Acta Cryst. (1999). C**55**, IUC9900111 [**doi:10.1107/S0108270199098807**]

(±)-(3'aα,3'bα,6'aα,7'aα)- and (±)-(3'aα,3'bβ,6'aβ,7'aα)-2,3,3a,3b,4,5,6,6a,7,7a-decahydro-5,5-dimethylspiro[1,3-dioxane-2,5'-pentaleno[2,1-*b*]furan]-2'-one

J. W. Bats, S. H. Öhlinger and J. Mulzer

Abstract

The crystal structures of the title compounds, $C_{15}H_{22}O_4$ have been determined at 134 K and 135 K respectively.. The pentaleno[2,1-*b*]furane group has an *endo*-conformation for the (3'aα,3'bα,6'aα,7'aα)-isomer and an *exo*-conformation for the (3'aα,3'bβ,6'aβ,7'aα)-isomer. The axial C—C bond attached to the spiro center is about 0.02 Å longer than the equatorial C—C bond.

Comment

The three five-membered rings are *cis*-connected about the C3—C4 and the C6—C7 bonds. The five-membered lactone ring approximately has a C3-envelope conformation in both compounds. The five-membered ring labeled C3 through C7 has conformation intermediate between a C4-envelope and a C4,C5-twist in (I) and a C5,C6-twist conformation in (II). The five-membered ring labeled C6 through C10 has a conformation intermediate between a C9-envelope and a C9,C10-twist in (I) and a conformation intermediate between a C8-envelope and a C7,C8-twist in (II). The C3—C7 (1.562 (1) Å) and the C6—C7 (1.573 (1) Å) bonds in (I), which have almost eclipsed conformations, are both slightly lengthened compared with a standard C—C single bond of 1.54 Å. The six-membered dioxane ring has a chair conformation in both compounds. The axial C—C bond attached to the spiro center [C9—C8 for (I) and C9—C10 for (II)] is about 0.02 Å longer than the equatorial C—C bond [C9—C10 for (I) and C9—C8 for (II)]. This bond length difference may result from a short repulsive steric interaction between H8A and H13B with a distance of 2.20 (2) Å in (I) or from a short repulsive steric interaction between H10A and H13B with a distance of only 2.02 (2) Å in (II). Similar differences between axial and equatorial bond lengths have been reported for other spiro-connected dioxane derivatives (Solans *et al.*, 1985, Bats *et al.*, 1999). The intramolecular H8B···C1 and H8B···C2 distances of 2.55 (1) Å and 2.57 (1) Å in (I) approach the van der Waals contact distance. The shortest intermolecular O···H distance is 2.58 (1) Å in (I) and 2.56 (1) Å in (II).

Experimental

The oily starting material (±)-(3'aα,4'β,6'aα)-3',3'a,4',6'a-tetrahydro- 5,5,*N*,*N*-tetramethyl-spiro[1,3-dioxane-2,2'(1'*H*)-pentalene]-4'-acetamide [for (I)] or the crystalline starting material (±)-(3'aα,4'α,6'aα)-3',3'a, 4',6'a-tetrahydro-5,5,*N*,*N*-tetramethyl-spiro[1,3-dioxane-2,2'(1'H)-pentalene]- 4'-acetamide [for (II)] (Bats *et al.*, 1999) was subjected to a iodolactonization reaction. The resulting iodolactone as main product was separated from the reaction mixture (Mulzer & Öhlinger, 1999) by column chromatography and was subsequently hydrogenolysed to the corresponding title compound (Deslongchamps *et al.*, 1978). Crystals suitable for data collection were obtained at 277 K from a solution in diethylether by slowly adding pentane.

CIF access

Refinement

H atoms of both compounds were refined with isotropic displacement parameters.

Computing details

For both compounds, data collection: *SMART* (Siemens, 1995); cell refinement: *SMART* (Siemens, 1995); data reduction: *SAINT* (Siemens, 1995); program(s) used to solve structure: *SHELXTL* (Sheldrick, 1996); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); software used to prepare material for publication: CIF in *SHELXL97* (Sheldrick, 1997)

(±)-(3'aα,3'bα,6'aα,7'aα)-octahydro-5,5-dimethyl-spiro[1,3-dioxane- 2,5'(2'*H*)pentaleno[2,1-*b*]furan]-2'-one

Crystal data

$C_{15}H_{22}O_4$	γ = 70.162 (11)°
M_r = 266.33	V = 687.98 (19) Å^3
Triclinic, $P\bar{1}$	Z = 2
a = 5.6779 (10) Å	Mo $K\alpha$
b = 6.3017 (11) Å	μ = 0.09 mm^{-1}
c = 20.480 (3) Å	T = 134 (2) K
α = 88.370 (11)°	0.85 × 0.36 × 0.34 mm
β = 86.483 (7)°	

Data collection

Siemens SMART diffractometer	4038 independent reflections
Absorption correction: numerical (Sheldrick, 1996)	3582 reflections with $I > 2\sigma(I)$
T_{min} = 0.943, T_{max} = 0.970	R_{int} = 0.038
11957 measured reflections	

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039	261 parameters
$wR(F^2)$ = 0.125	All H-atom parameters refined
S = 1.56	$\Delta\rho_{max}$ = 0.42 e Å^{-3}
4038 reflections	$\Delta\rho_{min}$ = −0.22 e Å^{-3}

(±)-(3'aα,3'bβ,6'aβ,7'aα)-octahydro-5,5-dimethyl-spiro[1,3-dioxane- 2,5'(2'*H*)pentaleno[2,1-*b*]furan]-2'-one

Crystal data

$C_{15}H_{22}O_4$	V = 1351.5 (4) Å^3
M_r = 266.33	Z = 4

Monoclinic, $P2_1/c$	Mo $K\alpha$
a = 6.6228 (15) Å	μ = 0.09 mm^{-1}
b = 21.651 (3) Å	T = 135 (2) K
c = 9.5738 (12) Å	0.85 × 0.40 × 0.08 mm
β = 100.106 (14)°	

Data collection

Siemens SMART diffractometer	3959 independent reflections
Absorption correction: numerical (Sheldrick, 1996)	2892 reflections with $I > 2\sigma(I)$
T_{min} = 0.953, T_{max} = 0.993	R_{int} = 0.052
22851 measured reflections	

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050	261 parameters
$wR(F^2)$ = 0.127	All H-atom parameters refined
S = 1.29	$\Delta\rho_{max}$ = 0.39 e Å^{-3}
3959 reflections	$\Delta\rho_{min}$ = −0.25 e Å^{-3}

References

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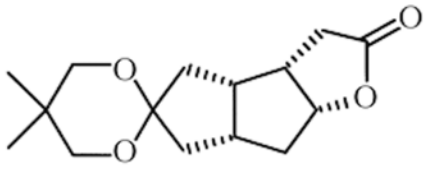
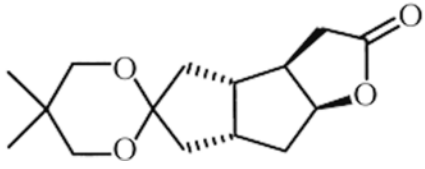
CIF access

Scheme 1



(I)



(II)

supplementary materials

(±)-(3'aα,3'bα,6'aα,7'aα)-octahydro-5,5-dimethyl-spiro[1,3-dioxane- 2,5'(2'*H*)pentaleno[2,1-*b*]furan]-2'-one

Crystal data

$C_{15}H_{22}O_4$	F_{000} = 288
M_r = 266.33	D_x = 1.286 Mg m^{-3}
Triclinic, $P\overline{1}$	Melting point: 358(1) K
a = 5.6779 (10) Å	Mo $K\alpha$ radiation λ = 0.71073 Å
b = 6.3017 (11) Å	Cell parameters from 186 reflections
c = 20.480 (3) Å	θ = 3–23º
α = 88.370 (11)º	μ = 0.09 mm^{-1}
β = 86.483 (7)º	T = 134 (2) K
γ = 70.162 (11)º	Prism, yellow
V = 687.98 (19) Å^3	0.85 × 0.36 × 0.34 mm
Z = 2	

Data collection

Siemens SMART diffractometer	4038 independent reflections
Radiation source: fine-focus sealed tube	3582 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.038
T = 134(2) K	θ_{max} = 30.6º
ω scans	θ_{min} = 2.0º
Absorption correction: numerical (Sheldrick, 1996)	h = −8→8
T_{min} = 0.943, T_{max} = 0.970	k = −9→8
11957 measured reflections	l = −30→26

Refinement

Refinement on F^2	Hydrogen site location: difference Fourier map
Least-squares matrix: full	All H-atom parameters refined
$R[F^2 > 2\sigma(F^2)]$ = 0.039	$w = 1/[\sigma^2(F_o^2) + (0.06P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.125	$(\Delta/\sigma)_{max}$ = 0.001
S = 1.56	$\Delta\rho_{max}$ = 0.42 e Å^{-3}
4038 reflections	$\Delta\rho_{min}$ = −0.22 e Å^{-3}
261 parameters	Extinction correction: SHELXL97, Fc^*=kFc[1+0.001xFc$^2\lambda^3$/sin(2θ)]$^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.019 (7)
Secondary atom site location: difference Fourier map	

Special details

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 $F^2 > 2\text{sigma}(F^2)$ 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 ($Å^2$)

	x	*y*	*z*	U_{iso}*/U_{eq}
O3	0.61349 (11)	0.34963 (10)	0.70213 (3)	0.01822 (15)
O4	0.73237 (11)	0.65651 (10)	0.66612 (3)	0.01993 (15)
O1	0.35795 (11)	0.66046 (11)	0.90795 (3)	0.02242 (16)
C12	0.93013 (16)	0.29285 (16)	0.61165 (4)	0.01990 (18)
C9	0.64785 (14)	0.55149 (14)	0.72087 (4)	0.01522 (17)
C10	0.39794 (14)	0.71640 (15)	0.74632 (4)	0.01813 (18)
C7	0.73411 (14)	0.76408 (14)	0.80838 (4)	0.01686 (17)
C11	0.83738 (16)	0.19124 (15)	0.67249 (4)	0.01944 (18)
C6	0.46926 (14)	0.89541 (14)	0.78180 (4)	0.01763 (18)
C8	0.81423 (14)	0.52473 (15)	0.77922 (4)	0.01761 (18)
O2	0.55222 (16)	0.30200 (13)	0.93613 (3)	0.03348 (19)
C13	0.95950 (17)	0.51212 (16)	0.63356 (4)	0.02159 (19)
C4	0.41960 (15)	0.86867 (15)	0.90052 (4)	0.01952 (18)
C3	0.70387 (14)	0.78402 (14)	0.88450 (4)	0.01828 (18)
C5	0.29863 (15)	1.00315 (15)	0.84195 (4)	0.02048 (19)
C1	0.56371 (18)	0.48610 (16)	0.92408 (4)	0.0229 (2)
C15	1.18409 (19)	0.12882 (19)	0.58659 (5)	0.0295 (2)
C2	0.78730 (17)	0.56475 (16)	0.92379 (4)	0.02267 (19)
C14	0.73848 (19)	0.3416 (2)	0.55886 (5)	0.0298 (2)
H10B	0.327 (2)	0.627 (2)	0.7773 (6)	0.024 (3)*
H13B	1.102 (2)	0.4811 (19)	0.6627 (6)	0.023 (3)*
H8B	0.772 (2)	0.416 (2)	0.8091 (6)	0.026 (3)*
H4	0.372 (2)	0.952 (2)	0.9436 (6)	0.024 (3)*
H7	0.855 (2)	0.8359 (18)	0.7906 (5)	0.016 (2)*
H6	0.479 (2)	1.0136 (19)	0.7501 (6)	0.019 (3)*
H3	0.779 (2)	0.892 (2)	0.8988 (6)	0.029 (3)*
H8A	0.997 (2)	0.464 (2)	0.7678 (6)	0.028 (3)*
H11B	0.977 (2)	0.144 (2)	0.7064 (6)	0.032 (3)*
H11A	0.788 (2)	0.064 (2)	0.6613 (6)	0.020 (3)*
H10A	0.285 (2)	0.772 (2)	0.7112 (6)	0.029 (3)*
H2B	0.814 (2)	0.597 (2)	0.9702 (7)	0.033 (3)*
H5B	0.123 (2)	1.005 (2)	0.8376 (7)	0.031 (3)*
H5A	0.294 (2)	1.161 (2)	0.8477 (6)	0.029 (3)*

H15A	1.242 (3)	0.195 (2)	0.5455 (7)	0.039 (3)*
H2A	0.941 (2)	0.447 (2)	0.9076 (6)	0.034 (3)*
H14C	0.576 (3)	0.442 (2)	0.5744 (7)	0.040 (4)*
H13A	0.990 (2)	0.596 (2)	0.5949 (7)	0.031 (3)*
H14B	0.711 (3)	0.209 (2)	0.5470 (7)	0.037 (3)*
H14A	0.797 (3)	0.411 (3)	0.5199 (9)	0.061 (5)*
H15C	1.318 (3)	0.101 (2)	0.6192 (7)	0.041 (4)*
H15B	1.159 (3)	−0.020 (3)	0.5766 (8)	0.056 (4)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O3	0.0178 (3)	0.0170 (3)	0.0207 (3)	−0.0076 (2)	0.0036 (2)	−0.0036 (2)
O4	0.0241 (3)	0.0173 (3)	0.0177 (3)	−0.0069 (2)	0.0027 (2)	0.0021 (2)
O1	0.0225 (3)	0.0234 (3)	0.0233 (3)	−0.0107 (3)	0.0019 (2)	−0.0008 (3)
C12	0.0197 (4)	0.0235 (4)	0.0170 (4)	−0.0083 (3)	0.0021 (3)	−0.0034 (3)
C9	0.0156 (3)	0.0147 (4)	0.0157 (4)	−0.0058 (3)	0.0000 (3)	0.0002 (3)
C10	0.0134 (3)	0.0180 (4)	0.0212 (4)	−0.0027 (3)	−0.0021 (3)	−0.0017 (3)
C7	0.0139 (3)	0.0176 (4)	0.0188 (4)	−0.0050 (3)	0.0001 (3)	−0.0013 (3)
C11	0.0202 (4)	0.0159 (4)	0.0208 (4)	−0.0048 (3)	0.0035 (3)	−0.0024 (3)
C6	0.0154 (3)	0.0151 (4)	0.0206 (4)	−0.0031 (3)	−0.0008 (3)	0.0004 (3)
C8	0.0151 (3)	0.0184 (4)	0.0167 (4)	−0.0020 (3)	−0.0015 (3)	−0.0012 (3)
O2	0.0562 (5)	0.0231 (4)	0.0231 (4)	−0.0168 (3)	0.0031 (3)	−0.0009 (3)
C13	0.0235 (4)	0.0243 (4)	0.0191 (4)	−0.0118 (3)	0.0052 (3)	−0.0017 (3)
C4	0.0183 (4)	0.0190 (4)	0.0208 (4)	−0.0059 (3)	0.0021 (3)	−0.0041 (3)
C3	0.0157 (3)	0.0195 (4)	0.0196 (4)	−0.0056 (3)	−0.0007 (3)	−0.0041 (3)
C5	0.0165 (4)	0.0171 (4)	0.0248 (4)	−0.0017 (3)	0.0000 (3)	−0.0028 (3)
C1	0.0322 (4)	0.0219 (4)	0.0138 (4)	−0.0085 (4)	0.0013 (3)	−0.0023 (3)
C15	0.0259 (4)	0.0313 (5)	0.0290 (5)	−0.0081 (4)	0.0097 (4)	−0.0076 (4)
C2	0.0236 (4)	0.0227 (4)	0.0183 (4)	−0.0029 (3)	−0.0035 (3)	−0.0017 (3)
C14	0.0314 (5)	0.0424 (6)	0.0182 (4)	−0.0151 (4)	−0.0032 (3)	−0.0027 (4)

Geometric parameters (Å, °)

O3—C9	1.4174 (10)	C9—C8	1.5388 (11)
O3—C11	1.4350 (10)	C10—C6	1.5353 (12)
O4—C9	1.4258 (10)	C7—C8	1.5462 (12)
O4—C13	1.4381 (11)	C7—C3	1.5618 (12)
O1—C1	1.3553 (11)	C7—C6	1.5725 (11)
O1—C4	1.4701 (11)	C6—C5	1.5400 (12)
C12—C15	1.5293 (13)	O2—C1	1.2017 (12)
C12—C13	1.5295 (13)	C4—C5	1.5089 (13)
C12—C11	1.5307 (13)	C4—C3	1.5356 (11)
C12—C14	1.5317 (12)	C3—C2	1.5233 (13)
C9—C10	1.5180 (11)	C1—C2	1.5111 (14)
C9—O3—C11	112.61 (6)	C3—C7—C6	106.61 (6)
C9—O4—C13	113.39 (6)	O3—C11—C12	111.14 (7)
C1—O1—C4	109.96 (7)	C10—C6—C5	115.88 (7)

C15—C12—C13	109.98 (7)	C10—C6—C7	104.73 (7)
C15—C12—C11	109.10 (8)	C5—C6—C7	105.99 (7)
C13—C12—C11	106.31 (7)	C9—C8—C7	104.33 (6)
C15—C12—C14	110.85 (8)	O4—C13—C12	111.25 (7)
C13—C12—C14	110.36 (8)	O1—C4—C5	110.62 (7)
C11—C12—C14	110.13 (8)	O1—C4—C3	103.80 (7)
O3—C9—O4	110.89 (6)	C5—C4—C3	106.89 (7)
O3—C9—C10	109.11 (6)	C2—C3—C4	101.49 (7)
O4—C9—C10	107.13 (7)	C2—C3—C7	116.96 (7)
O3—C9—C8	114.46 (7)	C4—C3—C7	105.05 (6)
O4—C9—C8	111.82 (6)	C4—C5—C6	106.18 (7)
C10—C9—C8	102.85 (6)	O2—C1—O1	121.14 (9)
C9—C10—C6	104.00 (6)	O2—C1—C2	129.18 (9)
C8—C7—C3	117.38 (7)	O1—C1—C2	109.69 (8)
C8—C7—C6	105.77 (6)	C1—C2—C3	103.31 (7)
C11—O3—C9—O4	57.48 (8)	C9—O4—C13—C12	56.35 (9)
C11—O3—C9—C10	175.25 (6)	C15—C12—C13—O4	−171.56 (7)
C11—O3—C9—C8	−70.16 (8)	C11—C12—C13—O4	−53.58 (9)
C13—O4—C9—O3	−56.47 (9)	C14—C12—C13—O4	65.83 (9)
C13—O4—C9—C10	−175.44 (6)	C1—O1—C4—C5	138.45 (7)
C13—O4—C9—C8	72.59 (8)	C1—O1—C4—C3	24.11 (9)
O3—C9—C10—C6	164.97 (7)	O1—C4—C3—C2	−33.34 (8)
O4—C9—C10—C6	−74.92 (8)	C5—C4—C3—C2	−150.31 (7)
C8—C9—C10—C6	43.06 (8)	O1—C4—C3—C7	88.91 (8)
C9—O3—C11—C12	−58.76 (9)	C5—C4—C3—C7	−28.06 (9)
C15—C12—C11—O3	173.45 (7)	C8—C7—C3—C2	7.45 (10)
C13—C12—C11—O3	54.89 (9)	C6—C7—C3—C2	125.73 (8)
C14—C12—C11—O3	−64.67 (10)	C8—C7—C3—C4	−104.15 (8)
C9—C10—C6—C5	−147.19 (7)	C6—C7—C3—C4	14.14 (9)
C9—C10—C6—C7	−30.83 (8)	O1—C4—C5—C6	−81.12 (8)
C8—C7—C6—C10	6.99 (9)	C3—C4—C5—C6	31.25 (9)
C3—C7—C6—C10	−118.66 (7)	C10—C6—C5—C4	94.00 (9)
C8—C7—C6—C5	130.00 (7)	C7—C6—C5—C4	−21.65 (9)
C3—C7—C6—C5	4.35 (9)	C4—O1—C1—O2	175.80 (8)
O3—C9—C8—C7	−156.50 (6)	C4—O1—C1—C2	−4.11 (9)
O4—C9—C8—C7	76.34 (8)	O2—C1—C2—C3	162.41 (9)
C10—C9—C8—C7	−38.30 (8)	O1—C1—C2—C3	−17.69 (9)
C3—C7—C8—C9	137.76 (7)	C4—C3—C2—C1	30.63 (8)
C6—C7—C8—C9	19.03 (8)	C7—C3—C2—C1	−82.98 (8)

(±)-(3'aα,3'bβ,6'aβ,7'aα)-octahydro-5,5-dimethyl-spiro[1,3-dioxane- 2,5'(2'*H*)pentaleno[2,1-*b*]furan]-2'-one

Crystal data

$C_{15}H_{22}O_4$	D_x = 1.309 Mg m^{-3}
M_r = 266.33	Melting point: 374(1) K
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å

a = 6.6228 (15) Å	Cell parameters from 148 reflections
b = 21.651 (3) Å	θ = 3–23º
c = 9.5738 (12) Å	μ = 0.09 mm^{-1}
β = 100.106 (14)º	T = 135 (2) K
V = 1351.5 (4) $Å^3$	Plate, colorless
Z = 4	0.85 × 0.40 × 0.08 mm
F_{000} = 576	

Data collection

Siemens SMART diffractometer	3959 independent reflections
Radiation source: fine-focus sealed tube	2892 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.052
T = 135(2) K	θ_{max} = 30.6º
ω scans	θ_{min} = 1.9º
Absorption correction: numerical (Sheldrick, 1996)	h = −9→9
T_{min} = 0.953, T_{max} = 0.993	k = −29→31
22851 measured reflections	l = −13→13

Refinement

Refinement on F^2	Hydrogen site location: difference Fourier map
Least-squares matrix: full	All H-atom parameters refined
$R[F^2 > 2\sigma(F^2)]$ = 0.050	$w = 1/[\sigma^2(F_o^2) + (0.06P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.127	$(\Delta/\sigma)_{max}$ = 0.001
S = 1.29	$\Delta\rho_{max}$ = 0.39 e $Å^{-3}$
3959 reflections	$\Delta\rho_{min}$ = −0.25 e $Å^{-3}$
261 parameters	Extinction correction: SHELXL97, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.0005 (18)
Secondary atom site location: difference Fourier map	

Special details

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 $F^2 > 2\text{sigma}(F^2)$ 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 ($Å^2$)

	x	*y*	*z*	U_{iso}*/U_{eq}
O4	0.54181 (13)	0.54729 (4)	0.71520 (9)	0.0210 (2)
O3	0.78971 (13)	0.58279 (4)	0.90071 (9)	0.0238 (2)
O1	1.14924 (13)	0.34868 (4)	0.62506 (9)	0.0237 (2)
O2	0.97058 (16)	0.28276 (5)	0.47314 (10)	0.0358 (3)
C9	0.74498 (18)	0.53869 (5)	0.79020 (13)	0.0184 (3)
C12	0.55589 (18)	0.65825 (5)	0.76797 (13)	0.0188 (3)
C8	0.7532 (2)	0.47388 (5)	0.85345 (13)	0.0190 (3)
C3	0.90956 (19)	0.37003 (6)	0.78066 (13)	0.0190 (3)
C6	0.94736 (19)	0.47047 (6)	0.66229 (14)	0.0213 (3)
C4	1.13314 (19)	0.38042 (6)	0.75839 (13)	0.0216 (3)
C7	0.79651 (19)	0.43145 (5)	0.73359 (12)	0.0171 (3)
C14	0.5553 (2)	0.72111 (6)	0.69549 (15)	0.0242 (3)
C11	0.7723 (2)	0.64492 (6)	0.84659 (15)	0.0229 (3)
C13	0.5047 (2)	0.60789 (6)	0.65563 (14)	0.0231 (3)
C5	1.1585 (2)	0.44859 (6)	0.73702 (15)	0.0248 (3)
C1	0.9857 (2)	0.31226 (6)	0.58153 (13)	0.0230 (3)
C2	0.8415 (2)	0.31441 (6)	0.68687 (14)	0.0230 (3)
C10	0.9027 (2)	0.53865 (6)	0.68848 (18)	0.0310 (3)
C15	0.4023 (2)	0.65664 (7)	0.87053 (17)	0.0302 (3)
H6	0.924 (2)	0.4606 (6)	0.5594 (14)	0.018 (3)*
H5B	1.196 (2)	0.4680 (7)	0.8316 (17)	0.032 (4)*
H11B	0.877 (2)	0.6516 (6)	0.7827 (15)	0.021 (3)*
H8B	0.626 (2)	0.4648 (6)	0.8863 (14)	0.020 (3)*
H3	0.905 (2)	0.3611 (7)	0.8809 (15)	0.023 (4)*
H7	0.671 (2)	0.4238 (6)	0.6717 (13)	0.018 (3)*
H13B	0.586 (2)	0.6133 (7)	0.5786 (15)	0.025 (4)*
H8A	0.871 (2)	0.4715 (6)	0.9375 (15)	0.021 (3)*
H13A	0.352 (2)	0.6079 (7)	0.6127 (16)	0.031 (4)*
H11A	0.813 (2)	0.6707 (7)	0.9292 (16)	0.028 (4)*
H14C	0.662 (2)	0.7215 (6)	0.6350 (15)	0.025 (4)*
H15B	0.404 (2)	0.6155 (8)	0.9168 (16)	0.034 (4)*
H2B	0.860 (2)	0.2753 (8)	0.7398 (16)	0.038 (4)*
H4	1.245 (2)	0.3622 (7)	0.8327 (16)	0.027 (4)*
H5A	1.271 (2)	0.4592 (7)	0.6876 (15)	0.030 (4)*
H14B	0.590 (2)	0.7531 (7)	0.7670 (16)	0.030 (4)*
H14A	0.420 (3)	0.7331 (8)	0.6386 (17)	0.039 (4)*
H2A	0.699 (2)	0.3166 (7)	0.6369 (15)	0.028 (4)*
H15A	0.257 (3)	0.6656 (7)	0.8157 (17)	0.039 (4)*
H10A	0.840 (3)	0.5602 (8)	0.5983 (18)	0.049 (5)*
H15C	0.442 (3)	0.6891 (9)	0.9488 (19)	0.050 (5)*
H10B	1.031 (3)	0.5596 (8)	0.7367 (19)	0.055 (5)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O4	0.0229 (5)	0.0160 (4)	0.0225 (5)	−0.0028 (3)	−0.0006 (4)	0.0012 (3)
O3	0.0291 (5)	0.0152 (4)	0.0240 (5)	0.0014 (4)	−0.0040 (4)	−0.0025 (3)
O1	0.0254 (5)	0.0214 (5)	0.0261 (5)	0.0020 (4)	0.0091 (4)	−0.0025 (4)
O2	0.0412 (6)	0.0359 (6)	0.0300 (6)	0.0025 (4)	0.0057 (4)	−0.0122 (5)
C9	0.0191 (6)	0.0162 (6)	0.0203 (6)	−0.0011 (5)	0.0042 (5)	−0.0021 (5)
C12	0.0197 (6)	0.0157 (6)	0.0210 (6)	0.0009 (4)	0.0038 (5)	0.0007 (5)
C8	0.0222 (6)	0.0158 (6)	0.0198 (6)	−0.0006 (5)	0.0055 (5)	0.0003 (5)
C3	0.0228 (6)	0.0157 (6)	0.0183 (6)	−0.0007 (5)	0.0032 (5)	0.0018 (5)
C6	0.0250 (6)	0.0163 (6)	0.0250 (7)	−0.0004 (5)	0.0110 (5)	0.0015 (5)
C4	0.0214 (6)	0.0218 (6)	0.0210 (7)	0.0019 (5)	0.0019 (5)	−0.0019 (5)
C7	0.0193 (6)	0.0151 (6)	0.0170 (6)	−0.0009 (5)	0.0037 (5)	0.0009 (4)
C14	0.0248 (7)	0.0180 (6)	0.0299 (7)	0.0003 (5)	0.0053 (6)	0.0032 (5)
C11	0.0254 (7)	0.0144 (6)	0.0270 (7)	−0.0003 (5)	−0.0006 (5)	−0.0025 (5)
C13	0.0276 (7)	0.0178 (6)	0.0220 (7)	0.0011 (5)	−0.0009 (5)	0.0030 (5)
C5	0.0219 (7)	0.0213 (7)	0.0326 (8)	−0.0039 (5)	0.0085 (5)	−0.0043 (6)
C1	0.0277 (7)	0.0156 (6)	0.0247 (7)	0.0038 (5)	0.0022 (5)	0.0002 (5)
C2	0.0283 (7)	0.0149 (6)	0.0261 (7)	−0.0015 (5)	0.0059 (5)	−0.0002 (5)
C10	0.0358 (8)	0.0172 (7)	0.0466 (9)	0.0016 (6)	0.0257 (7)	0.0039 (6)
C15	0.0346 (8)	0.0240 (7)	0.0365 (8)	0.0055 (6)	0.0187 (7)	0.0045 (6)

Geometric parameters (Å, °)

O4—C9	1.4219 (15)	C12—C14	1.5274 (17)
O4—C13	1.4345 (15)	C12—C15	1.5334 (18)
O3—C9	1.4173 (14)	C8—C7	1.5355 (17)
O3—C11	1.4387 (15)	C3—C2	1.5224 (17)
O1—C1	1.3453 (16)	C3—C4	1.5490 (18)
O1—C4	1.4700 (15)	C3—C7	1.5536 (17)
O2—C1	1.2076 (15)	C6—C5	1.5299 (19)
C9—C8	1.5256 (17)	C6—C10	1.5346 (18)
C9—C10	1.5480 (18)	C6—C7	1.5547 (16)
C12—C11	1.5249 (18)	C4—C5	1.5035 (18)
C12—C13	1.5273 (18)	C1—C2	1.5064 (18)
C9—O4—C13	113.66 (9)	C4—C3—C7	105.04 (10)
C9—O3—C11	111.59 (9)	C5—C6—C10	114.09 (12)
C1—O1—C4	111.46 (9)	C5—C6—C7	103.38 (10)
O3—C9—O4	110.13 (9)	C10—C6—C7	107.08 (10)
O3—C9—C8	109.58 (10)	O1—C4—C5	108.29 (10)
O4—C9—C8	106.60 (10)	O1—C4—C3	105.57 (10)
O3—C9—C10	113.22 (11)	C5—C4—C3	107.07 (10)
O4—C9—C10	111.44 (11)	C8—C7—C3	116.03 (10)
C8—C9—C10	105.54 (10)	C8—C7—C6	102.80 (10)
C11—C12—C13	106.60 (10)	C3—C7—C6	106.16 (10)
C11—C12—C14	108.66 (10)	O3—C11—C12	111.33 (10)

C13—C12—C14	109.49 (10)	O4—C13—C12	111.82 (10)
C11—C12—C15	110.57 (11)	C4—C5—C6	104.65 (10)
C13—C12—C15	110.15 (11)	O2—C1—O1	121.14 (12)
C14—C12—C15	111.24 (10)	O2—C1—C2	128.66 (12)
C9—C8—C7	104.60 (10)	O1—C1—C2	110.18 (11)
C2—C3—C4	102.99 (10)	C1—C2—C3	105.22 (10)
C2—C3—C7	115.36 (11)	C6—C10—C9	105.91 (10)
C11—O3—C9—O4	−59.75 (12)	C10—C6—C7—C3	−149.50 (11)
C11—O3—C9—C8	−176.71 (9)	C9—O3—C11—C12	60.23 (13)
C11—O3—C9—C10	65.77 (13)	C13—C12—C11—O3	−54.07 (13)
C13—O4—C9—O3	57.77 (12)	C14—C12—C11—O3	−171.99 (10)
C13—O4—C9—C8	176.57 (9)	C15—C12—C11—O3	65.66 (13)
C13—O4—C9—C10	−68.76 (12)	C9—O4—C13—C12	−55.26 (13)
O3—C9—C8—C7	−156.22 (10)	C11—C12—C13—O4	51.32 (13)
O4—C9—C8—C7	84.62 (11)	C14—C12—C13—O4	168.69 (10)
C10—C9—C8—C7	−33.99 (14)	C15—C12—C13—O4	−68.67 (14)
C1—O1—C4—C5	−126.17 (11)	O1—C4—C5—C6	81.17 (12)
C1—O1—C4—C3	−11.77 (13)	C3—C4—C5—C6	−32.24 (13)
C2—C3—C4—O1	19.78 (12)	C10—C6—C5—C4	153.34 (11)
C7—C3—C4—O1	−101.36 (10)	C7—C6—C5—C4	37.43 (13)
C2—C3—C4—C5	135.02 (11)	C4—O1—C1—O2	179.39 (11)
C7—C3—C4—C5	13.88 (13)	C4—O1—C1—C2	−1.76 (14)
C9—C8—C7—C3	152.86 (10)	O2—C1—C2—C3	−166.49 (13)
C9—C8—C7—C6	37.48 (12)	O1—C1—C2—C3	14.77 (14)
C2—C3—C7—C8	143.33 (11)	C4—C3—C2—C1	−20.57 (13)
C4—C3—C7—C8	−104.03 (12)	C7—C3—C2—C1	93.26 (12)
C2—C3—C7—C6	−103.20 (12)	C5—C6—C10—C9	−106.98 (13)
C4—C3—C7—C6	9.44 (12)	C7—C6—C10—C9	6.76 (15)
C5—C6—C7—C8	93.60 (11)	O3—C9—C10—C6	136.47 (12)
C10—C6—C7—C8	−27.19 (14)	O4—C9—C10—C6	−98.72 (13)
C5—C6—C7—C3	−28.70 (12)	C8—C9—C10—C6	16.61 (15)