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5-Methoxy-1,2′,3-trimethyl-4,6-dioxa-2-azaspiro[bicyclo[3.2.0]hept-2-ene-7,4′-isoquinoline]-1′,3′(2′*H*,4′*H*)-dione

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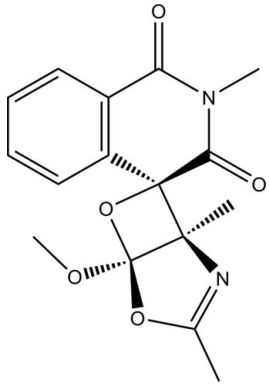
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.001 Å; R factor = 0.049; wR factor = 0.126; data-to-parameter ratio = 39.0.

In the isoquinoline ring system of the title molecule, $C_{16}H_{16}N_2O_5$, the *N*-heterocyclic ring is in a half-boat conformation. The dioxaazaspiro ring is essentially planar [maximum deviation = 0.022 (1) Å] and forms a dihedral angle of 24.56 (4)° with the benzene ring.

Related literature

For general background to and the potential biological activity of the title compound, see: Du *et al.* (2008); Chen *et al.* (2006); Yu *et al.* (2010); Harris *et al.* (2005); Zhang *et al.* (2004); Wang *et al.* (2010); Huang *et al.* (2011). For the stability of the temperature controller used in the data collection, see: Cosier & Glazer (1986). For standard bond-length data, see: Allen *et al.* (1987). For ring conformations, see: Cremer & Pople (1975). For related structures, see: Fun *et al.* (2011*a*,*b*,*c*).



Experimental

Crystal data

$C_{16}H_{16}N_2O_5$
M_r = 316.31
Monoclinic, $P2_1/c$
a = 7.3643 (1) Å
b = 29.8703 (6) Å
c = 6.7802 (1) Å
β = 105.294 (1)°
V = 1438.65 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 100 K
0.46 × 0.31 × 0.27 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.951, T_{max} = 0.971
44501 measured reflections
8265 independent reflections
6842 reflections with $I > 2\sigma(I)$
R_{int} = 0.033

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.126
S = 1.08
8265 reflections
212 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.50 e Å^{-3}
$\Delta\rho_{min}$ = −0.37 e Å^{-3}

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH5241).

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supporting information

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5-Methoxy-1,2′,3-trimethyl-4,6-dioxa-2-azaspiro[bicyclo[3.2.0]hept-2-ene-7,4′-isoquinoline]-1′,3′(2′*H*,4′*H*)-dione

Hoong-Kun Fun, Ching Kheng Quah, Chengmei Huang and Haitao Yu

S1. Comment

Isoquinoline-1,3,4-trione derivatives have been reported to be a type of small molecular inhibitor against caspase-3 which can promote apoptosis of the cells (Du *et al.*, 2008; Chen *et al.*, 2006). Photoreactions of isoquinoline-1,3,4-trione could lead to structurally important motifs (Yu *et al.*, 2010). Oxazole ring is found in some bioactive natural products such as Annuloline and Ostreogrycin A. Oxazoles can be used to inhibit the activity of malignant tumors (Harris *et al.*, 2005). Since a lot of natural products especially the alkaloids containing isoquinoline or oxazole ring are bioactive, convenient method to construct such moieties is of current research interest (Zhang *et al.*, 2004; Wang *et al.*, 2010). The title compound which was derived from isoquinoline-1,3,4-trione and oxazoles (Huang *et al.*, 2011) may has a potential use in biochemical and pharmaceutical fields. We report in this paper the crystal structure of the title compound with a relative configuration of (1S*, 4′S*, 5R*).

In the title racemic compound, Fig. 1, atoms C9, C10 and C12 are the stereo centers. The isoquinoline ring system (N1/C1-C9) is not completely planar, the *N*-heterocyclic ring (N1/C1-C3/C8/C9) being distorted towards a half-boat conformation with atom C9 deviating by 0.231 (1) Å from the mean plane through the remaining atoms, puckering parameters (Cremer & Pople, 1975) Q = 0.3501 (8) Å, Θ = 112.83 (13)° and φ = 282.17 (13)°. The dioxa-2-azaspiro ring (N2/O4/C10-C12) is essentially planar [maximum deviation of 0.022 (1) Å at atom C10] and it inclines at a dihedral angle of 24.56 (4)° with the benzene ring (C3-C8). Bond lengths (Allen *et al.*, 1987) and angles are within normal ranges and comparable to related structures (Fun *et al.*, 2011*a*,*b*,*c*). In the crystal of the title compound, unlike in our previously determined structures mentioned above, there are no significant intermolecular hydrogen bonds.

S2. Experimental

The title compound was the main product from the photoreaction between isoquinoline-1,3,4-trione and 4-methyl-5-methoxy-2-methyloxazole. The compound was purified by flash column chromatography with ethyl acetate/petroleum ether (1:3) as eluents. X-ray quality crystals of the title compound was obtained from slow evaporation of an acetone and petroleum ether solution (1:4) (*m.p.* 437-439 K).

S3. Refinement

All H atoms were positioned geometrically and refined using a riding model with C–H = 0.93 - 0.96 Å and U_{iso}(H) = 1.2 or 1.5 U_{eq}(C). A rotating-group model was applied for the methyl groups. The highest residual electron density peak is located at 0.77 Å from C9 and the deepest hole is located at 0.52 Å from C2.

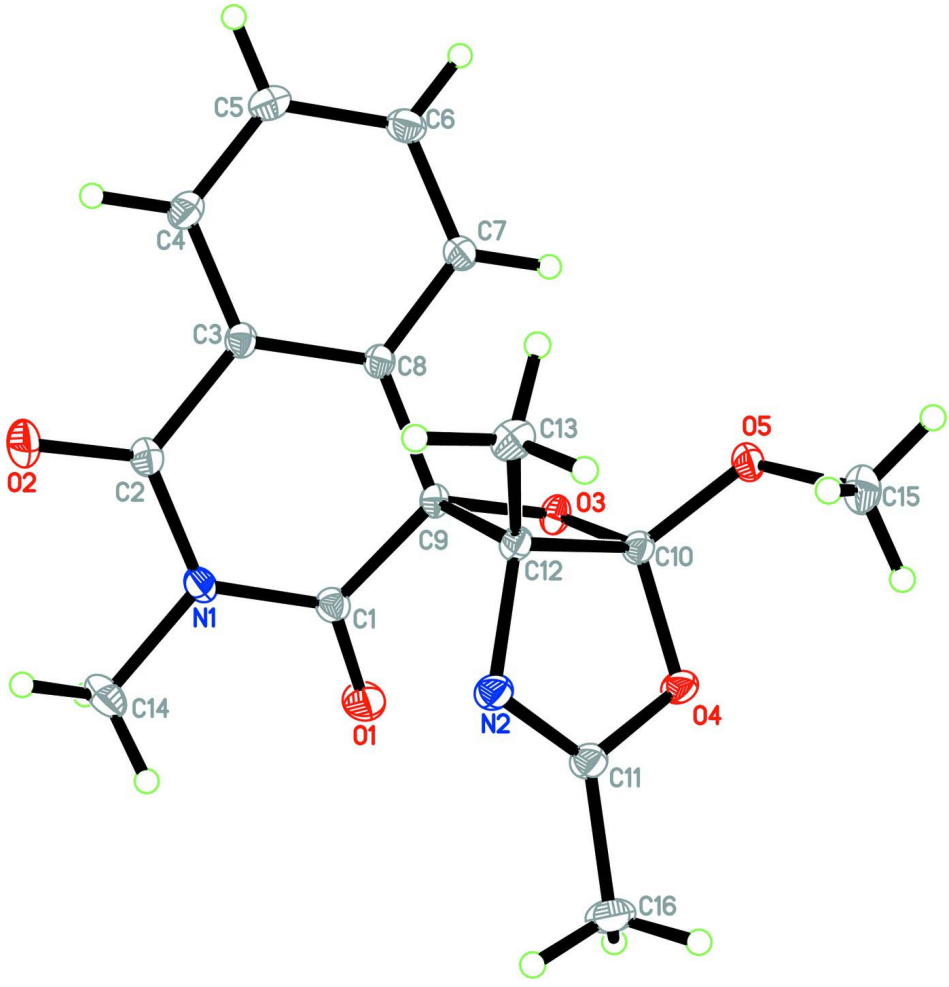

Figure 1

The molecular structure of the title compound showing 50% probability displacement ellipsoids for non-H atoms.

5-Methoxy-1,2′,3-trimethyl-4,6-dioxa-2-azaspiro[bicyclo[3.2.0]hept-2-ene- 7,4′-isoquinoline]-1′,3′(2′*H*,4′*H*)-dione

Crystal data

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Monoclinic, $P2_1/c$
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$a = 7.3643$ (1) Å
$b = 29.8703$ (6) Å
$c = 6.7802$ (1) Å
$\beta = 105.294$ (1)°
$V = 1438.65$ (4) Å^3
$Z = 4$

$F(000) = 664$
$D_x = 1.460$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 9541 reflections
$\theta = 2.7$–$38.9°$
$\mu = 0.11$ mm^{-1}
$T = 100$ K
Block, colourless
0.46 × 0.31 × 0.27 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.951, T_{max} = 0.971
44501 measured reflections
8265 independent reflections
6842 reflections with $I > 2\sigma(I)$
R_{int} = 0.033
θ_{max} = 39.1°, θ_{min} = 2.7°
h = −13→11
k = −52→52
l = −10→12

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.126
S = 1.08
8265 reflections
212 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
$w = 1/[\sigma^2(F_o^2) + (0.0561P)^2 + 0.4048P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.50 e Å^{-3}
$\Delta\rho_{min}$ = −0.37 e Å^{-3}

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cryosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 100.0 (1) K.

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > 2\text{sigma}(F^2)$ 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}
O1	0.98692 (10)	0.07763 (2)	1.21672 (11)	0.01961 (12)
O2	0.97185 (10)	0.22835 (2)	1.29761 (11)	0.02036 (12)
O3	0.64272 (8)	0.074342 (18)	0.93351 (9)	0.01245 (10)
O4	0.80974 (8)	0.037356 (19)	0.72911 (9)	0.01405 (10)
O5	0.50850 (8)	0.06418 (2)	0.59672 (9)	0.01442 (10)
N1	0.99366 (9)	0.15373 (2)	1.23860 (10)	0.01288 (11)
N2	0.98329 (9)	0.10159 (2)	0.78175 (11)	0.01329 (11)
C1	0.91387 (11)	0.11323 (3)	1.15734 (11)	0.01266 (12)
C2	0.89437 (11)	0.19420 (3)	1.22005 (11)	0.01288 (12)
C3	0.68983 (10)	0.19200 (2)	1.11676 (11)	0.01123 (11)
C4	0.57505 (11)	0.22751 (2)	1.14283 (12)	0.01418 (12)
H4A	0.6279	0.2526	1.2171	0.017*
C5	0.38157 (12)	0.22526 (3)	1.05764 (13)	0.01604 (13)
H5A	0.3048	0.2489	1.0743	0.019*

C6	0.30314 (11)	0.18742 (3)	0.94723 (12)	0.01532 (13)
H6A	0.1739	0.1860	0.8893	0.018*
C7	0.41660 (11)	0.15171 (3)	0.92290 (11)	0.01323 (12)
H7A	0.3630	0.1264	0.8506	0.016*
C8	0.61080 (10)	0.15391 (2)	1.00719 (10)	0.01055 (11)
C9	0.73909 (10)	0.11699 (2)	0.97884 (11)	0.01041 (11)
C10	0.67607 (10)	0.07134 (2)	0.73762 (11)	0.01101 (11)
C11	0.98054 (11)	0.05895 (3)	0.76219 (12)	0.01374 (12)
C12	0.79021 (10)	0.11558 (2)	0.76495 (11)	0.01075 (11)
C13	0.72971 (12)	0.15463 (3)	0.62183 (12)	0.01482 (13)
H13A	0.7495	0.1475	0.4909	0.022*
H13B	0.8025	0.1806	0.6771	0.022*
H13C	0.5986	0.1607	0.6063	0.022*
C14	1.18102 (12)	0.15176 (3)	1.38319 (13)	0.01961 (15)
H14A	1.2585	0.1311	1.3338	0.029*
H14B	1.1695	0.1420	1.5143	0.029*
H14C	1.2376	0.1809	1.3961	0.029*
C15	0.51787 (13)	0.05535 (3)	0.39061 (13)	0.02037 (15)
H15A	0.3927	0.0530	0.3024	0.031*
H15B	0.5840	0.0278	0.3874	0.031*
H15C	0.5831	0.0794	0.3447	0.031*
C16	1.14282 (12)	0.02931 (3)	0.76728 (17)	0.02177 (17)
H16A	1.2575	0.0462	0.8101	0.033*
H16B	1.1314	0.0173	0.6333	0.033*
H16C	1.1448	0.0053	0.8618	0.033*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0194 (3)	0.0141 (2)	0.0220 (3)	0.0030 (2)	−0.0004 (2)	0.0036 (2)
O2	0.0186 (3)	0.0147 (3)	0.0249 (3)	−0.0053 (2)	0.0006 (2)	−0.0044 (2)
O3	0.0150 (2)	0.0102 (2)	0.0132 (2)	−0.00369 (17)	0.00555 (17)	−0.00184 (17)
O4	0.0112 (2)	0.0101 (2)	0.0215 (3)	−0.00064 (17)	0.00536 (18)	−0.00282 (18)
O5	0.0113 (2)	0.0169 (2)	0.0144 (2)	−0.00187 (18)	0.00209 (17)	−0.00453 (19)
N1	0.0106 (2)	0.0138 (3)	0.0126 (2)	−0.00073 (19)	0.00022 (18)	−0.0006 (2)
N2	0.0113 (2)	0.0118 (2)	0.0179 (3)	−0.00080 (19)	0.0059 (2)	−0.0016 (2)
C1	0.0126 (3)	0.0126 (3)	0.0121 (3)	0.0000 (2)	0.0022 (2)	0.0005 (2)
C2	0.0132 (3)	0.0125 (3)	0.0123 (3)	−0.0019 (2)	0.0022 (2)	−0.0002 (2)
C3	0.0124 (3)	0.0104 (3)	0.0106 (2)	−0.0008 (2)	0.0026 (2)	0.0001 (2)
C4	0.0174 (3)	0.0106 (3)	0.0146 (3)	0.0006 (2)	0.0044 (2)	−0.0004 (2)
C5	0.0169 (3)	0.0143 (3)	0.0176 (3)	0.0039 (2)	0.0058 (2)	0.0005 (2)
C6	0.0117 (3)	0.0175 (3)	0.0164 (3)	0.0022 (2)	0.0031 (2)	0.0001 (2)
C7	0.0113 (3)	0.0146 (3)	0.0133 (3)	0.0000 (2)	0.0022 (2)	−0.0016 (2)
C8	0.0114 (3)	0.0107 (3)	0.0095 (2)	−0.0003 (2)	0.00277 (19)	−0.0006 (2)
C9	0.0110 (3)	0.0094 (2)	0.0107 (2)	−0.0013 (2)	0.00248 (19)	−0.0008 (2)
C10	0.0104 (3)	0.0102 (3)	0.0127 (3)	−0.0006 (2)	0.0035 (2)	−0.0015 (2)
C11	0.0116 (3)	0.0123 (3)	0.0183 (3)	−0.0008 (2)	0.0056 (2)	−0.0020 (2)
C12	0.0112 (3)	0.0098 (3)	0.0117 (3)	−0.0006 (2)	0.0040 (2)	−0.0009 (2)

C13	0.0187 (3)	0.0124 (3)	0.0145 (3)	0.0010 (2)	0.0064 (2)	0.0021 (2)
C14	0.0131 (3)	0.0241 (4)	0.0178 (3)	−0.0014 (3)	−0.0026 (2)	0.0012 (3)
C15	0.0215 (4)	0.0237 (4)	0.0149 (3)	−0.0007 (3)	0.0030 (3)	−0.0056 (3)
C16	0.0140 (3)	0.0152 (3)	0.0374 (5)	0.0019 (3)	0.0092 (3)	−0.0041 (3)

Geometric parameters (Å, º)

O1—C1	1.2116 (10)	C6—C7	1.3914 (11)
O2—C2	1.2183 (9)	C6—H6A	0.9300
O3—C10	1.4158 (9)	C7—C8	1.3946 (10)
O3—C9	1.4512 (9)	C7—H7A	0.9300
O4—C11	1.3784 (9)	C8—C9	1.4972 (10)
O4—C10	1.4257 (9)	C9—C12	1.5921 (10)
O5—C10	1.3636 (9)	C10—C12	1.5508 (10)
O5—C15	1.4416 (10)	C11—C16	1.4804 (11)
N1—C1	1.3933 (10)	C12—C13	1.5075 (10)
N1—C2	1.4012 (10)	C13—H13A	0.9600
N1—C14	1.4679 (10)	C13—H13B	0.9600
N2—C11	1.2799 (10)	C13—H13C	0.9600
N2—C12	1.4574 (10)	C14—H14A	0.9600
C1—C9	1.5206 (10)	C14—H14B	0.9600
C2—C3	1.4857 (10)	C14—H14C	0.9600
C3—C4	1.3961 (10)	C15—H15A	0.9600
C3—C8	1.3990 (10)	C15—H15B	0.9600
C4—C5	1.3907 (12)	C15—H15C	0.9600
C4—H4A	0.9300	C16—H16A	0.9600
C5—C6	1.3941 (12)	C16—H16B	0.9600
C5—H5A	0.9300	C16—H16C	0.9600
C10—O3—C9	93.33 (5)	O5—C10—O4	111.53 (6)
C11—O4—C10	105.68 (6)	O3—C10—O4	112.06 (6)
C10—O5—C15	116.23 (6)	O5—C10—C12	125.39 (6)
C1—N1—C2	123.91 (6)	O3—C10—C12	93.25 (5)
C1—N1—C14	116.97 (7)	O4—C10—C12	104.67 (6)
C2—N1—C14	118.08 (7)	N2—C11—O4	118.12 (7)
C11—N2—C12	106.79 (6)	N2—C11—C16	127.02 (7)
O1—C1—N1	121.77 (7)	O4—C11—C16	114.85 (7)
O1—C1—C9	122.53 (7)	N2—C12—C13	112.88 (6)
N1—C1—C9	115.52 (6)	N2—C12—C10	104.58 (6)
O2—C2—N1	120.65 (7)	C13—C12—C10	121.57 (6)
O2—C2—C3	122.79 (7)	N2—C12—C9	113.37 (6)
N1—C2—C3	116.38 (6)	C13—C12—C9	117.72 (6)
C4—C3—C8	120.27 (7)	C10—C12—C9	83.13 (5)
C4—C3—C2	118.63 (7)	C12—C13—H13A	109.5
C8—C3—C2	120.95 (6)	C12—C13—H13B	109.5
C5—C4—C3	119.93 (7)	H13A—C13—H13B	109.5
C5—C4—H4A	120.0	C12—C13—H13C	109.5
C3—C4—H4A	120.0	H13A—C13—H13C	109.5

C4—C5—C6	119.74 (7)	H13B—C13—H13C	109.5
C4—C5—H5A	120.1	N1—C14—H14A	109.5
C6—C5—H5A	120.1	N1—C14—H14B	109.5
C7—C6—C5	120.57 (7)	H14A—C14—H14B	109.5
C7—C6—H6A	119.7	N1—C14—H14C	109.5
C5—C6—H6A	119.7	H14A—C14—H14C	109.5
C6—C7—C8	119.88 (7)	H14B—C14—H14C	109.5
C6—C7—H7A	120.1	O5—C15—H15A	109.5
C8—C7—H7A	120.1	O5—C15—H15B	109.5
C7—C8—C3	119.60 (7)	H15A—C15—H15B	109.5
C7—C8—C9	121.75 (6)	O5—C15—H15C	109.5
C3—C8—C9	118.62 (6)	H15A—C15—H15C	109.5
O3—C9—C8	112.58 (6)	H15B—C15—H15C	109.5
O3—C9—C1	111.58 (6)	C11—C16—H16A	109.5
C8—C9—C1	112.49 (6)	C11—C16—H16B	109.5
O3—C9—C12	90.22 (5)	H16A—C16—H16B	109.5
C8—C9—C12	116.44 (6)	C11—C16—H16C	109.5
C1—C9—C12	111.66 (6)	H16A—C16—H16C	109.5
O5—C10—O3	108.56 (6)	H16B—C16—H16C	109.5

C2—N1—C1—O1	−161.40 (8)	O1—C1—C9—C12	−81.94 (9)
C14—N1—C1—O1	6.68 (11)	N1—C1—C9—C12	93.26 (7)
C2—N1—C1—C9	23.35 (10)	C15—O5—C10—O3	−173.45 (6)
C14—N1—C1—C9	−168.56 (7)	C15—O5—C10—O4	−49.51 (9)
C1—N1—C2—O2	178.82 (8)	C15—O5—C10—C12	78.38 (9)
C14—N1—C2—O2	10.86 (11)	C9—O3—C10—O5	−126.97 (6)
C1—N1—C2—C3	3.52 (10)	C9—O3—C10—O4	109.41 (6)
C14—N1—C2—C3	−164.44 (7)	C9—O3—C10—C12	2.15 (6)
O2—C2—C3—C4	−12.66 (11)	C11—O4—C10—O5	142.09 (6)
N1—C2—C3—C4	162.54 (7)	C11—O4—C10—O3	−95.97 (7)
O2—C2—C3—C8	171.75 (7)	C11—O4—C10—C12	3.77 (7)
N1—C2—C3—C8	−13.05 (10)	C12—N2—C11—O4	0.91 (9)
C8—C3—C4—C5	−0.74 (11)	C12—N2—C11—C16	179.69 (8)
C2—C3—C4—C5	−176.36 (7)	C10—O4—C11—N2	−3.21 (9)
C3—C4—C5—C6	0.27 (12)	C10—O4—C11—C16	177.86 (7)
C4—C5—C6—C7	0.53 (12)	C11—N2—C12—C13	−132.59 (7)
C5—C6—C7—C8	−0.85 (12)	C11—N2—C12—C10	1.60 (8)
C6—C7—C8—C3	0.38 (11)	C11—N2—C12—C9	90.33 (7)
C6—C7—C8—C9	−177.87 (7)	O5—C10—C12—N2	−133.99 (7)
C4—C3—C8—C7	0.41 (10)	O3—C10—C12—N2	110.46 (6)
C2—C3—C8—C7	175.93 (7)	O4—C10—C12—N2	−3.35 (7)
C4—C3—C8—C9	178.71 (7)	O5—C10—C12—C13	−4.83 (10)
C2—C3—C8—C9	−5.77 (10)	O3—C10—C12—C13	−120.39 (7)
C10—O3—C9—C8	116.84 (6)	O4—C10—C12—C13	125.81 (7)
C10—O3—C9—C1	−115.57 (6)	O5—C10—C12—C9	113.59 (7)
C10—O3—C9—C12	−2.09 (5)	O3—C10—C12—C9	−1.97 (5)
C7—C8—C9—O3	−23.21 (9)	O4—C10—C12—C9	−115.78 (6)
C3—C8—C9—O3	158.52 (6)	O3—C9—C12—N2	−101.04 (6)

C7—C8—C9—C1	−150.32 (7)	C8—C9—C12—N2	143.45 (6)
C3—C8—C9—C1	31.42 (9)	C1—C9—C12—N2	12.37 (8)
C7—C8—C9—C12	78.99 (8)	O3—C9—C12—C13	124.09 (7)
C3—C8—C9—C12	−99.28 (7)	C8—C9—C12—C13	8.58 (9)
O1—C1—C9—O3	17.35 (10)	C1—C9—C12—C13	−122.51 (7)
N1—C1—C9—O3	−167.44 (6)	O3—C9—C12—C10	1.92 (5)
O1—C1—C9—C8	144.99 (8)	C8—C9—C12—C10	−113.59 (6)
N1—C1—C9—C8	−39.81 (9)	C1—C9—C12—C10	115.32 (6)