden.
Sheldrick, G. M. (2004). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Shi, Y.-C., Cheng, H.-J., Sui, C.-X. & Zhu, B.-B. (2007). *J. Chem. Crystallogr.* **37**, 399–405.
Shi, Y.-C., Cheng, H.-J., Wu, S. & Hu, Y.-Y. (2012). *J. Chem. Crystallogr.* **42**, 633–638.
Shi, Y.-C., Sui, C.-X., Cheng, H.-J. & Zhu, B.-B. (2007). *J. Chem. Crystallogr.* **37**, 407–413.
Shi, Y.-C., Sui, C.-X. & Zhu, B.-B. (2006). *Acta Cryst.* E**62**, m2781–m2783.
Shi, Y.-C. & Zhang, S.-H. (2007). *Acta Cryst.* E**63**, o138–o140.
Shi, Y.-C., Zhang, S.-H., Cheng, H.-J. & Sun, W.-P. (2006). *Acta Cryst.* C**62**, m407–m410.
Shi, Y.-C., Zhu, B.-B. & Ng, S. W. (2007). *Acta Cryst.* E**63**, m1385–m1387.
Shi, Y.-C., Zhu, B.-B. & Sui, C.-X. (2006*a*). *Acta Cryst.* E**62**, m2389–m2391.
Shi, Y.-C., Zhu, B.-B. & Sui, C.-X. (2006*b*). *Acta Cryst.* E**62**, m2461–m2463.
Shi, Y.-C., Zhu, B.-B. & Sui, C.-X. (2006*c*). *Acta Cryst.* C**62**, m577–m580.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.
Zhu, B.-B., Shi, Y.-C., Shen, W.-B. & Li, Q.-K. (2011). *Acta Cryst.* E**67**, m1533.
Zotov, A. Y., Palyulin, V. A. & Zefirov, N. S. (1997). *J. Chem. Inf. Comput. Sci.* **37**, 766–773.

supplementary materials

Acta Cryst. (2013). C**69**, 1177-1180 [doi:10.1107/S0108270113023792]

Two seven-membered heterocycles with 1,2-diaza ring N atoms: 3,5,7-triphenyl-1,2-diazacyclohepta-1(7),2-diene and 3,7-bis(2-hydroxyphenyl)-5-phenyl-1,2-diazacyclohepta-1(7),2-diene

Yao-Cheng Shi and Hong-Yang Duan

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: *SIR2004* (Burla *et al.*, 2005); program(s) used to refine structure: *SHELXTL* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009) and *WinGX* (Farrugia, 2012); software used to prepare material for publication: *publCIF* (Westrip, 2010).

(I) 3,5,7-triphenyl-1,2-diazacyclohepta-1(7),2-diene

Crystal data

$C_{23}H_{20}N_2$
$M_r = 324.41$
Triclinic, $P\overline{1}$
$a = 9.4245\ (14)$ Å
$b = 10.6605\ (16)$ Å
$c = 18.405\ (3)$ Å
$\alpha = 80.2989\ (19)°$
$\beta = 85.094\ (2)°$
$\gamma = 78.1155\ (19)°$
$V = 1781.1\ (5)$ Å^3
$Z = 4$
$F(000) = 688$
$D_x = 1.210$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 5062 reflections
$\theta = 2.0–27.7°$
$\mu = 0.07$ mm^{-1}
$T = 296$ K
Block, white
0.24 × 0.20 × 0.15 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω and φ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)
$T_{min} = 0.967$, $T_{max} = 0.982$
15860 measured reflections
8141 independent reflections
5062 reflections with $I > 2\sigma(I)$
$R_{int} = 0.030$
$\theta_{max} = 27.7°$, $\theta_{min} = 2.0°$
$h = -11\rightarrow12$
$k = -13\rightarrow13$
$l = -24\rightarrow24$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.049$
$wR(F^2) = 0.149$
$S = 1.02$
8141 reflections
451 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0725P)^2 + 0.0625P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.18$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ e Å^{-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.

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

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.45206 (19)	0.35100 (18)	0.06052 (10)	0.0567 (5)
H1	0.4049	0.4128	0.0240	0.068*
C2	0.5909 (2)	0.2851 (2)	0.04550 (11)	0.0630 (5)
H2	0.6366	0.3030	−0.0008	0.076*
C3	0.6618 (2)	0.19324 (19)	0.09872 (12)	0.0650 (5)
H3	0.7548	0.1481	0.0883	0.078*
C4	0.5946 (2)	0.1683 (2)	0.16752 (13)	0.0693 (6)
H4	0.6432	0.1071	0.2039	0.083*
C5	0.4549 (2)	0.23370 (17)	0.18309 (11)	0.0589 (5)
H5	0.4102	0.2154	0.2296	0.071*
C6	0.38163 (17)	0.32626 (15)	0.12960 (9)	0.0459 (4)
C7	0.23121 (17)	0.39697 (16)	0.14432 (9)	0.0461 (4)
C8	0.12814 (18)	0.33644 (16)	0.19997 (9)	0.0478 (4)
H8A	0.0805	0.3993	0.2311	0.057*
H8B	0.1826	0.2630	0.2312	0.057*
C9	0.01134 (17)	0.29026 (15)	0.16181 (9)	0.0442 (4)
H9	−0.0735	0.2906	0.1965	0.053*
C10	0.05610 (17)	0.15394 (15)	0.14295 (9)	0.0444 (4)
C11	0.1572 (2)	0.12425 (17)	0.08598 (11)	0.0596 (5)
H11	0.2032	0.1885	0.0597	0.071*
C12	0.1908 (2)	0.00012 (19)	0.06769 (12)	0.0703 (6)
H12	0.2587	−0.0179	0.0292	0.084*
C13	0.1246 (2)	−0.09664 (18)	0.10582 (12)	0.0658 (5)
H13	0.1455	−0.1792	0.0926	0.079*
C14	0.0278 (2)	−0.06981 (17)	0.16334 (11)	0.0619 (5)
H14	−0.0157	−0.1352	0.1902	0.074*
C15	−0.00653 (18)	0.05436 (17)	0.18225 (10)	0.0517 (4)
H15	−0.0723	0.0708	0.2218	0.062*
C16	−0.03654 (18)	0.39067 (15)	0.09335 (9)	0.0474 (4)
H16A	0.0364	0.3792	0.0533	0.057*
H16B	−0.1269	0.3767	0.0776	0.057*
C17	−0.05721 (18)	0.52700 (15)	0.11026 (9)	0.0463 (4)
C18	−0.20589 (18)	0.60540 (16)	0.11874 (9)	0.0475 (4)

C19	−0.32386 (19)	0.54717 (19)	0.14197 (11)	0.0592 (5)
H19	−0.3103	0.4572	0.1529	0.071*
C20	−0.4617 (2)	0.6214 (2)	0.14902 (12)	0.0688 (5)
H20	−0.5402	0.5814	0.1649	0.083*
C21	−0.4828 (2)	0.7542 (2)	0.13257 (11)	0.0690 (6)
H21	−0.5757	0.8038	0.1372	0.083*
C22	−0.3676 (2)	0.8138 (2)	0.10936 (11)	0.0650 (5)
H22	−0.3827	0.9037	0.0980	0.078*
C23	−0.2289 (2)	0.74058 (17)	0.10276 (10)	0.0553 (4)
H23	−0.1509	0.7816	0.0877	0.066*
C24	0.64207 (19)	0.85674 (17)	0.43180 (10)	0.0542 (4)
H24	0.5895	0.8962	0.4698	0.065*
C25	0.7791 (2)	0.78340 (18)	0.44365 (11)	0.0612 (5)
H25	0.8178	0.7736	0.4895	0.073*
C26	0.8592 (2)	0.72444 (18)	0.38795 (12)	0.0638 (5)
H26	0.9515	0.6750	0.3962	0.077*
C27	0.8017 (2)	0.73931 (19)	0.32052 (12)	0.0664 (5)
H27	0.8558	0.7007	0.2826	0.080*
C28	0.6633 (2)	0.81159 (18)	0.30837 (10)	0.0594 (5)
H28	0.6247	0.8195	0.2626	0.071*
C29	0.58134 (18)	0.87253 (16)	0.36372 (9)	0.0473 (4)
C30	0.43378 (18)	0.95174 (16)	0.35081 (9)	0.0472 (4)
C31	0.33390 (18)	0.91588 (17)	0.30130 (9)	0.0492 (4)
H31A	0.3893	0.8541	0.2714	0.059*
H31B	0.2906	0.9927	0.2684	0.059*
C32	0.21158 (18)	0.85520 (15)	0.34815 (9)	0.0455 (4)
H32	0.1286	0.8720	0.3169	0.055*
C33	0.25497 (17)	0.70859 (15)	0.36832 (9)	0.0458 (4)
C34	0.2912 (2)	0.64605 (18)	0.43816 (11)	0.0702 (6)
H34	0.2901	0.6944	0.4759	0.084*
C35	0.3293 (3)	0.5110 (2)	0.45268 (13)	0.0803 (6)
H35	0.3527	0.4704	0.5001	0.096*
C36	0.3329 (2)	0.43795 (19)	0.39851 (14)	0.0700 (6)
H36	0.3590	0.3480	0.4085	0.084*
C37	0.2974 (2)	0.4986 (2)	0.32940 (13)	0.0720 (6)
H37	0.2996	0.4495	0.2919	0.086*
C38	0.2583 (2)	0.63236 (19)	0.31423 (11)	0.0625 (5)
H38	0.2337	0.6717	0.2667	0.075*
C39	0.16124 (19)	0.92650 (16)	0.41463 (9)	0.0510 (4)
H39A	0.0696	0.9055	0.4359	0.061*
H39B	0.2323	0.8984	0.4521	0.061*
C40	0.14324 (19)	1.07091 (16)	0.39051 (9)	0.0478 (4)
C41	−0.00265 (18)	1.15585 (16)	0.38144 (9)	0.0465 (4)
C42	−0.1257 (2)	1.10590 (18)	0.37626 (10)	0.0570 (5)
H42	−0.1177	1.0166	0.3806	0.068*
C43	−0.2598 (2)	1.1862 (2)	0.36478 (12)	0.0700 (6)
H43	−0.3410	1.1507	0.3617	0.084*
C44	−0.2728 (2)	1.3181 (2)	0.35797 (12)	0.0739 (6)
H44	−0.3623	1.3724	0.3491	0.089*

C45	−0.1526 (2)	1.36985 (19)	0.36436 (11)	0.0679 (5)
H45	−0.1619	1.4593	0.3605	0.082*
C46	−0.0187 (2)	1.29029 (17)	0.37643 (10)	0.0543 (4)
H46	0.0612	1.3263	0.3813	0.065*
N1	0.19129 (15)	0.50891 (14)	0.10536 (8)	0.0554 (4)
N2	0.05077 (15)	0.57917 (13)	0.11899 (9)	0.0547 (4)
N3	0.39270 (16)	1.04815 (14)	0.38622 (8)	0.0543 (4)
N4	0.25420 (15)	1.12582 (13)	0.37384 (8)	0.0514 (4)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0509 (11)	0.0633 (12)	0.0548 (11)	−0.0083 (9)	−0.0072 (8)	−0.0070 (9)
C2	0.0505 (11)	0.0795 (14)	0.0624 (12)	−0.0121 (10)	−0.0010 (9)	−0.0225 (10)
C3	0.0481 (11)	0.0632 (13)	0.0866 (15)	−0.0011 (9)	−0.0098 (10)	−0.0279 (11)
C4	0.0582 (12)	0.0601 (12)	0.0836 (15)	−0.0014 (10)	−0.0164 (11)	−0.0009 (10)
C5	0.0550 (11)	0.0534 (11)	0.0650 (12)	−0.0087 (9)	−0.0076 (9)	0.0000 (9)
C6	0.0441 (9)	0.0425 (9)	0.0546 (10)	−0.0130 (7)	−0.0082 (7)	−0.0090 (7)
C7	0.0454 (9)	0.0448 (9)	0.0504 (9)	−0.0134 (7)	−0.0042 (7)	−0.0070 (7)
C8	0.0502 (10)	0.0491 (10)	0.0445 (9)	−0.0110 (8)	−0.0026 (7)	−0.0069 (7)
C9	0.0428 (9)	0.0434 (9)	0.0463 (9)	−0.0112 (7)	0.0026 (7)	−0.0060 (7)
C10	0.0430 (9)	0.0414 (9)	0.0486 (9)	−0.0091 (7)	−0.0064 (7)	−0.0033 (7)
C11	0.0628 (11)	0.0466 (10)	0.0699 (12)	−0.0158 (9)	0.0144 (9)	−0.0132 (9)
C12	0.0742 (13)	0.0548 (12)	0.0814 (14)	−0.0092 (10)	0.0142 (11)	−0.0226 (10)
C13	0.0676 (13)	0.0435 (10)	0.0878 (15)	−0.0076 (9)	−0.0116 (11)	−0.0148 (10)
C14	0.0664 (12)	0.0436 (10)	0.0767 (13)	−0.0210 (9)	−0.0164 (10)	0.0069 (9)
C15	0.0498 (10)	0.0514 (10)	0.0527 (10)	−0.0135 (8)	−0.0073 (8)	0.0018 (8)
C16	0.0480 (9)	0.0452 (9)	0.0494 (9)	−0.0062 (7)	−0.0049 (7)	−0.0108 (7)
C17	0.0496 (10)	0.0416 (9)	0.0467 (9)	−0.0092 (8)	−0.0022 (7)	−0.0035 (7)
C18	0.0487 (10)	0.0470 (10)	0.0476 (9)	−0.0058 (8)	−0.0070 (7)	−0.0113 (7)
C19	0.0520 (11)	0.0558 (11)	0.0716 (12)	−0.0107 (9)	0.0001 (9)	−0.0165 (9)
C20	0.0516 (11)	0.0788 (15)	0.0807 (14)	−0.0135 (10)	0.0033 (10)	−0.0277 (11)
C21	0.0540 (12)	0.0847 (16)	0.0662 (13)	0.0106 (11)	−0.0097 (10)	−0.0324 (11)
C22	0.0721 (14)	0.0549 (11)	0.0639 (12)	0.0058 (10)	−0.0127 (10)	−0.0149 (9)
C23	0.0574 (11)	0.0481 (10)	0.0602 (11)	−0.0065 (9)	−0.0081 (9)	−0.0096 (8)
C24	0.0575 (11)	0.0530 (11)	0.0543 (10)	−0.0146 (9)	−0.0031 (8)	−0.0099 (8)
C25	0.0620 (12)	0.0569 (11)	0.0665 (12)	−0.0160 (9)	−0.0165 (10)	−0.0027 (9)
C26	0.0508 (11)	0.0505 (11)	0.0872 (15)	−0.0095 (9)	−0.0068 (10)	−0.0017 (10)
C27	0.0614 (12)	0.0599 (12)	0.0740 (14)	−0.0066 (10)	0.0123 (10)	−0.0139 (10)
C28	0.0632 (12)	0.0630 (12)	0.0515 (10)	−0.0114 (9)	−0.0018 (9)	−0.0092 (9)
C29	0.0495 (10)	0.0440 (9)	0.0499 (10)	−0.0143 (8)	−0.0007 (8)	−0.0064 (7)
C30	0.0520 (10)	0.0442 (9)	0.0470 (9)	−0.0146 (8)	−0.0015 (7)	−0.0055 (7)
C31	0.0558 (10)	0.0499 (10)	0.0424 (9)	−0.0112 (8)	−0.0014 (7)	−0.0078 (7)
C32	0.0475 (9)	0.0455 (9)	0.0455 (9)	−0.0116 (7)	−0.0052 (7)	−0.0076 (7)
C33	0.0406 (9)	0.0437 (9)	0.0554 (10)	−0.0121 (7)	−0.0008 (7)	−0.0099 (8)
C34	0.1031 (16)	0.0477 (11)	0.0613 (12)	−0.0147 (11)	−0.0170 (11)	−0.0067 (9)
C35	0.1083 (18)	0.0515 (12)	0.0784 (15)	−0.0149 (12)	−0.0161 (13)	0.0026 (11)
C36	0.0651 (13)	0.0423 (11)	0.1010 (17)	−0.0113 (9)	0.0053 (12)	−0.0101 (11)
C37	0.0768 (14)	0.0593 (13)	0.0890 (16)	−0.0194 (11)	0.0038 (12)	−0.0340 (12)
C38	0.0697 (13)	0.0589 (12)	0.0612 (11)	−0.0102 (10)	−0.0049 (9)	−0.0178 (9)

C39	0.0580 (10)	0.0457 (10)	0.0477 (9)	−0.0094 (8)	0.0025 (8)	−0.0058 (7)
C40	0.0567 (10)	0.0416 (9)	0.0450 (9)	−0.0094 (8)	0.0012 (8)	−0.0086 (7)
C41	0.0530 (10)	0.0446 (9)	0.0423 (9)	−0.0118 (8)	0.0047 (7)	−0.0092 (7)
C42	0.0567 (11)	0.0503 (10)	0.0646 (12)	−0.0125 (9)	0.0048 (9)	−0.0121 (9)
C43	0.0530 (12)	0.0730 (14)	0.0861 (15)	−0.0146 (10)	0.0016 (10)	−0.0181 (11)
C44	0.0553 (12)	0.0725 (15)	0.0855 (15)	0.0038 (11)	0.0031 (11)	−0.0126 (11)
C45	0.0683 (13)	0.0482 (11)	0.0821 (14)	−0.0042 (10)	0.0078 (11)	−0.0102 (10)
C46	0.0572 (11)	0.0457 (10)	0.0590 (11)	−0.0097 (8)	0.0054 (8)	−0.0101 (8)
N1	0.0458 (8)	0.0458 (9)	0.0715 (10)	−0.0101 (7)	0.0000 (7)	−0.0009 (7)
N2	0.0477 (8)	0.0429 (8)	0.0719 (10)	−0.0101 (7)	−0.0020 (7)	−0.0035 (7)
N3	0.0531 (9)	0.0462 (8)	0.0660 (10)	−0.0096 (7)	−0.0065 (7)	−0.0137 (7)
N4	0.0524 (9)	0.0442 (8)	0.0592 (9)	−0.0085 (7)	−0.0068 (7)	−0.0116 (7)

Geometric parameters (Å, º)

C1—C2	1.381 (2)	C24—C29	1.392 (2)
C1—C6	1.393 (2)	C24—H24	0.9300
C1—H1	0.9300	C25—C26	1.380 (3)
C2—C3	1.373 (3)	C25—H25	0.9300
C2—H2	0.9300	C26—C27	1.368 (3)
C3—C4	1.376 (3)	C26—H26	0.9300
C3—H3	0.9300	C27—C28	1.387 (3)
C4—C5	1.388 (3)	C27—H27	0.9300
C4—H4	0.9300	C28—C29	1.392 (2)
C5—C6	1.389 (2)	C28—H28	0.9300
C5—H5	0.9300	C29—C30	1.486 (2)
C6—C7	1.488 (2)	C30—N3	1.284 (2)
C7—N1	1.286 (2)	C30—C31	1.508 (2)
C7—C8	1.508 (2)	C31—C32	1.562 (2)
C8—C9	1.555 (2)	C31—H31A	0.9700
C8—H8A	0.9700	C31—H31B	0.9700
C8—H8B	0.9700	C32—C33	1.521 (2)
C9—C10	1.517 (2)	C32—C39	1.538 (2)
C9—C16	1.543 (2)	C32—H32	0.9800
C9—H9	0.9800	C33—C34	1.380 (2)
C10—C15	1.387 (2)	C33—C38	1.382 (2)
C10—C11	1.387 (2)	C34—C35	1.395 (3)
C11—C12	1.387 (3)	C34—H34	0.9300
C11—H11	0.9300	C35—C36	1.359 (3)
C12—C13	1.375 (3)	C35—H35	0.9300
C12—H12	0.9300	C36—C37	1.362 (3)
C13—C14	1.365 (3)	C36—H36	0.9300
C13—H13	0.9300	C37—C38	1.384 (3)
C14—C15	1.392 (3)	C37—H37	0.9300
C14—H14	0.9300	C38—H38	0.9300
C15—H15	0.9300	C39—C40	1.507 (2)
C16—C17	1.508 (2)	C39—H39A	0.9700
C16—H16A	0.9700	C39—H39B	0.9700
C16—H16B	0.9700	C40—N4	1.291 (2)
C17—N2	1.289 (2)	C40—C41	1.488 (2)

C17—C18	1.487 (2)	C41—C42	1.388 (2)
C18—C19	1.385 (2)	C41—C46	1.398 (2)
C18—C23	1.396 (2)	C42—C43	1.384 (3)
C19—C20	1.384 (3)	C42—H42	0.9300
C19—H19	0.9300	C43—C44	1.371 (3)
C20—C21	1.372 (3)	C43—H43	0.9300
C20—H20	0.9300	C44—C45	1.380 (3)
C21—C22	1.370 (3)	C44—H44	0.9300
C21—H21	0.9300	C45—C46	1.380 (2)
C22—C23	1.385 (2)	C45—H45	0.9300
C22—H22	0.9300	C46—H46	0.9300
C23—H23	0.9300	N1—N2	1.4056 (19)
C24—C25	1.379 (2)	N3—N4	1.4083 (19)

C2—C1—C6	120.96 (17)	C29—C24—H24	119.6
C2—C1—H1	119.5	C24—C25—C26	120.53 (18)
C6—C1—H1	119.5	C24—C25—H25	119.7
C3—C2—C1	120.23 (18)	C26—C25—H25	119.7
C3—C2—H2	119.9	C27—C26—C25	119.50 (18)
C1—C2—H2	119.9	C27—C26—H26	120.2
C2—C3—C4	119.67 (18)	C25—C26—H26	120.2
C2—C3—H3	120.2	C26—C27—C28	120.37 (19)
C4—C3—H3	120.2	C26—C27—H27	119.8
C3—C4—C5	120.53 (18)	C28—C27—H27	119.8
C3—C4—H4	119.7	C27—C28—C29	120.92 (18)
C5—C4—H4	119.7	C27—C28—H28	119.5
C4—C5—C6	120.38 (18)	C29—C28—H28	119.5
C4—C5—H5	119.8	C24—C29—C28	117.80 (16)
C6—C5—H5	119.8	C24—C29—C30	120.94 (15)
C5—C6—C1	118.22 (16)	C28—C29—C30	121.26 (16)
C5—C6—C7	121.65 (15)	N3—C30—C29	117.17 (15)
C1—C6—C7	120.12 (15)	N3—C30—C31	121.53 (15)
N1—C7—C6	117.17 (14)	C29—C30—C31	121.22 (15)
N1—C7—C8	121.65 (15)	C30—C31—C32	110.55 (13)
C6—C7—C8	121.11 (14)	C30—C31—H31A	109.5
C7—C8—C9	111.65 (13)	C32—C31—H31A	109.5
C7—C8—H8A	109.3	C30—C31—H31B	109.5
C9—C8—H8A	109.3	C32—C31—H31B	109.5
C7—C8—H8B	109.3	H31A—C31—H31B	108.1
C9—C8—H8B	109.3	C33—C32—C39	114.17 (13)
H8A—C8—H8B	108.0	C33—C32—C31	112.31 (13)
C10—C9—C16	111.73 (13)	C39—C32—C31	109.82 (13)
C10—C9—C8	114.79 (13)	C33—C32—H32	106.7
C16—C9—C8	109.01 (13)	C39—C32—H32	106.7
C10—C9—H9	107.0	C31—C32—H32	106.7
C16—C9—H9	107.0	C34—C33—C38	117.41 (17)
C8—C9—H9	107.0	C34—C33—C32	123.80 (16)
C15—C10—C11	117.68 (16)	C38—C33—C32	118.78 (16)
C15—C10—C9	120.10 (15)	C33—C34—C35	120.62 (19)

C11—C10—C9	122.21 (14)	C33—C34—H34	119.7
C12—C11—C10	120.91 (17)	C35—C34—H34	119.7
C12—C11—H11	119.5	C36—C35—C34	121.0 (2)
C10—C11—H11	119.5	C36—C35—H35	119.5
C13—C12—C11	120.68 (19)	C34—C35—H35	119.5
C13—C12—H12	119.7	C35—C36—C37	118.90 (19)
C11—C12—H12	119.7	C35—C36—H36	120.6
C14—C13—C12	119.08 (18)	C37—C36—H36	120.6
C14—C13—H13	120.5	C36—C37—C38	120.83 (19)
C12—C13—H13	120.5	C36—C37—H37	119.6
C13—C14—C15	120.72 (17)	C38—C37—H37	119.6
C13—C14—H14	119.6	C33—C38—C37	121.25 (19)
C15—C14—H14	119.6	C33—C38—H38	119.4
C10—C15—C14	120.88 (17)	C37—C38—H38	119.4
C10—C15—H15	119.6	C40—C39—C32	109.97 (13)
C14—C15—H15	119.6	C40—C39—H39A	109.7
C17—C16—C9	110.90 (13)	C32—C39—H39A	109.7
C17—C16—H16A	109.5	C40—C39—H39B	109.7
C9—C16—H16A	109.5	C32—C39—H39B	109.7
C17—C16—H16B	109.5	H39A—C39—H39B	108.2
C9—C16—H16B	109.5	N4—C40—C41	116.91 (14)
H16A—C16—H16B	108.0	N4—C40—C39	121.36 (15)
N2—C17—C18	117.66 (15)	C41—C40—C39	121.68 (14)
N2—C17—C16	122.19 (15)	C42—C41—C46	117.92 (16)
C18—C17—C16	120.14 (14)	C42—C41—C40	121.87 (15)
C19—C18—C23	118.65 (16)	C46—C41—C40	120.19 (15)
C19—C18—C17	121.36 (15)	C43—C42—C41	121.42 (18)
C23—C18—C17	119.99 (15)	C43—C42—H42	119.3
C20—C19—C18	120.67 (18)	C41—C42—H42	119.3
C20—C19—H19	119.7	C44—C43—C42	119.85 (19)
C18—C19—H19	119.7	C44—C43—H43	120.1
C21—C20—C19	120.03 (19)	C42—C43—H43	120.1
C21—C20—H20	120.0	C43—C44—C45	119.79 (19)
C19—C20—H20	120.0	C43—C44—H44	120.1
C22—C21—C20	120.26 (18)	C45—C44—H44	120.1
C22—C21—H21	119.9	C44—C45—C46	120.65 (19)
C20—C21—H21	119.9	C44—C45—H45	119.7
C21—C22—C23	120.28 (19)	C46—C45—H45	119.7
C21—C22—H22	119.9	C45—C46—C41	120.33 (17)
C23—C22—H22	119.9	C45—C46—H46	119.8
C22—C23—C18	120.11 (18)	C41—C46—H46	119.8
C22—C23—H23	119.9	C7—N1—N2	118.48 (13)
C18—C23—H23	119.9	C17—N2—N1	117.55 (14)
C25—C24—C29	120.87 (17)	C30—N3—N4	118.10 (14)
C25—C24—H24	119.6	C40—N4—N3	117.54 (14)

C6—C1—C2—C3	0.2 (3)	C25—C24—C29—C30	−179.90 (16)
C1—C2—C3—C4	−0.8 (3)	C27—C28—C29—C24	−1.0 (3)
C2—C3—C4—C5	1.0 (3)	C27—C28—C29—C30	179.10 (16)

C3—C4—C5—C6	−0.6 (3)	C24—C29—C30—N3	31.3 (2)
C4—C5—C6—C1	0.0 (3)	C28—C29—C30—N3	−148.76 (17)
C4—C5—C6—C7	179.31 (17)	C24—C29—C30—C31	−145.51 (16)
C2—C1—C6—C5	0.2 (3)	C28—C29—C30—C31	34.4 (2)
C2—C1—C6—C7	−179.12 (16)	N3—C30—C31—C32	−71.6 (2)
C5—C6—C7—N1	155.10 (17)	C29—C30—C31—C32	105.09 (17)
C1—C6—C7—N1	−25.6 (2)	C30—C31—C32—C33	−89.81 (17)
C5—C6—C7—C8	−27.9 (2)	C30—C31—C32—C39	38.37 (18)
C1—C6—C7—C8	151.36 (16)	C39—C32—C33—C34	−19.5 (2)
N1—C7—C8—C9	71.8 (2)	C31—C32—C33—C34	106.31 (19)
C6—C7—C8—C9	−105.05 (17)	C39—C32—C33—C38	160.18 (16)
C7—C8—C9—C10	87.21 (17)	C31—C32—C33—C38	−73.96 (19)
C7—C8—C9—C16	−38.97 (18)	C38—C33—C34—C35	0.0 (3)
C16—C9—C10—C15	−126.52 (16)	C32—C33—C34—C35	179.74 (18)
C8—C9—C10—C15	108.72 (17)	C33—C34—C35—C36	0.5 (3)
C16—C9—C10—C11	52.3 (2)	C34—C35—C36—C37	−0.4 (3)
C8—C9—C10—C11	−72.5 (2)	C35—C36—C37—C38	−0.1 (3)
C15—C10—C11—C12	2.0 (3)	C34—C33—C38—C37	−0.5 (3)
C9—C10—C11—C12	−176.81 (17)	C32—C33—C38—C37	179.76 (16)
C10—C11—C12—C13	−0.2 (3)	C36—C37—C38—C33	0.5 (3)
C11—C12—C13—C14	−1.6 (3)	C33—C32—C39—C40	171.24 (14)
C12—C13—C14—C15	1.6 (3)	C31—C32—C39—C40	44.09 (18)
C11—C10—C15—C14	−2.1 (2)	C32—C39—C40—N4	−72.9 (2)
C9—C10—C15—C14	176.75 (15)	C32—C39—C40—C41	104.24 (17)
C13—C14—C15—C10	0.3 (3)	N4—C40—C41—C42	160.69 (16)
C10—C9—C16—C17	−170.82 (13)	C39—C40—C41—C42	−16.5 (2)
C8—C9—C16—C17	−42.90 (17)	N4—C40—C41—C46	−18.1 (2)
C9—C16—C17—N2	72.4 (2)	C39—C40—C41—C46	164.70 (15)
C9—C16—C17—C18	−106.57 (16)	C46—C41—C42—C43	1.5 (3)
N2—C17—C18—C19	−151.71 (17)	C40—C41—C42—C43	−177.30 (17)
C16—C17—C18—C19	27.3 (2)	C41—C42—C43—C44	0.3 (3)
N2—C17—C18—C23	28.7 (2)	C42—C43—C44—C45	−1.5 (3)
C16—C17—C18—C23	−152.37 (16)	C43—C44—C45—C46	1.0 (3)
C23—C18—C19—C20	0.2 (3)	C44—C45—C46—C41	0.8 (3)
C17—C18—C19—C20	−179.48 (17)	C42—C41—C46—C45	−2.0 (3)
C18—C19—C20—C21	0.3 (3)	C40—C41—C46—C45	176.79 (16)
C19—C20—C21—C22	−0.2 (3)	C6—C7—N1—N2	−178.77 (14)
C20—C21—C22—C23	−0.4 (3)	C8—C7—N1—N2	4.2 (2)
C21—C22—C23—C18	0.9 (3)	C18—C17—N2—N1	−175.19 (14)
C19—C18—C23—C22	−0.8 (3)	C16—C17—N2—N1	5.9 (2)
C17—C18—C23—C22	178.86 (16)	C7—N1—N2—C17	−60.5 (2)
C29—C24—C25—C26	0.3 (3)	C29—C30—N3—N4	178.94 (13)
C24—C25—C26—C27	0.1 (3)	C31—C30—N3—N4	−4.2 (2)
C25—C26—C27—C28	−0.9 (3)	C41—C40—N4—N3	176.38 (14)
C26—C27—C28—C29	1.3 (3)	C39—C40—N4—N3	−6.4 (2)
C25—C24—C29—C28	0.2 (2)	C30—N3—N4—C40	61.8 (2)

(II) 3,7-bis(2-hydroxyphenyl)-5-phenyl-1,2-diazacyclohepta-1(7),2-diene

Crystal data

$C_{23}H_{20}N_2O_2$
M_r = 356.41
Monoclinic, *C*2/*c*
a = 23.9715 (14) Å
b = 9.5669 (16) Å
c = 18.2303 (12) Å
β = 119.256 (3)°
V = 3647.5 (7) Å^3
Z = 8
F(000) = 1504
D_x = 1.298 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 1649 reflections
θ = 2.0–25.0°
μ = 0.08 mm^{-1}
T = 296 K
Block, yellow
0.30 × 0.28 × 0.26 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω and *φ* scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)
T_{min} = 0.967, T_{max} = 0.972
12998 measured reflections
3224 independent reflections
1647 reflections with $I > 2\sigma(I)$
R_{int} = 0.071
θ_{max} = 25.0°, θ_{min} = 2.0°
h = −27→28
k = −11→11
l = −21→21

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.109
S = 0.96
3224 reflections
253 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0426P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.13 e Å^{-3}
$\Delta\rho_{min}$ = −0.13 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0014 (2)

Special details

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

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

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.37760 (14)	0.7121 (3)	0.13321 (16)	0.0607 (7)
C2	0.42387 (14)	0.8122 (4)	0.14804 (18)	0.0827 (10)
H2	0.4647	0.7840	0.1606	0.099*

C3	0.41027 (18)	0.9513 (4)	0.14444 (19)	0.0923 (11)
H3	0.4421	1.0168	0.1552	0.111*
C4	0.34995 (17)	0.9960 (3)	0.12501 (17)	0.0795 (9)
H4	0.3406	1.0909	0.1217	0.095*
C5	0.30379 (13)	0.8978 (3)	0.11052 (14)	0.0595 (7)
H5	0.2631	0.9280	0.0973	0.071*
C6	0.31590 (12)	0.7540 (3)	0.11501 (14)	0.0500 (6)
C7	0.26667 (11)	0.6507 (3)	0.10285 (13)	0.0450 (6)
C8	0.20110 (11)	0.6912 (2)	0.08683 (14)	0.0479 (6)
H8A	0.1692	0.6360	0.0408	0.058*
H8B	0.1933	0.7890	0.0709	0.058*
C9	0.19542 (10)	0.6666 (2)	0.16681 (14)	0.0420 (6)
H9	0.2233	0.7356	0.2084	0.050*
C10	0.12857 (11)	0.6920 (2)	0.15289 (15)	0.0415 (6)
C11	0.12080 (12)	0.7586 (2)	0.21469 (16)	0.0545 (7)
H11	0.1567	0.7863	0.2641	0.065*
C12	0.06055 (14)	0.7850 (3)	0.20441 (19)	0.0654 (8)
H12	0.0564	0.8293	0.2469	0.079*
C13	0.00712 (14)	0.7458 (3)	0.1318 (2)	0.0647 (8)
H13	−0.0334	0.7644	0.1245	0.078*
C14	0.01365 (13)	0.6789 (3)	0.06980 (17)	0.0626 (7)
H14	−0.0226	0.6514	0.0207	0.075*
C15	0.07382 (13)	0.6522 (2)	0.07994 (17)	0.0576 (7)
H15	0.0776	0.6070	0.0374	0.069*
C16	0.22259 (11)	0.5200 (2)	0.20419 (13)	0.0451 (6)
H16A	0.2685	0.5272	0.2400	0.054*
H16B	0.2044	0.4902	0.2389	0.054*
C17	0.20836 (11)	0.4110 (2)	0.13753 (14)	0.0435 (6)
C18	0.16451 (11)	0.2931 (2)	0.12071 (14)	0.0425 (6)
C19	0.12935 (11)	0.2789 (2)	0.16312 (15)	0.0523 (7)
H19	0.1327	0.3478	0.2011	0.063*
C20	0.09007 (12)	0.1661 (3)	0.15024 (16)	0.0640 (7)
H20	0.0677	0.1584	0.1798	0.077*
C21	0.08399 (13)	0.0638 (3)	0.09293 (17)	0.0646 (8)
H21	0.0576	−0.0128	0.0841	0.077*
C22	0.11670 (13)	0.0752 (3)	0.04939 (17)	0.0648 (8)
H22	0.1119	0.0066	0.0106	0.078*
C23	0.15679 (12)	0.1872 (2)	0.06208 (16)	0.0535 (7)
H1O	0.3599 (15)	0.519 (3)	0.1286 (18)	0.105 (12)*
H2O	0.2146 (13)	0.272 (3)	0.0362 (18)	0.098 (10)*
N1	0.28238 (9)	0.5199 (2)	0.10805 (12)	0.0511 (5)
N2	0.23654 (9)	0.41738 (19)	0.09215 (12)	0.0506 (5)
O1	0.39492 (9)	0.5766 (3)	0.13736 (12)	0.0776 (6)
O2	0.18745 (11)	0.1904 (2)	0.01661 (13)	0.0808 (6)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.058 (2)	0.080 (2)	0.0454 (18)	−0.0123 (17)	0.0258 (15)	0.0017 (14)
C2	0.061 (2)	0.113 (3)	0.070 (2)	−0.027 (2)	0.0293 (17)	−0.0015 (19)

C3	0.093 (3)	0.111 (3)	0.073 (2)	−0.057 (2)	0.040 (2)	−0.014 (2)
C4	0.106 (3)	0.077 (2)	0.064 (2)	−0.033 (2)	0.048 (2)	−0.0091 (16)
C5	0.072 (2)	0.0653 (18)	0.0450 (17)	−0.0161 (16)	0.0318 (15)	−0.0030 (13)
C6	0.0528 (18)	0.0622 (16)	0.0366 (16)	−0.0098 (14)	0.0232 (14)	−0.0013 (12)
C7	0.0452 (17)	0.0569 (16)	0.0330 (15)	−0.0032 (13)	0.0191 (13)	−0.0001 (12)
C8	0.0475 (16)	0.0485 (14)	0.0474 (16)	−0.0001 (12)	0.0229 (13)	0.0071 (11)
C9	0.0419 (15)	0.0406 (13)	0.0412 (15)	−0.0041 (11)	0.0186 (12)	−0.0032 (11)
C10	0.0382 (15)	0.0380 (13)	0.0462 (16)	−0.0021 (11)	0.0190 (13)	0.0007 (11)
C11	0.0516 (18)	0.0677 (16)	0.0495 (17)	−0.0051 (13)	0.0288 (15)	−0.0052 (13)
C12	0.065 (2)	0.0805 (19)	0.070 (2)	−0.0008 (16)	0.0474 (19)	−0.0015 (16)
C13	0.054 (2)	0.0690 (18)	0.083 (2)	0.0010 (15)	0.042 (2)	0.0118 (17)
C14	0.0443 (18)	0.0701 (18)	0.065 (2)	−0.0063 (14)	0.0197 (16)	−0.0028 (15)
C15	0.0498 (18)	0.0599 (16)	0.0629 (19)	−0.0030 (14)	0.0274 (16)	−0.0118 (14)
C16	0.0449 (15)	0.0500 (14)	0.0377 (14)	0.0002 (12)	0.0181 (12)	0.0001 (11)
C17	0.0422 (16)	0.0469 (14)	0.0406 (15)	0.0086 (12)	0.0195 (13)	0.0044 (11)
C18	0.0435 (15)	0.0420 (13)	0.0395 (15)	0.0028 (11)	0.0184 (13)	0.0038 (11)
C19	0.0556 (18)	0.0541 (15)	0.0464 (17)	−0.0047 (13)	0.0242 (15)	0.0023 (12)
C20	0.067 (2)	0.0693 (18)	0.0589 (19)	−0.0088 (16)	0.0328 (16)	0.0064 (15)
C21	0.064 (2)	0.0550 (17)	0.064 (2)	−0.0111 (14)	0.0231 (17)	0.0076 (15)
C22	0.078 (2)	0.0436 (15)	0.069 (2)	−0.0040 (15)	0.0328 (17)	−0.0041 (14)
C23	0.0638 (18)	0.0455 (15)	0.0562 (18)	0.0039 (13)	0.0331 (16)	−0.0023 (13)
N1	0.0500 (14)	0.0574 (13)	0.0521 (14)	−0.0032 (12)	0.0300 (11)	−0.0019 (11)
N2	0.0545 (14)	0.0487 (12)	0.0572 (14)	−0.0033 (11)	0.0339 (12)	−0.0040 (10)
O1	0.0528 (14)	0.0947 (16)	0.0912 (16)	0.0010 (13)	0.0398 (12)	0.0054 (12)
O2	0.1081 (17)	0.0676 (13)	0.1017 (16)	−0.0172 (12)	0.0786 (15)	−0.0309 (11)

Geometric parameters (Å, º)

C1—O1	1.351 (3)	C12—H12	0.9300
C1—C2	1.389 (4)	C13—C14	1.372 (3)
C1—C6	1.407 (3)	C13—H13	0.9300
C2—C3	1.364 (4)	C14—C15	1.386 (3)
C2—H2	0.9300	C14—H14	0.9300
C3—C4	1.378 (4)	C15—H15	0.9300
C3—H3	0.9300	C16—C17	1.509 (3)
C4—C5	1.375 (3)	C16—H16A	0.9700
C4—H4	0.9300	C16—H16B	0.9700
C5—C6	1.400 (3)	C17—N2	1.300 (3)
C5—H5	0.9300	C17—C18	1.468 (3)
C6—C7	1.471 (3)	C18—C19	1.401 (3)
C7—N1	1.297 (3)	C18—C23	1.418 (3)
C7—C8	1.503 (3)	C19—C20	1.375 (3)
C8—C9	1.547 (3)	C19—H19	0.9300
C8—H8A	0.9700	C20—C21	1.386 (3)
C8—H8B	0.9700	C20—H20	0.9300
C9—C10	1.514 (3)	C21—C22	1.366 (3)
C9—C16	1.556 (3)	C21—H21	0.9300
C9—H9	0.9800	C22—C23	1.381 (3)
C10—C11	1.384 (3)	C22—H22	0.9300
C10—C15	1.389 (3)	C23—O2	1.351 (3)

C11—C12	1.386 (3)	N1—N2	1.394 (2)
C11—H11	0.9300	O1—H1O	0.95 (3)
C12—C13	1.370 (3)	O2—H2O	0.97 (3)
O1—C1—C2	117.2 (3)	C11—C12—H12	120.0
O1—C1—C6	123.1 (2)	C12—C13—C14	119.6 (3)
C2—C1—C6	119.8 (3)	C12—C13—H13	120.2
C3—C2—C1	121.0 (3)	C14—C13—H13	120.2
C3—C2—H2	119.5	C13—C14—C15	120.4 (3)
C1—C2—H2	119.5	C13—C14—H14	119.8
C2—C3—C4	120.7 (3)	C15—C14—H14	119.8
C2—C3—H3	119.6	C14—C15—C10	120.8 (2)
C4—C3—H3	119.6	C14—C15—H15	119.6
C5—C4—C3	118.9 (3)	C10—C15—H15	119.6
C5—C4—H4	120.6	C17—C16—C9	112.89 (18)
C3—C4—H4	120.6	C17—C16—H16A	109.0
C4—C5—C6	122.4 (3)	C9—C16—H16A	109.0
C4—C5—H5	118.8	C17—C16—H16B	109.0
C6—C5—H5	118.8	C9—C16—H16B	109.0
C5—C6—C1	117.3 (2)	H16A—C16—H16B	107.8
C5—C6—C7	121.6 (2)	N2—C17—C18	116.4 (2)
C1—C6—C7	121.2 (2)	N2—C17—C16	119.8 (2)
N1—C7—C6	117.0 (2)	C18—C17—C16	123.8 (2)
N1—C7—C8	120.1 (2)	C19—C18—C23	117.0 (2)
C6—C7—C8	122.8 (2)	C19—C18—C17	121.4 (2)
C7—C8—C9	110.07 (18)	C23—C18—C17	121.5 (2)
C7—C8—H8A	109.6	C20—C19—C18	121.9 (2)
C9—C8—H8A	109.6	C20—C19—H19	119.1
C7—C8—H8B	109.6	C18—C19—H19	119.1
C9—C8—H8B	109.6	C19—C20—C21	119.6 (2)
H8A—C8—H8B	108.2	C19—C20—H20	120.2
C10—C9—C8	113.01 (18)	C21—C20—H20	120.2
C10—C9—C16	113.57 (17)	C22—C21—C20	120.2 (2)
C8—C9—C16	109.80 (17)	C22—C21—H21	119.9
C10—C9—H9	106.7	C20—C21—H21	119.9
C8—C9—H9	106.7	C21—C22—C23	121.0 (2)
C16—C9—H9	106.7	C21—C22—H22	119.5
C11—C10—C15	117.7 (2)	C23—C22—H22	119.5
C11—C10—C9	119.3 (2)	O2—C23—C22	117.2 (2)
C15—C10—C9	122.9 (2)	O2—C23—C18	122.5 (2)
C10—C11—C12	121.3 (2)	C22—C23—C18	120.3 (2)
C10—C11—H11	119.3	C7—N1—N2	119.52 (19)
C12—C11—H11	119.3	C17—N2—N1	120.17 (19)
C13—C12—C11	120.1 (3)	C1—O1—H1O	108.9 (18)
C13—C12—H12	120.0	C23—O2—H2O	105.1 (16)
O1—C1—C2—C3	179.5 (3)	C13—C14—C15—C10	0.2 (4)
C6—C1—C2—C3	−0.6 (4)	C11—C10—C15—C14	0.2 (3)
C1—C2—C3—C4	−0.8 (5)	C9—C10—C15—C14	−179.4 (2)

C2—C3—C4—C5	1.0 (4)	C10—C9—C16—C17	91.1 (2)
C3—C4—C5—C6	0.2 (4)	C8—C9—C16—C17	−36.5 (3)
C4—C5—C6—C1	−1.5 (4)	C9—C16—C17—N2	69.7 (3)
C4—C5—C6—C7	177.4 (2)	C9—C16—C17—C18	−111.5 (2)
O1—C1—C6—C5	−178.4 (2)	N2—C17—C18—C19	−177.2 (2)
C2—C1—C6—C5	1.7 (4)	C16—C17—C18—C19	4.0 (3)
O1—C1—C6—C7	2.7 (4)	N2—C17—C18—C23	4.1 (3)
C2—C1—C6—C7	−177.1 (2)	C16—C17—C18—C23	−174.7 (2)
C5—C6—C7—N1	179.9 (2)	C23—C18—C19—C20	1.4 (3)
C1—C6—C7—N1	−1.3 (3)	C17—C18—C19—C20	−177.4 (2)
C5—C6—C7—C8	−1.4 (3)	C18—C19—C20—C21	−0.9 (4)
C1—C6—C7—C8	177.4 (2)	C19—C20—C21—C22	−0.2 (4)
N1—C7—C8—C9	73.4 (3)	C20—C21—C22—C23	0.7 (4)
C6—C7—C8—C9	−105.2 (2)	C21—C22—C23—O2	179.9 (2)
C7—C8—C9—C10	−174.46 (18)	C21—C22—C23—C18	−0.2 (4)
C7—C8—C9—C16	−46.5 (2)	C19—C18—C23—O2	179.1 (2)
C8—C9—C10—C11	−140.0 (2)	C17—C18—C23—O2	−2.1 (4)
C16—C9—C10—C11	94.1 (2)	C19—C18—C23—C22	−0.8 (3)
C8—C9—C10—C15	39.5 (3)	C17—C18—C23—C22	178.0 (2)
C16—C9—C10—C15	−86.4 (3)	C6—C7—N1—N2	−176.21 (19)
C15—C10—C11—C12	0.0 (3)	C8—C7—N1—N2	5.0 (3)
C9—C10—C11—C12	179.5 (2)	C18—C17—N2—N1	−175.34 (19)
C10—C11—C12—C13	−0.4 (4)	C16—C17—N2—N1	3.5 (3)
C11—C12—C13—C14	0.8 (4)	C7—N1—N2—C17	−59.8 (3)
C12—C13—C14—C15	−0.6 (4)		

Hydrogen-bond geometry (Å, º)

Cg2 is the centroid of the C10–C15 benzene ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*O*···N1	0.95 (3)	1.71 (3)	2.539 (3)	144 (3)
O2—H2*O*···N2	0.97 (3)	1.65 (3)	2.532 (3)	150 (2)
C21—H21···*Cg*2^{i}	0.93	2.74	3.495 (3)	138

Symmetry code: (i) *x*, *y*−1, *z*.