Powder study of 3-azabicyclo[3.3.1]nonane-2,4-dione 1-methylnaphthalene hemisolvate

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Key indicators

Powder X-ray study
T = 295 K
Mean σ(C–C) = 0.002 Å
R factor = 0.046
wR factor = 0.057

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.
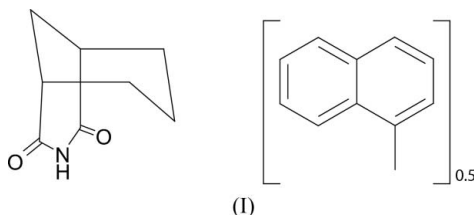
The crystal structure of the title compound, $C_8H_{11}N_1O_2\cdot0.5C_{11}H_{10}$, was solved by simulated annealing from laboratory X-ray powder diffraction data, collected at room temperature. Subsequent Rietveld refinement, using data collected to 1.51 Å resolution, yielded an R_{wp} value of 0.057. The compound crystallizes with two molecules of 3-azabicyclo-nonane-2,4-dione and one molecule of 1-methylnaphthalene in the asymmetric unit.
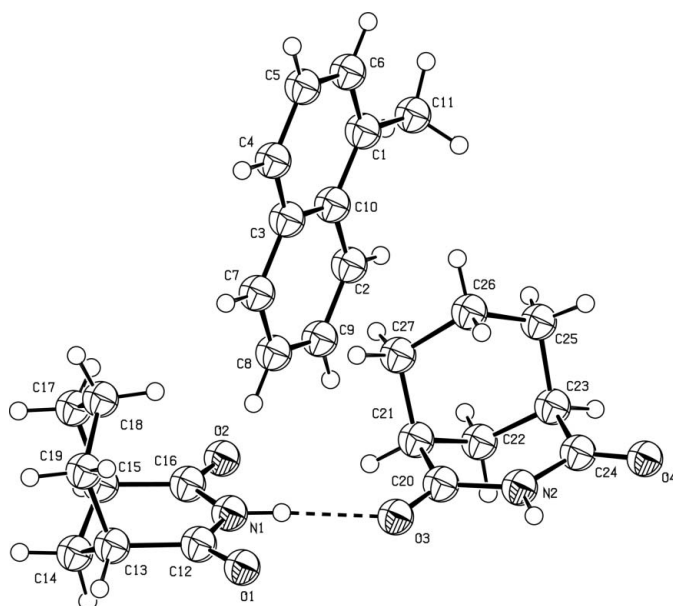
Comment

The title compound, (I), was crystallized from 1-methyl-naphthalene during a preliminary solvent screen in preparation for an automated parallel crystallization study of 3-azabicyclo[3.3.1]nonane-2,4-dione. The sample was identified as a new form using multi-sample foil transmission X-ray powder diffraction analysis (Florence *et al.*, 2003).
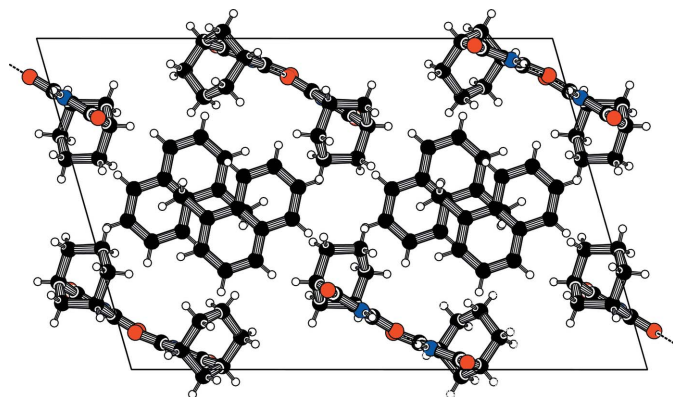

(I)

The crystal structure of (I) was solved by simulated annealing using laboratory X-ray powder diffraction data. The compound crystallizes in space group $P2_1/c$ with two molecules of 3-azabicyclononane-2,4-dione and one molecule of 1-methylnaphthalene in the asymmetric unit (Fig. 1). In the crystal structure, intermolecular N—H···O hydrogen bonds (Table 1) link two independent 3-azabicyclononane-2,4-dione molecules into a chain (Fig. 1) running along the *b* axis. The structure of this chain is very similar to that found in the un-solvated form 1 of 3-azabicyclononane-2,4-dione (Howie & Skakle, 2001). However, in form 1 the chain is propagated by a glide symmetry operation, whereas in (I) it propagates *via* a 2_1 screw axis. The paired hydrogen-bonded chains in (I) constitute a pseudo-layer parallel to the *bc* plane. Between two pseudo-layers related by translation along the *a* axis, channels parallel to the *b* axis are observed. These channels are filled by stacks of 1-methylnaphthalene solvent molecules, which are oriented nearly perpendicular to the *b* axis (Fig. 2).

Experimental

A polycrystalline sample of (I) was recrystallized by cooling a saturated 1-methylnaphthalene solution from 313 to 283 K. The sample

Figure 1
The asymmetric unit of (I), with the atom-numbering scheme. The dashed line indicates the N—H···O hydrogen bond between 3-azabicyclo-nonane-2,4-dione molecules. Displacement spheres are shown at the 50% probability level.
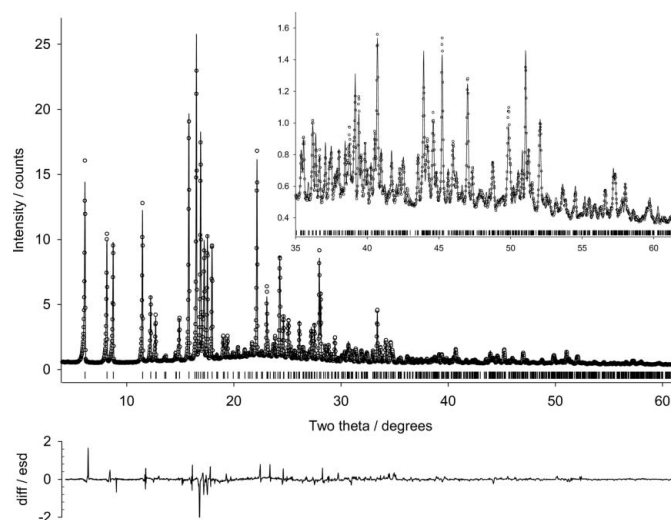

Figure 2
The crystal packing of (I) viewed down the *b* axis and showing the stacks of solvent molecules situated between the pseudo-layers formed by 3-azabicyclononane-2,4-dione molecules. Illustrated using *PLATON* (Spek, 2003).

was loaded into a 0.7 mm borosilicate glass capillary and rotated throughout the data collection to minimize preferred orientation effects. Data were collected using a variable count time (VCT) scheme in which the step time is increased with 2θ (Shankland *et al.*, 1997; Hill & Madsen, 2002).

Crystal data

$C_8H_{11}NO_2 \cdot 0.5C_{11}H_{10}$
$M_r = 224.28$
Monoclinic, $P2_1/c$
$a = 15.02360\ (18)$ Å
$b = 7.32295\ (12)$ Å
$c = 22.5164\ (3)$ Å
$\beta = 106.0201\ (6)°$
$V = 2380.99\ (6)$ Å^3
$Z = 8$

$D_x = 1.251$ Mg m^{-3}
Cu $K\alpha_1$ radiation
$\mu = 0.67$ mm^{-1}
$T = 295$ K
Specimen shape: cylinder
$12 \times 0.7 \times 0.7$ mm
Specimen prepared at 298 K
Particle morphology: needle, white

Figure 3
Final observed (points), calculated (line) and difference $[(y_{obs}\text{-}y_{calc})/\sigma(y_{obs})]$ profiles for the Rietveld refinement of the title compound.

Data collection

Bruker AXS D8 Advance
   diffractometer
Specimen mounting: 0.7 mm
   borosilicate capillary
Specimen mounted in transmission
   mode

Scan method: step
Absorption correction: none
$2\theta_{min} = 4.0$, $2\theta_{max} = 62.5°$
Increment in $2\theta = 0.017°$

Refinement

$R_p = 0.046$
$R_{wp} = 0.057$
$R_{exp} = 0.013$
$R_B = 3.499$
$S = 2.00$
Profile function: Fundamental
   parameters with axial divergence
   correction.
211 parameters

Only H-atom coordinates refined
$w = 1/\sigma(Y_{obs})^2$
$(\Delta/\sigma)_{max} = 0.01$
Preferred orientation correction:
   A spherical harmonics-based
   preferred orientation correction
   (Järvinen, 1993) was applied with
   TOPAS (Coelho, 2003) during
   the Rietveld refinement

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H11···O3	0.900 (5)	1.966 (6)	2.865 (2)	175.8 (4)
N2—H22···O2^i	0.901 (5)	1.921 (10)	2.812 (8)	170.1 (5)

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

The diffraction pattern indexed to a monoclinic cell [$M(20) = 44.5$, $F(20) = 146.5$; *DICVOL91*; Boultif & Louer, 1991] and the space group $P2_1/c$ was assigned from volume considerations and a statistical consideration of the systematic absences (Markvardsen *et al.*, 2001). The data set was background subtracted and truncated to 51.8° 2θ for Pawley fitting (Pawley, 1981; $\chi^2_{Pawley} = 15.77$) and the structure solved using the simulated annealing (SA) global optimization procedure, described previously (David *et al.*, 1998), that is now implemented in the *DASH* computer program (David *et al.*, 2001). The SA structure solution used 453 reflections and involved the optimization of three fragments totaling 18 degrees of freedom (six positional and orientational for each fragment present in the asymmetric unit). All

degrees of freedom were assigned random values at the start of the simulated annealing. The best SA solution had a favourable $\chi^2_{SA}/\chi^2_{Pawley}$ ratio of 3.47 and a chemically reasonable lattice packing arrangement, with no significant misfit to the diffraction data.

The solved structure was then refined against the data in the range 4–61.4° 2θ using a restrained Rietveld (1969) method as implemented in *TOPAS* (Coelho, 2003), with R_{wp} falling to 0.0566 during the refinement. All atomic positions (including H atoms) for the structure of (I) were refined, subject to a series of restraints on bond lengths, bond angles and planarity.

The restraints were set such that bonds and angles did not deviate more than 0.01 Å and 0.8°, respectively, from their initial values during the refinement. Atoms C12, C13, C15, C16, O1, N1, O2 and H1 (first molecule) and atoms C20, C21, C23, C24, O3, N2, O4 and H22 (second molecule) of 3-azabicyclo[3.3.1]nonane-2,4-dione were restrained to be coplanar. Restraints were also applied to the methylnaphthalene molecule, for planarity purposes. A spherical harmonics (4th order) correction of intensities for preferred orientation was applied in the final refinement (Järvinen, 1993). The observed and calculated diffraction patterns for the refined crystal structure are shown in Fig. 3. $U_{iso}(H)$ values were fixed at 0.076 Å^2.

Data collection: *DIFFRAC plus XRD Commander* (Kienle & Jacob, 2003); cell refinement: *TOPAS* (Coelho, 2003); data reduction: *DASH* (David *et al.*, 2001); program(s) used to solve structure: *DASH*; program(s) used to refine structure: *TOPAS*; molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *PLATON*, *enCIFer* (Allen *et al.*, 2004) and *SHELXL97* (Sheldrick, 1997).

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supporting information

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Crystal data

$C_8H_{11}NO_2 \cdot 0.5C_{11}H_{10}$	$Z = 8$
$M_r = 224.28$	$F(000) = 960$
Monoclinic, $P2_1/c$	$D_x = 1.251$ Mg m^{-3}
Hall symbol: -P 2ybc	Cu $K\alpha_1$ radiation, $\lambda = 1.54056$ Å
$a = 15.02360$ (18) Å	$\mu = 0.67$ mm^{-1}
$b = 7.32295$ (12) Å	$T = 295$ K
$c = 22.5164$ (3) Å	white
$\beta = 106.0201$ (6)°	cylinder, 12×0.7 mm
$V = 2380.99$ (6) Å^3	Specimen preparation: Prepared at 298 K

Data collection

Bruker AXS D8 Advance diffractometer	Specimen mounting: 0.7 mm borosilicate capillary
Radiation source: sealed X-ray tube, Bruker-AXS D8	Data collection mode: transmission
Primary focussing, Ge 111 monochromator	Scan method: step
	$2\theta_{min} = 4.0°$, $2\theta_{max} = 62.5°$, $2\theta_{step} = 0.017°$

Refinement

Least-squares matrix: selected elements only	1 constraint
$R_p = 0.046$	Only H-atom coordinates refined
$R_{wp} = 0.057$	Weighting scheme based on measured s.u.'s $1/\sigma(Y_{obs})^2$
$R_{exp} = 0.013$	$(\Delta/\sigma)_{max} = 0.01$
$R_{Bragg} = 3.499$	Background function: Chebyshev polynomial
3387 data points	Preferred orientation correction: A spherical harmonics-based preferred orientation correction (Järvinen, 1993) was applied with Topas during the Rietveld refinement.
Profile function: Fundamental parameters with axial divergence correction.	
211 parameters	
201 restraints	

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.54315 (6)	0.4443 (3)	0.76875 (4)	0.0321 (8)*
C2	0.51206 (6)	0.4415 (5)	0.70448 (4)	0.0321 (8)*
C3	0.57567 (6)	0.4424 (5)	0.66958 (4)	0.0321 (8)*
C4	0.67010 (6)	0.4485 (4)	0.69889 (4)	0.0321 (8)*
C5	0.70105 (6)	0.4487 (4)	0.76294 (4)	0.0321 (8)*
C6	0.63774 (5)	0.4471 (4)	0.79780 (4)	0.0321 (8)*
C7	0.54443 (6)	0.4412 (4)	0.60537 (4)	0.0321 (8)*
C8	0.45010 (6)	0.4434 (4)	0.57598 (4)	0.0321 (8)*
C9	0.38669 (6)	0.4450 (4)	0.61066 (4)	0.0321 (8)*
C10	0.41743 (6)	0.4445 (4)	0.67475 (4)	0.0321 (8)*
H1	0.6592 (2)	0.4473 (9)	0.84165 (15)	0.0760*
H2	0.7135 (2)	0.4490 (9)	0.67509 (16)	0.0760*
H3	0.7657 (2)	0.4497 (9)	0.78299 (16)	0.0760*
H4	0.5877 (2)	0.4411 (9)	0.58152 (16)	0.0760*
H5	0.4290 (2)	0.4438 (9)	0.53217 (15)	0.0760*
H6	0.3222 (2)	0.4450 (9)	0.59045 (17)	0.0760*
H7	0.3738 (2)	0.4450 (9)	0.69837 (18)	0.0760*
C11	0.47485 (6)	0.44543 (14)	0.80686 (4)	0.0321 (8)*
H8	0.5076 (2)	0.4393 (8)	0.84951 (16)	0.0760*
H9	0.4346 (2)	0.3432 (7)	0.79622 (16)	0.0760*
H10	0.4393 (3)	0.5547 (7)	0.79910 (17)	0.0760*
N1	0.17867 (8)	0.20902 (12)	0.47851 (5)	0.0321 (8)*
H11	0.1574 (2)	0.3045 (8)	0.49575 (17)	0.0760*
C12	0.22707 (9)	0.2494 (13)	0.43608 (5)	0.0321 (8)*
O1	0.23991 (19)	0.4086 (2)	0.42479 (10)	0.0321 (8)*
C13	0.26293 (6)	0.09128 (13)	0.40697 (5)	0.0321 (8)*
H12	0.2652 (2)	0.1316 (8)	0.36728 (16)	0.0760*
C14	0.20052 (6)	−0.07379 (13)	0.40282 (5)	0.0321 (8)*
H13	0.2243 (2)	−0.1756 (8)	0.38588 (17)	0.0760*
H14	0.1404 (2)	−0.051 (8)	0.3763 (16)	0.0760*
C15	0.19727 (6)	−0.12209 (13)	0.46805 (4)	0.0321 (8)*
H15	0.156 (2)	−0.2206 (8)	0.46813 (17)	0.0760*
C16	0.15968 (9)	0.03559 (14)	0.49644 (5)	0.0321 (8)*
O2	0.1167 (12)	0.0194 (3)	0.53435 (8)	0.0321 (8)*
C17	0.29412 (6)	−0.17195 (12)	0.50824 (4)	0.0321 (8)*
H16	0.3104 (2)	−0.2788 (8)	0.48964 (17)	0.0760*
H17	0.288 (2)	−0.2007 (8)	0.54817 (18)	0.0760*
C18	0.36461 (6)	−0.02279 (13)	0.50985 (4)	0.0321 (8)*
H18	0.3513 (2)	0.0791 (8)	0.53214 (15)	0.0760*
H19	0.4252 (2)	−0.0652 (8)	0.53002 (16)	0.0760*
C19	0.36203 (6)	0.04481 (13)	0.44539 (4)	0.0321 (8)*
H20	0.3999 (2)	0.1491 (8)	0.44618 (17)	0.0760*
H21	0.3852 (2)	−0.0438 (9)	0.42271 (16)	0.0760*
N2	0.06436 (9)	0.71279 (13)	0.59201 (4)	0.0321 (8)*
H22	0.0757 (3)	0.8074 (8)	0.56961 (17)	0.0760*

C20	0.08087 (10)	0.53992 (14)	0.57266 (5)	0.0321 (8)*
O3	0.11144 (19)	0.5238 (3)	0.52816 (7)	0.0321 (8)*
C21	0.05876 (7)	0.38030 (13)	0.60806 (4)	0.0321 (8)*
H23	0.0424 (3)	0.2824 (8)	0.57937 (15)	0.0760*
C22	−0.01946 (6)	0.42824 (13)	0.63579 (4)	0.0321 (8)*
H24	−0.0324 (2)	0.3294 (7)	0.65950 (16)	0.0760*
H25	−0.0752 (2)	0.4521 (8)	0.60452 (15)	0.0760*
C23	0.01063 (7)	0.59259 (13)	0.67805 (4)	0.0321 (8)*
H26	−0.0373 (3)	0.6329 (8)	0.69504 (16)	0.0760*
C24	0.03020 (10)	0.75294 (13)	0.64179 (5)	0.0321 (8)*
O4	0.0208 (2)	0.9113 (2)	0.65479 (10)	0.0321 (8)*
C25	0.09695 (6)	0.54694 (13)	0.73074 (4)	0.0321 (8)*
H27	0.0770 (2)	0.4561 (7)	0.75432 (17)	0.0760*
H28	0.1135 (2)	0.6545 (7)	0.75490 (17)	0.0760*
C26	0.17607 (6)	0.47383 (13)	0.70790 (4)	0.0321 (8)*
H29	0.2010 (2)	0.5698 (8)	0.68908 (16)	0.0760*
H30	0.2239 (2)	0.4276 (8)	0.74153 (16)	0.0760*
C27	0.14519 (7)	0.32465 (12)	0.65954 (4)	0.0321 (8)*
H31	0.1923 (2)	0.2904 (8)	0.64094 (17)	0.0760*
H32	0.1301 (2)	0.2148 (8)	0.67711 (17)	0.0760*

Geometric parameters (Å, º)

O1—C12	1.220 (9)	C25—C26	1.5171 (13)
O2—C16	1.210 (13)	C26—C27	1.5217 (13)
O3—C20	1.218 (2)	C21—H23	0.951 (5)
O4—C24	1.2138 (19)	C22—H24	0.951 (5)
N1—C16	1.3860 (14)	C22—H25	0.950 (3)
N1—C12	1.383 (3)	C23—H26	0.951 (5)
N1—H11	0.900 (5)	C25—H27	0.950 (4)
N2—C24	1.3870 (17)	C25—H28	0.951 (5)
N2—C20	1.3833 (15)	C26—H29	0.949 (5)
N2—H22	0.901 (5)	C26—H30	0.951 (4)
C12—C13	1.502 (7)	C27—H31	0.950 (4)
C13—C14	1.5168 (13)	C27—H32	0.951 (5)
C13—C19	1.5395 (14)	C1—C2	1.3927 (12)
C14—C15	1.5246 (14)	C1—C6	1.3909 (12)
C15—C17	1.5306 (13)	C1—C11	1.5084 (13)
C15—C16	1.5034 (15)	C2—C3	1.3948 (13)
C17—C18	1.5148 (13)	C2—C10	1.3945 (13)
C18—C19	1.5238 (12)	C3—C4	1.3905 (13)
C13—H12	0.951 (4)	C3—C7	1.3914 (12)
C14—H14	0.95 (3)	C4—C5	1.3878 (12)
C14—H13	0.951 (5)	C5—C6	1.3903 (12)
C15—H15	0.95 (2)	C7—C8	1.3889 (13)
C17—H16	0.951 (5)	C8—C9	1.3885 (13)
C17—H17	0.952 (11)	C9—C10	1.3885 (12)
C18—H19	0.950 (4)	C4—H2	0.951 (3)

C18—H18	0.951 (5)	C5—H3	0.951 (3)
C19—H21	0.949 (5)	C6—H1	0.950 (3)
C19—H20	0.950 (5)	C7—H4	0.951 (3)
C20—C21	1.5027 (15)	C8—H5	0.949 (3)
C21—C27	1.5379 (14)	C9—H6	0.950 (3)
C21—C22	1.5163 (14)	C10—H7	0.951 (4)
C22—C23	1.5234 (13)	C11—H8	0.952 (4)
C23—C24	1.5048 (14)	C11—H9	0.951 (5)
C23—C25	1.5336 (13)	C11—H10	0.951 (5)
C12—N1—C16	125.9 (4)	C21—C27—C26	112.11 (8)
C12—N1—H11	116.7 (5)	C20—C21—H23	106.2 (3)
C16—N1—H11	117.4 (3)	C22—C21—H23	111.7 (3)
C20—N2—C24	125.95 (10)	C27—C21—H23	108.6 (3)
C24—N2—H22	117.3 (3)	C21—C22—H24	110.9 (2)
C20—N2—H22	116.7 (3)	C21—C22—H25	111.2 (2)
O1—C12—N1	119.5 (5)	C23—C22—H24	108.8 (3)
N1—C12—C13	117.2 (6)	C23—C22—H25	111.5 (3)
O1—C12—C13	123.33 (18)	H24—C22—H25	106.3 (4)
C12—C13—C19	109.45 (9)	C22—C23—H26	111.7 (3)
C14—C13—C19	110.12 (8)	C24—C23—H26	105.0 (3)
C12—C13—C14	110.6 (2)	C25—C23—H26	109.2 (2)
C13—C14—C15	108.01 (8)	C23—C25—H27	104.8 (2)
C14—C15—C17	110.30 (8)	C23—C25—H28	107.1 (2)
C14—C15—C16	110.31 (8)	C26—C25—H27	109.9 (3)
C16—C15—C17	109.83 (8)	C26—C25—H28	112.4 (2)
O2—C16—C15	124.12 (16)	H27—C25—H28	109.5 (4)
N1—C16—C15	116.63 (10)	C25—C26—H29	109.4 (3)
O2—C16—N1	119.22 (16)	C25—C26—H30	110.3 (2)
C15—C17—C18	112.73 (7)	C27—C26—H29	107.0 (3)
C17—C18—C19	112.19 (7)	C27—C26—H30	109.2 (3)
C13—C19—C18	111.93 (8)	H29—C26—H30	108.3 (4)
C12—C13—H12	106.3 (4)	C21—C27—H31	108.3 (2)
C14—C13—H12	111.8 (3)	C21—C27—H32	106.8 (2)
C19—C13—H12	108.5 (2)	C26—C27—H31	112.8 (3)
C13—C14—H13	110.8 (3)	C26—C27—H32	111.7 (3)
H13—C14—H14	106 (3)	H31—C27—H32	104.6 (4)
C13—C14—H14	111 (3)	C2—C1—C6	119.67 (8)
C15—C14—H13	108.7 (3)	C2—C1—C11	120.36 (8)
C15—C14—H14	111.6 (18)	C6—C1—C11	119.97 (8)
C14—C15—H15	111.8 (3)	C1—C2—C3	119.99 (8)
C16—C15—H15	105.1 (13)	C1—C2—C10	120.25 (8)
C17—C15—H15	109.4 (11)	C3—C2—C10	119.72 (8)
C18—C17—H17	112.7 (11)	C2—C3—C4	120.05 (8)
H16—C17—H17	109.7 (7)	C2—C3—C7	119.89 (8)
C15—C17—H16	104.7 (2)	C4—C3—C7	120.05 (8)
C15—C17—H17	106.7 (17)	C3—C4—C5	119.91 (8)
C18—C17—H16	109.9 (3)	C4—C5—C6	120.10 (8)

C17—C18—H19	110.3 (3)	C1—C6—C5	120.26 (8)
H18—C18—H19	108.5 (4)	C3—C7—C8	120.15 (8)
C19—C18—H18	107.1 (3)	C7—C8—C9	120.02 (8)
C17—C18—H18	109.3 (3)	C8—C9—C10	120.09 (8)
C19—C18—H19	109.3 (2)	C2—C10—C9	120.11 (8)
C18—C19—H21	111.9 (3)	C3—C4—H2	120.1 (2)
H20—C19—H21	104.7 (4)	C5—C4—H2	120.0 (2)
C18—C19—H20	112.7 (2)	C4—C5—H3	119.9 (2)
C13—C19—H20	108.3 (3)	C6—C5—H3	120.0 (2)
C13—C19—H21	106.9 (2)	C1—C6—H1	119.9 (2)
O3—C20—N2	119.28 (14)	C5—C6—H1	119.9 (2)
N2—C20—C21	117.37 (10)	C3—C7—H4	120.0 (2)
O3—C20—C21	123.36 (14)	C8—C7—H4	119.9 (2)
C20—C21—C27	109.98 (9)	C7—C8—H5	120.0 (2)
C22—C21—C27	109.88 (7)	C9—C8—H5	120.0 (2)
C20—C21—C22	110.38 (9)	C8—C9—H6	119.9 (2)
C21—C22—C23	108.06 (8)	C10—C9—H6	120.0 (2)
C22—C23—C25	110.66 (8)	C2—C10—H7	120.0 (2)
C22—C23—C24	110.26 (8)	C9—C10—H7	119.9 (2)
C24—C23—C25	109.86 (9)	C1—C11—H8	109.3 (2)
O4—C24—C23	124.16 (15)	C1—C11—H9	109.7 (2)
N2—C24—C23	116.38 (9)	C1—C11—H10	109.6 (3)
O4—C24—N2	119.41 (15)	H8—C11—H9	109.5 (4)
C23—C25—C26	112.90 (7)	H8—C11—H10	109.4 (4)
C25—C26—C27	112.54 (8)	H9—C11—H10	109.3 (4)
C16—N1—C12—O1	−179.26 (18)	C22—C21—C27—C26	−57.27 (10)
C16—N1—C12—C13	−0.40 (18)	C21—C22—C23—C24	60.46 (11)
C12—N1—C16—O2	179.8 (7)	C21—C22—C23—C25	−61.29 (10)
C12—N1—C16—C15	1.51 (18)	C22—C23—C24—N2	−31.85 (15)
C20—N2—C24—O4	178.34 (19)	C25—C23—C24—O4	−87.1 (2)
C24—N2—C20—C21	0.9 (2)	C22—C23—C25—C26	54.33 (10)
C24—N2—C20—O3	−179.72 (18)	C24—C23—C25—C26	−67.65 (10)
C20—N2—C24—C23	0.7 (2)	C25—C23—C24—N2	90.38 (12)
O1—C12—C13—C14	−151.34 (18)	C22—C23—C24—O4	150.67 (19)
N1—C12—C13—C19	−91.62 (15)	C23—C25—C26—C27	−47.76 (10)
N1—C12—C13—C14	29.85 (14)	C25—C26—C27—C21	49.14 (10)
O1—C12—C13—C19	87.2 (2)	C6—C1—C2—C3	−0.3 (5)
C12—C13—C14—C15	−58.83 (16)	C6—C1—C2—C10	−178.0 (3)
C12—C13—C19—C18	64.9 (3)	C11—C1—C2—C3	179.3 (3)
C19—C13—C14—C15	62.25 (10)	C11—C1—C2—C10	1.7 (4)
C14—C13—C19—C18	−56.88 (11)	C2—C1—C6—C5	0.6 (4)
C13—C14—C15—C16	59.90 (10)	C11—C1—C6—C5	−179.1 (2)
C13—C14—C15—C17	−61.60 (10)	C1—C2—C3—C4	−0.8 (5)
C17—C15—C16—O2	−88.1 (7)	C1—C2—C3—C7	−179.4 (3)
C16—C15—C17—C18	−66.00 (10)	C10—C2—C3—C4	176.8 (3)
C14—C15—C17—C18	55.79 (10)	C10—C2—C3—C7	−1.8 (5)
C14—C15—C16—N1	−31.77 (13)	C1—C2—C10—C9	178.9 (3)

C14—C15—C16—O2	150.1 (7)	C3—C2—C10—C9	1.2 (5)
C17—C15—C16—N1	90.02 (11)	C2—C3—C4—C5	1.7 (5)
C15—C17—C18—C19	−49.32 (10)	C7—C3—C4—C5	−179.7 (3)
C17—C18—C19—C13	49.63 (10)	C2—C3—C7—C8	1.5 (5)
O3—C20—C21—C22	−150.43 (18)	C4—C3—C7—C8	−177.1 (3)
N2—C20—C21—C27	−92.44 (13)	C3—C4—C5—C6	−1.5 (5)
N2—C20—C21—C22	28.97 (14)	C4—C5—C6—C1	0.4 (4)
O3—C20—C21—C27	88.17 (19)	C3—C7—C8—C9	−0.6 (5)
C27—C21—C22—C23	62.69 (9)	C7—C8—C9—C10	0.0 (4)
C20—C21—C22—C23	−58.77 (10)	C8—C9—C10—C2	−0.3 (5)
C20—C21—C27—C26	64.43 (11)		

Hydrogen-bond geometry (Å, º)

D—H···A	D—H	H···A	D···A	D—H···A
N1—H11···O3	0.90 (1)	1.97 (1)	2.865 (2)	176 (1)
N2—H22···O2[i]	0.90 (1)	1.92 (1)	2.812 (8)	170 (1)

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