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2-*tert*-Butoxy-1-phenyl-1-(2,2,6,6-tetramethylpiperidin-1-yloxy)ethane

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Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.001 Å
R factor = 0.039
wR factor = 0.114
Data-to-parameter ratio = 28.8

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

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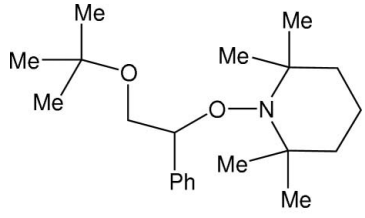
The title compound, $C_{21}H_{35}NO_2$, contains a piperidine ring in a chair conformation, with a pyramidal N atom and a single (exocyclic) N—O bond in an equatorial orientation.

Comment

Nitroxide-mediated polymerization (NMP) has emerged in recent years as a successful controlled or 'living' radical polymerization technique which can be used to prepare polymers of target molecular weight, narrow polydispersity and complex architecture (block and graft copolymers, star polymers, *etc.*) (Matyjaszewski, 2003). Successful NMP requires the use of a monomolecular initiator, identified as an alkoxyamine, which is derived from a nitroxide. This alkoxyamine should be prepared separately and then added in a known concentration to the monomer to be polymerized. Synthetic routes to alkoxyamines include the trapping of alkyl radicals by free nitroxides at moderate temperatures (Braslau *et al.*, 1997; Miura *et al.*, 1998) and a catalytic route involving Mn–salen complexes [H_2salen is bis(salicylidene)ethylenediamine; Dao *et al.*, 1998]. The title compound, (I), has been synthesized in the course of these studies (Cameron *et al.*, 2000).



(I)

The molecular structure of (I) (Fig. 1) is similar in its main features to the other *N*-oxy-2,2,6,6-tetramethylpiperidinyl derivatives reported by Ermert & Vasella (1993), Jahn *et al.* (2001, 2002) and Leitich *et al.* (2002). The piperidine ring adopts a chair conformation with the N—O1 bond in an equatorial orientation. The N—O1 distance is typical for a single bond (Allen *et al.*, 1987), while the N atom has pyramidal geometry, corresponding to sp^3 hybridization.

Experimental

A solution of 2,2,6,6-tetramethylpiperidinyloxy (TEMPO; 0.187 g, 1.20 mmol) and di-*tert*-butylperoxalate (0.148 g, 0.63 mmol) in styrene (5 ml) was prepared. The mixture was degassed by three freeze/pump/thaw cycles and backflushed with argon after the final cycle. The resulting solution was sealed, then heated overnight at 313 K. After cooling, the solution was dried *in vacuo* to constant

mass. The crude solid product obtained was then purified by flash chromatography on silica gel, eluting with a mixture of 40–60 petroleum ether/ethyl acetate (90:10 *v*/*v*). After removal of the solvent, a white crystalline solid was obtained in 79% yield. The characterization data of the isolated product were in agreement with those given in the literature (Bon *et al.*, 1999). ^{1}H NMR (300 MHz): δ 1.04 (*s*, 9H, $CH_3 \times 3$), 0.58, 1.02, 1.19, 1.37 (*br s*, 3H, CH_3), 0.9–1.7 (*br m*, 6H, $CH_2 \times 3$), 3.41 (*m*, 1H, H1), 3.89 (*m*, 1H, H171), 4.74 (*m*, 1H, H172), 7.18–7.35 (*m*, 5H, Ph) p.p.m. (for the H-atom numbering, see Fig. 1).

Crystal data

$C_{21}H_{35}NO_2$
M_r = 333.50
Orthorhombic, *Pbca*
a = 15.532 (4) Å
b = 11.119 (3) Å
c = 23.247 (5) Å
V = 4014.8 (17) Å^3
Z = 8
D_x = 1.104 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 902 reflections
θ = 12.1–24.3°
μ = 0.07 mm^{-1}
T = 100 (2) K
Block, colourless
1.00 × 0.66 × 0.14 mm

Data collection

Bruker SMART 6000 CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.579, T_{max} = 1.000
60196 measured reflections
7229 independent reflections
6298 reflections with $I > 2\sigma(I)$
R_{int} = 0.020
θ_{max} = 32.5°
$h = -23 \rightarrow 23$
$k = -16 \rightarrow 16$
$l = -34 \rightarrow 35$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.114
S = 1.06
7229 reflections
251 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0633P)^2 + 0.7171P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.52 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.4422 (8)	C2—C3	1.5399 (11)
O1—N	1.4595 (7)	C3—C4	1.5198 (14)
O2—C17	1.4158 (8)	C4—C5	1.5153 (13)
O2—C18	1.4446 (8)	C5—C6	1.5312 (10)
N—C6	1.4987 (9)	C6—C10	1.5280 (10)
N—C2	1.5033 (9)	C6—C9	1.5353 (11)
C1—C11	1.5168 (9)	C18—C20	1.5187 (10)
C1—C17	1.5228 (9)	C18—C21	1.5200 (11)
C2—C8	1.5354 (11)	C18—C19	1.5238 (11)
C2—C7	1.5377 (12)		
C1—O1—N	112.33 (5)	C5—C4—C3	108.36 (7)
C17—O2—C18	116.92 (5)	C4—C5—C6	112.78 (6)
O1—N—C6	106.17 (5)	C10—C6—C5	107.62 (6)
O1—N—C2	107.19 (5)	O2—C17—C1	108.48 (5)
C6—N—C2	117.35 (5)	O2—C18—C20	103.38 (6)
O1—C1—C11	114.45 (5)	O2—C18—C21	111.07 (6)
O1—C1—C17	105.37 (5)	C20—C18—C21	110.35 (6)
C11—C1—C17	110.04 (5)	O2—C18—C19	111.02 (6)
N—C2—C3	107.44 (6)	C20—C18—C19	110.12 (7)
C4—C3—C2	114.33 (7)	C21—C18—C19	110.68 (7)

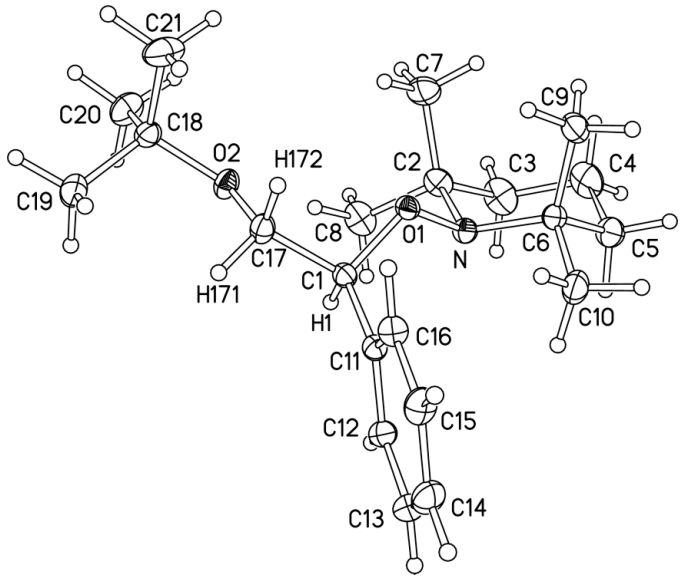


Figure 1
The molecular structure of (I), showing atomic displacement ellipsoids at the 50% probability level.

Methyl groups were treated as threefold symmetrical bodies rotating around C—C bonds, with a refined common U_{iso} for the three H atoms. Other H atoms were treated as riding on the corresponding C atoms, with refined U_{iso} values. C—H distances are 0.95–1.00 Å.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 2001); program(s) used to solve structure: *SHELXTL* (Bruker, 2001); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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supporting information

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Crystal data

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Orthorhombic, *Pbca*
Hall symbol: -P 2ac 2ab
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$c = 23.247$ (5) Å
$V = 4014.8$ (17) Å^3
$Z = 8$
$F(000) = 1472$
$D_x = 1.104$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 902 reflections
$\theta = 12.1$–$24.3°$
$\mu = 0.07$ mm^{-1}
$T = 100$ K
Block, colourless
1.00 × 0.66 × 0.14 mm

Data collection

Bruker SMART 6000 CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 5.6 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2001)
$T_{min} = 0.579$, $T_{max} = 1.000$
60196 measured reflections
7229 independent reflections
6298 reflections with $I > 2\sigma(I)$
$R_{int} = 0.020$
$\theta_{max} = 32.5°$, $\theta_{min} = 1.8°$
$h = -23 \rightarrow 23$
$k = -16 \rightarrow 16$
$l = -34 \rightarrow 35$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.114$
$S = 1.06$
7229 reflections
251 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0633P)^2 + 0.7171P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.52$ e Å^{-3}
$\Delta\rho_{min} = -0.17$ e Å^{-3}

Special details

Experimental. The data collection nominally covered full sphere of reciprocal space, by a combination of 4 sets of ω scans; each set at different φ and/or 2θ angles and each scan (20 sec exposure) covering 0.3° in ω. Crystal to detector distance 4.85 cm.
Crystals are shattering when cut, therefore a crystal larger than the beam diameter was used and the intensities were corrected by *SADABS* program (actual absorption is negligible).

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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.50726 (3)	0.34047 (4)	0.41290 (2)	0.01533 (9)
O2	0.33509 (3)	0.38797 (5)	0.37666 (2)	0.01928 (10)
N	0.56521 (4)	0.25032 (5)	0.38875 (2)	0.01604 (10)
C1	0.43261 (4)	0.28730 (6)	0.43963 (3)	0.01552 (11)
H1	0.4152	0.2145	0.4172	0.016 (2)*
C2	0.55469 (5)	0.25419 (7)	0.32449 (3)	0.02115 (13)
C3	0.62458 (6)	0.17267 (8)	0.29828 (4)	0.03198 (17)
H31	0.6109	0.0880	0.3078	0.040 (3)*
H32	0.6228	0.1808	0.2559	0.044 (3)*
C4	0.71538 (6)	0.20006 (9)	0.31880 (4)	0.03276 (18)
H41	0.7564	0.1415	0.3021	0.044 (3)*
H42	0.7326	0.2818	0.3064	0.038 (3)*
C5	0.71683 (5)	0.19182 (7)	0.38386 (4)	0.02631 (15)
H51	0.7755	0.2106	0.3977	0.032 (3)*
H52	0.7032	0.1083	0.3955	0.035 (3)*
C6	0.65274 (4)	0.27758 (6)	0.41254 (3)	0.01839 (12)
C7	0.55652 (6)	0.38093 (8)	0.29805 (3)	0.02855 (16)
H71	0.5145 (5)	0.4323 (4)	0.3179 (3)	0.0386 (13)*
H72	0.5418 (5)	0.37606 (12)	0.2570 (3)	0.0386 (13)*
H73	0.6144 (4)	0.4154 (4)	0.3024 (3)	0.0386 (13)*
C8	0.46745 (5)	0.19737 (8)	0.30943 (3)	0.02742 (15)
H81	0.4625 (2)	0.1184 (7)	0.3284 (3)	0.0375 (18)*
H82	0.4632 (2)	0.1870 (6)	0.2674 (3)	0.0375 (18)*
H83	0.4207 (3)	0.2502 (5)	0.3228 (3)	0.0375 (18)*
C9	0.68364 (5)	0.40811 (7)	0.40643 (4)	0.02434 (14)
H91	0.7018 (5)	0.4226 (2)	0.3668 (3)	0.0405 (19)*
H92	0.7321 (5)	0.4220 (2)	0.4324 (3)	0.0405 (19)*
H93	0.6367 (4)	0.4628 (4)	0.4162 (3)	0.0405 (19)*
C10	0.65061 (5)	0.24755 (8)	0.47666 (3)	0.02721 (15)
H101	0.6137 (5)	0.3078 (6)	0.49731 (15)	0.0381 (18)*
H102	0.7106 (4)	0.2503 (7)	0.49259 (14)	0.0381 (18)*
H103	0.6261 (5)	0.1649 (6)	0.48219 (6)	0.0381 (18)*
C11	0.44530 (4)	0.25205 (6)	0.50208 (3)	0.01565 (11)
C12	0.43806 (4)	0.13184 (6)	0.51850 (3)	0.01924 (12)
H12	0.4270	0.0723	0.4901	0.025 (3)*
C13	0.44677 (5)	0.09773 (7)	0.57597 (3)	0.02422 (14)
H13	0.4423	0.0154	0.5865	0.033 (3)*

C14	0.46198 (5)	0.18453 (8)	0.61768 (3)	0.02565 (15)
H14	0.4674	0.1618	0.6569	0.038 (3)*
C15	0.46920 (5)	0.30477 (7)	0.60196 (3)	0.02317 (14)
H15	0.4796	0.3642	0.6305	0.036 (3)*
C16	0.46121 (4)	0.33830 (6)	0.54450 (3)	0.01906 (12)
H16	0.4666	0.4205	0.5340	0.025 (3)*
C17	0.36194 (4)	0.38169 (6)	0.43478 (3)	0.01849 (12)
H171	0.3127	0.3595	0.4596	0.024 (2)*
H172	0.3840	0.4609	0.4475	0.024 (2)*
C18	0.26850 (4)	0.47410 (6)	0.36304 (3)	0.01854 (12)
C19	0.18898 (5)	0.45399 (8)	0.40020 (4)	0.02978 (17)
H191	0.1726 (3)	0.3670 (7)	0.3991 (3)	0.0434 (19)*
H192	0.1403 (4)	0.5037 (6)	0.3852 (2)	0.0434 (19)*
H193	0.20186 (18)	0.4780 (7)	0.4408 (3)	0.0434 (19)*
C20	0.24793 (6)	0.44747 (8)	0.30046 (3)	0.02784 (15)
H201	0.3015 (4)	0.4557 (6)	0.27679 (17)	0.0386 (13)*
H202	0.2035 (4)	0.5057 (6)	0.28632 (14)	0.0386 (13)*
H203	0.2252 (5)	0.3634 (6)	0.29697 (6)	0.0386 (13)*
C21	0.30130 (6)	0.60218 (7)	0.36949 (4)	0.03203 (18)
H211	0.3145 (5)	0.6183 (3)	0.4105 (3)	0.045 (2)*
H212	0.2565 (4)	0.6593 (4)	0.3560 (3)	0.045 (2)*
H213	0.3542 (5)	0.6126 (2)	0.3461 (3)	0.045 (2)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.01407 (19)	0.01375 (19)	0.0182 (2)	0.00091 (15)	0.00335 (15)	0.00001 (15)
O2	0.0180 (2)	0.0243 (2)	0.0155 (2)	0.00446 (17)	−0.00207 (16)	0.00068 (17)
N	0.0160 (2)	0.0153 (2)	0.0169 (2)	0.00227 (17)	0.00319 (17)	−0.00101 (17)
C1	0.0140 (2)	0.0174 (3)	0.0151 (2)	−0.00090 (19)	0.00074 (18)	0.0011 (2)
C2	0.0229 (3)	0.0245 (3)	0.0160 (3)	−0.0010 (2)	0.0043 (2)	−0.0023 (2)
C3	0.0312 (4)	0.0365 (4)	0.0283 (4)	0.0017 (3)	0.0104 (3)	−0.0114 (3)
C4	0.0266 (4)	0.0364 (4)	0.0352 (4)	0.0043 (3)	0.0136 (3)	−0.0058 (3)
C5	0.0201 (3)	0.0224 (3)	0.0365 (4)	0.0065 (2)	0.0073 (3)	0.0021 (3)
C6	0.0149 (2)	0.0178 (3)	0.0225 (3)	0.0018 (2)	0.0017 (2)	0.0026 (2)
C7	0.0322 (4)	0.0336 (4)	0.0199 (3)	−0.0027 (3)	0.0016 (3)	0.0078 (3)
C8	0.0280 (3)	0.0342 (4)	0.0200 (3)	−0.0048 (3)	0.0009 (3)	−0.0077 (3)
C9	0.0187 (3)	0.0198 (3)	0.0345 (4)	−0.0017 (2)	0.0003 (3)	−0.0003 (3)
C10	0.0185 (3)	0.0396 (4)	0.0235 (3)	0.0032 (3)	−0.0020 (2)	0.0076 (3)
C11	0.0140 (2)	0.0175 (3)	0.0155 (2)	0.0000 (2)	0.00058 (19)	0.00121 (19)
C12	0.0195 (3)	0.0183 (3)	0.0199 (3)	−0.0009 (2)	−0.0012 (2)	0.0025 (2)
C13	0.0243 (3)	0.0248 (3)	0.0235 (3)	0.0001 (3)	−0.0014 (2)	0.0078 (2)
C14	0.0249 (3)	0.0343 (4)	0.0177 (3)	0.0030 (3)	−0.0017 (2)	0.0053 (3)
C15	0.0228 (3)	0.0299 (3)	0.0169 (3)	0.0027 (3)	−0.0019 (2)	−0.0024 (2)
C16	0.0191 (3)	0.0200 (3)	0.0181 (3)	0.0001 (2)	0.0001 (2)	−0.0011 (2)
C17	0.0157 (3)	0.0247 (3)	0.0151 (3)	0.0034 (2)	−0.0002 (2)	0.0011 (2)
C18	0.0189 (3)	0.0184 (3)	0.0184 (3)	0.0004 (2)	−0.0027 (2)	0.0026 (2)
C19	0.0210 (3)	0.0351 (4)	0.0333 (4)	0.0090 (3)	0.0043 (3)	0.0111 (3)

C20	0.0322 (4)	0.0299 (4)	0.0214 (3)	0.0024 (3)	−0.0091 (3)	0.0007 (3)
C21	0.0422 (5)	0.0208 (3)	0.0332 (4)	−0.0059 (3)	−0.0137 (3)	0.0053 (3)

Geometric parameters (Å, º)

O1—C1	1.4422 (8)	C9—H92	0.980
O1—N	1.4595 (7)	C9—H93	0.980
O2—C17	1.4158 (8)	C10—H101	1.000
O2—C18	1.4446 (8)	C10—H102	1.000
N—C6	1.4987 (9)	C10—H103	1.000
N—C2	1.5033 (9)	C11—C12	1.3945 (10)
C1—C11	1.5168 (9)	C11—C16	1.3975 (9)
C1—C17	1.5228 (9)	C12—C13	1.3955 (10)
C1—H1	1.000	C12—H12	0.950
C2—C8	1.5354 (11)	C13—C14	1.3883 (12)
C2—C7	1.5377 (12)	C13—H13	0.950
C2—C3	1.5399 (11)	C14—C15	1.3906 (12)
C3—C4	1.5198 (14)	C14—H14	0.950
C3—H31	0.990	C15—C16	1.3924 (10)
C3—H32	0.990	C15—H15	0.950
C4—C5	1.5153 (13)	C16—H16	0.950
C4—H41	0.990	C17—H171	0.990
C4—H42	0.990	C17—H172	0.990
C5—C6	1.5312 (10)	C18—C20	1.5187 (10)
C5—H51	0.990	C18—C21	1.5200 (11)
C5—H52	0.990	C18—C19	1.5238 (11)
C6—C10	1.5280 (10)	C19—H191	1.000
C6—C9	1.5353 (11)	C19—H192	1.000
C7—H71	0.980	C19—H193	1.000
C7—H72	0.980	C20—H201	1.000
C7—H73	0.980	C20—H202	1.000
C8—H81	0.990	C20—H203	1.000
C8—H82	0.990	C21—H211	0.990
C8—H83	0.990	C21—H212	0.990
C9—H91	0.980	C21—H213	0.990
C1—O1—N	112.33 (5)	H91—C9—H93	109.5
C17—O2—C18	116.92 (5)	H92—C9—H93	109.5
O1—N—C6	106.17 (5)	C6—C10—H101	109.5
O1—N—C2	107.19 (5)	C6—C10—H102	109.5
C6—N—C2	117.35 (5)	H101—C10—H102	109.5
O1—C1—C11	114.45 (5)	C6—C10—H103	109.5
O1—C1—C17	105.37 (5)	H101—C10—H103	109.5
C11—C1—C17	110.04 (5)	H102—C10—H103	109.5
O1—C1—H1	108.9	C12—C11—C16	118.62 (6)
C11—C1—H1	108.9	C12—C11—C1	119.94 (6)
C17—C1—H1	108.9	C16—C11—C1	121.40 (6)
N—C2—C8	108.10 (5)	C11—C12—C13	120.97 (7)

N—C2—C7	114.93 (6)	C11—C12—H12	119.5
C8—C2—C7	107.59 (7)	C13—C12—H12	119.5
N—C2—C3	107.44 (6)	C14—C13—C12	119.75 (7)
C8—C2—C3	106.83 (6)	C14—C13—H13	120.1
C7—C2—C3	111.61 (6)	C12—C13—H13	120.1
C4—C3—C2	114.33 (7)	C13—C14—C15	119.90 (7)
C4—C3—H31	108.7	C13—C14—H14	120.0
C2—C3—H31	108.7	C15—C14—H14	120.1
C4—C3—H32	108.7	C14—C15—C16	120.16 (7)
C2—C3—H32	108.7	C14—C15—H15	119.9
H31—C3—H32	107.6	C16—C15—H15	119.9
C5—C4—C3	108.36 (7)	C15—C16—C11	120.59 (7)
C5—C4—H41	110.0	C15—C16—H16	119.7
C3—C4—H41	110.0	C11—C16—H16	119.7
C5—C4—H42	110.0	O2—C17—C1	108.48 (5)
C3—C4—H42	110.0	O2—C17—H171	110.0
H41—C4—H42	108.4	C1—C17—H171	110.0
C4—C5—C6	112.78 (6)	O2—C17—H172	110.0
C4—C5—H51	109.0	C1—C17—H172	110.0
C6—C5—H51	109.0	H171—C17—H172	108.4
C4—C5—H52	109.1	O2—C18—C20	103.38 (6)
C6—C5—H52	109.0	O2—C18—C21	111.07 (6)
H51—C5—H52	107.8	C20—C18—C21	110.35 (6)
N—C6—C10	107.23 (5)	O2—C18—C19	111.02 (6)
N—C6—C5	107.65 (6)	C20—C18—C19	110.12 (7)
C10—C6—C5	107.62 (6)	C21—C18—C19	110.68 (7)
N—C6—C9	116.15 (5)	C18—C19—H191	109.5
C10—C6—C9	107.67 (6)	C18—C19—H192	109.5
C5—C6—C9	110.19 (6)	H191—C19—H192	109.5
C2—C7—H71	109.5	C18—C19—H193	109.5
C2—C7—H72	109.5	H191—C19—H193	109.5
H71—C7—H72	109.5	H192—C19—H193	109.5
C2—C7—H73	109.5	C18—C20—H201	109.5
H71—C7—H73	109.5	C18—C20—H202	109.5
H72—C7—H73	109.5	H201—C20—H202	109.5
C2—C8—H81	109.5	C18—C20—H203	109.5
C2—C8—H82	109.5	H201—C20—H203	109.5
H81—C8—H82	109.5	H202—C20—H203	109.5
C2—C8—H83	109.5	C18—C21—H211	109.5
H81—C8—H83	109.5	C18—C21—H212	109.5
H82—C8—H83	109.5	H211—C21—H212	109.5
C6—C9—H91	109.5	C18—C21—H213	109.5
C6—C9—H92	109.5	H211—C21—H213	109.5
H91—C9—H92	109.5	H212—C21—H213	109.5
C6—C9—H93	109.5		

C1—O1—N—C6	−128.03 (5)	O1—N—C6—C5	−174.94 (5)
C1—O1—N—C2	105.78 (6)	C2—N—C6—C5	−55.17 (7)

N—O1—C1—C11	86.82 (6)	O1—N—C6—C9	−50.91 (7)
N—O1—C1—C17	−152.16 (5)	C2—N—C6—C9	68.86 (8)
O1—N—C2—C8	−72.99 (7)	C4—C5—C6—N	56.34 (8)
C6—N—C2—C8	167.78 (6)	C4—C5—C6—C10	171.63 (7)
O1—N—C2—C7	47.18 (7)	C4—C5—C6—C9	−71.23 (8)
C6—N—C2—C7	−72.05 (8)	O1—C1—C11—C12	−117.89 (6)
O1—N—C2—C3	172.05 (5)	C17—C1—C11—C12	123.70 (7)
C6—N—C2—C3	52.82 (8)	O1—C1—C11—C16	64.45 (8)
N—C2—C3—C4	−52.39 (9)	C17—C1—C11—C16	−53.96 (8)
C8—C2—C3—C4	−168.19 (7)	C18—O2—C17—C1	−179.32 (5)
C7—C2—C3—C4	74.46 (9)	O1—C1—C17—O2	72.26 (6)
C2—C3—C4—C5	56.39 (10)	C11—C1—C17—O2	−163.88 (5)
C3—C4—C5—C6	−57.99 (9)	C17—O2—C18—C20	−173.50 (6)
O1—N—C6—C10	69.52 (6)	C17—O2—C18—C21	68.17 (8)
C2—N—C6—C10	−170.72 (6)	C17—O2—C18—C19	−55.45 (8)