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(1*RS*,2*SR*,7*RS*,8*RS*)-*N*-Benzoyltricyclo[6.2.2.0^{2,7}]-dodeca-9,11-diene-1,10-dicarboximide

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.005 Å
R factor = 0.088
wR factor = 0.192
Data-to-parameter ratio = 17.2

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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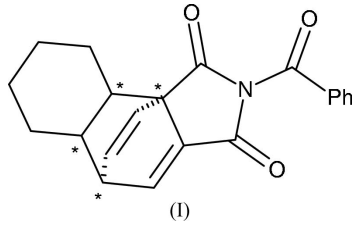
The title 1,4-photoadduct, $C_{21}H_{19}NO_3$, was formed on irradiation of *N*-benzoylphthalimide in dichloromethane containing cyclohexene. The bond lengths and angles are generally within the normal ranges. A notable feature of the molecule is the presence within it of four contiguous chiral centres.

Comment

The photochemistry of phthalimides has been studied extensively and has been reviewed by Kanaoka (1978), Coyle (1984) and Oelgemöller & Griesbeck (2002). Schwack (1987) has reported the photo-induced *para*-cycloaddition of cyclohexene to *N*-trichloromethylthio-, *N*-methyl- and *N*-phenylphthalimides. Suau *et al.* (1989) have reported the *ortho*- and *para*-photocycloaddition of 3-methoxy-*N*-methylphthalimide to 1-hexene and Kubo *et al.* (1989) have reported analogous *ortho*- and *para*-cycloadditions of *N*-methylphthalimide to allyltrimethylsilane. In each case, the *para*-cycloaddition products are structurally analogous to the title compound, (I). However, the structures were only elucidated by spectroscopic means and lack stereochemical certainty. The determination of the structure of (I) presented here was undertaken in the context of a study of the photochemistry of *N*-benzoylphthalimide but is clearly of significance in relation to the analogous compounds.



(I)

The molecule of (I) is shown in Fig. 1. Selected bond lengths and angles are given in Table 1. The bond lengths, along with those of the phenyl group *R*1 defined by C16–C21 in the range 1.361 (6)–1.389 (5) Å, are not unusual excepting, perhaps, the C2—C3 and C6—C7 bond lengths of 1.493 (5) and 1.481 (5) Å, respectively. Likewise, with the sole exception of the angle C9—C10—C14 of 134.1 (3)°, the bond angles, including the internal angles of the phenyl group in the range 117.9 (3)–121.1 (4)°, are as expected. The cyclohexane ring, *R*3, defined by C2–C7, adopts the chair conformation, with puckering parameters (Cremer & Pople, 1975) Q = 0.564 (4) Å, θ = 168.5 (5) and φ = 151 (2)°. The dihedral angle between the least-squares planes of phenyl group *R*1 (r.m.s. displacement = 0.0006 Å) and five-membered ring *R*2, defined by C1/C10/C13–C14/N1 (r.m.s. displacement = 0.0143 Å) is 61.97 (15)°. Atom O3 is displaced from the least-squares

planes of *R*1 and *R*2 by 0.145 (7) and 1.134 (6) Å, respectively. The packing of the molecules of (I) creates layers parallel to ($\bar{1}02$) (Fig. 2) in such a way as to generate the first two C—H···π interactions given in Table 2 (shown as dashed lines in Fig. 2). The only contact between the layers, other than van der Waals interactions, is the third, longer, C—H···π contact given in Table 2.

The racemic nature of (I), a prerequisite for the refinement of the structure in the centrosymmetric space group $P2_1/c$, is a natural consequence of the manner in which the compound has been formed from achiral reactants. In principle, given that the unsymmetrical 1,4-addition across the aromatic ring must of necessity be *cis*, there are four possible racemic products, two involving *trans* ring junctions at C2—C7 and two involving *cis* junctions at C2—C7. Formation of the single unsymmetrical product, (I), suggests a favoured approach by the cyclohexene to the excited phthalimide, possibly involving minimization of steric interactions between the *N*-benzoylimide and cyclohexene rings in the transition state. The stereochemistry at the C2—C7 ring junction is the outcome of overall *trans* addition across the cyclohexene double bond.

Experimental

Compound (I) was one of the products of irradiation for 40 h of *N*-benzoylphthalimide (2.90 g, 11.5 mmol) and cyclohexene (19.60 g, 239.0 mmol) in dichloromethane (300 ml) by a 400 W medium-pressure mercury vapour lamp fitted with a Pyrex filter. After removal of solvents under vacuum three products (previously detected by thin-layer chromatography) were isolated by means of a Chromatotron and a 4 mm silica plate with a mixture of dichloromethane and light petroleum (b.p. 313–333 K) (2:98 increased stepwise to 60:40) as eluant to yield: (i) recovered *N*-benzoylphthalimide (2.75 g); (ii) a mixture of minor products as a colourless oil (12 mg); (iii) compound (I), a white crystalline solid [160 mg, 80%; m.p. 411–413 K (from chloroform/light petroleum, b.p. 363–373 K)], λ_{max} (MeCN): 251 (ε 20,208 dm^3 mol^{-1} cm^{-1}); ν_{max} 2929 (aliphatic CH), 1717 and 1694 (C═O), 1297 and 1252 cm^{-1}; δ_H (270 MHz, $CDCl_3$): 7.89–7.47 (5H, *m*, ArH), 7.10 (1H, *d, J* 6.0 Hz, vinylic H), 6.82 (1*H*, *d* of *d*, *J* 6.0 Hz, *J* 7.0 Hz, vinylic H), 6.16 (1*H*, *d*, *J* 7.0 Hz, vinylic H), 3.76 (1H, *t, J* 6.0 Hz), 2.10–1.11 (10H, *m*, cyclohexane derived moiety); δ_C (67.8 MHz, $CDCl_3$): 173.0, 167.1, 162.3 (carbonyl C), 143.3, 141.7, 137.3, 134.8, 131.9, 130.4, 128.8, 123.5 (aromatic and vinylic C), 56.2, 52.5, 50.7, 45.7, 32.7, 30.2, 27.7 and 27.4 (aliphatic C); analysis found: C 75.3, H 5.8, N 3.9%; $C_{21}H_{19}NO_3$ requires: C 75.7, H 5.8, N 4.2%; m/e: 333 (1), 265 (47), 264 (31), 252 (56), 105 (100), 77 (63) and 67 (45%).

Crystal data

$C_{21}H_{19}NO_3$
M_r = 333.37
Monoclinic, $P2_1/c$
a = 8.111 (3) Å
b = 12.999 (7) Å
c = 16.256 (5) Å
β = 100.76 (3)°
V = 1683.8 (12) Å^3
Z = 4
D_x = 1.315 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 14 reflections
θ = 11.0–13.0°
μ = 0.09 mm^{-1}
T = 298 (2) K
Block, colourless
0.60 × 0.40 × 0.26 mm

Data collection

Nicolet *P*3 four-circle diffractometer
θ–2θ scans
Absorption correction: none
3882 measured reflections
3882 independent reflections
1880 reflections with $I > 2\sigma(I)$
θ_{max} = 30.1°
$h = 0 \rightarrow 11$
$k = 0 \rightarrow 18$
$l = -22 \rightarrow 22$
2 standard reflections
every 50 reflections
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.088
$wR(F^2)$ = 0.192
S = 1.03
3882 reflections
226 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0735P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—C13	1.408 (4)	C2—C3	1.493 (5)
N1—C14	1.425 (4)	C2—C7	1.514 (5)
N1—C15	1.444 (4)	C6—C7	1.481 (5)
O1—C13	1.203 (4)	C7—C8	1.584 (5)
O2—C14	1.200 (4)	C8—C9	1.506 (5)
O3—C15	1.194 (4)	C8—C12	1.519 (5)
C1—C11	1.500 (5)	C9—C10	1.330 (4)
C1—C13	1.504 (5)	C10—C14	1.462 (5)
C1—C10	1.504 (4)	C11—C12	1.323 (5)
C1—C2	1.571 (4)	C15—C16	1.473 (5)
C13—N1—C14	113.0 (3)	C12—C8—C7	100.8 (3)
C13—N1—C15	121.3 (3)	C10—C9—C8	112.1 (3)
C14—N1—C15	125.0 (3)	C9—C10—C14	134.1 (3)
C11—C1—C13	118.0 (3)	C9—C10—C1	115.4 (3)
C11—C1—C10	108.2 (3)	C14—C10—C1	110.3 (3)
C13—C1—C10	103.4 (3)	C12—C11—C1	113.2 (3)
C11—C1—C2	109.4 (3)	C11—C12—C8	114.5 (3)
C13—C1—C2	114.3 (3)	O1—C13—N1	124.0 (3)
C10—C1—C2	102.0 (3)	O1—C13—C1	127.9 (3)
C3—C2—C7	110.4 (3)	N1—C13—C1	108.1 (3)
C3—C2—C1	124.6 (3)	O2—C14—N1	123.9 (3)
C7—C2—C1	107.4 (3)	O2—C14—C10	130.9 (3)
C6—C7—C2	110.8 (3)	N1—C14—C10	105.1 (3)
C6—C7—C8	122.4 (3)	O3—C15—N1	118.4 (3)
C2—C7—C8	109.0 (3)	O3—C15—C16	123.8 (3)
C9—C8—C12	108.3 (3)	N1—C15—C16	117.7 (3)
C9—C8—C7	109.1 (3)		
C11—C1—C2—C3	−86.5 (4)	C6—C7—C8—C9	−87.4 (4)
C13—C1—C2—C3	48.3 (5)	C2—C7—C8—C9	44.1 (4)
C10—C1—C2—C3	159.1 (4)	C6—C7—C8—C12	158.8 (4)
C11—C1—C2—C7	44.9 (4)	C2—C7—C8—C12	−69.7 (4)
C13—C1—C2—C7	179.7 (3)	C8—C9—C10—C14	178.1 (3)
C10—C1—C2—C7	−69.5 (3)	C8—C9—C10—C1	3.3 (4)
C1—C2—C3—C4	−171.5 (3)	C13—C1—C10—C9	179.5 (3)
C5—C6—C7—C8	−170.5 (4)	C13—C1—C10—C14	3.4 (3)
C3—C2—C7—C6	−65.8 (4)	C1—C11—C12—C8	3.4 (4)
C1—C2—C7—C6	155.5 (3)	C9—C8—C12—C11	−54.9 (4)
C3—C2—C7—C8	156.7 (3)	C7—C8—C12—C11	59.5 (4)
C1—C2—C7—C8	18.0 (4)		

Table 2
Geometry (Å,°) of C—H···π contacts in (I).

C—H···*Cg*[a]	C—H	H···Cg	H_{perp}[b]	γ[c]	C—H···*Cg*	C···*Cg*
C6—H6*A*···*Cg*1^{i}	0.97	2.80	2.69	16	149	3.68
C6—H6*B*···*Cg*1^{ii}	0.97	3.03	2.93	15	135	3.79
C4—H4*B*···*Cg*2^{iii}	0.97	3.34	3.28	11	120	3.92

Notes: (*a*) *Cg*1 and *Cg*2 are the centroids of the rings defined by C16–C21 and C1/C10/C13–C14/N1, respectively; (*b*) H_{perp} is the perpendicular distance of the H atom from the mean plane of the ring; (*c*) γ is the angle at hydrogen between H_{perp} and H···*Cg*. Symmetry codes (i) $1-x, 1-y, 1-z$; (ii) $1+x, \frac{3}{2}-y, \frac{1}{2}+z$; (iii) $1+x, y, z$.

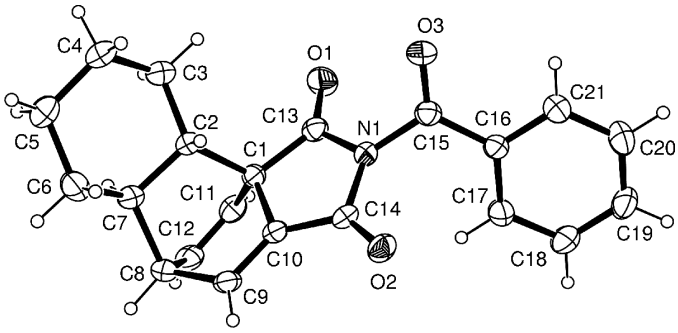


Figure 1
A view of (I). Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small circles of arbitrary radii.

The incompleteness (84.9% complete for $\theta_{full} = 25^\circ$) of the mid-1980s data set upon which this refinement is based is due to the suppression, during data reduction and contrary to current practice, of reflections with intensities measured as negative. As a consequence, the omissions are scattered throughout the data set although they are more prevalent at high θ. In the final stages of refinement, H atoms were introduced in calculated positions with C—H set at 0.93, 0.97 and 0.98 Å for aryl/alkene, methylene and tertiary H atoms, respectively, and refined with a riding model, with $U_{iso}(H) = 1.2U_{eq}(C)$ in all cases.

Data collection: *Nicolet P3 Software* (Nicolet, 1980); cell refinement: *Nicolet P3 Software*; data reduction: *RDNIC* (Howie, 1980); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2003).

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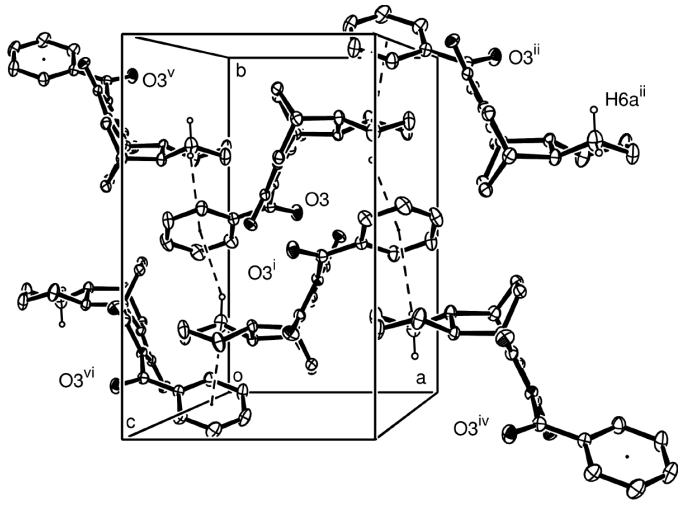

Figure 2
A layer of molecules of (I). Displacement ellipsoids are drawn at the 20% probability level and H atoms involved in C—H..π contacts (dashed lines) are shown as small circles of arbitrary radii. [Symmetry codes (i) $1-x$, $1-y$, $1-z$; (ii) $1+x$, $\frac{3}{2}-y$, $\frac{1}{2}+z$; (iv) $2-x$, $y-\frac{1}{2}$, $\frac{3}{2}-z$; (v) $x-1$, $\frac{3}{2}-y$, $z-\frac{1}{2}$; (vi) $-x$, $y-\frac{1}{2}$, $\frac{1}{2}-z$.]

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supporting information

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(1*RS*,2*SR*,7*RS*,8*RS*)-*N*-Benzoyltricyclo[6.2.2.0^{2,7}]dodeca-9,11-diene-1,10-dicarboximide

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(1RS,2SR,7RS,8RS)-*N*-Benzoyltricyclo[6.2.2.0^{2,7}]dodeca-9,11-diene-1,10- dicarboximide

Crystal data

$C_{21}H_{19}NO_3$
M_r = 333.37
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 8.111 (3) Å
b = 12.999 (7) Å
c = 16.256 (5) Å
β = 100.76 (3)°
V = 1683.8 (12) Å^3
Z = 4
$F(000)$ = 704
D_x = 1.315 Mg m^{-3}
Melting point = 411–413 K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 14 reflections
θ = 11.0–13.0°
μ = 0.09 mm^{-1}
T = 298 K
Block, colourless
0.60 × 0.40 × 0.26 mm

Data collection

Nicolet P3 four-circle diffractometer
Radiation source: normal-focus sealed tube
Graphite monochromator
θ–2θ scans
3882 measured reflections
3882 independent reflections
1880 reflections with $I > 2\sigma(I)$
R_{int} = 0.000
θ_{max} = 30.1°, θ_{min} = 2.0°
h = 0→11
k = 0→18
l = −22→22
2 standard reflections every 50 reflections
intensity decay: none

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.088
$wR(F^2)$ = 0.192
S = 1.03
3882 reflections
226 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
$w = 1/[\sigma^2(F_o^2) + (0.0735P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

Special details

Experimental. Scan rates, dependent on prescan intensity (Ip), were in the range 58.6 (Ip>2500) to 5.33 (Ip<150) degrees 2-theta per min. Scan widths, dependent on 2-theta, were in the range 2.4 to 2.7 degrees 2-theta. Stationary crystal, stationary counter background counts were taken on either side of the peak each for 25% of the total (peak plus background) count time.

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.
Least-squares planes (*x*,*y*,*z* in crystal coordinates) and deviations from them (* indicates atom used to define plane)
- 7.2306 (0.0071) *x* + 5.8226 (0.0186) *y* + 1.6136 (0.0260) *z* = 1.6557 (0.0174)
* -0.0185 (0.0020) C1 * 0.0197 (0.0020) C10 * 0.0112 (0.0020) C13 * -0.0131 (0.0019) C14 * 0.0007 (0.0020) N1 0.0528 (0.0052) O1 - 0.0531 (0.0047) O2 - 1.1343 (0.0056) O3 - 0.1943 (0.0055) C15
Rms deviation of fitted atoms = 0.0143
- 0.6803 (0.0143) *x* + 10.2618 (0.0152) *y* - 9.4566 (0.0246) *z* = 2.4682 (0.0136)
Angle to previous plane (with approximate e.s.d.) = 61.97 (0.15)
* -0.0002 (0.0026) C16 * -0.0003 (0.0028) C17 * 0.0002 (0.0031) C18 * 0.0005 (0.0032) C19 * -0.0010 (0.0032) C20 * 0.0009 (0.0029) C21 - 0.0135 (0.0068) N1 0.1449 (0.0070) O3 0.0297 (0.0059) C15
Rms deviation of fitted atoms = 0.0006

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}
N1	0.3644 (3)	0.6274 (2)	0.39508 (16)	0.0392 (7)
O1	0.4530 (3)	0.7657 (2)	0.32558 (15)	0.0603 (8)
O2	0.3050 (3)	0.51618 (19)	0.49727 (15)	0.0500 (7)
O3	0.4287 (3)	0.5436 (2)	0.28269 (16)	0.0619 (8)
C1	0.4954 (4)	0.7640 (3)	0.47789 (19)	0.0373 (8)
C2	0.6906 (4)	0.7617 (3)	0.5097 (2)	0.0414 (8)
H2	0.7172	0.6884	0.5171	0.050*
C3	0.8167 (5)	0.8014 (4)	0.4613 (2)	0.0713 (13)
H3A	0.8087	0.8757	0.4567	0.086*
H3B	0.7957	0.7725	0.4053	0.086*
C4	0.9903 (5)	0.7707 (4)	0.5070 (3)	0.0714 (13)
H4A	1.0038	0.6972	0.5005	0.086*
H4B	1.0731	0.8049	0.4807	0.086*
C5	1.0262 (5)	0.7962 (4)	0.5997 (3)	0.0722 (13)
H5A	1.0452	0.8696	0.6063	0.087*
H5B	1.1286	0.7615	0.6257	0.087*
C6	0.8865 (5)	0.7655 (4)	0.6452 (2)	0.0690 (13)
H6A	0.8822	0.6912	0.6498	0.083*
H6B	0.9075	0.7943	0.7012	0.083*
C7	0.7247 (4)	0.8045 (3)	0.5978 (2)	0.0476 (9)
H7	0.7447	0.8779	0.5904	0.057*
C8	0.5568 (4)	0.8011 (3)	0.6343 (2)	0.0489 (10)
H8	0.5752	0.8166	0.6944	0.059*

C9	0.4719 (4)	0.6989 (3)	0.6132 (2)	0.0442 (9)
H9	0.4473	0.6528	0.6530	0.053*
C10	0.4362 (4)	0.6823 (3)	0.5311 (2)	0.0375 (8)
C11	0.4270 (4)	0.8654 (3)	0.4999 (2)	0.0481 (9)
H11	0.3711	0.9111	0.4602	0.058*
C12	0.4543 (4)	0.8834 (3)	0.5814 (2)	0.0523 (10)
H12	0.4139	0.9416	0.6044	0.063*
C13	0.4398 (4)	0.7243 (3)	0.3902 (2)	0.0423 (8)
C14	0.3603 (4)	0.5969 (3)	0.4789 (2)	0.0387 (8)
C15	0.3223 (4)	0.5619 (3)	0.3224 (2)	0.0428 (9)
C16	0.1475 (4)	0.5261 (3)	0.2993 (2)	0.0418 (8)
C17	0.0221 (4)	0.5567 (3)	0.3415 (2)	0.0548 (11)
H17	0.0475	0.6006	0.3873	0.066*
C18	−0.1412 (5)	0.5220 (4)	0.3155 (3)	0.0674 (12)
H18	−0.2250	0.5426	0.3440	0.081*
C19	−0.1789 (5)	0.4577 (4)	0.2485 (3)	0.0709 (13)
H19	−0.2884	0.4346	0.2311	0.085*
C20	−0.0557 (6)	0.4274 (4)	0.2069 (3)	0.0730 (13)
H20	−0.0822	0.3833	0.1612	0.088*
C21	0.1079 (5)	0.4610 (3)	0.2314 (2)	0.0594 (11)
H21	0.1905	0.4399	0.2024	0.071*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0374 (15)	0.0425 (17)	0.0380 (16)	−0.0070 (14)	0.0082 (12)	−0.0007 (14)
O1	0.0765 (19)	0.0623 (18)	0.0420 (15)	−0.0096 (15)	0.0103 (13)	0.0145 (14)
O2	0.0512 (15)	0.0449 (15)	0.0555 (16)	−0.0120 (13)	0.0138 (12)	0.0067 (13)
O3	0.0498 (16)	0.084 (2)	0.0578 (17)	−0.0057 (14)	0.0247 (13)	−0.0184 (15)
C1	0.0352 (18)	0.042 (2)	0.0345 (18)	−0.0005 (16)	0.0055 (14)	0.0031 (16)
C2	0.0350 (18)	0.048 (2)	0.043 (2)	−0.0049 (16)	0.0131 (15)	−0.0030 (17)
C3	0.055 (3)	0.114 (4)	0.050 (2)	−0.020 (3)	0.022 (2)	−0.007 (2)
C4	0.043 (2)	0.102 (4)	0.075 (3)	−0.012 (2)	0.026 (2)	−0.016 (3)
C5	0.038 (2)	0.103 (4)	0.076 (3)	−0.004 (2)	0.011 (2)	−0.013 (3)
C6	0.054 (3)	0.104 (4)	0.047 (2)	0.002 (3)	0.0052 (19)	−0.015 (2)
C7	0.0389 (19)	0.063 (3)	0.043 (2)	−0.0021 (18)	0.0137 (16)	−0.0008 (19)
C8	0.048 (2)	0.060 (3)	0.042 (2)	−0.0055 (19)	0.0191 (17)	−0.0104 (19)
C9	0.042 (2)	0.053 (2)	0.041 (2)	−0.0024 (17)	0.0180 (16)	0.0046 (18)
C10	0.0336 (17)	0.041 (2)	0.0398 (19)	−0.0024 (16)	0.0119 (14)	0.0032 (17)
C11	0.039 (2)	0.043 (2)	0.062 (3)	0.0039 (17)	0.0101 (17)	0.007 (2)
C12	0.045 (2)	0.050 (2)	0.068 (3)	0.0012 (19)	0.0238 (19)	−0.011 (2)
C13	0.0377 (19)	0.049 (2)	0.041 (2)	0.0005 (17)	0.0084 (15)	0.0081 (18)
C14	0.0303 (17)	0.040 (2)	0.048 (2)	−0.0008 (16)	0.0134 (15)	0.0039 (17)
C15	0.044 (2)	0.047 (2)	0.039 (2)	−0.0005 (17)	0.0102 (17)	0.0019 (17)
C16	0.0396 (19)	0.051 (2)	0.0351 (19)	−0.0026 (17)	0.0070 (15)	0.0000 (17)
C17	0.043 (2)	0.072 (3)	0.049 (2)	−0.003 (2)	0.0085 (17)	−0.014 (2)
C18	0.039 (2)	0.100 (4)	0.065 (3)	−0.011 (2)	0.0138 (19)	−0.008 (3)
C19	0.050 (2)	0.099 (4)	0.060 (3)	−0.030 (3)	0.001 (2)	−0.001 (3)

C20	0.075 (3)	0.088 (4)	0.051 (3)	−0.023 (3)	−0.001 (2)	−0.016 (2)
C21	0.064 (3)	0.068 (3)	0.047 (2)	−0.015 (2)	0.0128 (19)	−0.009 (2)

Geometric parameters (Å, º)

N1—C13	1.408 (4)	C6—H6B	0.9700
N1—C14	1.425 (4)	C7—C8	1.584 (5)
N1—C15	1.444 (4)	C7—H7	0.9800
O1—C13	1.203 (4)	C8—C9	1.506 (5)
O2—C14	1.200 (4)	C8—C12	1.519 (5)
O3—C15	1.194 (4)	C8—H8	0.9800
C1—C11	1.500 (5)	C9—C10	1.330 (4)
C1—C13	1.504 (5)	C9—H9	0.9300
C1—C10	1.504 (4)	C10—C14	1.462 (5)
C1—C2	1.571 (4)	C11—C12	1.323 (5)
C2—C3	1.493 (5)	C11—H11	0.9300
C2—C7	1.514 (5)	C12—H12	0.9300
C2—H2	0.9800	C15—C16	1.473 (5)
C3—C4	1.518 (6)	C16—C21	1.380 (5)
C3—H3A	0.9700	C16—C17	1.387 (5)
C3—H3B	0.9700	C17—C18	1.389 (5)
C4—C5	1.516 (6)	C17—H17	0.9300
C4—H4A	0.9700	C18—C19	1.361 (6)
C4—H4B	0.9700	C18—H18	0.9300
C5—C6	1.518 (6)	C19—C20	1.366 (6)
C5—H5A	0.9700	C19—H19	0.9300
C5—H5B	0.9700	C20—C21	1.383 (5)
C6—C7	1.481 (5)	C20—H20	0.9300
C6—H6A	0.9700	C21—H21	0.9300
C13—N1—C14	113.0 (3)	C9—C8—C12	108.3 (3)
C13—N1—C15	121.3 (3)	C9—C8—C7	109.1 (3)
C14—N1—C15	125.0 (3)	C12—C8—C7	100.8 (3)
C11—C1—C13	118.0 (3)	C9—C8—H8	112.7
C11—C1—C10	108.2 (3)	C12—C8—H8	112.7
C13—C1—C10	103.4 (3)	C7—C8—H8	112.7
C11—C1—C2	109.4 (3)	C10—C9—C8	112.1 (3)
C13—C1—C2	114.3 (3)	C10—C9—H9	123.9
C10—C1—C2	102.0 (3)	C8—C9—H9	123.9
C3—C2—C7	110.4 (3)	C9—C10—C14	134.1 (3)
C3—C2—C1	124.6 (3)	C9—C10—C1	115.4 (3)
C7—C2—C1	107.4 (3)	C14—C10—C1	110.3 (3)
C3—C2—H2	104.1	C12—C11—C1	113.2 (3)
C7—C2—H2	104.1	C12—C11—H11	123.4
C1—C2—H2	104.1	C1—C11—H11	123.4
C2—C3—C4	108.4 (3)	C11—C12—C8	114.5 (3)
C2—C3—H3A	110.0	C11—C12—H12	122.8
C4—C3—H3A	110.0	C8—C12—H12	122.8

C2—C3—H3B	110.0	O1—C13—N1	124.0 (3)
C4—C3—H3B	110.0	O1—C13—C1	127.9 (3)
H3A—C3—H3B	108.4	N1—C13—C1	108.1 (3)
C5—C4—C3	114.7 (3)	O2—C14—N1	123.9 (3)
C5—C4—H4A	108.6	O2—C14—C10	130.9 (3)
C3—C4—H4A	108.6	N1—C14—C10	105.1 (3)
C5—C4—H4B	108.6	O3—C15—N1	118.4 (3)
C3—C4—H4B	108.6	O3—C15—C16	123.8 (3)
H4A—C4—H4B	107.6	N1—C15—C16	117.7 (3)
C4—C5—C6	113.8 (3)	C21—C16—C17	119.3 (3)
C4—C5—H5A	108.8	C21—C16—C15	117.9 (3)
C6—C5—H5A	108.8	C17—C16—C15	122.8 (3)
C4—C5—H5B	108.8	C16—C17—C18	120.1 (4)
C6—C5—H5B	108.8	C16—C17—H17	119.9
H5A—C5—H5B	107.7	C18—C17—H17	119.9
C7—C6—C5	109.1 (4)	C19—C18—C17	120.2 (4)
C7—C6—H6A	109.9	C19—C18—H18	119.9
C5—C6—H6A	109.9	C17—C18—H18	119.9
C7—C6—H6B	109.9	C18—C19—C20	119.8 (4)
C5—C6—H6B	109.9	C18—C19—H19	120.1
H6A—C6—H6B	108.3	C20—C19—H19	120.1
C6—C7—C2	110.8 (3)	C19—C20—C21	121.1 (4)
C6—C7—C8	122.4 (3)	C19—C20—H20	119.4
C2—C7—C8	109.0 (3)	C21—C20—H20	119.4
C6—C7—H7	104.3	C16—C21—C20	119.5 (4)
C2—C7—H7	104.3	C16—C21—H21	120.3
C8—C7—H7	104.3	C20—C21—H21	120.3

C11—C1—C2—C3	−86.5 (4)	C7—C8—C12—C11	59.5 (4)
C13—C1—C2—C3	48.3 (5)	C14—N1—C13—O1	−178.3 (3)
C10—C1—C2—C3	159.1 (4)	C15—N1—C13—O1	10.9 (5)
C11—C1—C2—C7	44.9 (4)	C14—N1—C13—C1	0.9 (4)
C13—C1—C2—C7	179.7 (3)	C15—N1—C13—C1	−169.8 (3)
C10—C1—C2—C7	−69.5 (3)	C11—C1—C13—O1	57.3 (5)
C7—C2—C3—C4	58.4 (5)	C10—C1—C13—O1	176.6 (4)
C1—C2—C3—C4	−171.5 (3)	C2—C1—C13—O1	−73.4 (5)
C2—C3—C4—C5	−49.9 (5)	C11—C1—C13—N1	−121.9 (3)
C3—C4—C5—C6	46.7 (6)	C10—C1—C13—N1	−2.6 (3)
C4—C5—C6—C7	−49.5 (5)	C2—C1—C13—N1	107.4 (3)
C5—C6—C7—C2	58.7 (5)	C13—N1—C14—O2	−178.3 (3)
C5—C6—C7—C8	−170.5 (4)	C15—N1—C14—O2	−7.9 (5)
C3—C2—C7—C6	−65.8 (4)	C13—N1—C14—C10	1.3 (3)
C1—C2—C7—C6	155.5 (3)	C15—N1—C14—C10	171.6 (3)
C3—C2—C7—C8	156.7 (3)	C9—C10—C14—O2	1.5 (6)
C1—C2—C7—C8	18.0 (4)	C1—C10—C14—O2	176.6 (3)
C6—C7—C8—C9	−87.4 (4)	C9—C10—C14—N1	−178.0 (4)
C2—C7—C8—C9	44.1 (4)	C1—C10—C14—N1	−2.9 (3)
C6—C7—C8—C12	158.8 (4)	C13—N1—C15—O3	51.8 (5)

C2—C7—C8—C12	−69.7 (4)	C14—N1—C15—O3	−117.8 (4)
C12—C8—C9—C10	50.0 (4)	C13—N1—C15—C16	−125.1 (3)
C7—C8—C9—C10	−58.8 (4)	C14—N1—C15—C16	65.3 (4)
C8—C9—C10—C14	178.1 (3)	O3—C15—C16—C21	5.2 (6)
C8—C9—C10—C1	3.3 (4)	N1—C15—C16—C21	−178.1 (3)
C11—C1—C10—C9	−54.7 (4)	O3—C15—C16—C17	−173.4 (4)
C13—C1—C10—C9	179.5 (3)	N1—C15—C16—C17	3.3 (5)
C2—C1—C10—C9	60.6 (4)	C21—C16—C17—C18	0.0 (6)
C11—C1—C10—C14	129.2 (3)	C15—C16—C17—C18	178.6 (4)
C13—C1—C10—C14	3.4 (3)	C16—C17—C18—C19	0.0 (7)
C2—C1—C10—C14	−115.4 (3)	C17—C18—C19—C20	0.1 (7)
C13—C1—C11—C12	166.6 (3)	C18—C19—C20—C21	−0.2 (7)
C10—C1—C11—C12	49.9 (4)	C17—C16—C21—C20	−0.1 (6)
C2—C1—C11—C12	−60.5 (4)	C15—C16—C21—C20	−178.8 (4)
C1—C11—C12—C8	3.4 (4)	C19—C20—C21—C16	0.2 (7)
C9—C8—C12—C11	−54.9 (4)		