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t-3-Ethyl-*r*-2,*c*-6-bis(2-furyl)piperidin-4-one

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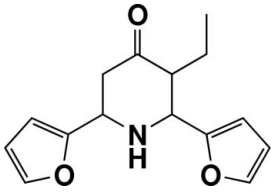
Key indicators: single-crystal X-ray study; T = 200 K; mean σ(C–C) = 0.002 Å; R factor = 0.059; wR factor = 0.194; data-to-parameter ratio = 25.0.

In the title molecule, $C_{15}H_{17}NO_3$, the piperidine ring adopts a chair conformation. The dihedral angle between the two furyl rings is 72.4 (1)°. The ethyl group and the furyl rings have equatorial orientations. Molecules are linked by N—H···O hydrogen bonds.

Related literature

For a related crystal structure, see Thiruvalluvar *et al.* (2007).



Experimental

Crystal data

$C_{15}H_{17}NO_3$
M_r = 259.30
Monoclinic, $P2_1/c$
a = 5.1620 (2) Å
b = 20.2855 (9) Å
c = 12.9825 (5) Å
β = 91.128 (3)°
V = 1359.18 (10) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 200 (2) K
0.41 × 0.36 × 0.18 mm

Data collection

Oxford Diffraction Gemini diffractometer
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2007)
T_{min} = 0.965, T_{max} = 1.000 (expected range = 0.950–0.984)
11183 measured reflections
4354 independent reflections
2961 reflections with $I > 2\sigma(I)$
R_{int} = 0.072

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.059
$wR(F^2)$ = 0.194
S = 1.07
4354 reflections
174 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.41 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O4^{i}	0.867 (17)	2.234 (17)	3.0991 (14)	176.2 (15)

Symmetry code: (i) $x, -y+\frac{1}{2}, z-\frac{1}{2}$.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2007); cell refinement: *CrysAlis CCD*; data reduction: *CrysAlis RED* (Oxford Diffraction, 2007); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *PLATON* (Spek, 2003).

RJB acknowledges the NSF–MRI program for funding to purchase the X-ray CCD diffractometer. JJ is grateful to the UGC [F. No. 30–71/2004(SR)], New Delhi, India, for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RN2035).

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Thiruvalluvar, A., Balamurugan, S., Jayabharathi, J. & Manimekalai, A. (2007). *Acta Cryst.* E**63**, o2910.

supporting information

Acta Cryst. (2008). E**64**, o59 [https://doi.org/10.1107/S1600536807062204]

t-3-Ethyl-*r*-2,*c*-6-bis(2-furyl)piperidin-4-one

S. Balamurugan, A. Thiruvalluvar, R. J. Butcher, A. Manimekalai and J. Jayabharathi

S1. Comment

Thiruvalluvar *et al.* (2007) have reported a crystal structure of t3-benzyl-r2,c-6-di-2-furylpiperidin-4-one wherein the piperidine ring is in chair form. In the title molecule, $C_{15}H_{17}NO_3$, Fig.1., the piperidine ring adopts a chair conformation. The dihedral angle between the two furyl rings is 72.4 (1)°. The ethyl group in the 3-position and the furyl rings at positions 2 and 6 have equatorial orientations. Molecules are linked by N1—H1···O4 hydrogen bonds, Fig.2., forming an infinite one-dimensional chain with the base vector (0 0 1).

S2. Experimental

A mixture of ammonium acetate (100 mmol, 7.7 g), furfuraldehyde (200 mmol, 16.5 ml) and methyl propyl ketone (100 mmol, 10.6 ml) in distilled ethanol was heated first to boiling. After cooling, the viscous liquid obtained was dissolved in ether (200 ml) and shaken with 10 ml of conc. hydrochloric acid. The precipitated hydrochloride of the title compound was removed by filtration and washed first with 40 cc mixture of ethanol and ether (1:1 *v*/*v*) and then with ether to remove most of the coloured impurities. The base was liberated from an alcoholic solution by adding aqueous ammonia and then diluted with water. It was recrystallized from alcohol. The yield of the isolated product was 13.5 g (70%).

S3. Refinement

The N-bound H atom was found in a difference Fourier map and refined isotropically. The remaining H atoms were positioned geometrically and allowed to ride on their parent atoms with C—H = 0.95–1.00 Å and U_{iso} = 1.2–1.5U_{eq}(C).

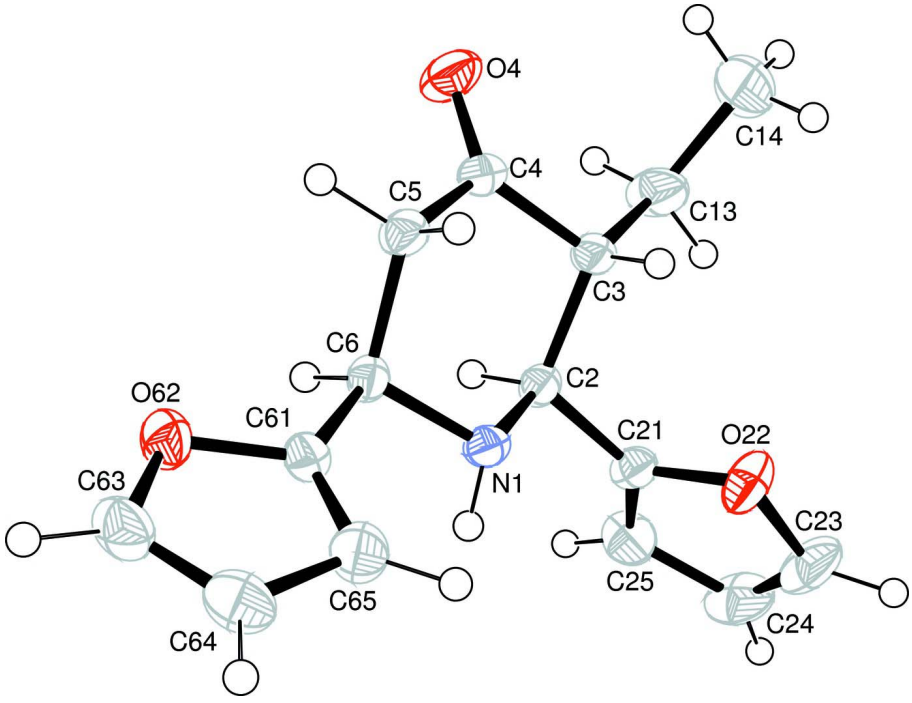

Figure 1

The molecular structure of the title compound with the atomic numbering and 50% probability displacement ellipsoids. H atoms are shown as small spheres of arbitrary radius.

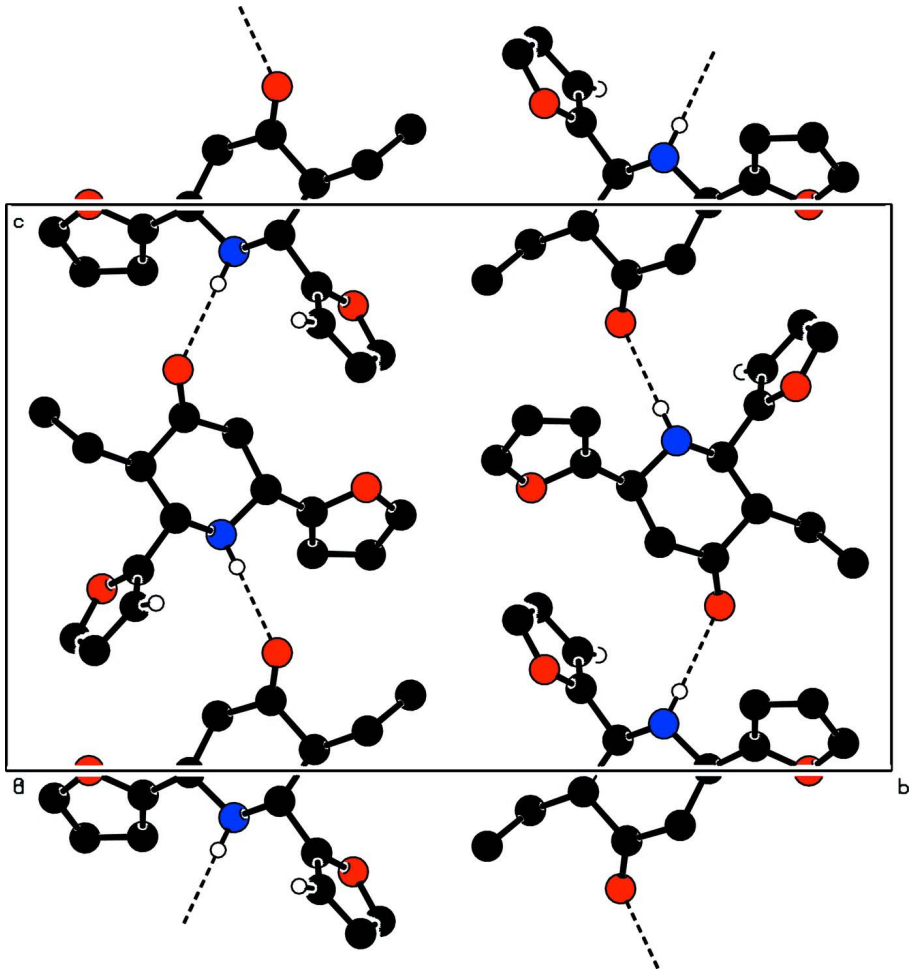

Figure 2

The molecular packing of the title compound, viewed down the *a* axis showing the N—H···O (dashed lines) hydrogen bonds.

t-3-Ethyl-r-2,c-6-bis(2-furyl)piperidin-4-one

Crystal data

$C_{15}H_{17}NO_3$
$M_r = 259.30$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 5.1620\ (2)$ Å
$b = 20.2855\ (9)$ Å
$c = 12.9825\ (5)$ Å
$\beta = 91.128\ (3)°$
$V = 1359.18\ (10)$ Å^3
$Z = 4$

$F(000) = 552$
$D_x = 1.267$ Mg m^{-3}
Melting point: 320 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 5794 reflections
$\theta = 4.7–32.4°$
$\mu = 0.09$ mm^{-1}
$T = 200$ K
Plate, colourless
0.41 × 0.36 × 0.18 mm

Data collection

Oxford Diffraction Gemini diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 10.5081 pixels mm^{-1}
phi and ω scans
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2007)
T_{min} = 0.965, T_{max} = 1.000

11183 measured reflections
4354 independent reflections
2961 reflections with $I > 2\sigma(I)$
R_{int} = 0.072
θ_{max} = 32.4°, θ_{min} = 4.7°
$h = -7 \rightarrow 7$
$k = -29 \rightarrow 23$
$l = -18 \rightarrow 18$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.059
$wR(F^2)$ = 0.194
S = 1.07
4354 reflections
174 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
$w = 1/[\sigma^2(F_o^2) + (0.1195P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.41 e $Å^{-3}$
$\Delta\rho_{min}$ = −0.26 e $Å^{-3}$

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

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}
O4	0.7516 (2)	0.19399 (5)	0.70845 (7)	0.0423 (3)
O22	0.65339 (19)	0.10804 (6)	0.32132 (8)	0.0390 (3)
O62	0.43126 (18)	0.40669 (5)	0.50031 (7)	0.0332 (3)
N1	0.6097 (2)	0.24278 (5)	0.41720 (8)	0.0246 (3)
C2	0.7958 (2)	0.19114 (6)	0.44599 (9)	0.0236 (3)
C3	0.6869 (3)	0.15193 (6)	0.53734 (9)	0.0262 (3)
C4	0.6510 (2)	0.20108 (6)	0.62400 (9)	0.0273 (3)
C5	0.4922 (3)	0.26132 (6)	0.59650 (9)	0.0278 (3)
C6	0.5885 (2)	0.29338 (6)	0.49665 (9)	0.0237 (3)
C13	0.8496 (4)	0.09209 (8)	0.57028 (12)	0.0435 (5)
C14	0.6945 (5)	0.04322 (9)	0.63338 (16)	0.0622 (7)
C21	0.8466 (2)	0.14932 (6)	0.35453 (9)	0.0259 (3)
C23	0.7409 (3)	0.07793 (9)	0.23394 (12)	0.0468 (5)
C24	0.9769 (4)	0.09963 (9)	0.21229 (12)	0.0485 (5)
C25	1.0469 (3)	0.14554 (8)	0.29020 (12)	0.0382 (4)
C61	0.4080 (2)	0.34542 (6)	0.45684 (9)	0.0258 (3)

C63	0.2484 (2)	0.44582 (5)	0.45159 (9)	0.0374 (4)
C64	0.1172 (2)	0.41118 (5)	0.38123 (9)	0.0405 (5)
C65	0.2180 (3)	0.34610 (7)	0.38376 (11)	0.0356 (4)
H1	0.657 (3)	0.2606 (8)	0.3599 (13)	0.028 (4)*
H2	0.96189	0.21250	0.46873	0.0282*
H3	0.51127	0.13546	0.51602	0.0314*
H5A	0.50437	0.29363	0.65353	0.0333*
H5B	0.30808	0.24859	0.58727	0.0333*
H6	0.76302	0.31334	0.51011	0.0284*
H13A	1.00135	0.10725	0.61164	0.0521*
H13B	0.91449	0.06968	0.50816	0.0521*
H14A	0.80508	0.00579	0.65282	0.0932*
H14B	0.54586	0.02745	0.59223	0.0932*
H14C	0.63285	0.06498	0.69568	0.0932*
H23	0.64657	0.04619	0.19461	0.0561*
H24	1.07879	0.08681	0.15554	0.0582*
H25	1.20538	0.16918	0.29591	0.0458*
H63	0.22022	0.49111	0.46625	0.0449*
H64	−0.01859	0.42700	0.33741	0.0486*
H65	0.16260	0.31003	0.34216	0.0427*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O4	0.0628 (7)	0.0404 (6)	0.0232 (5)	0.0127 (5)	−0.0095 (4)	−0.0034 (4)
O22	0.0381 (5)	0.0437 (6)	0.0353 (5)	−0.0029 (4)	0.0006 (4)	−0.0178 (5)
O62	0.0413 (6)	0.0228 (4)	0.0352 (5)	0.0053 (4)	−0.0039 (4)	−0.0016 (4)
N1	0.0316 (5)	0.0236 (5)	0.0185 (4)	0.0023 (4)	0.0002 (3)	−0.0006 (4)
C2	0.0274 (6)	0.0222 (6)	0.0210 (5)	0.0013 (4)	−0.0016 (4)	−0.0024 (4)
C3	0.0372 (7)	0.0208 (6)	0.0205 (5)	0.0027 (5)	−0.0012 (4)	0.0015 (4)
C4	0.0352 (6)	0.0271 (6)	0.0197 (5)	0.0005 (5)	0.0015 (4)	0.0006 (4)
C5	0.0369 (7)	0.0249 (6)	0.0217 (5)	0.0054 (5)	0.0018 (4)	−0.0024 (4)
C6	0.0285 (6)	0.0194 (5)	0.0230 (5)	0.0009 (4)	−0.0026 (4)	−0.0022 (4)
C13	0.0684 (10)	0.0324 (7)	0.0295 (6)	0.0188 (7)	0.0000 (6)	0.0034 (6)
C14	0.1052 (16)	0.0309 (8)	0.0497 (10)	−0.0023 (9)	−0.0165 (10)	0.0132 (8)
C21	0.0292 (6)	0.0262 (6)	0.0221 (5)	0.0047 (5)	−0.0009 (4)	−0.0013 (4)
C23	0.0644 (11)	0.0453 (9)	0.0303 (7)	0.0166 (8)	−0.0064 (7)	−0.0178 (7)
C24	0.0660 (11)	0.0504 (10)	0.0295 (7)	0.0227 (8)	0.0118 (7)	−0.0069 (6)
C25	0.0408 (8)	0.0346 (7)	0.0397 (8)	0.0044 (6)	0.0124 (6)	−0.0004 (6)
C61	0.0324 (6)	0.0200 (5)	0.0250 (5)	0.0007 (5)	0.0019 (4)	0.0008 (4)
C63	0.0446 (8)	0.0267 (7)	0.0410 (8)	0.0102 (6)	0.0034 (6)	0.0049 (5)
C64	0.0420 (8)	0.0370 (8)	0.0423 (8)	0.0091 (6)	−0.0049 (6)	0.0100 (6)
C65	0.0396 (7)	0.0304 (7)	0.0364 (7)	0.0023 (6)	−0.0083 (5)	0.0008 (5)

Geometric parameters (Å, º)

O4—C4	1.2124 (15)	C61—C65	1.3512 (19)
O22—C21	1.3656 (16)	C63—C64	1.3276 (15)

O22—C23	1.3727 (19)	C64—C65	1.4192 (18)
O62—C61	1.3693 (16)	C2—H2	1.0000
O62—C63	1.3775 (14)	C3—H3	1.0000
N1—C2	1.4648 (15)	C5—H5A	0.9900
N1—C6	1.4608 (16)	C5—H5B	0.9900
N1—H1	0.867 (17)	C6—H6	1.0000
C2—C21	1.4868 (17)	C13—H13A	0.9900
C2—C3	1.5432 (17)	C13—H13B	0.9900
C3—C4	1.5174 (17)	C14—H14A	0.9800
C3—C13	1.532 (2)	C14—H14B	0.9800
C4—C5	1.5104 (18)	C14—H14C	0.9800
C5—C6	1.5414 (17)	C23—H23	0.9500
C6—C61	1.4936 (16)	C24—H24	0.9500
C13—C14	1.523 (3)	C25—H25	0.9500
C21—C25	1.3440 (19)	C63—H63	0.9500
C23—C24	1.331 (3)	C64—H64	0.9500
C24—C25	1.417 (2)	C65—H65	0.9500

O4···C14	3.222 (2)	H1···C65	2.875 (16)
O4···C65^{i}	3.3796 (18)	H1···O4^{vi}	2.234 (17)
O4···N1^{ii}	3.0991 (14)	H2···H5B^{vii}	2.4500
O22···C25^{iii}	3.2394 (19)	H2···H6	2.3600
O22···C13	3.3835 (19)	H3···O22	2.7000
O22···N1	3.0137 (16)	H3···H14B	2.4100
O62···C23^{ii}	3.4148 (18)	H5A···C23^{ii}	3.0500
O4···H65^{i}	2.7200	H5A···H25^{v}	2.5500
O4···H1^{ii}	2.234 (17)	H5B···H2^{iii}	2.4500
O4···H14C	2.6900	H6···C65^{vii}	2.9700
O4···H13A	2.5300	H6···H2	2.3600
O22···H3	2.7000	H13A···O4	2.5300
O22···H25^{iii}	2.6400	H13B···O22	2.8600
O22···H13B	2.8600	H13B···C21	2.5900
O62···H63^{iv}	2.7700	H14A···C24^{ix}	2.9700
O62···H23^{ii}	2.9000	H14B···H3	2.4100
O62···H24^{v}	2.7400	H14C···O4	2.6900
N1···O22	3.0137 (16)	H14C···C4	2.9200
N1···O4^{vi}	3.0991 (14)	H14C···H64^{i}	2.5500
N1···H65	2.8400	H23···C63^{xii}	2.8400
C13···O22	3.3835 (19)	H23···H63^{xii}	2.4800
C14···O4	3.222 (2)	H23···O62^{vi}	2.9000
C23···O62^{vi}	3.4148 (18)	H24···O62^{xiii}	2.7400
C25···O22^{vii}	3.2394 (19)	H24···C63^{xiii}	2.8800
C65···O4^{viii}	3.3796 (18)	H25···O22^{vii}	2.6400
C4···H14C	2.9200	H25···H5A^{xiii}	2.5500
C14···H64^{i}	3.0700	H63···H23^{x}	2.4800
C21···H13B	2.5900	H63···O62^{iv}	2.7700
C23···H5A^{vi}	3.0500	H63···C63^{xi}	2.9500
C24···H14A^{ix}	2.9700	H63···H63^{xi}	2.4800

C63···H23^{x}	2.8400	H64···C14^{viii}	3.0700
C63···H63^{xi}	2.9500	H64···H14C^{viii}	2.5500
C63···H24^{v}	2.8800	H65···N1	2.8400
C65···H1	2.875 (16)	H65···O4^{viii}	2.7200
C65···H6^{iii}	2.9700		

C21—O22—C23	106.48 (11)	C2—C3—H3	108.00
C61—O62—C63	106.26 (9)	C4—C3—H3	108.00
C2—N1—C6	112.35 (9)	C13—C3—H3	108.00
C2—N1—H1	108.8 (10)	C4—C5—H5A	109.00
C6—N1—H1	109.8 (11)	C4—C5—H5B	109.00
N1—C2—C3	108.52 (9)	C6—C5—H5A	109.00
N1—C2—C21	109.23 (9)	C6—C5—H5B	109.00
C3—C2—C21	113.10 (10)	H5A—C5—H5B	108.00
C2—C3—C4	106.43 (10)	N1—C6—H6	109.00
C4—C3—C13	112.82 (11)	C5—C6—H6	109.00
C2—C3—C13	114.64 (12)	C61—C6—H6	109.00
O4—C4—C5	122.04 (11)	C3—C13—H13A	109.00
O4—C4—C3	122.42 (11)	C3—C13—H13B	109.00
C3—C4—C5	115.47 (10)	C14—C13—H13A	109.00
C4—C5—C6	110.96 (11)	C14—C13—H13B	109.00
N1—C6—C5	109.10 (10)	H13A—C13—H13B	108.00
C5—C6—C61	112.33 (10)	C13—C14—H14A	109.00
N1—C6—C61	107.89 (9)	C13—C14—H14B	109.00
C3—C13—C14	112.00 (16)	C13—C14—H14C	109.00
O22—C21—C25	109.56 (11)	H14A—C14—H14B	109.00
O22—C21—C2	117.57 (9)	H14A—C14—H14C	109.00
C2—C21—C25	132.75 (12)	H14B—C14—H14C	109.00
O22—C23—C24	110.18 (14)	O22—C23—H23	125.00
C23—C24—C25	106.76 (15)	C24—C23—H23	125.00
C21—C25—C24	107.02 (14)	C23—C24—H24	127.00
O62—C61—C65	109.68 (11)	C25—C24—H24	127.00
C6—C61—C65	133.59 (12)	C21—C25—H25	126.00
O62—C61—C6	116.74 (10)	C24—C25—H25	126.00
O62—C63—C64	110.28 (9)	O62—C63—H63	125.00
C63—C64—C65	107.13 (10)	C64—C63—H63	125.00
C61—C65—C64	106.65 (12)	C63—C64—H64	126.00
N1—C2—H2	109.00	C65—C64—H64	126.00
C3—C2—H2	109.00	C61—C65—H65	127.00
C21—C2—H2	109.00	C64—C65—H65	127.00

C21—O22—C23—C24	−0.64 (18)	C4—C3—C13—C14	77.18 (17)
C23—O22—C21—C2	176.79 (12)	C2—C3—C13—C14	−160.78 (13)
C23—O22—C21—C25	0.33 (16)	C2—C3—C4—O4	−123.63 (12)
C63—O62—C61—C65	0.00 (14)	C13—C3—C4—C5	179.97 (13)
C63—O62—C61—C6	179.71 (9)	C3—C4—C5—C6	−49.62 (14)
C61—O62—C63—C64	−0.05 (14)	O4—C4—C5—C6	127.45 (12)
C6—N1—C2—C21	−168.73 (9)	C4—C5—C6—N1	50.06 (13)

C6—N1—C2—C3	67.56 (12)	C4—C5—C6—C61	169.65 (10)
C2—N1—C6—C5	−61.28 (12)	C5—C6—C61—C65	−97.83 (16)
C2—N1—C6—C61	176.43 (9)	C5—C6—C61—O62	82.53 (12)
C21—C2—C3—C13	53.53 (14)	N1—C6—C61—O62	−157.19 (10)
C3—C2—C21—O22	49.48 (14)	N1—C6—C61—C65	22.46 (18)
N1—C2—C21—C25	103.97 (16)	C2—C21—C25—C24	−175.66 (14)
N1—C2—C3—C4	−59.65 (12)	O22—C21—C25—C24	0.07 (16)
C3—C2—C21—C25	−135.07 (15)	O22—C23—C24—C25	0.7 (2)
N1—C2—C3—C13	174.89 (11)	C23—C24—C25—C21	−0.46 (19)
C21—C2—C3—C4	178.99 (9)	O62—C61—C65—C64	0.07 (16)
N1—C2—C21—O22	−71.49 (13)	C6—C61—C65—C64	−179.60 (12)
C13—C3—C4—O4	2.92 (18)	O62—C63—C64—C65	0.09 (13)
C2—C3—C4—C5	53.42 (14)	C63—C64—C65—C61	−0.10 (14)

Symmetry codes: (i) *x*+1, −*y*+1/2, *z*+1/2; (ii) *x*, −*y*+1/2, *z*+1/2; (iii) *x*−1, *y*, *z*; (iv) −*x*+1, −*y*+1, −*z*+1; (v) *x*−1, −*y*+1/2, *z*+1/2; (vi) *x*, −*y*+1/2, *z*−1/2; (vii) *x*+1, *y*, *z*; (viii) *x*−1, −*y*+1/2, *z*−1/2; (ix) −*x*+2, −*y*, −*z*+1; (x) −*x*+1, *y*+1/2, −*z*+1/2; (xi) −*x*, −*y*+1, −*z*+1; (xii) −*x*+1, *y*−1/2, −*z*+1/2; (xiii) *x*+1, −*y*+1/2, *z*−1/2.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O4^{vi}	0.867 (17)	2.234 (17)	3.0991 (14)	176.2 (15)

Symmetry code: (vi) *x*, −*y*+1/2, *z*−1/2.